Sludge rheology: semi – empirical correlations to predict the apparent viscosity and yield stress of sludge mixtures

A thesis submitted in fulfilment of the requirements for the degree of Doctor of Philosophy

Flora Markis

B. Eng. (Chemical)

B. Sc. (AppChe)

School of Civil Environmental and Chemical Engineering

College of Science Engineering and Health

RMIT University

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Declaration

I certify that except where due acknowledgement has been made, the work is that of the author alone; the work has not been submitted previously, in whole or in part, to qualify for any other academic award; the content of the thesis is the result of work which has been carried out since the official commencement date of the approved research program; any editorial work, paid or unpaid, carried out by a third party is acknowledged; and, ethics procedures and guidelines have been followed.

Flora Markis

December, 2015

DEDICATION

I, Flora Markis, dedicate this thesis in honor of my Father – Sefardon Markis and in celebration of the sacrifice he made 20 years ago to leave his motherland in search of freedom, hope and a future in a new unknown land

"A father is always making his baby into a little woman. And when she is a woman he turns her back again."

Enid Bagnold.

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"Le temps est un grand maître, dit-on, le malheur est qu'il tue ses élèves"

Berlioz.

PUBLICATIONS

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NOMENCLATURE

Symbol	Description	Unit
α, β etc.	Fitting parameters	
α_0	Plateau viscosity	Pa.s
γ	Shear strain	(%)
Γ	Dimensionless shear rate	
$\dot{\gamma}$	Shear rate	1/s
γ_c	Critical shear strain	(%)
$\dot{\gamma}_c$	Critical shear rate	1/s
δ	Phase angle	°
η	Apparent viscosity	Pa.s
η_0	zero shear viscosity or viscosity of liquid medium	Pa.s
η_∞	Infinite viscosity	Pa.s
η_L	Limiting viscosity	Pa.s
η'	dynamic viscosity	Pa.s
η_{mix}	Apparent viscosity of mixture	Pa.s
θ	Deflection angle	rad
λ	Structural parameter	
ρ	Density	Kg/m^3
τ	Shear stress	Pa
τ_c or τ_y	critical stress or yield stress	Pa
τ_B	Bingham yield stress	Pa
$\tau_{calculated}$	Calculated stress	Pa
τ_{HB}	Herschel – Bulkley yield stress	Pa
τ_{master}	Master curve yield stress	Pa
$\tau_{measured}$	Measured stress	Pa

τ_{mix}	Yield stress of mixture	Pa
φ	Volume fraction	
ω	Angular velocity	Rad/s
A	Area	m^2
a, b, c, d etc.	Fitting parameters	
C	Total solids concentration	(%)
C_{min}	Minimum total solids concentration, below which, no yield stress	(%)
C_p	Mixed liquor suspended solids	(%)
CSS	Suspended solids concentration	(%)
CSS_{max}	Maximum suspended solids concentration	(%)
D	Diameter	m
D_c	Cup diameter	mm
D_i	Impeller diameter	m
D_D	Diameter of digester	m
D_v	Vane diameter	mm
E_a	Activation energy	kJ/mol
F	Shearing force	N/m
f	Friction factor	
G	Shear modulus	Pa
G’	Elastic modulus	Pa
G”	Viscous modulus	Pa
G_0	Critical modulus	Pa
G_0^*	Complex modulus	Pa
H	Height of cup	mm

H_D	Height of digester	
H_{loss}	Head loss	M
K	Fluid consistency	$Pa.s^n$
$k_{(master)}$	Master curve fluid consistency	Pa.s
L	Length	m
M or Ω	Torque	Nm
N	Impeller speed	RPS
n or m	Flow index	
NP	Power number	
P	Pumping or mixing power	W or kW
P_s	Specific power input	W/m^3
Q	Flow rate	m^3/s
R	Gas constant	J/K.mol
r	Radius	m
Re_m	Mixing Reynolds number	
R_e	Radius of rotating cup	mm
R_i	Radius of rotating vane	mm
S_x and S_y	Shift factor in x and y axes	
T	Temperature	°C
t	Duration of creep	s
T_0	Reference temperature	°C
T_a	Applied temperature	°C
U_g	Aeration intensity	
v	Velocity	m/s
V_m	Volume of digester	mL
$V_{mixture}$	Volume of the mixture	mL

V_P, V_S, V_D,	Volume of primary (P), secondary (S), digested (D)	mL
W	Water content	(%)

SUMMARY

Municipal sludge is the by-product of the wastewater treatment process. The wastewater process produces three different types of sludge – primary and secondary sludge. Primary sludge is the product of the primary clarification process and is known as the most difficult of the three to handle whilst secondary sludge is the product of the secondary treatment process. The primary and secondary sludges undergo further treatment using the anaerobic digestion process whereby the sludge feed is continuously mixed in the absence of oxygen using a constant re – circulation loop of digested sludge to produce biogas (methane gas – CH_4), carbon dioxide (CO_2) and anaerobic digested sludge. In this process, the sludge feed which is primary and secondary sludge or a mixture of the two sludges is treated and stabilized so that its volatile solids are reduced by 40%. The anaerobic digestion process is the most commonly used technique to stabilize and reduce the volatile solids of the sludge feed. However, the exponential growth of sludge volume due to the increasing world population has made the anaerobic digestion process an inefficient technique in treating sludge.

Anaerobic digestion requires efficient mixing of the primary and secondary sludge entering the digester using a re –circulation of digested sludge to provide an optimum environment for digestion. Efficient mixing is essential to transfer substrates to microorganisms, to maintain process stability, to maintain a uniform pH and temperature for bacterial growth, to prevent short circuiting and solids deposition at the bottom of the digester as well as to minimize scum and foam formation. However, the additional sludge loads due to increased wastewater volume has led to inefficient mixing resulting in the formation of dead zones, otherwise known as inactive volumes within the digester. This creates a poor microbial environment for biogas production such that anaerobic digesters fail. Hence, it is essential to understand how and why anaerobic digesters can be optimized so that efficient mixing is achieved.

The first step in understanding how anaerobic digesters can be optimized is to study the flow behaviour of the sludge entering the anaerobic digester prior to and after it is mixed. In this way, any changes to the flow behaviour may be detected. In this study, the impact of volume fraction and total solids concentration on the apparent viscosity and yield stress of sludge mixtures is investigated because these are the two most important parameters that influence anaerobic digester performance.

This study aims to investigate the impact of total solids concentration on the rheology (solid, transitional and liquid regime) of individual primary and secondary sludge so that any changes which may influence the rheology may be detected. Another objective is to investigate the rheology of primary and secondary sludge mixtures. This aims at investigating how and why the volume fraction of secondary sludge influences the rheology, most notably, the apparent viscosity and yield stress of primary – secondary sludge mixtures. Next, the impact of volume fraction of digested sludge on the apparent viscosity and yield stress of primary – secondary – digested sludge mixtures was investigated. As such, the flow behaviour within the digesters may be predicted. The impact of total solids concentration on sludge mixtures was also investigated.

Experiments were performed in France and Australia in two seasons (summer and winter) so that any changes to the rheology of sludge due to different treatment processes and environmental conditions may be detected. Creep tests were performed by applying a pre – shear to obtain a material that is always in the same initial state of destructuration. Then a short period of rest was provided allowing the structure to rebuild. A constant stress was then applied for a duration of time. Creep tests were carried out in solid, transitional and liquid regimes using an Anton Paar rheometer (France) and a HR – 3 rheometer (Australia). To study the shear and time dependent behaviour of sludge, the creep tests were altered by changing the period of rest between the pre – shear and creep. In all cases, the wide gap vane geometry was employed to reduce inertia effects. Tool surfaces were roughened to reduce wall slip. The flow curves were reconstructed using the torque and deflection angle data.

The experimental results on the rheological behaviour of primary sludge (with 2.8, 3.7, 5.5, 6.8 and 8.2% TS) and secondary sludge (with 2.8, 4.0, 5.0, 6.5 and 9.2% TS) showed that both primary and secondary sludges behaved as non – Newtonian, shear thinning materials. For stresses below the yield stress, primary and secondary sludge exhibited viscoelastic behaviour similar to colloidal suspensions or gels. Primary sludge experienced viscosity bifurcation and yielded abruptly similar to highly thixotropic colloidal suspensions whilst secondary sludge yielded smoothly, similar to gels. In the liquid regime, both primary and secondary sludge behaved as shear thinning materials. A dimensionless form of the Herschel – Bulkely model was employed to develop the master curve for primary and secondary sludge. The apparent viscosity and yield stress of primary and secondary sludge increased with increasing total solids concentration and was attributed to the strengthening of the hydrodynamic and non-hydrodynamic interactions within the sludge. The apparent viscosity and yield stress followed an exponential and power law model as a function of total solids concentration, respectively.

Mixtures of primary and secondary sludges as well as mixtures of primary – secondary – digested sludges with 2.5 – 7% TS also behaved as non – Newtonian, shear thinning yield stress materials. In all cases, a dimensionless form of the Herschel – Bulkley model was used to predict the flow behaviour of mixed sludge so that the apparent viscosity and yield stress of mixed sludge also followed exponential and power law models as a function of the total solids concentration of the mixture, respectively.

The experimental results for primary – secondary sludge mixtures showed that the apparent viscosity and yield stress of mixed sludge depended on the volume fraction of secondary sludge and total solids concentration. The apparent viscosity and yield stress of mixed sludge prepared by mixing primary sludge (with 3, 4, 5, 6.5 and 7.1%TS) and secondary sludge (with 3, 4, 5, 6.5 and 7.1%TS) increased with increasing volume fraction of secondary sludge. This was attributed to the deflocculation of the weak structure of primary sludge. The weak colloidal like particles of primary sludge became trapped and entangled in the gel like network structure of secondary sludge so that the resulting mixed sludge exhibited elevated apparent viscosity and yield stress values. When thickened primary sludge (5.4% TS) was mixed with dilute secondary sludge (2.8%TS) and vice – versa, the apparent viscosity and yield stress of the mixed sludge increased with increasing volume fraction of thickened sludge – regardless of sludge type. This trend was attributed to the strengthening of hydrodynamic and non-hydrodynamic interactions within concentrated sludge. The apparent viscosity and yield stress of primary – secondary sludge mixtures were predicted using a power law model as a function of volume fraction of secondary sludge.

The experimental results on primary – secondary – digested sludge mixtures showed that the apparent viscosity and yield stress of a primary – secondary sludge mixture (50:50 v/v) mixed with digested sludge depended on the volume fraction of digested sludge and total solids concentration of the mixture.

The apparent viscosity and yield stress of primary – secondary – digested sludge prepared by mixing a 50:50 (v/v) primary – secondary sludge mixture (with 3, 4, 5.1, 6.3, 7.1%TS) to digested sludge (with 3, 4, 5.1, 6.3, 7.1%TS) increased with increasing volume fraction of digested sludge so that the solid interactions within the sludge mixture increased. This was highlighted using the shear compliance and shear modulus of sludge mixtures. When a thickened primary – secondary sludge mixture (5%TS) was mixed with dilute digested sludge (1.8%), the apparent viscosity and yield stress decreased as the volume fraction of digested sludge increased. This was attributed to the dilution effect so that the hydrodynamic and non-hydrodynamic interactions of the sludge mixture were reduced when dilute digested sludge was added. The apparent viscosity and yield stress were predicted using a power law model as a function of volume fraction of digested sludge whilst the parameters of these models were estimated using the pH of the sludge mixture. The shear compliance and complex modulus followed an exponential relationship with increasing digested sludge volume fraction.

Finally, procedures are outlined to demonstrate how the developed knowledge in this thesis can be used to estimate the Herschel – Bulkley model of different sludge mixtures (within the studied range) using the master curves as well as the developed apparent viscosity and yield stress correlations for different types of sludge mixtures. Additionally, a procedure is outlined to demonstrate how the developed correlations can be used to optimize the power and energy requirements for unit operations such as pumps and mixing systems.

CHAPTER 1

INTRODUCTION

Chapter 1: Introduction

1.1 Project rationale

The design and optimization of wastewater treatment facilities require the accurate prediction and estimation of the flow behaviour of sludge in order to gain a better understanding of the hydrodynamics of the various unit operations such as pumps (pressure loss calculations), heat exchangers and mixing systems (digester hydrodynamics). Over the years, Slatter (1997, 2001, 2008) has consistently shown that the rheology of sludge plays a fundamental role in understanding the hydrodynamic behaviour of sludge as it flows through the treatment process.

The legal banning of conventional sludge disposal methods, increasing urban populations, urban land shortages and economical aspects associated with the expansion of treatment plants has led to the treatment of a more concentrated and complex sludge in the sludge treatment line (Eshtiaghi et al., 2012a). Current wastewater treatment plants are reaching full capacity and cannot handle any additional load without concentrating the input sludge or expanding the current treatment process which in turn is costly. A better understanding of the flow properties of concentrated sludge is required as they influence the efficient operation and optimization of wastewater treatment facilities.

In sludge treatment lines, primary and secondary sludges are stabilized by entering the anaerobic digester for further pathogen reduction and biogas production. Whilst studies have focused on the effect of mixing primary and secondary sludge on biogas production and digester performance (Bouallagui et al., 2010), a few studies have investigated how and why the flow behaviour prior to and after mixing changes. Additionally, the current literature on sludge focuses on the rheology of activated and digested sludge with few studies on primary sludge.

Currently, research is focused on the rheological characterisation of low to medium concentrations of secondary or activated sludge (Baudez and Coussot, 2001, Baudez, 2008, Forster, 1981, Forster, 1982, Forster, 2002, Mikkelsen, 2001, Mori et al., 2006, Seyssiecq et al., 2008, Tixier et al., 2003b, Tixier et al., 2003a, Guibaud et al., 2004) as well as digested sludge (Baudez et al., 2011b, Baudez et al., 2013a, Bhattacharya, 1981, Campbell and Crescuolo, 1982, Eshtiaghi et al., 2012b, Forster, 1982, Forster, 2002, Wang et al., 2011). In these studies, sludge is always described as a non – Newtonian shear thinning material. Secondary and digested sludge exhibit viscoelasticity at low shear stresses (Baudez and Coussot, 2001, Baudez et al., 2011b, Baudez et al., 2013a) and exhibit temperature dependent flow behaviour (Dieudé-Fauvel et al., 2009, Baudez et al., 2013b, Farno et al., 2014). Tabuteau et al (2006) and Baudez (2008) showed that activated sludge displayed thixotropy whereby it undergoes physical aging and shear rejuvenation. Baudez et al (2011b) demonstrated that digested sludge experienced shear banding. On the other hand, there are two studies on the rheological characterisation of primary sludge (Bhattacharya, 1981, Moeller and Torres, 1997) in which the results contradict each other. Whilst Bhattacharya (1981) described primary sludge as a shear thinning, yield stress material, Moeller and Torres (1997) detected no yield stress. The contradictory results between the works of Bhattacharya (1981) and Moeller and Torres (1997) emphasise that an accurate procedure and estimation of the flow behaviour of primary sludge is required within the field of sludge rheology.

There are no studies investigating how and why the rheology of different types of sludge changes if concentrations or the volume fraction of one type of sludge varies. In this way, the flow behaviour prior and after mixing may be studied so that the flow behaviour within anaerobic digesters may be simulated.

1.2 Aim of project

This study aims to:

- Investigate the rheology (solid, liquid and yielding) of individual primary and secondary sludge over a wide range of total solids concentration,
- Gain an in – depth knowledge on the rheology of mixtures of primary and secondary sludge if primary and secondary sludge are mixed together at the same total solids concentration or by mixing thickened primary to dilute secondary (and vice versa) while varying the volume fraction of secondary sludge from 0 – 1.

- Gain an in – depth knowledge on the rheology of mixtures of primary, secondary and digested sludge by adding digested sludge to a mixture of primary and secondary sludge. The mixtures are prepared at the same total solids concentration or by mixing dilute digested sludge to thickened primary – secondary sludge at different volume fractions of digested sludge (0 – 1). In this way, the rheology of the primary – secondary – digested sludge mixtures prior to and after mixing may be investigated so that anaerobic digester conditions may be simulated.
- Develop correlations that predict the rheology of sludge mixtures based on rheological properties of individual sludge in the mixture.

To achieve the abovementioned aims, the following research questions will be addressed:

- What is the rheological behaviour of primary and secondary sludge? How and why does the rheological behaviour of primary sludge differ from secondary sludge? How and why does the rheological behaviour change with total solids concentration?

- What is the rheological behaviour of mixtures of primary and secondary sludge? How and why does the rheology of mixture change when the volume fraction of secondary sludge varies? Which correlation may be used to predict the rheological behaviour of mixtures of primary and secondary sludge?

- What is the rheological behaviour of mixtures of primary, secondary and digested sludge? How and why does the rheology of the three sludge mixture change when digested sludge is added? What is the correlation when a third material (digested sludge) is introduced?

1.3 Thesis outline:

Chapter 2 will provide a detailed literature review on the available sludge rheology, rheometry and modelling.

Chapter 3 contains the methodology that was used in this study. This will be separated into two sections, the first, containing an outline of the experimental procedure and set up; the second containing an outline of the master curve development.

Chapter 4 will answer the first research question on the rheological characterisation of primary and secondary sludge. In this chapter, we will investigate the solid, liquid and yielding characteristics of primary and secondary sludge over a wide range of concentrations. Any significant differences in the rheological behaviour of these two different types of sludge will be presented. The result of this chapter was published in Chem. Eng. J. Vol.253, P. 526–537 (2014).

Chapter 5 will answer the second research question on the rheological characterisation of mixtures of primary and secondary sludge. In this chapter, we will investigate how the volume fraction of secondary sludge influences the rheological behaviour, most notably, the apparent viscosity and yield stress of mixtures of primary and secondary sludge. We will also investigate how and why the rheological behaviour of primary sludge changes after it is mixed with secondary sludge. Correlations are presented to predict the apparent viscosity and yield stress of primary – secondary sludge mixtures. The result of this chapter was published in Chem. Eng. J., DOI: 10.1016/j.cej.2015.11.107.

Chapter 6 will answer the third research question on the rheological characterisation of primary – secondary – digested sludge mixtures. Here, we will focus on how and why the apparent viscosity and yield stress of the primary and secondary sludge mixture is influenced by the addition of digested sludge. Correlations are presented to predict the apparent viscosity and yield stress of primary – secondary sludge – digested mixtures. The result of this chapter has been submitted to Water Research J., Ref. No.: WR33452.

Chapter 7 provides a summary of the industrial implication of the fundamental knowledge developed in this thesis and how wastewater treatment industry may benefit from the outcome of this study.

Chapter 8 provides a summary of the contribution of knowledge made through this thesis and recommends in this area that needs to be further developed and studied in the future.

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CHAPTER 2

LITERATURE REVIEW

Chapter 2: Sludge rheology literature review

2.1 Introduction

Due to the increasing urban population, urban land shortages and economics, wastewater treatment plants have been treating more concentrated and complex sludge. As such, the sustainable management of wastewater treatment plants has become a major issue. Predicting the rheology of sludge as it flows through various unit operations (e.g. pumping and transportation, storage and handling, mixing, chemical conditioning and dewatering) of the wastewater treatment process is essential for the design and optimization of the various unit operations. However, as the sludge flows through the different unit operations, its flow behaviour and characteristics are altered, indicating that the flow behaviour depends on the unit operation the sludge has experienced as well as various factors such as the total solids concentration, temperature, water content and particle interactions governing the structure of the sludge. This chapter contains a review on the current research that has been conducted on the rheology of different types of sludge such as primary, secondary and digested sludge.

This chapter shows that the apparent viscosity, yield stress, thixotropy and viscoelastic characteristics of sludge are influenced by several factors such as the total solids concentration, temperature, water content and particle interactions governing the structure of the sludge. Whilst several researchers have attempted to model these rheological properties, it is shown that there are inconsistencies in the literature due to the sampling technique, rheometric measuring technique or the data analysis technique. To prevent inconsistencies within the literature, an appropriate procedure, rheometric technique and data analysis technique should be selected for the specific sludge.

This chapter highlights that there are extensive studies focusing on the rheology of activated and digested sludge with few contradictory studies on primary sludge. These studies are aimed at improving unit operations such as pipelines, heat exchangers, storage tanks or dewatering units. However, there are no known studies investigating the rheology of different types of sludge as a feed to the anaerobic digestion process. Furthermore, there are no known studies focusing on the rheology of sludge mixtures so that the flow behaviour prior to and after mixing may be determined. As such, there are no known studies which focus on how and why the flow behaviour prior to and after mixing changes and how it influences digester performance.

2.2 What is sludge?

Sewage sludge or more commonly, "sludge", is the by-product of the treatment of municipal wastewater, that is, the water and a semi-solid residue produced from human and residential waste, industrial waste and hospital waste, runoff from streets, farmlands and landfill leachates (Sanin et al., 2011).

Prior to undergoing sludge treatment, "fresh" sewage sludge is an odorous suspension of organic flocs suspended in water (Sanin et al., 2011). It is composed of water (more than 95%), mineral particles, dead and alive bacteria (polymeric and dissolved) (Bhattacharya, 1981, Baudez et al., 2013b). There are two types of sludge produced by the wastewater treatment process: primary sludge (also referred to as raw sludge) or secondary sludge (also referred to as "waste activated sludge") (Sanin et al., 2011).

Primary sludge is the product of the primary treatment process, whereby settleable (large and heavy) solids are removed from wastewater using a primary clarifier. The primary clarifier, also known as a settling tank or sedimentation tank, operates by allowing the heavier solids to settle to the bottom, whilst the lighter, smaller solids remain afloat. The bottoms of the clarifier are known as "raw" primary sludge and are objectionable, highly pathogenic and contain a high amount of water. These characteristics make it very difficult to handle. It is sent to the anaerobic digester for further treatment so that a more desirable and disposable material is produced (Sanin et al., 2011).

"Waste activated sludge" or simply "secondary sludge" is the product of the secondary treatment process. In this process, air is injected into a mixed liquor (i.e. mixture of suspended solid in liquid) using porous diffusers such as surface aerators or brushes or aspirators. The microorganisms produced in the aeration tank are then removed (via a final clarifier) and recycled to the beginning of the aeration system. This system produces more biological waste material than required by the actual system. The biological waste material produced from the secondary sludge process is referred to as "waste activated sludge" and is commonly mixed with raw primary sludge and sent to anaerobic digesters for further processing prior to disposal (Sanin et al., 2011).

In general, sludge is sent to the sewage sludge treatment process to be physically, chemically and biologically treated to eliminate odour as well as remove suspended and dissolved organics, pathogens and bacteria. Anaerobic digestion is the most notable sewage sludge treatment process employed to stabilize sewage sludge and reduce its percentage volatile solids by about 40% (Sanin et al., 2011).

Figure 1 illustrates the typical primary and secondary sludge wastewater treatment systems, leading to anaerobic digestion.

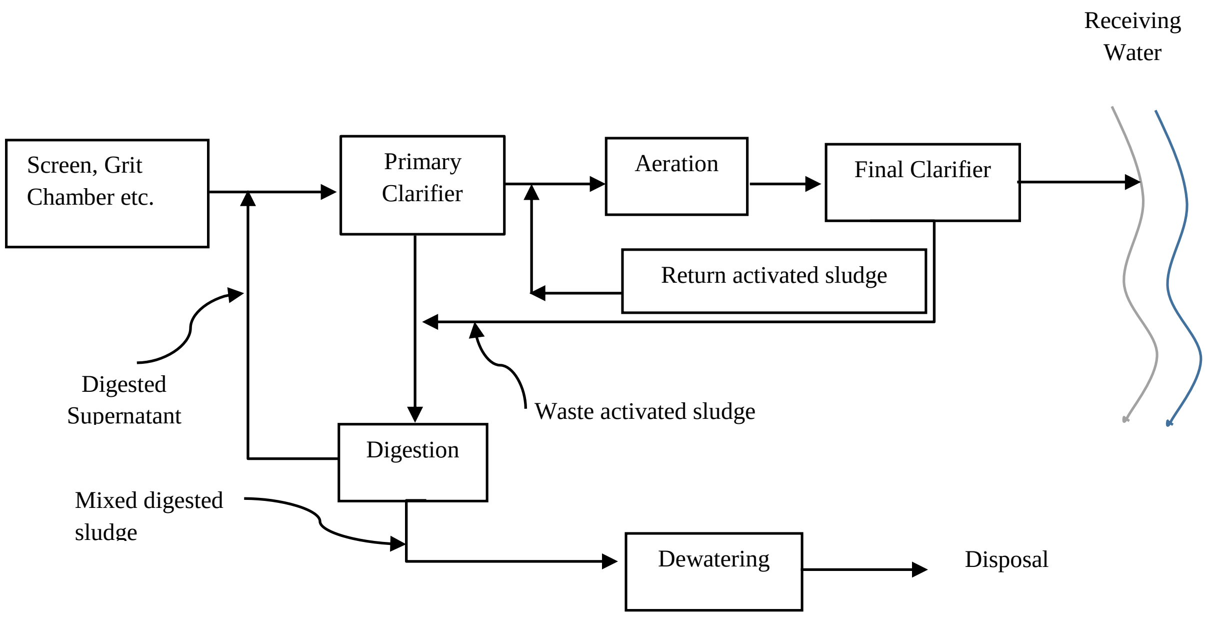

Figure 1: Typical primary and secondary sludge wastewater treatment systems, leading to anaerobic digestion (Sanin et al., 2011)

The anaerobic digestion process requires the microbial degradation of organic matter through the constant mixing of microorganisms (in the absence of oxygen) to produce methane gas (CH_4), carbon dioxide (CO_2) and anaerobic digested sludge. The organic matter to be digested is primary and secondary sludge, or a mixture of the two sludges. The methane gas produced during the anaerobic digestion process is used to either heat other treatment units or produce electricity. Anaerobic digesters operate under mesophilic conditions (temperature range between 30 – 38 °C, operating temperature = 35°C) or thermophilic conditions (temperature range between 50 – 60°C, operating temperature = 55°C) and require efficient mixing of the primary and secondary sludge entering the digester to provide the optimum environment for digestion. The anaerobic digested sludge is thickened or dewatered (i.e. further treatment) to reduce its volume prior to disposal (Sanin et al., 2011).

The quantity and quality of different types of sludge are dependent on the treatment process they have experienced. Also, each sludge is biologically different (depending on the treatment process) such that the interactions that govern the network structure of the sludge are different. As such, the different types of sludge and the governing interactions must be defined correctly.

2.2.1 Primary sludge

Primary sludge is defined as a flocculated mixture of organic and inorganic, alive and dead bacteria with gas bubbles trapped within the suspension (Bhattacharya, 1981). Cui et al (2011) and Bayoudh et al (2009) explained that the bacteria in primary sludge are held together by nonspecific Lif-shitz van der Waals forces as well as hydrogen and chemical bonds.

2.2.2 Secondary sludge

Secondary sludge is made up of polysaccharide and protein rich bacteria and micro – organisms so that extracellular polymeric substances (i.e. EPS) are formed. The EPS are said to form a three dimensional gel like structure with a negative surface charge (Wingender et al., 1999). Keilding (2001) and Sutherland (2001) state that when secondary sludge interacts with water, it behaves as a gel to form flocs. The network structure of secondary sludge is held together by electrostatic and hydrogen bonds (Flemming 1996).

2.2.3 Digested sludge

Digested sludge is the by – product of the anaerobic digested process whereby primary and secondary sludge, as substrates, are degraded and biogas is formed by the action of micro – organisms in the absence of air. This means that the large flocs are broken down to smaller flocs via constant mixing (Mahmoud et al., 2006) and therefore the organic matter is also broken down so that smaller, more uniform flocs form with a homogenous grain structure (Turovskiy and Mathai, 2006). The anaerobic digestion process reduces the EPS content whilst increasing the amount of colloidal particles (Karapanagiotis et al., 1989). Forster (1983) explained that the digested sludge contained proteins and lipopolysaccharides; these are amphiphile lipids with both hydrophobic and hydrophilic heads. Na'mar and Ganczarczyk, (1993) found that digested sludge was a more stable suspension (relative to primary and secondary sludge) with a low settlingability and Forster (2002) found that digested sludge exhibited a low surface charge such that steric rather than electrostatic interactions dominate. Mikkelsen and Keiding (2002) demonstrated that the structure of sludge changes prior to and after anaerobic digestion through the ratio between the protein and polysaccharides. This ratio was constant for secondary and (mesophilic) digested sludge, however, the degree of dispersion was 20 times higher after digestion. As such, the structure of sludge was altered after digestion.

2.3 What is rheology?

Rheology is the study of flow of matter mainly in liquids but can be extended to "soft solids" in addition to complex materials. These materials cannot be described by a single value of viscosity and thus rheological measurements must be carried out to determine the rheological behaviour of these materials (Chhabra and Richardson, 2008). Rheology has been described by (Sanin et al., 2011) as "the science that deals with the relationship between an imposed shear stress and the resultant shear rate under different conditions".

The behaviour of a material under an applied force is illustrated in Figure 2 whereby a fluid is contained between two parallel plates of area, (A) and a specific distance apart (d_y).The upper plate is subjected to a Force (F) to give it a velocity(dV_x), whilst the lower plate remains stationary. This means that the fluid next to the upper plate moves at a velocity of dV_x whilst the fluid next to the lower plate has a velocity of zero. As such, a uniform velocity gradient of magnitude dV_x/d_y is developed within the fluid due to the uniform shearing Force, F, across the distance dy (Chhabra and Richardson, 2008, Sanin et al., 2011).

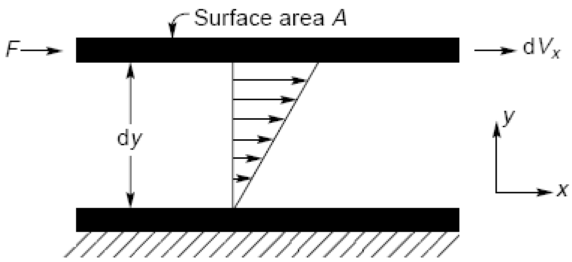

Figure 2: Unidirectional flow (Chhabra & Richardson, 2008)

The experiment illustrated in Figure 2 can be described using Eq. 1 whereby the velocity gradient dV_x/d_y is known as the shear rate or rate of shear, $\dot{\gamma}$, whilst the shearing force per unit area (F/A) is known as the shear stress, τ (Chhabra and Richardson, 2008, Sanin et al., 2011).

$$\frac{F}{A} = \tau = \frac{\eta dV_x}{d_y} = \eta\dot{\gamma} \qquad \text{Eq. 1}$$

The constant of proportionality, known as η in Eq. 1 is the Newtonian viscosity.

2.3.1 Classification of fluid behaviour

Fluids are classified in two categories according to their response to an applied stress (at a constant pressure and temperature). The first of the two are Newtonian fluids; these fluids exhibit a direct proportionality between the shear stress and shear rate under the laminar flow conditions such that Eq. 1 becomes (Chhabra and Richardson, 2008, Sanin et al., 2011):

$$\eta = \frac{\tau}{\dot{\gamma}} \qquad \text{Eq. 2}$$

The Newtonian viscosity (η) is a measure of the resistance to flow and can be defined as the ratio of the shear stress to shear rate. The Newtonian viscosity depends only on the material, temperature and pressure of the fluid. The plot of the shear stress versus shear rate (i.e. flow curve, see Figure 3) consists of a straight line with the slope η and passes through the origin for a Newtonian fluid. Thus the η, obtained from the flow curve, describes the flow behaviour at a fixed temperature and pressure for a Newtonian fluid (Chhabra and Richardson, 2008).

A non – Newtonian fluid is defined as a fluid whose apparent viscosity (i.e. shear stress/ shear rate) is not constant at a given temperature and pressure, but differs depending on the flow conditions such as flow geometry, shear rate and/ or the kinematic history of the fluid. Thus the flow curve (shear rate versus shear stress) of such a fluid is described as non-linear and (may or may not pass through the origin) (Chhabra and Richardson, 2008).

Non – Newtonian fluids can be identified according to the different fluid models that can be used to describe their fluid behaviour. Non – Newtonian fluids are classified in three categories – time independent fluids, time dependent fluids and viscoelastic fluids (Chhabra and Richardson, 2008).

2.3.2 Non – Newtonian fluid models

The most commonly used non – Newtonian fluid models to describe the behaviour (under steady state laminar flow regime) are the power law (or Ostwald model; Eq. 3), the Bingham model (Eq. 4), the Herschel – Bulkley model (Eq. 5), the Truncated power law model (Eq. 6), the sisko model (Eq. 7), the casson model (Eq. 8) and the cross model (Eq. 9) (Chhabra and Richardson, 2008).

$$\tau = K\dot{\gamma}^n \qquad \text{Eq. 3}$$

$$\tau = \tau_y + K\dot{\gamma} \qquad \text{Eq. 4}$$

$$\tau = \tau_y + K\dot{\gamma^n} \qquad \text{Eq. 5}$$

$$\frac{\tau}{\tau_y} = \left(\frac{\dot{\gamma}}{\dot{\gamma_Y}}\right)^n \qquad \text{Eq. 6}$$

$$\tau = \eta_\infty\dot{\gamma} + K\dot{\gamma^m} \qquad \text{Eq. 7}$$

$$\sqrt{\tau} = \sqrt{\tau_y} + \sqrt{\eta\dot{\gamma}} \qquad \text{Eq. 8}$$

$$\eta = \frac{\eta_0}{1+(K\dot{\gamma})^m} \qquad \text{Eq. 9}$$

The power law model (Eq. 1) is a pseudoplastic model that describes the shear thinning behaviour (i.e. decrease in apparent viscosity with increasing shear rate) of a material where τ is the shear stress, $\dot{\gamma}$ is the shear rate, K is the fluid consistency, n is the flow index. The fluid consistency is a measure of the proportionality and has the units $Pa.s^n$ and the flow index is a dimensionless measure of the material behaviour (Chhabra and Richardson, 2008).

The power law model is illustrated in Figure 3 as the "pseudoplastic" curve which passes through the origin. When n equals 1($n = 1$), the curve reduces to a straight line that passes through the origin and the fluid is said to be Newtonian. When n is less than 1($n < 1$), the fluid is known as shear thinning and the flow curve will exhibit an upward concave curve. When n is greater than 1 ($n > 1$), the fluid is known as shear thickening (i.e. dilatant) and the resulting flow curve will exhibit a downward concave curve (Chhabra and Richardson, 2008).

The Bingham model is a two parameter modification of the power law model that describes the viscoplastic behaviour of the fluid. The typical Bingham fluid behaves as an elastic solid at low shear stresses, however, when a critical stress, known as the yield stress is overcome, the fluid flows like a viscous fluid. The Bingham fluid exhibits a linear flow curve as illustrated in Figure 3 (Chhabra and Richardson, 2008).

The Herschel – Bulkley model is a three parameter combination of the power law model and the Bingham model that describes the viscoplastic plus the shear thinning or thickening behaviour of a fluid. A fluid that is represented by the Herschel – Bulkley model behaves as a solid at low stresses, until a yield stress is reached, then flows like a shear thinning fluid. Once the applied stress is removed, it returns back to its original solid state. As such the Herschel – Bulkley model describes both viscous and plastic fluid properties. In Figure 3, the Herschel – Bulkley model is shown as the "yield pseudoplastic" concave curve (Chhabra and Richardson, 2008).

The Truncated power law, Sisko, Casson and Cross fluid models are variations of the power law, Bingham and Herschel – Bulkley models. The type of fluid model selected to represent the fluid depends on the non – Newtonian fluid behaviour displayed by the fluid (Chhabra and Richardson, 2008).

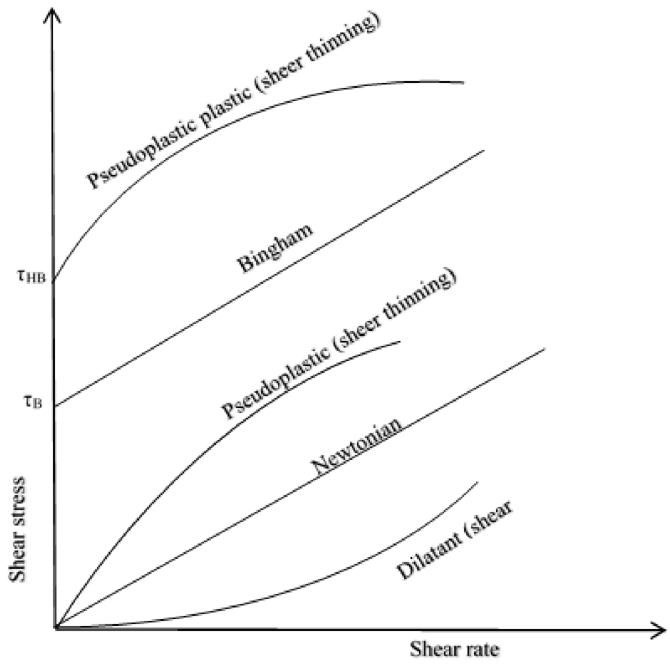

Figure 3: Fluid models (Chhabra & Richardson, 2008)

2.3.3 Non – Newtonian fluid behaviour

As stated earlier, non – Newtonian fluid behaviour is classified in three categories: time independent, time dependent, and viscoelastic. Time independent fluids are defined as "purely viscous", "inelastic" or "generalized Newtonian fluids" so that the behaviour of time dependent fluids is dependent on the applied stress only. Time independent fluid behaviour is divided into three types: shear thinning (i.e. pseudoplastic), shear thickening (i.e. dilatant) and viscoplastic. Time dependent fluid behaviour is not only dependent on the applied stress, but also on the time of shear and the kinematic history of the fluid. Time dependent behaviour is divided into two types: thixotropic and rheopectic. In contrast to both time independent and dependent behaviour, viscoelastic fluids display both viscous (i.e. liquid) and elastic (i.e. solid) behaviour (Chhabra and Richardson, 2008).

2.3.3.1 Time independent fluids

Fluids that display shear thinning behaviour demonstrate a decrease in the apparent viscosity with increasing shear rate. Shear thinning fluids do not exhibit a yield stress. However, shear thinning fluids are often classified as Newtonian fluids at very low and very high shear rates. The flow curve contains upper and lower limits known as the zero shear rate Newtonian viscosity and infinite shear rate Newtonian viscosity, depicted as η_o and η_∞ respectively (as shown in Figure 4). Shear thinning behaviour can be described using the power law model, the sisko model and the cross model (Chhabra and Richardson, 2008).

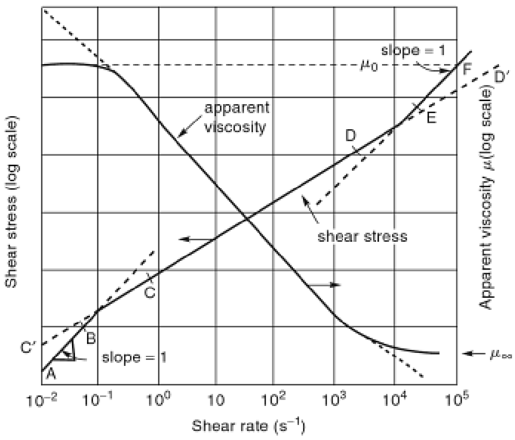

Figure 4: Shear thinning fluid behaviour (Chhabra & Richardson, 2008)

Shear thickening fluids also known as dilatant fluids are characterized by an increasing apparent viscosity with increasing shear rate as well as an absence of the yield stress. Shear thickening behaviour is defined as a liquid filling the void of a material when at rest, however, when the material is subjected to high shear, the liquid expands and insufficient liquid is present to fill the increasing void space indicating an increase in the solid – solid interactions. As such, higher stresses are applied and the resulting viscosity increases with increasing shear rate. The power law model with a power law index, n, greater than 1 ($n > 1$), is used to describe the shear thickening behaviour (Chhabra and Richardson, 2008).

Viscoplastic fluids, also known as “yield stress” fluids behave as a solid for stresses below the yield stress, however, flow as viscous liquids when the yield stress is exceeded. The flow curve of such fluids can be described as linear, modelled using the Binhgam fluid model (i.e. Bingham plastic fluids) indicating a constant, plastic viscosity or non-linear, modelled using the Herschel – Bulkley fluid model (i.e. yield psuedoplastic fluids) indicating shear thinning or shear thickening behaviour. In both cases, a yield stress is detected (Chhabra and Richardson, 2008).

2.3.3.2 Time dependent fluids

Time dependent fluid behaviour cannot be described by the mathematical fluid models. The apparent viscosity of these fluids not only depends on the applied stress but also on the time of shear in addition to their kinematic history. Time dependent fluids are broken down into two categories: thixotropic or rheopectic fluids (Chhabra and Richardson, 2008).

Thixotropic fluids are observed when a fluid is subjected to a constant stress or shear, and the resulting apparent viscosity decreases with the time of shearing. A hysteresis loop describes thixotropy for time dependent fluids; this is obtained by employing a increasing the shear rate constantly from zero to a maximum value then decreasing the rate constantly. The area within the hysteresis loop depends on the time in which the fluid is subjected to shear, the rate of shearing (increasing/ decreasing) and past kinematic history of the fluid. Thus, a larger area means a stronger time dependent fluid behaviour for such fluids. In contract, time independent fluids do not display a hysteresis loop with an enclosed area of zero (Chhabra and Richardson, 2008).

Figure 5 below illustrates the hysteresis loop for a thixotropic fluid in addition to a rheopectic fluid.

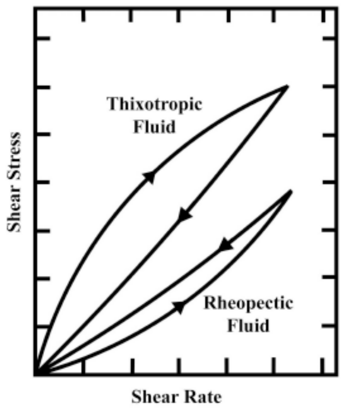

Figure 5: Time dependent fluids (Chhabra & Richardson, 2008)

For viscoplastic fluids, the term "false body" describes the thixotropic behaviour whereby the original structure of the fluid is regained after long periods of time with the presence of a yield stress. This is due to the solid like properties of such fluids (Chhabra and Richardson, 2008).

Rheopectic fluids demonstrate an increasing apparent viscosity with time of shearing. In contrast to thixotropic fluids, rheopectic fluids exhibit structural build up under shear and breakdown at rest. Figure 5 illustrates the hysteresis curve for a rheopectic fluid (Chhabra and Richardson, 2008).

In order for a fluid to be either a thixotropic or rheopectic fluid, it must obey Freundlich's definition (1928) whereby the fluid must be inelastic and depend on the shear rate; the fluid must also recover its structure when the applied stress or shear is removed over a period of time. The flow curve for thixotropic and rheopectic fluids consist of a hysteresis loop in addition to a decay (thixotropic) or build up (rheopexy) of stress under constant shear (Chhabra and Richardson, 2008).

Thixotropic and rheopectic behaviour can be illustrated using three different graphs, the first consisting of a hysteresis loop obtained by increasing the shear rate (or stress) and then decreasing it. Secondly, the shear rate (or stress) against time on a linear scale determined by applying a constant shear stress or rate for a specific time and recording the resulting deformation or stress; An example of this is shown in Figure 6 (Chhabra and Richardson, 2008).

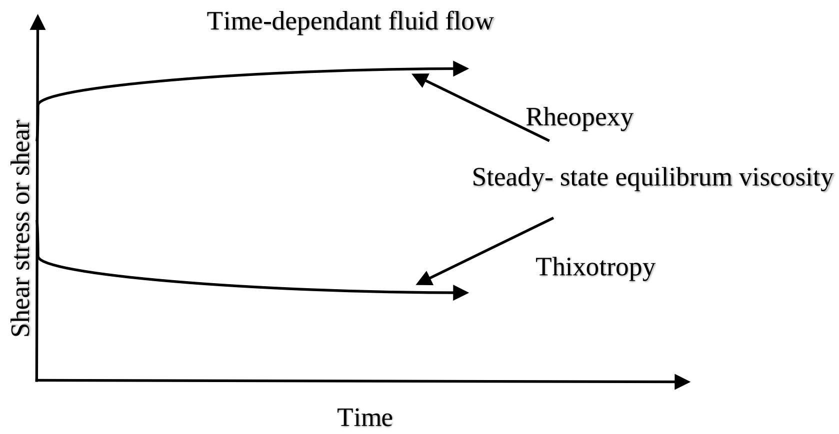

Figure 6: Thixotropic and rheopectic behaviour of Non-Newtonian fluids (Chhabra & Richardson, 2008)

As shown in Figure 6, the viscosity curve for both time dependent fluids can be also illustrated by measuring the apparent viscosity under a constant rate or stress for a specific time (Chhabra and Richardson, 2008).

2.3.4 Viscoelastic fluids

The classic theory of elasticity states that

"The stress in a sheared body is directly proportional to the strain"

Tension is therefore described by Hooke's law with the constant of proportionality known as Young's modulus, G (Chhabra and Richardson, 2008):

$$\tau_{yx} = -G\frac{d_x}{d_y} = G(\gamma_{yx}) \qquad \text{Eq. 10}$$

, where d_x is described as the shear displacement of two elements separated by a distance d_y (Chhabra and Richardson, 2008).

Elasticity is described by considering the elastic deformation of an ideal solid, whereby the original structure is recovered once the applied stress is removed. The solid is said to flow "creep" when the applied stress is greater than the yield stress and the original structure of the fluid does not recover. An ideal fluid will flow under an applied stress; however, it will not flow when the stress is removed. Viscoelastic fluids are said to contain both elastic and viscous properties, that is, the fluid can store and recover shear energy. Gel structures exhibit viscoelastic properties.

Oscillatory measurements are carried out to determine the rheological behaviour of complex fluids, in particular, the viscoelastic properties, whereby a fluid is subjected to an oscillatory strain (γ) and the response is determined. This response describes the elastic and viscous or damping characteristics of a fluid (Coussot, 2005).

For a fluid in which a sinusoidal strain (γ) has been applied (Eq. 11) at an angular frequency of (ω), a steady sinusoidal stress (τ) is determined as shown in Eq. 12 (Coussot, 2005).

$\gamma = \gamma_0 \sin \omega t$ Eq. 11

$\tau = G\gamma_0 \sin \omega t + \eta\gamma_0 \cos \omega t$ Eq. 12

Thus the storage (elastic) modulus, G' and loss (viscous) modulus, G" are determined in addition to the dynamic viscosity ($\mu\eta' = (G"/\eta$) (Chhabra and Richardson, 2008).

G' and G" are determined by measuring the amplitude and phase shift of an oscillating strain signal. Therefore for a viscoelastic fluid $G' = G$ and $G" = \eta\omega$ (Chhabra and Richardson, 2008).

For other fluid types, an estimation of the viscous and elastic properties is determined by employing Eq. 13 (Chhabra and Richardson, 2008).

$\tan \delta = G"/G'$ Eq. 13

For other materials, the G' and G" often depend on the stress (or strain) amplitude and frequency and can be determined by both applying a frequency and varying the deformation and stress amplitude or by applying a stress or amplitude and varying the frequency. Such measurements describe the behaviour of materials (Chhabra and Richardson, 2008).

2.4 Sludge Rheology

2.4.1 Flow measurements

Rheology is the study of the flow and deformation of matter (Chhabra and Richardson, 2008) for which the fluid behaviour can be described as either Newtonian or non – Newtonian. Dilute suspensions including dilute sewage sludge behave as Newtonian fluids, however, at higher concentrations, sewage sludge exhibits, complex, non – Newtonian behaviour (Mori et al., 2006, Wang et al., 2011, Seyssiecq et al., 2003, Novarino et al., 2010, Spinosa and Lotito, 2003, Baudez and Coussot, 2001, Baudez et al., 2004, Baudez et al., 2011b, Lotito and Lotito, 2014b, Tang and Zhang, 2014, Liu et al., 2012, Dai et al., 2014, Ségalen et al., 2015, Ma et al., 2014, Ruiz-Hernando et al., 2015, Feng et al., 2014) such that the rheological behaviour is dependent on the treatment process (Lotito et al., 1997, Battistoni, 1997). Eshtiaghi et al (2013b) outlines the various complex fluid models that are used to describe the behaviour of sewage sludge as well as complex suspensions in the steady state laminar flow regime such as the power law or Ostwald model (Moeller and Torres, 1997, Bougrier et al., 2006, Wu et al., 2011, Feng et al., 2014), the Bingham model (Sozanski et al., 1997, Guibaud et al., 2004, Mu and Yu, 2006, Lotito and Lotito, 2014a), the sisko model (Mori et al., 2006), the Herschel – Bulkley model (Slatter, 1997, Eshtiaghi et al., 2012b, Baudez and Coussot, 2001, Baudez et al., 2011b, Markis et al., 2014, Liu et al., 2012, Dai et al., 2014, Ma et al., 2014, Jiang et al., 2014, Urrea et al., 2014, Urrea et al., 2015, Seyssiecq et al., 2015), the truncated power law model (Baudez et al., 2011b, Baudez et al., 2013b) and the cross model (Sybiliski, 2011). The power law and Sisko models are most commonly used to describe the shear thinning properties of sludge whilst the Bingham, Herschel – Bulkley and Casson models have been commonly used to describe both the shear thinning properties and yield stress.

The power law, Bingham and Herschel – Bulkley models are the simplest and most commonly used of the above mentioned models depending on the existence of the yield stress (Chhabra and Richardson, 2008). Seyssiecq et al (2003)'s review on activated sludge rheology as well as Ratkovich et al (2013)'s review emphasises that the selection of an appropriate model is highly subjective and dependent on the experimental conditions such as the applied shear stress or shear rate as well as the type of sludge. Eshtiaghi et al (2013b) reviewed various studies whereby various rheological models were used to describe the flow behaviour of different types of sludge depending on factors such as yield stress, solids concentration and shear stress or shear rate range. Indeed, this is true, Khalili – Garakani et al (2011) used different fluid models to characterize the flow behaviour of sludge in a submerged reactor such that the Herschel – Bulkley model was used at high concentrations of activated sludge whilst the Bingham model was used to characterise dilute sludge; the power law model was used to determine the viscosity in the low shear rate range. At an intermediate shear rate range, Martin et al (2011) used the Bingham model to characterize membrane bioreactor and anaerobic digested sludge at the intermediate shear rate range. Feng et al (2014) showed that the rheological behaviour of activated sludge may be modelled using a Herschel – Bulkley, Bingham or power law model prior to thermal hydrolysis, and by a Newtonian model after thermal hydrolysis such that the behaviour changes from non – Newtonian to Newtonian. Urrea et al (2015) employed the Bingham model to investigate the rheological behaviour of activated sludge treated by thermal hydrolysis such that the fluid behaviour was transformed from a Bingham plastic to a Newtonian fluid after treatment. A modified combination of the Herschel – Bulkley model and the Bingham model was used by Baudez et al (2011b) and Ségalen et al (2015) as it takes into account the rheological behaviour of sludge over a wide shear rate range. This is fundamental because the non – Newtonian fluid behaviour cannot be described by the power law model in the high shear rate range where the viscosity remains higher than the water viscosity (Baudez et al., 2011b). The modified model (Eq. 14) includes a new parameter, α_o known as the "plateau viscosity" which describes when apparent viscosity tends to a plateau (i.e. limit) at high shear rates.

$$\tau = \tau_y + (K\dot{\gamma}^{(m-1)} + \alpha_0)\dot{\gamma} \qquad \text{Eq. 14}$$

In addition to employing the mathematical fluid models to describe the behaviour of sludge, several researchers have attempted to develop correlations between the rheological parameters such as the apparent viscosity, η, the yield stress, τ_y the flow index, n and the fluid consistency, K with characteristics such as total solids concentration (%TS), temperature (T) and bound water content. This is extensively reviewed by Eshtiaghi et al (2013b).

2.4.1.1 Apparent viscosity

Viscosity is defined as the ratio between shear stress to shear rate (Chhabra and Richardson, 2008) and is the fundamental parameter by which the physical characteristics of sludge are measured because it relates the deformation and flow properties of sludge (Chhabra and Richardson, 2008). The viscosity of Newtonian or highly diluted suspensions at a constant temperature and pressure can be described by Einstein's viscosity model (Seyssiecq et al., 2003, Eshtiaghi et al., 2013b, Genovese et al., 2007):

$$\eta = \eta_0(1 + 2.5\varphi) \qquad \text{Eq. 15}$$

, where η is defined as the apparent viscosity, η_o is the viscosity of the fluid phase and φ is the particle volume fraction.

The Einstein viscosity model has been used to describe the relationship between viscosity and particle concentration (Genovese et al., 2007, Marti et al., 2005). Einstein's viscosity equation means that the viscosity of a suspension increases with increasing particle concentration (Marti et al., 2005). Seyssiecq et al (2003) explains that increasing the particle concentration and reducing water content leads to an increase in the viscosity of sludges which means that theoretically, the Einstein viscosity model can be applied to sludge thickening/ dewaterability. Indeed, Eshtiaghi et al (2013b) highlights that whilst dilute sewage sludge exhibits Newtonian behaviour (Sanin, 2002), concentrated sludge exhibits non – Newtonian behaviour (Markis et al., 2014) whose flow properties including viscosity is highly dependent on the treatment process (Lotito et al., 1997, Battistoni, 1997).

Currently, most researchers have focused on relating what is known as the "limiting viscosity" to solids concentration (Tixier et al., 2003a, Pevere et al., 2006, Pevere et al., 2009) except Baudez et al (2011b) whom investigated the relationship between the Bingham viscosity and total solids concentration. Eshtiaghi et al (2013b) describes the limiting viscosity as the asymptotic value of the viscosity versus time curve at high shear rates whereby the apparent viscosity plateaus to a constant value. Similarly, Tixier et al (2003a) explains that the limiting viscosity is the viscosity of sludge corresponding to the maximum dispersion of flocs under shear. The limiting viscosity has been used to characterise various sludges such as digested sludge (Pevere et al., 2007, Pevere et al., 2009, Pevere et al., 2006), aerobic sludge (Riley and Forster, 2002, Su and Yu, 2005) , bioreactor sludge (Abu-Jdayil et al., 2010) and activated sludge (Tixier et al., 2003a, Tixier et al., 2003b).

2.4.1.1.1 Relationship between apparent viscosity and solids concentration

Various studies (Forster, 2002, Tixier et al., 2003a, Pevere et al., 2006, Mu and Yu, 2006, Moreau et al., 2009, Ma et al., 2014) have investigated the limiting viscosity as a function of solids concentration and found that it increased with solids concentration suggesting that increasing solids concentration led to an increase in structural units within the suspension, resulting in the formation of stronger inter – particle interactions. This led to a higher apparent viscosity experienced by sludge. Pevere et al (2006) also highlighted the importance of particle – particle interactions between the limiting viscosity and solids concentration by explaining that the limiting viscosity of sludge increased with decreasing particle size at a constant solids concentration due to the increased surface area allowing particles to interact with each other. Battistoni et al (1993), Tixier et al (2003a) and Abu –Jdayil et al (2010) modelled the limiting viscosity as a function of solids concentration using an exponential function. Ma et al (2014) studied the rheological behaviour of aerobic granular sludge and demonstrated that the limiting viscosity followed an exponential growth as a function of total suspended solids concentration. Ma et al (2014) employed an exponential model to demonstrate that the rheological behaviour reflected the internal structure in dense aggregated suspensions such as aerobic granular sludge and explained that the weak links in the flocs of aerobic granular sludge lead to a higher elasticity compared to those between neighbouring flocs. Battistoni et al (1993), Baudez et al (2011b) and Baudez et al (2013b) modelled the relationship between the Bingham viscosity and total solids concentration using an exponential model. Recently, Markis et al (2014) found that the apparent viscosity of primary and secondary sludge followed an exponential model. Lotito and Lotito (2014a) modelled the apparent viscosity using both an exponential model similar to Sanin (2002) and Tixier et al (2003a). Lotito et al (1997), Khalili – Garakani et al (2011) and Rosenberger et al (2002) found that it followed a power law function. Liu et al (2012) found that the apparent viscosity and fluid consistency of slurry fuel prepared by mixing municipal wastewater sludge and coal increased after the addition of sludge. Moreover, Liu et al (2012) found that the apparent viscosity increased with increasing concentration of the coal – sludge slurry. Dai et al (2014) studied the evolution of the rheology of sludge which has been through anaerobic digestion. Dai et al (2014) employed the fluid consistency as a measure of the apparent viscosity of sludge before and after it was anaerobically digested and demonstrated that it decreased after anaerobic digestion was carried out. No correlation was employed by Liu et al (2012) and Dai et al (2014) to predict the apparent viscosity.

Table 1 contains a summary of the equations as well as the type of sludge each correlation was applied to. As illustrated Table 1, the majority of research focuses on activated sludge.

Table 1: Summary equations describing the viscosity as a function of solids concentration

Author	Sludge type	Equation
Battistoni et al (1993), Baudez et al (2011, 2013), Markis et al (2014), Tixier et al (2003b), and Abu –Jdayil et al (2010), Tixier et al (2003a), Sanin (2002), Lotito and Lotito (2014a)	Activated sludge, digested sludge, primary, secondary sludge, granular sludge and rotating biological contactor (RBC) sludge	$\eta = a.e^{(b.C)}$ or $\eta_B = a.e^{(b.C)}$
Garakani et al (2011)	Activated sludge	$\eta = a.(C_p^b/\dot{\gamma})$
Rosenberger et al (2002) and Yang et al (2009)	Activated sludge	$\eta = a/c.(C_p^b/U_g)$
Saffarian et al (2011)	Activated sludge	$\eta_p = [1 - e^{(-m\,\gamma)}]^n\,\tau_B\,\dot{\gamma} + \eta_B$
Ma et al (2014)	Aerobic granular sludge	$\eta_\infty \sim CSS^{\frac{1}{(3-D_f)}}$

2.4.1.1.2 Relationship between apparent viscosity and Temperature

The temperature dependent behaviour of sludge is highlighted by Eshtiaghi et al (2013b) whereby an increase in temperature leads to a decrease in apparent viscosity (Battistoni et al., 1993, Abu-Jdayil et al., 2010) and (Sozanski et al., 1997, Mu et al., 2007, Baudez et al., 2013b, Farno et al., 2014, Farno et al., 2015, Lotito and Lotito, 2014a). However, Moreau et al (2009) explains that if the temperature range is not large, than the impact of temperature is negligible. The temperature dependent behaviour of sludge follows an Arrhenius type model:

$$\eta_\infty = Ke^{\left(\frac{E_a}{RT}\right)} \qquad \text{Eq. 16}$$

The Arrhenius model has been used to describe the relationship between temperature and limiting viscosity of various types of sludge including bioreactor sludge (Yang et al., 2009, Abu-Jdayil et al.,2010), anaerobic digested sludge (Battistoni et al., 1993) and diluted sludge (Sozanski et al., 1997). Eshtiaghi et al (2013b) explains that various modifications have been made to the Arrhenius model and to predict the limiting viscosity of sludge as a function of temperature. These are presented in Table 2. Baudez et al (2013b), Farno et al (2014) and Farno et al (2015) have recently objected to using the Arrhenuis equation to measure tempreature effects on viscsoity as the composition of sludge changes with temperature.

Table 2: Summary of equations describing the relationship between apparent viscosity and temperature

Author	Sludge type	Equation
Sozanski et al (1997)	Dilute sludge	$(WT)_1 = 1/(T - 273.45).[(\eta_B)_{273.45}/(\eta_B) - 1].100$
Dieude – Fauvel et al (2009)	Activated sludge	$\eta = a.\, e^{(b/T - T_a)} + c$
Jiang et al (2007)	sludge	$Ln(\eta/\eta_\infty) \approx a + b\,(T_o/T) + (T_o/T)^2$
Yang et al (2009)	Bioreactor sludge	$\eta = a.CSS_p^b.e^{E_a/R(T+273.15)}$
Garakani et al (2011)	sludge	$\eta = a.(CSS_p^b/\dot{\gamma}).e^{E_a/R(T+273.15)}$
Manoliadis and Bishop (1984), Lotito and Lotito (2014a)	Raw, activated and digested sludge	$\eta = a.e^{(-b.T)}$

Sozanski et al (1997) studied the impact of temperature on the Bingham viscosity and yield stress whilst Dieude – Fauvel et al (2009) developed an Arrhenius type model to predict the viscosity of sludge as a function of temperature. Ségalen et al (2015) validated the model developed by Dieude – Fauvel et al (2009) so that the infinite viscosity was proportional to the water viscosity allowing for it to be modelled using the Arrhenius type model with temperature. Feng et al (2014) and Urrea et al (2015) demonstrated that when activated sludge was thermally treated using thermal hydrolysis, the apparent viscosity was reduced due to the destruction of flocs and organic matter. Urrea et al (2015) showed that the apparent viscosity was reduced by two orders of magnitude and was modelled using an Arrhenius type equation for Newtonian fluids. In contrast, Jiang et al (2007) developed a model to estimate the relationship between temperature and viscosity in order to develop a hydrodynamic model for a membrane reactor. The relationship between viscosity, mixed liquor suspended solids of bioreactor sludge and temperature at a constant shear rate were describe using a correlation developed by Yang et al (2009). Khalil – Garakani et al (2011) modified this model to take into account the effect of shear rate on the apparent viscosity. Lotito and Lotito (2014a) observed that the fluid consistency, which is a measure of the apparent viscosity followed an exponential decay as a function of temperature at different concentrations. This was found to be in agreement with the work of Manoliadis and Bishop (1984). Most recently, Baudez et al (2013b) and Farno et al (2014) found that the thermal history experienced by sludge had a great impact on the viscosity such that the Bingham viscosity after heating and cooling increased. The proposed explanation by Baudez et al (2013b) suggests that the solids have dissolved, which is a partially irreversible process indicating that the Arrhenius type model cannot describe the relationship between temperature and apparent viscosity.

2.4.1.1.3 Relationship between apparent viscosity and bound water

As highlighted by Eshtiaghi et al (2013b), few researchers have studied the effect of bound water content on the limiting viscosity. Sozanski et al (1997) found that the viscosity decreased as the bound water content increased. Such behaviour was described by Forster (1983) and followed an exponential function:

$$\eta_L = \eta e^{(b.W_{kr} - W)} \quad \text{Eq. 17}$$

Liao et al (2000) explained that this behaviour was due to a change in floc structure and presence of extracellular polymeric substances (EPS) on the sludge surface.

2.4.2.2 Yield stress

In a review carried out by Barnes (1999) the definition of the yield stress as well as its existence was investigated. In two separate review papers focusing on sludge, Eshtiaghi et al (2013b) as well as Seyssiecq et al (2003) investigated the current literature on yield stress. The yield stress is generally defined as the minimum stress that is required in order for the material to flow continuously. Materials exhibiting a yield stress are defined as either viscoplastic or viscoelastic. The definition and existence of the yield stress is often debated in literature mainly due to the lack of equipment and experimental protocol required in determining its existence. Sludge has been described by various researchers as displaying a yield stress (Bhattacharya, 1981, Slatter, 1997, Baudez and Coussot, 2001, Baudez, 2008, Baudez et al., 2011b, Eshtiaghi et al., 2012b, Baudez et al., 2013a, Markis et al., 2014), however, there are still few researchers such as Moeller and Torres (1997) and Valioulis (1980) who demonstrate that sludge does not exhibit a yield stress. Barnes (1999) explains that whilst several rheological models such as the Bingham or the Herschel – Bulkley model can be used to predict the flow behaviour of sludge over a specific shear rate, it does not indicate that sludge is a yield stress fluid. In their review, Seyssiecq et al (2003) explained with current rheometric devices being more advanced, aggregated sludge does exhibit a yield stress and its quantitative knowledge is required in the design and optimization of various wastewater treatment unit operations such as pumping and mixing. Spinosa and Lotito (2003) highlighted the importance of the yield stress on various treatment operations such as stabilization, storage and transportation, dewatering and conditioning, agricultural use, land filling and incineration whereby the yield stress has an impact on storage and transportation whatever the type of sludge – liquid, paste or solid.

As summarized by Eshtiaghi et al (2013b), when investigating the sludge flow behaviour, two types of yield stresses have been observed – the first been the static yield stress and the second been the dynamic yield stress. The static yield stress has been defined as the stress corresponding to the transition between fully elastic and viscoelastic behaviour whereas the dynamic yield stress has been defined as the stress corresponding to the transition between viscoelastic and viscous behaviour. Eshtiaghi et al (2013b) explained that different types of yield stress were determined by researchers investigating rheology of sludge and state that the dynamic yield stress should be of most interest as it is the stress required for fully continuous flow.

Several rheometric techniques have been investigated regarding the determination of the yield stress. Nguyen and Boger (1992) and Liddel and Boger (1996) summarized the various techniques. Flow or dynamic measurements are the most common techniques on determining the yield stress. The yield stress is extrapolated from the flow curve measurements using rheological models such as the Herschel – Bulkely model (Slatter, 1997) or Bingham model (Mikkelsen, 2001, Lotito and Lotito, 2014a, Manoliadis and Bishop, 1984), or measured by performing an oscillatory strain or stress sweep tests at a constant frequency in the dynamic mode or, or by performing creep tests (Coussot, 2005, Baudez and Coussot, 2001, Baudez et al., 2011b, Markis et al., 2014). However, the former technique relies heavily on the accuracy of measurements, which is difficult to obtain due to wall slip, end and inertia effects.

Supata and Prost (1996), Baudez and Coussot (2001), and Baudez and Coussot (2004) have determined the yield stress using a combination of the flow and dynamic measurements whereby Supata and Prost (1996) found that the yield stress determined through oscillatory measurements was higher than that determined through flow measurements. In contrast, Mori et al (2006) found that static yield stress was higher (although in the same order of magnitude) and reasoned that the static yield stress corresponds to the start of flow whereas the dynamic yield stress corresponds to the point just before flow starts. In a study conducted using a similar experimental procedure, Wang et al (2011) demonstrated that the static and dynamic yield stress of conditioned and unconditioned sludge determined from flow and dynamic measurements correlated well. Similarly, Ayol et al (2006) performed flow and dynamic measurements on conditioned and unconditioned sludge. The dynamic yield stress was calculated by determining the critical amplitude of deformation, γ_c above which the linear viscoelastic region ends. Below this region, the complex modulus,

G_O^* is constant (i.e plateaus), however, above this region, the complex modulus, G_O^* is no longer constant and decays. As such the yield stress is calculated at the intercept between the complex modulus on the plateau and the critical deformation (i.e. $\tau_c = \gamma_c . G_O^*$). In a recent study, Jiang et al (2014) studied the effect of higher total solids concentration and temperature on the yield stress obtained from both dynamic and flow measurements. Jiang et al (2014) demonstrated that the yield stress obtained from the flow measurements (defined as the flow yield stress) increased following a power law as a function of total solids concentration. Similarly, Jiang et al (2014) demonstrated that the dynamic yield stress (also known as the critical modulus, G_o), determined from the intersection of the G' and G" curves increased with the total solids concentration following a power law model. By comparing the two different yield stresses (obtained from flow and dynamic measurements), Jiang et al (2014) observed that both yield stresses were within the same order of magnitude. Furthermore, Jiang et al (2014) showed that the dynamic yield stress was higher than the flow yield stress and attributed the difference to when flow begins in each mode. In the flow measurements, Jiang et al (2014) explained that the yield stress is determined when the sludge begins to flow, whereas in the dynamic mode, flow has already begun when the crossover between G' and G" occurs (due to dynamic solicitation). Furthermore, Jiang et al (2014) used the work of Baudez and Coussot (2004) to explain the small difference between the two different yield stresses whereby the transition between elastic solid to viscous liquid (when the imposed to a stress, strain or frequency increases) cannot be associated with the crossover region between G' and G". It is due spatial propagation of the interface between the solid and liquid phases (Baudez and Coussot, 2004).

Baudez et al (2008) and Markis et al (2014) performed creep tests on various sludge types using the procedure: pre – shear, rest, creep and the resulting behaviour was examined using the strain – time curve (i.e. creep curve). Different stresses were applied below and above the yield stress. For stresses below the yield stress (i.e. $\tau < \tau_c$), the creep curve remained constant with a decreasing slope. For stresses above the yield stress (i.e. $\tau > \tau_c$), the creep curve increased with a finite slope indicating that sludge flows steadily. Creep tests have been employed by Coussot (2005) to determine the yield stress of various suspensions, gels and pastes.

In fact, in the various techniques that have been outlined to determine the yield stress, the most accurate is the creep test. Coupled with the vane geometry, creep tests are able to estimate the yield stress accurately because wall slip, end and inertia effects are reduced. This indicates that it is essential to establish the appropriate technique as well as measuring apparatus, which is highlighted by Barnes (1999), Seyssiecq et al (2003) and Eshtiaghi et al (2013b).

2.4.2.2.1 Relationship between yield stress and solids concentration

The relationship between the yield stress and solids concentration of sludge has been investigated by several researchers and in all cases, the yield stress increases with increasing solids concentration (Mikkelsen, 2001, Riley and Forster, 2002, Forster, 2002, Spinosa and Lotito, 2003, Slatter, 1997, Baudez, 2008, Baudez et al., 2011b, Eshtiaghi et al., 2012b, Markis et al., 2014). Several correlations have been developed to describe the relationship between yield stress and solids concentration. Slatter (1997) used the correlation below in order to link the yield stress and the suspended solids concentration of digested sludge. Mori et al (2006) developed a correlation to describe the exponential increase of the dynamic yield stress of activated sludge over a wide range of solids concentrations. Lotito and Lotito (2014a), Battistoni et al (1997), Forster (2002), and Abu – jdayil et al (2010) employed an exponential correlation similar to Mori et al (2006). Baudez et al (2011b) showed that the relationship between yield stress and solids concentration of digested sludge followed a power law model. Markis et al (2014) verified the model employed by Baudez et al (2011b) experimentally using primary and secondary sludge over a wide range of total solids concentrations. Lotito and Lotito (2014a) demonstrated that a simpler power law model may be used. Liu et al (2012) found that the yield stress of fuel slurry prepared by mixing municipal sludge and coal increased after addition of sludge. Furthermore, Liu et al (2012) demonstrated that the yield stress increased with increasing concentration of coal – sludge sludge. Dai et al (2014) demonstrated that the yield stress of sludge decreased after it had been through anaerobic digestion. No correlations were employed by Liu et al (2012) and Dai et al (2014) to predict the yield stress. Jiang et al (2014) employed a power law correlation to predict the yield stress determined from two different measurements – flow and dynamic. Urrea et al (2015) showed that when activated sludge was treated using thermal hydrolysis, the increase in temperature reduced the total suspended solids leading to a reduction in the yield stress.

The correlations between yield stress and solids concentration are summarized in Table 3.

Table 3: Summary of equations describing the relationship between yield stress and solids concentration

Author	Sludge type	Equation
Slatter (2007)	Digested sludge	$\tau_y = a.(CSS^3/CSS_{max} - CSS)$
Mori et al (2006), Lotito and Lotito (2014a), Urrea et al (2015)	Digested sludge	$\tau_y = a.e^{(b.CSS)}$
Baudez et al (2011), Markis et al (2014), Lotito and Lotito (2014a), Jiang et al (2014)	Primary, secondary and digested sludge	$\tau_y = \alpha\,(C - C_{min})^m$, $\tau_y = a\,C^b$

2.4.2.1.2 Relationship between yield stress, temperature and bound water content

The relationship between yield stress and temperature is often described using an exponential model. Abu – Jdayil et al (2010) and Battistoni et al (1993) used an Arrhenius type equation (Eq. 18) to describe the effect of temperature on the yield stress of bioreactor sludge and anaerobic digested sludge.

$$\tau_y = c.e^{E_a/RT} \qquad \text{Eq. 18}$$

Ségalen et al. (2015) demonstrated that the yield stress followed a non – Arrhenius Vogel Tamman Fulcher (VTF) equation with temperature with the same activation energy:

$$\tau_y = A.e^{E_a/R(T-T_0)} \qquad \text{Eq. 19}$$

Manoliadis and Bishop (1984) modelled the relationship between the yield stress and temperature using an exponential model. This was validated by Lotito and Lotito (2014a), whom also demonstrated that the yield stress may be modeled using a simple power law model ($\tau_y = A.T^b$).

$$\tau_y = A.e^{-bT} \qquad \text{Eq. 20}$$

The relationship between the Bingham yield stress and temperature was developed by Sozanski et al (1997) and also demonstrated that the relationship between yield stress and bound water content followed an exponential model.

$$(WT)_2 = \frac{1}{T - 273.45}\left[\frac{(\eta_B)_{273.45}}{\eta_{B_T}} - 1\right]100 \qquad \text{Eq. 21}$$

$$\tau_y = c.e^{[d(W_{kr}-W)]} \qquad \text{Eq. 22}$$

, where T is the temperate, η_{B_T} is the Bingam viscosity at a known temperature, $(\eta_B)_{273.45}$ is the Bingham viscosity at the reference temperature, $(WT)_1$ is the water content, W_{kr} is the critical water content, W is the water content.

Forster (2002) studied the rheological and physico-chemical characteristics of sewage and was able to develop a rule that described the influence of water content and yield stress.

$$Water\ content = a.\ln\tau_y + b \qquad \text{Eq. 23}$$

, where τ_y is the yield stress and a and b are model parameters.

2.4.2.1.3 Impact of sludge surface charge on apparent viscosity and yield stress of sludge

The works of Forster (1981, 1982, 2002) illustrate the relationship between the surface chemistry and rheological properties. According to Forster (1982) and (2002), the non – Newtonian behaviour of sewage sludges is related to the materials surface chemistry, so the surface charge carried by the component particles. Forster (1982) studied activated, anaerobically digested and aerobically digested sludges and found that the relationship between surface charge and rheological properties is controlled by the ionic strength of the suspending fluid as well as the chemical nature of the sludge surfaces. For activated sludge, Forster (1982) found that polysaccharides influenced the surface charge. By studying the surface polysaccharide content, Forster (1982) found that the viscosity was reduced (through the addition of cellulose). Hence, the influence of polysaccharide on surface charges is significant. Forster (1982) was unsuccessful in determining the relationship between surface charge and rheological properties for other sludge types and emphasised the need to research the surface chemistry of sludge and its influence on the rheological properties. No model was developed to describe the relationship between surface charge and viscosity of activated sludge. Forster (2002) developed a model that linked the surface charge and yield stress of sludge.

$$Surface\ charge = -a \ln \tau_y + b \qquad \text{Eq. 24}$$

, where τ_y is the yield stress and a and b are model parameters.

Sanin (2002) examined the influence of various properties such as pH, conductivity, solids concentration and flocculation on the rheology of activated sludge. The rheograms were fit using the power law model (Ostwald model). Sanin (2002) observed that the viscosity increased with increasing solids concentration. It was also found that the viscosity increased with increasing pH. This was due to the increased negative charge on flocs which increases repulsion leading to the expansion of floc matrix. Increasing the conductivity meant that the apparent viscosity decreased. Sanin (2002) argued that this was due to the compression of the electrical double layer around particles which results in a more compact floc structure.

2.4.3 Thixotropy

As explained in section 2.3.3 non – Newtonian fluid behaviour, thixotropy refers to the time dependent shear thinning behaviour of fluids. Barnes (1997) refers to thixotropy as the "reversible changes from a flowable fluid to a solid like elastic gel". Eshtiaghi et al (2013b) summarized that various researchers (Battistoni, 1997, Tixier et al., 2003b, Tixier et al., 2003a, Baudez, 2006, Baudez, 2008) have defined thixotropy as the time dependent disintegration of the internal structure of sludge as a result of applied shear stress.

Baudez (2008) explained that below a critical shear stress, colloidal forces dominate resulting in the restructuration (i.e. physical aging) of structure of sludge whilst shearing forces result in the breakdown of the solid structure (shear rejuvenation). Baudez (2008) demonstrated that once the critical shear stress has been reached, the solid structure breaks down completely so that sludge flows steadily following a truncated power law fluid model. More recently, Markis et al (2014) demonstrated that when primary sludge is subjected to a stress below the yield stress and once the applied stress is removed (and the sludge is allowed to rest), it undergoes physical aging. The behaviour is dependent on the strength of the network structure (where colloidal forces dominate) as well as the applied stress, regardless of the amount of time it is at rest. However, Markis et al (2014) found that secondary sludge exhibited shear rejuvenation and physical aging. When secondary sludge is allowed to rest (i.e. applied stress is removed) for a short period of time, hydrodynamic forces are dominant which keep the structure broken (i.e. sludge exhibits shear rejuvenation) and result in deflocculation. In contrast, after a prolonged time of rest, colloidal forces dominate; the secondary sludge undergoes physical aging whereby the network structure becomes stronger which results in flocculation. In the same study, Markis et al (2014) performed hysteresis loop on dilute and thickened primary and secondary sludge. The hysteresis loop of both dilute primary and dilute secondary sludge were superimposed illustrating that shear dependent thixotropic behaviour was non-existent. Thickened primary sludge displayed a large area of the hysteresis loop; in contrast, the hysteresis curve of thickened secondary sludge remained superimposed. Such behaviour suggests that primary sludge exhibits shear dependent tendencies which increase with increasing solids concentration. Secondary sludge did not display shear dependent behaviour. Tixier et al (2003a) and (2003b) studied the thixotropic behaviour of sludge using the hysteresis loop and found that it varied depending on the type of sludge. In another study, Baudez (2006) reconstructed the velocity profile of sewage sludge such that the solid and liquid behaviour was modelled. The correlation used by Baudez (2006) contained a structural parameter, λ (measured as a function of time) which was used to characterise the time dependent behaviour of sludge. In fact, several researchers (Labanda et al., 2004, Dullaert and Mewis, 2005, Rosales and Hernández, 2006, Mewis and Wagner, 2009) have employed this parameter when investigating the time dependency of yield stress fluids. The structural parameter λ is known as the extent of structural build up such that when $\lambda = 0$ the structure is completely broken and when $\lambda = 1$ the structure has rebuilt itself completely (Cheng and Evans, 1965). Baudez (2006) demonstrated that the measured stress (which reflects the strength of the sludge) increased with increasing time of rest (between pre – shear and creep). Baudez (2006) compared the velocity profile data with that obtained from the hysteresis curve and illustrated that the hysteresis loop was an artefact of the rheometric procedure and its accuracy depends on the rheometer. More recently, Eshtiaghi et al (2012b) performed step stress experiments to compare the thixotropic behaviour of digested sludge with model fluids first by applying a pre – shear (900s, stress corresponding to $200s^{-1}$) then imposing a high stress (corresponding to $100s^{-1}$), followed by a low stress (corresponding to $5s^{-1}$); the previous stress (corresponding to a 100^{-1}) was then reapplied. Eshtiaghi et al (2012b) found that there was a 17% difference between the shear rate data obtained from the same applied stress (corresponding to $100s^{-1}$). This illustrates the shear dependency of digested sludge, especially when it is subjected to different shear stresses.

The above mentioned research highlights the inconsistency associated with investigating the thixotropic behaviour of sludge. Ruiz – Hernando et al (2015) developed a model to describe the thixotropic behaviour of waste activated sludge after it was thermally treated using a thermal specific energies (E_s). The thixotropic model was based on the time dependent structural parameter; S. This parameter could measure any changes in the internal structure and arrangement of the flocs at any time and shear rate. Ruiz – Hernando et al (2015) demonstrated that the kinetic coefficients of the break up and build up process were used to measure the thixotropic behaviour of sludge. Furthermore, Ruiz – Hernando et al (2015) showed that the application of E_s reduced the steady state viscosity and the kinetic coefficients of the break up and build up process indicating that the thixotropy increased. The inconsistencies in the literature suggest that the thixotropic behaviour of sludge is difficult measure as there are various techniques which can employed, as highlighted above (i.e. creep tests at various time of rest, hysteresis loops, reconstructing the velocity profile or step stress tests) and no exact protocol. The above mentioned studies also highlight that it is difficult to interpret whether sludge is time dependent, shear dependent or both. In fact, Seyssiecq et al (2003) showed that although several researchers have studied the thixotropic behaviour of sludge, few have attempted to model it as it is difficult to due to the variations in the rheometric technique. In reality, thixotropy is a very difficult characteristic to understand and control and has a direct impact of real life applications such as sludge transportation and mixing.

Eshtiaghi et al (2013b) and Ratkovich et al (2014) have highlighted that thixotropy has a major impact on the transportation of sludge in pipelines mainly causing blockage throughout the line when the shear stress is not sufficient enough to maintain continuous, homogenous flow. When sludge is pumped and transported for long periods of time, its behaviour is altered dramatically. As such, changes in the flow behaviour as a function of time must be taken into account in the design of pipelines and pumping systems (i.e. transportation units). Baudez (2006) emphasizes that the time dependency of sludge also influences the hydraulics of mixing tanks and reactors because the sludge undergoes restructuration at prolonged retention times. This results in an increase in size of unmixed regions (known as dead zones) which is detrimental and hinders the mixing process (i.e. efficient mixing). As such, understanding the thixotropic behaviour is vital in optimizing unit operations of the sludge treatment process.

2.4.3 Oscillatory measurements

The viscoelastic behaviour of sludge was investigated by applying a sinusoidal deformation and measuring the resulting sinusoidal stress (or strain) such that the storage (G') and loss modulus (G"). As such, the amount of energy stored and dissipated during deformation was obtained (Chhabra and Richardson, 2008).

Ayol et al (2006) completed oscillatory measurements on conditioned and unconditioned sludge and showed that in the linear, viscoelastic region, G'>G", and that beyond this region, the elasticity was reduced such that G" > G'. Chen et al (2005) focused on the influence of polymer addition on the coagulation of sludge and found that the addition of coagulant polymer influenced the complex modulus of sludge. In fact this is true, Wang et al (2011) illustrated that the addition of polymer caused a change in the network strength of the flocs leading to the formation of bridges between the cationic polymers and negatively charged particles resulting in a more rigid and solid like sludge structure. This caused an increase in the storage modulus. Wang et al (2011) used frequency sweeps to demonstrate a cross over region whereby G'>G" followed by G">G' indicating a transition of elastic to viscous; this trend was also found in solids and pastes. Low viscous sludges displayed gel like behaviour (and therefore a higher elasticity) at high shear rates in the linear viscoelastic region such that more energy was stored in the rigid structure of the conditioned anaerobic sludge (Wang et al., 2011). Eshtiaghi et al (2013a) observed similar behaviour on unconditioned digested sludge so that when G’>G”, digested sludge behaves as an elastic solid, however, when G”>G’, the flocs breakdown and digested sludge behaves as a viscous liquid. The similarities between sludge (anaerobic digested and raw) and soft glassy materials was investigated by Baudez et al (2011a) and (2013a) whereby G’ and G” were constant in the linear viscoelastic region and G’>G” followed by a crossover region in which G” reached a maximum then G”>G’. Such behaviour is the hallmark of soft glassy materials indicating that the viscoelastic behaviour of sludge can be modelled using model fluids such as gels, emulsions and suspensions (Baudez et al., 2013a). More recently, Jiang et al (2014) performed oscillatory measurements on highly concentrated sludge (at different total solids concentrations and temperatures). Jiang et al (2014) demonstrate that when highly concentrated anaerobic sludge is subjected to low strain, G’ and G” were nearly constant corresponding to the linear, viscoelastic regime. However, beyond a critical strain, G’ decreases whilst G” follows an initial peak then decreases corresponding to the viscous (liquid) regime. Jiang et al (2014) showed that the critical strain corresponds to the transition between the viscoelastic solid and viscous liquid regimes and used the critical modulus, G_c (cross over between G’ and G”) to observe the impact of total solids concentration on the viscoelastic behaviour. Jiang et al (2014) demonstrated that G’ and G” increased with increasing total solids concentration following the same trend such that the G_c increased with total solids concentration following a power law. Furthermore, Jiang et al (2014) calculated the energy of adhesion and showed that it increased with total solids concentration indicating that the interactions within the structure of anaerobic digested sludge increased with increasing concentration. As such, G’ and G” increased with total solids concentration. Jiang et al (2014) demonstrated that the temperature only had a small impact on the viscoelastic behaviour (compared to the impact of total solids concentration) and attributed this to the strengthened network structure arising from to increased internal interactions and steric links when anaerobic sludge is highly concentrated (>8%TS). Ma et al (2014) observed a similar to Jiang et al (2014) such that below a critical strain, aerobic granular sludge behaved as a linear viscoelastic solid (i.e. LVE) and above the critical strain, it behaved as a viscous liquid. Ma et al (2014) demonstrated that in the non – linear viscoelastic regime (non – LVE), G’ and G" followed a power law type function ($G' \infty\, \gamma^{-2n} and\, G" \infty\, \gamma^{-n}$), which is known as a hallmark of soft glassy materials and has been observed by Eshtiaghi et al (2013a) and Baudez et al (2013a). Ma et al (2014) performed frequency sweeps in the line LVE and non – LVE regimes and showed that in the LVE regime, G' and G" followed a similar power law response whereby G' >G" indicating that it was in the solid regime. In the non – LVE regime, G">G' indicating that that the granular sludge was in the liquid regime. The decrease in the viscosity in the LVE and non – LVE regimes indicated that the granular sludge exhibited both viscoelastic and shear thinning behaviour (Ma et al., 2014). Moreover, aerobic granular sludge was found to be thermally stable since there was no change in G' and G". Ma et al (2014) showed an optimum condition for cultivating aerobic granular sludge at 25 °C corresponding to maximum structural strength. Lastly, Ma et al (2014) employed a Wagner type constitutive model incorporating the relaxation and damping functions to predict the viscosity. Ségalen et al (2015) studied the impact of temperature on the relationship between electrical and rheological properties and observed that the storage modulus followed a Vogel Tamman Fulcher (VTF) model.

In contrast, Seviour et al (2009) developed a protocol in order to characterize granular sludge indicating that the macromolecular association was responsible for the formation of granular sludge under various environmental conditions as well as the yield point; this meant that the hydrogel gel properties of granular sludge were identified. This protocol could be used in flocculation in wastewater treatment and could explain the difference between granular and floccular sludge based on the solgel transition of the EPS obtained from sludge (Seviour et al., 2009b).

The reviewed literature, also presented in Eshtiaghi et al (2013b) indicates that the current literature linking sludge and its viscoelastic properties is indeed limited and that the available literature is not reliable. Whilst the above mentioned literature attempts to investigate the viscoelastic behaviour of sludge in the linear and nonlinear regimes, other than the work completed by Seviour et al (2009), little is known on the exact protocol required to correctly measure such complex behaviour. There is also little knowledge on how to interpret and model the obtained data. This is also explained by Eshtiaghi et al (2013b), who states that oscillatory measurements should be incorporated into the research on sludge rheology, however; an in – depth analysis into the experimental protocols as well as data analysis is required prior to making any conclusions on the research.

2.5 Sludge rheometry

The rheological properties of non-Newtonian fluids such as suspensions and sludge are determined using various rheometers. Flow measurements are used to determine the rheogram (plot of shear rate versus shear stress); this is linked to the viscosity of the fluid. The various rheometers that can be used to determine the rheological properties of a fluid are rotational, tube or systemic rheometers. However, due to the complex nature of sludge as well as rheometer sensitivity, no universal rheometer and technique has been used to characterise rheology of sludge.

The concentric cylinder geometry is the most commonly used rotational rheometer for sludge rheology. The concentric cylinder geometry consists of a cup and a bob with one of the two rotating at a constant rate. The shear stress is determined by measuring the resistant torque of one of two cylinders. Rotational rheometers equipped with the concentric geometry are most commonly employed for sludge studies. Campbell and Crescuolo (1982), Forester (1982) and Monteiro (1997) used the rotational rheometers to analyse the flow properties of various types of sludge. Mori et al (2006) studied the influence of geometry on the rheological characterisation of sludge and found that the concentric cylinders were the most suitable geometry compared to the double concentric cylinders. The double concentric cylinder geometry could not characterise sludge due to the size of its measuring gaps (Mori et al., 2006). Novarino et al (2010) employ the Anton Paar Physica rheometer equipped with the coaxial cylinders to determine the rheology of sludge whilst taking into account heterogeneous composition and interaction between solid-solid and solid-water particles. For their study of thixotropic behaviour of sludge, Tixier et al (2003a) employed the Anton Paar Physica rheometer equipped with the double gap measuring system. Spinosa and Lotito (2003) develop procedures for yield stress determination using the conventional rotational viscometer (Haaka Rheotest RV

2.1). Baudez et al (2004) employed the Paar Physica MC1+ equipped with either parallel plates or large coaxial cylinder geometry. Both these geometries were fitted with rough surfaces to avoid wall slip. Lotito et al (1997) worked with the rotational rheometer equipped with the concentric cylinder geometry for their study of sludge at different steps of treatment. Baudez and Coussot (2001) works with a larger annular gap to avoid the problems associated with size of suspended particles. However, from the above literature there is still little consistency between the geometry, sludge type and its rheological parameters.

Tube rheometers such as a capillary rheometer have also been employed for sludge analysis such as Slatter (1997) and Seyssiecq et al (2015). Slatter (1997) and Eshtiaghi et al (2013b) reviews the advantages and disadvantages of using a rotational or tube rheometer for sludge characterisation. This is summarised in Table 4.

Table 4: Advantages and disadvantages of rotational or tube rheometer (Slatter, 1997, Eshtiaghi et al., 2013b)

	Advantages	**Disadvantages**
Tube rheometers	Mechanically simple	Shear rate of a sample varies across the tube cross section
	Performs as a miniature pipeline	Time dependent fluids cannot be measured
	Operates at high shear rates	Large sample volumes required
	Measures laminar/ turbulent transition	
	Measures the effect of different diameter size on material	
Rotational rheometer	Rheology of time dependent fluids can be measured	Annular gap must be greater than the size of the largest particle. However, the annular gap is usually small in order to avoid correction factors as well as avoid turbulence
	Commercially accepted and most common form of rheometer. Installed as a "bench top" instrument that can be connected to the PC. Hence, the rheogram is obtained directly.	Non Newtonian fluid effects must be taken into account
	Small sample volumes are required	Laminar/ turbulent transition
		Shear migration of particles within the measuring gap

Tube viscometers have been employed by Slatter (1997) and Seyssiecq et al (2015) in order to correlate sludge rheological properties with sludge pumping processes. The balance beam rheometer has been used by Slatter (1997) as it reduces the problems associated with size of flocculated structures. Slatter (1997) discusses the advantages of using a balance beam rheoemeter such that the flow is measured more accurately from mass. Battacharya (1981) employed a pipe loop in order to rheologically characterise primary and digested sludge. Poitou et al (1997) studied the rheological and mechanical behaviour of pasty sludges using a capillary rheometer. Poitou et al (1997) also used parallel plates in their studies. The rheological properties of filamentous microorganism's sludges were studied by Allen and Robinson (1990) using three different capillary rheometers of varying sizes in order to determine rheometer performance. This literature suggests that tube viscometers are can be used to determine the flow properties of sludge samples, although the particle size of sludge can cause blockage in capillary tubes, hence upscaling to pipe loops is a suggestion.

Systemic rheometers are rheoreactor vessels equipped with either a vane or helical ribbon stirring device. In the vane geometry, a vane rotates at a rate N in a volume of fluid V. The relationship between the power drawn from the stirrer and the shear rate is defined according to the equation below (Seyssiecq et al., 2003).

$$\dot{\gamma} = \sqrt{\frac{P}{\eta V}} \qquad \text{Eq. 25}$$

, where η is the apparent viscosity. $\dot{\gamma}$ is the shear rate, P is the power drawn by the stirrer and V is the volume of the fluid.

Currently, there is no specific rheometer used to determine the rheological properties of sludge mainly due to the complex nature of sludge as well as the errors associated with each of the rheometers. Hence, there isn't a specific "universal" rheometer that can be used to determine the rheological properties of sludge. For rotational and systemic rheometers equipped with wide gap geometry, there are errors associated with the calculation of shear rate and shear stress within the gap especially for yield stress fluids whereby the fluid is not completely flowing because the yield stress has not been overcome. The calculated viscosity is an incorrect representation of the fluid viscosity. In order to avoid such problems, small gap geometries are used. However, these geometries themselves lead to errors when measuring rheological properties of sludge because the floc size is often greater than the gap size. This also leads to incorrect shear stress/ shear rate data. Another problem is the settling of particles in suspension during rheological measurements. This means that incorrect data is measured. Settling can be avoided through the use of vane geometry. Therefore, due to the nature of the sludge and sensitivity of the rheometer, it is almost impossible to rheologically characterize sludge using a universal technique. The rheology depends on the material history as well as the sensitivity of the rheometer.

2.6 Knowledge gap

The presented literature review on sludge rheology highlights that there is extensive research focusing on investigating the rheological behaviour of secondary or activated sludge as well as digested sludge. There are a few studies on the rheological characterization of primary sludge (Bhattacharya, 1981, Moeller and Torres, 1997) and both sets of research provide contradictory results. Whilst Bhattacharya (1981) described primary sludge as a shear thinning, yield stress material, Moeller and Torres (1997) detected no yield stress. Further inconsistencies are presented by the work of Bhattacharya (1981) as digested sludge is described as a dilatant material, which contradicts current literature (Baudez et al., 2011b, Eshtiaghi et al., 2012b, Slatter, 1997). The contradictory results between the works of Bhattacharya (1981) and Moeller and Torres (1997) emphasizes that an accurate procedure is required to estimate the flow behaviour of primary sludge. Additionally, there are no known correlations to accurately predict the flow behaviour, most notably, the apparent viscosity and yield stress of primary sludge over a wide range of total solids concentrations. Furthermore, no comparisons have been made between the rheology of primary sludge with secondary or digested sludge so that universal correlations may be employed to predict the apparent viscosity and yield stress of any sludge (regardless of type) as a function of total solids concentration. In this, way, the correlations will allow the flow behaviour of any sludge (and known total solids concentration) to be accurately predicted and estimated regardless of the treatment process it has experienced.

The presented literature review highlights that the current research focuses on applying the flow parameters of individual secondary and digested sludge to design and optimize unit operations such as pipelines, heat exchangers, storage tanks or dewatering units. Currently, there is no known study which investigates the flow behaviour of different types of sludge as a feed to the anaerobic digestion process. Additionally, except for the experimental work of Barourian et al (2013) on primary and secondary sludge mixtures, there are no known studies focusing the rheology of different types of sludge mixtures. As such, there are no known studies investigating how and why the flow behaviour of different types of sludges changes prior to and after mixing whereby primary and secondary sludges are used as the feed to the anaerobic digestion process. Any changes to this feed prior to and after mixing may influence the digester performance. No correlations have been developed to predict the apparent viscosity and yield stress of sludge mixtures. This highlights that it is necessary to study the flow behaviour of sludge mixtures, focusing on the evolution of the apparent viscosity and yield stress as these are the two most important parameters influencing the efficiency of anaerobic digestion. In return, the research will provide an insight on how and why the flow behaviour changes prior to and after mixing and whether or not these changes alter the efficiency of digestion by monitoring the apparent viscosity and yield stress of sludge mixtures.

In the project which was undertaken, experimental procedures have been developed to characterize the flow behaviour of primary, secondary and digested sludge as well as sludge mixtures. Master curves were developed to predict the flow behaviour of sludge as well as apparent viscosity and yield stress models to predict the flow behaviour of individual sludges as well sludge mixtures from the flow behaviour of the individual sludge.

2.7 Conclusion

In this chapter, sludge is defined as the by – product of the treatment of municipal wastewaters and explained that there are three different types of sludge depending on the treatment process the sludge has been subjected to – primary, secondary and digested sludge. It is explained that the rheological properties of sludge change as the sludge flows through the sludge treatment process and that these properties are also influenced by various factors such as total solids concentration, temperature, and water content and particle interactions. As such, predicting the flow behaviour of sludge accurately is essential, however, whilst several researchers have attempted to model the apparent viscosity, yield stress, thixotropic and viscoelastic properties as a function of total solids concentration, temperature, and water content and particle interactions, there are many inconsistencies. These inconsistencies are mainly a combination of rheometric technique and data analysis of the results. However, in all cases, sludge is defined as a complex, non – Newtonian, shear thinning fluid, exhibiting a yield stress. The non – Newtonian behaviour increases with increasing total solids concentration and decreases with temperature.

The presented literature shows that there are no known studies investigating the rheology of different types of sludge prior to and after it is mixed so that any changes may be observed and accounted for. The presented literature also highlights that there are no studies linking how these rheological changes may influence the performance of anaerobic digestion.

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CHAPTER 3

MATERIALS AND METHOD

Chapter 3: Materials and Method

3.1 Sample preparation

3.1.1 Sludge sampling

Sludge was sampled from various locations in Australia and France. In Australia, primary secondary and digested sludge was sampled from either Mount Martha wastewater treatment plant (WWTP, Mornington Peninsula, Victoria) or from the Eastern Treatment Plant (ETP, Bangholme, Victoria). In France, samples of primary sludge were obtained from Bessay (Allier, France) and secondary sludge was obtained from Vichy (Allier, France). A summary of the samples and their location is provided in Table 5.

Table 5: Summary of sludge type and location

	France	Australia
Primary sludge	Bessay (Allier)	Eastern Treatment plant (ETP, Bangholme, Victoria)
Secondary sludge	Vichy (Allier)	Mount Martha WWTP (Mornington Peninsula, Australia)
Digested sludge	N/A	Mount Martha WWTP (Mornington Peninsula, Australia)

Due to the organic nature of sludge, the samples were stored at 4 °C for 30 days prior to experiments to avoid any changes in the composition during rheological tests and to ensure the same material is always used throughout all our experiments (Baudez et al., 2011b).

3.1.1.1 Sludge thickening

In both Australia and France, sludge (at a low total solids concentration) was thickened to various concentrations using the vacuum filtration technique (Figure 7).

Figure 7: Vacuum filtration technique

In France, secondary sludge was collected with a total solid concentration of 22% at the outlet of the dewatering centrifuge step of a wastewater treatment plant. These samples were diluted to various concentrations.

3.1.1.2 Total solids concentration measurements

A standard operating procedure was used (APHA, 1992) to measure the total solids concentration of the sludge sampled in both Australia and France. This test was performed in triplicate to ensure reproducible total solids concentration was measured. To ensure a representative sample was selected for this test, the bulk of sludge was thoroughly hand mixed for sixty seconds prior to taking sample.

The total solids concentration of the sludge was measured as follows:

1. The mass of an empty weighing dish ($m_{(dish,\ empty)}$) was measured using a balance.
2. The balance was then zeroed.
3. The sample was hand mixed for sixty seconds, and then a sub – sample was placed into the dish (using a spatula).

4. The mass of the wet sludge ($m_{(wet\ sludge)}$) was measured using the balance (remembering that the balance was zeroed).
5. The weighing dish containing the wet sludge was then placed in a standard industrial oven (FD Series, BINDER Inc.) operating at 105 °C to dry overnight.
6. After drying, the weighing dish was placed in a desiccator to equilibrate to room temperature. Once, at room temperature, the mass of the dry sludge plus the weighing dish ($m_{(dry\ sludge+dish)}$) was measured.
7. This procedure performed three times

The following equation was used to calculate the total solids concentration:

$$\%\ total\ solids = \left[\frac{m_{(dry\ sludge+dish)} - m_{(dish,empt)}}{m_{(wet\ sludge)}}\right].100 \qquad \text{Eq. 26}$$

3.1.1.3 Sludge mixing

This section contains a summary of the procedure in which sludge samples were mixed together.

3.1.1.3.1 Primary and secondary sludge mixtures

First mixtures of primary and secondary sludge were prepared by adding secondary sludge to primary sludge over a wide range of volume fraction (between 0 – 1.0). The mixtures of primary and secondary sludge were first prepared at the same total solids concentration of individual primary and secondary sludge; this was followed by altering the total solids concentration of primary and secondary sludge in the mixture. In this way, the influence of total solids concentration on the apparent viscosity and yield stress of sludge mixtures was determined. Table 6 and 7 contains the total solids concentration and volume fraction required to make up the mixtures.

Table 6: Summary of total solids concentration required for primary and secondary sludge

	Primary sludge (%TS)	Secondary sludge (%TS)
France	5.0	5.0
	5.4	2.8
	2.8	5.4
Australia	3	3
	4	4
	5.1	5.1
	6.5	6.5
	7.1	7.1
	2.5	5.3

Table 7: Summary of volume (and resulting volume fraction) required to mix primary and secondary sludge

Primary sludge (mL)	Secondary sludge (mL)	Volume fraction (follow secondary sludge)
0	100	1
10	90	0.9
30	70	0.7
50	50	0.5
70	30	0.3
90	10	0.1
100	0	0

3.1.1.3.2 Primary and secondary sludge mixed with digested sludge

First, an equal volume (mL) of primary sludge (V_P) was mixed with an equal volume (mL) of secondary sludge(V_S), to make up a final volume of the mixture (In other words: $V_P + V_S = V_{mixture}$ mL). Then, the mixture was added to digested sludge (V_D) over a wide range of volume fraction (0-1.0) of digested sludge.

Similar to the mixtures of primary and secondary sludge, first mixtures of a 50:50 (v/v) primary and secondary sludge were added to digested sludge at the same total solids concentration of the individual sludge. Then the total solids concentration of the mixtures was varied so that the impact of total solids concentration and volume fraction on the yield stress and apparent viscosity could be studied.

Table 8 and 9 contains a summary of the total solids concentration and volume fractions required to make up the blends.

Table 8: Summary of total solids concentration required for primary and secondary sludge

	Primary sludge (%TS)	Secondary sludge (%TS)	Digested sludge (%TS)
Batch 1	1.8	1.8	1.8
	4.2	4.2	4.2
	7.0	7.0	7.0
	5.4	5.4	1.8
	1.8	1.8	5.4
Batch 2	3	3	3
	4	4	4.
	5.1	5.1	5.1
	6.3	6.3	6.3
	7.1	7.1	7.1
	4.5	4.5	1.6

Table 9: Summary of volume (and resulting volume fraction) required to mix primary, secondary and digested sludge

Primary sludge (mL)	Digested sludge (mL)	Volume fraction (follow digested sludge)
0	100	1
10	90	0.9
30	70	0.7
50	50	0.5
70	30	0.3
90	10	0.1
100	0	0

3.2 Rheometric technique

3.2.1 Rheometer

Two types of rheometers were used to perform the rheological measurements. In Australia, a discovery hybrid rheometer (HR – 3) was used; in France, an Anton Paar physica MCR 300 dynamic stress rheometer was used.

The discovery hybrid rheometer (HR – 3) was equipped with a wide gap vane geometry (vane diameter, D_v = 15 mm, cup diameter, D_c = 30 mm and vane height, H = 38 mm). Similarly, the Anton Paar physica MCR 300 dynamic stress rheometer was equipped with the wide gap vane geometry (diameter of the cup, D_c = 39.0 mm; diameter of the vane, D_v = 25.0 mm, height of vane, H = 70.0 mm). Figure 8 illustrates the vane geometry.

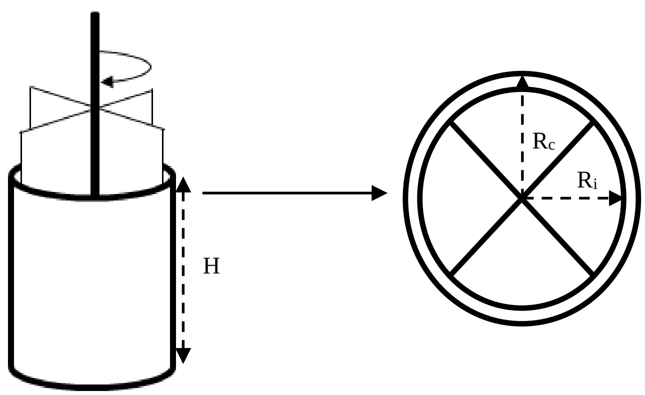


Figure 8: Illustration of vane geometry

In both cases, the vane geometry was used to reduce inertia and end effects (Dzuy and Boger, 1985). Tool surfaces were roughened to avoid wall slip (Baudez, 2008).

3.2.2 Rheometric measurement

Various rheometric measurements were performed to investigate the solid, liquid, and yielding and shear and time dependent behavior of sludge.

3.2.2.1 Creep test

Creep tests were performed in order to investigate the solid, liquid and yielding behaviour of sludge.

First, the sludge was pre – sheared for a specific amount of time at a shear stress corresponding to a high shear rate, depending on the solids concentration of sludge in order to obtain a homogenous material that is always in the same initial state of destructuration (Baudez, 2008, Coussot, 2005). This was followed by a certain period of rest to allow the structure to rebuilt (Coussot, 2005). Then, a stress (below the yield stress) was applied for duration of time. This was followed by succussive increasing steps of creep (at a constant increasing stress) to cover the solid and liquid regime. Figure 9 illustrates a typical creep curve. Table 10 contains the typical test conditions for sludge sampled in France and Australia.

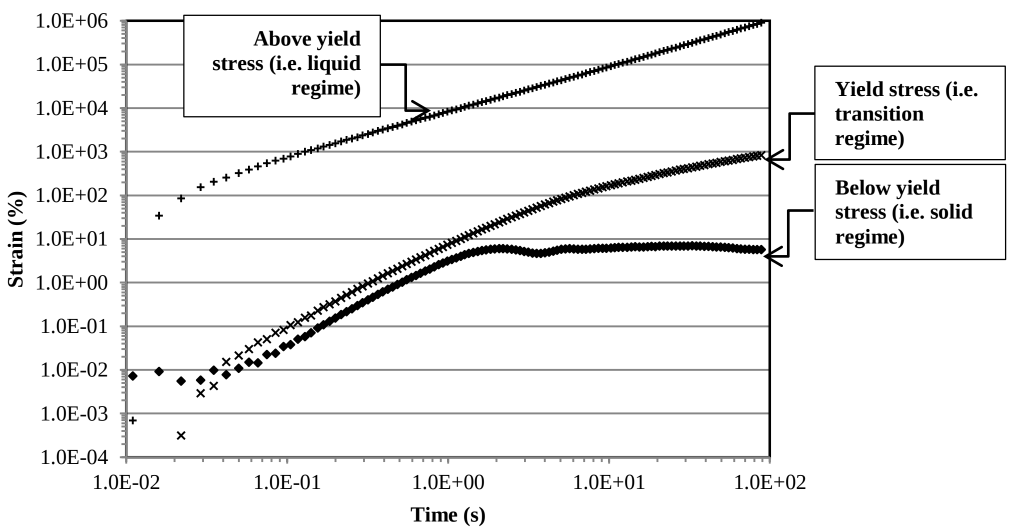


Figure 9: Typical creep curve for sludge

Table 10: Summary of typical operating conditions required to perform the rheological measurements

Location of rheological measurements	Duration of pre-shear (s)	Duration of rest (s)	Duration of creep (s)
France	150	150	88.6 or 154
Australia	150	150	120

The flow curves were obtained from the creep curve data for applied stresses above the yield stress (i.e. corresponding to the liquid regime). The shear stress and shear rate were recalculated using the derivatives of the torque (M) – deflection angle (θ) curve because the wide gap vane geometry was employed. First, the angular velocity (ω) was calculated using:

$$\omega = \frac{\theta}{t} \qquad \text{Eq. 27}$$

The shear stress at the surface of the rotating vane (τ_{Ri}) and the shear rate at the surface of the rotating vane (γ_{Ri}) were then calculated using:

$$\tau_{R_i} = \frac{M}{2\pi H R_i^2} \qquad \text{Eq. 28}$$

$$\dot{\gamma}_{R_i} = \left[2\left[\frac{R_0^2}{\left(R_0^2 - R_i^2\right)^2}\right]\omega\right] \qquad \text{Eq. 29}$$

, where D is the deflection angle (rad), t is the time (s), M is the torque (Nm), H is the height of the vane (m), R_i and R_o are the radii of the rotating vane and cup (Mezgar, 2006).

3.2.2.2 Shear dependent behaviour

The shear dependent behaviour of a fluid is typically measured using increasing and decreasing steps of stress. The shear stress and shear rate data required to plot the flow curve (corresponding to the increasing and decreasing steps of stress) was than recalculated (using Eq.27 – 29) so that a hysteresis loop was developed. The area within the hysteresis loop depends on the shearing time and shear hysteresis that the fluid has been subjected to. As such, the larger the area within the loop, the more shear – dependent (and thixotropic) the fluid is. However, if the increasing and decreasing flow curves are superimposed, then the fluid is not shear dependent (Chhabra and Richardson, 2008).

The shear dependent behaviour of sludge sampled in France was measured by successive increasing and decreasing steps of stress (i.e. creep) for a specific duration of time (88.6s); the ranges of applied stress for different concentrations of primary and secondary sludge are listed in Table 11. Before measurement, the sludge sample were pre – sheared (150s) at a shear stress corresponding to a high shear rate, and allowed to rest for duration of (150s) for primary sludge and (150s) for secondary sludge. The comparison of increasing and decreasing steps allowed us to analyse the impact of shear history on the flow behaviour of both sludges. From these increasing and decreasing steps, the shear stress versus shear rate required for the flow curves were recalculated using Eq. 27, 28, and 29.

Table 11: Summary of increasing and decreasing steps of stress required for primary and secondary sludge

Solids concentration, primary sludge, %TS	**2.80%**	**3.70%**	**5.50%**	**6.80%**	**8.20%**
Applied stress (Pa)	0.014	0.73	0.72	2.91	72.76
	0.049	1	1.4	3.6	80.3
	0.064	1.3	2.2	4.4	87
	0.109	1.5	2.5	5.1	91.5
	0.154	1.5	2.9	5.4	94.7
	0.192	1.5	3.1	5.8	97.5
	0.215	1.5	3.4	6.3	100.4
	0.25	1.6	3.6	6.6	101.7
	0.263	1.8	3.9	6.8	109.4
	0.278	1.9	4.2	7.3	116.4
	0.301	2	4.4	8.7	123.8
	0.327	2.2	4.5	9.4	131
	0.361	2.5	4.8	10.1	145.4
	0.359	2.7	5.1	10.9	176.1
	0.429	2.7	5.4	11.6	202.3
	0.584	3	5.8	13.1	228.5
Solids concentration, secondary sludge, %TS	**2.80%**	**4.00%**	**5.00%**	**6.50%**	**9.20%**
Applied stress (Pa)	0.03	1.46	0.72	2.91	101.86
	0.1	2	1.4	4.4	116.4
	0.3	2.6	2.2	5.8	120.8
	0.5	3	2.9	7.3	123.7
	0.6	3.1	3.6	7.7	126.7
	0.7	3	4.4	8	131
	0.9	3.1	4.8	8.3	135.3
	0.9	3.2	4.9	8.7	138.2
	1.2	3.5	5.4	10.2	141.1
	1.3	3.8	5.8	11.6	145.5
	1.4	4	6.5	13.1	160.1
	1.9	4.4	7.3	14.6	189.2
	2.2	4.2	8.8	18.9	218.3
	2.5	5.3	10.2	21.8	247.4
	2.7	5.5	12.9	24.7	276.5
	2.9	5.8	14.6	29.1	305.6

3.2.2.3 Time dependent behaviour

The time dependent behaviour of sludge was investigated by applying a pre – shear for 150s (at a constant stress corresponding to a high shear rate) followed by a period of rest which was varied from 60s, 120s, 300s, 900s, 1800s to 3600s. The same constant stress below the yield stress was then applied for all samples with a different duration of rest. Then the impact of different time of rest on the creep behaviour of each sludge sample was analysed.

3.3 Master curve development

This section provides a step by step procedure of the master curve development. The master curve was developed using a dimensionless form of the Herschel – Bulkley fluid model. A master curve was developed for different mixtures of primary and secondary sludge as well as mixtures of primary and secondary sludge mixed with digested sludge. These master curves were used for predicting the yield stress (τ_c), fluid consistency (K) and apparent viscosity (η) of any mixture at any volume fraction and concentrations of sludge.

The master curve was developed using the following steps:

1. Assign the raw data in specific cells.
2. Choose flow curve data of one of the sludge samples as a reference curve (in this example, 7% digested sludge was selected as shown in Figure 10). The chosen reference curve should cover the widest shear stress and shear rate range allowing for any flow curve whether in the low or high shear stress/shear rate range to be superimposed.

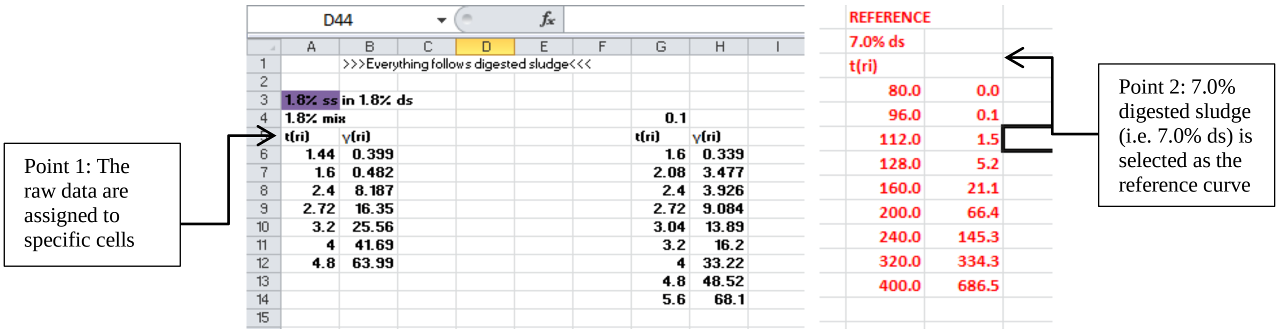


Figure 10: Snapshot of the excel spread sheet; summary of points 1 and 2

3. Plot the flow curve of each mixture and the flow curve of the reference sludge in one spreadsheet as shown in Figure 10.
4. Assign specific cells as shift factors and start with the value of 1 value.
5. Shift the flow curve of all the sludge mixtures (all different volume fractions at different concentrations) until they superimposes on the reference curve (7% digested sludge); in this example, the shear stress (y axis) and shear rate (x axis) of the 1.8% mixture of primary and secondary sludge are divided by the shift factors in the Y axis (S_y) and X axis (S_x), respectively, to superimpose on the reference curve.

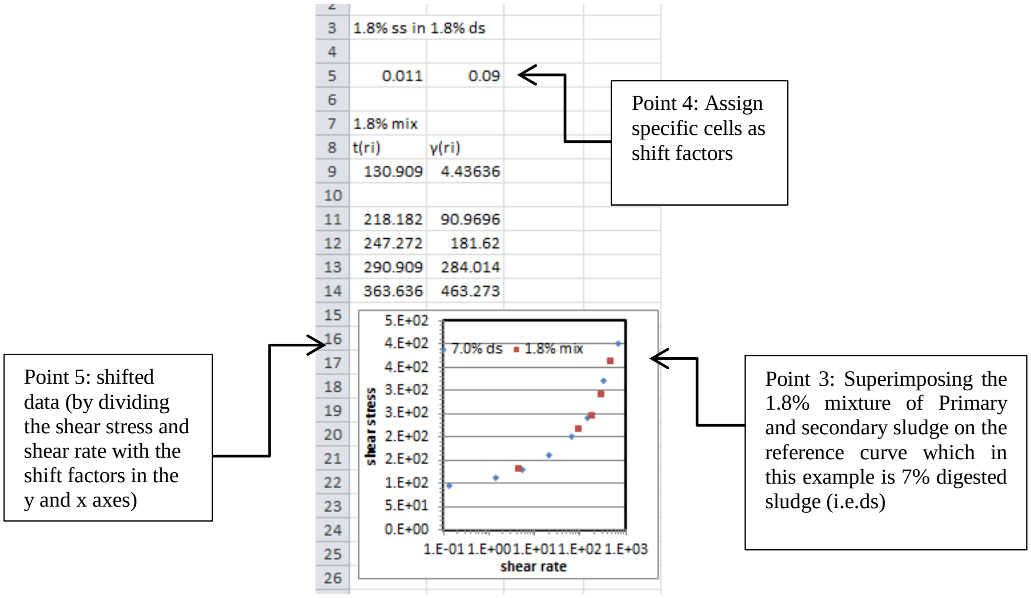


Figure 11: Snapshot of the excel spread sheet; summary of points 3, 4, and 5

6. The parameters of the Herschel – Bulkley model required for the reference curve are calculated (n, K τ_y) by minimising the error between the measured and calculated shear stress using excel solver function. The equation used to calculate the minimum error is:

$$minimum\ error = (\tau_{measured} - \tau_{calculated})^2/\tau_{calculated}{}^2 \qquad \text{Eq. 30}$$

Random values of these parameters were chosen which were reduced to zero once the error was minimized (see Figure 12).

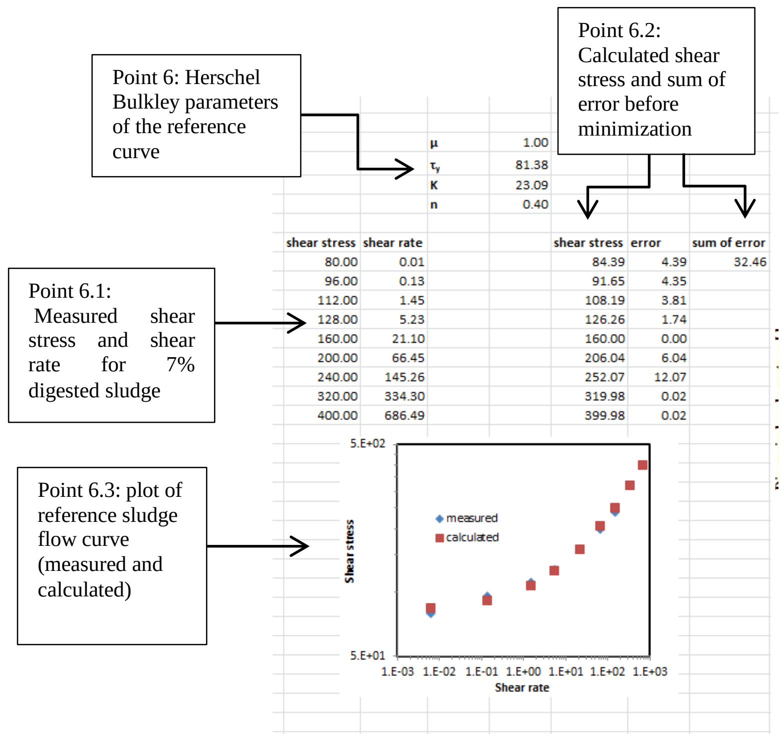


Figure 12: Snapshot of the excel spread sheet; summary of point 6

7. Convert the flow curve of reference sludge sample (in this example 7% digested sludge) and all other different sludge mixtures at different volume fractions and concentrations to a dimensionless form by plotting τ/τ_c against Γ.

   τ_c, K, and n are the Herschel Bulkley parameters of the reference sludge sample and μ=1 Pa.s as shown in Figure 13.

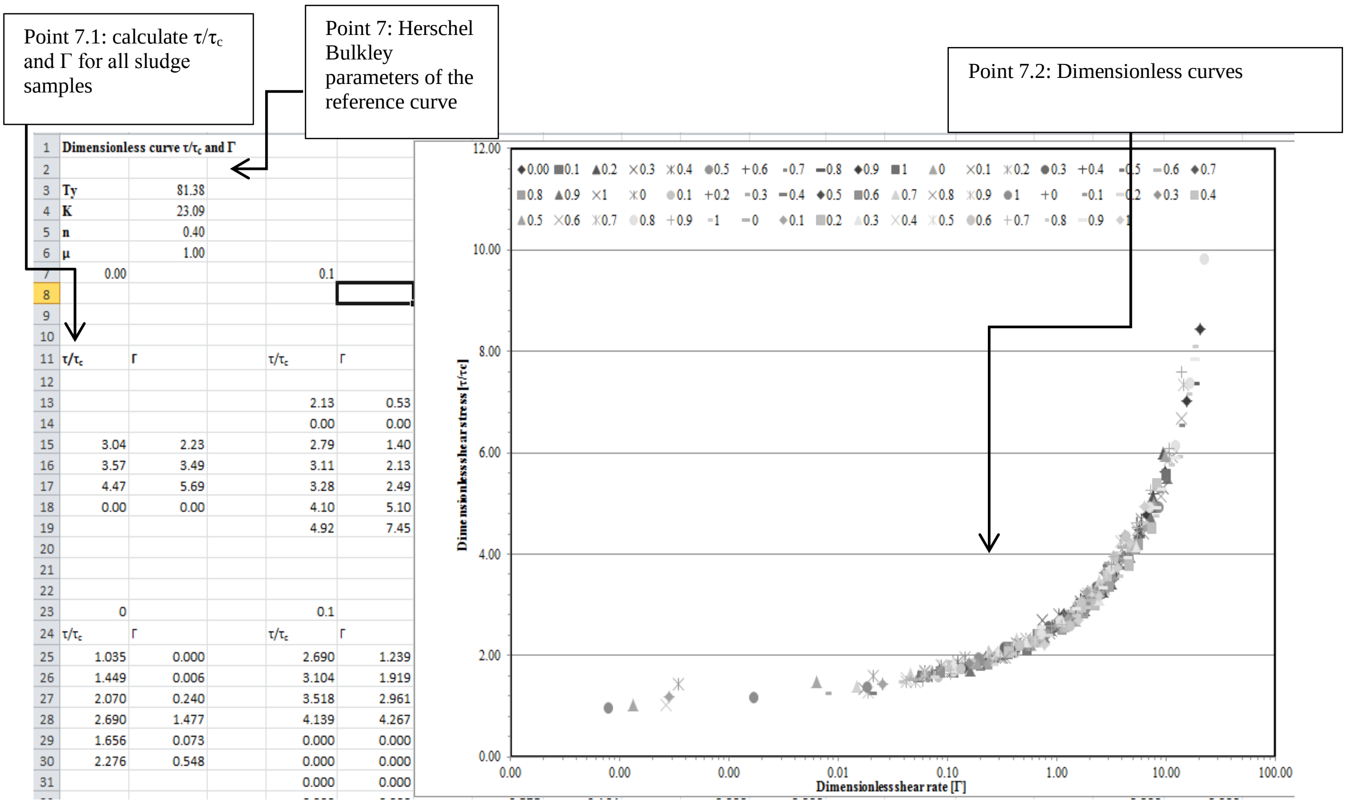


Figure 13: Snapshot of the excel spread sheet; summary of point 7

8. Recalculate the individual Herschel – Bulkley parameters of each sludge sample mixture using the shift factors and the master curve parameters (Figure 14). The flow index (n) was kept constant for all sludge mixtures so that the apparent viscosity and yield may be calculated.

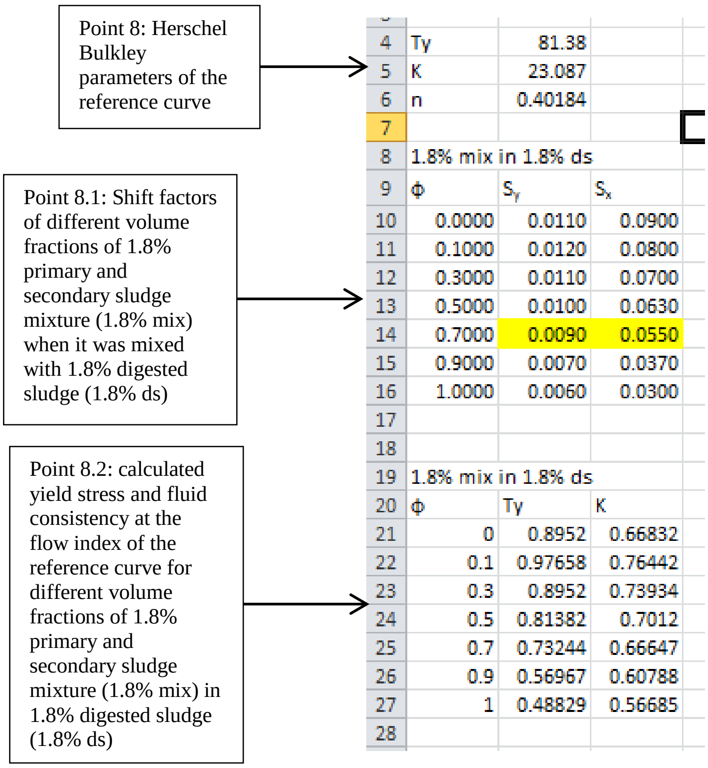
So, using $\tau = k\dot{\gamma}^{n} + \tau_{c\ (master)}$

$$\tau / S_y = k_{(master)} . (\dot{\gamma} / S_x)^n + \tau_{c\ (master)}$$

$$\tau = \left[k_{(master)} . (\dot{\gamma} / S_x)^n + \tau_{c\ (master)} \right] . S_y$$

$$\tau = k_{(master)} . (S_y / {S_x}^n) . \dot{\gamma}^n + \tau_{c\ (master)} . Sy$$

$$Where\ K = k_{(master)} . (S_y / {S_x}^n)\ and\ \tau_c = \tau_{c\ (master)} . Sy$$



4	Ty	81.38	
5	K	23.087	
6	n	0.40184	
7			
8	1.8% mix in 1.8% ds		
9	Φ	S_y	S_x
10	0.0000	0.0110	0.0900
11	0.1000	0.0120	0.0800
12	0.3000	0.0110	0.0700
13	0.5000	0.0100	0.0630
14	0.7000	0.0090	0.0550
15	0.9000	0.0070	0.0370
16	1.0000	0.0060	0.0300
17			
18			
19	1.8% mix in 1.8% ds		
20	Φ	Ty	K
21	0	0.8952	0.66832
22	0.1	0.97658	0.76442
23	0.3	0.8952	0.73934
24	0.5	0.81382	0.7012
25	0.7	0.73244	0.66647
26	0.9	0.56967	0.60788
27	1	0.48829	0.56685
28			

Figure 14: Snapshot of the excel spread sheet; summary of point 8

3.4 References

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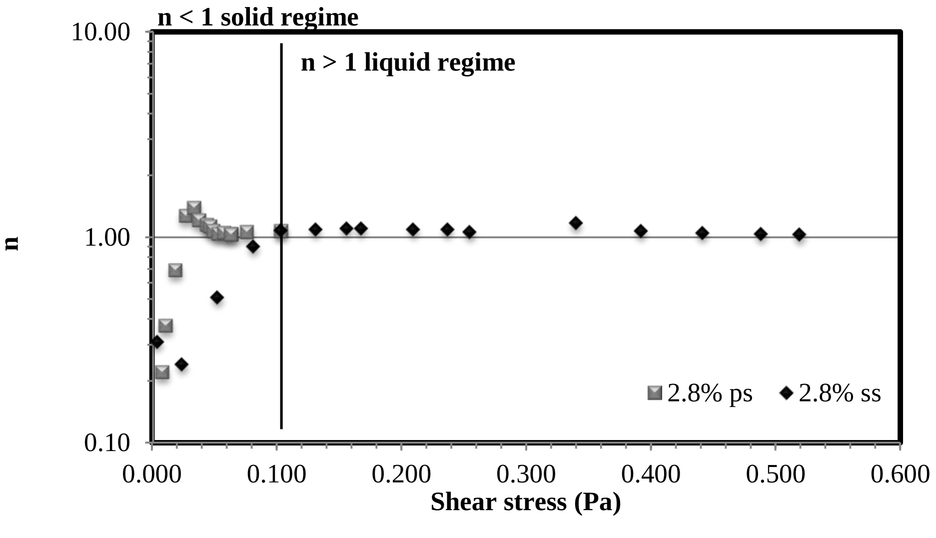
CHAPTER 4

RHEOLOGICAL CHARACTERIZATION OF PRIMARY AND SECONDARY SLUDGE: IMPACT OF TOTAL SOLIDS CONCENTRATION



Chapter 4: Rheological characterization of primary and secondary sludge: impact of total solids concentration

Abstract

Predicting the rheological behaviour of sludge is essential in the design and optimization of various unit operations of waste water treatment, most notably anaerobic digestion whereby the efficient mixing of sludge feed produces biogas and digested sludge. In this paper, the rheological behaviour of primary sludge (2.8%, 3.7%, 5.5%, 6.8% and 8.2% TS) and secondary sludge (2.8%, 4.0%, 5.0%, 6.5% and 9.2% TS) has been investigated. At low stress, below the yield stress, sludge behaved as a visco-elastic solid, whereby primary sludge yielded abruptly whilst secondary sludge flowed smoothly to steady state. In the steady state, both sludges behaved as shear thinning, yield stress fluids with primary sludge exhibiting highly thixotropic behaviour. The apparent viscosity, yield stress and fluid consistency of both primary and secondary sludge increase with increasing total solids concentration and followed the Herschel-Bulkley model. A master curve was developed based on the dimensionless form of the Herschel-Bulkley model allowing the rheology of primary and secondary sludge at any concentration to be determined.

Key words: Primary sludge, secondary sludge, rheology, Herschel-Bulkley model, viscoelasticity

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4.1 Introduction

Due to increasing urban populations, urban land shortages and economics, waste water treatment plants are under considerable pressure to treat higher loads without increase in plant size. This results in the treatment of a more concentrated and complex sludge. Therefore, an understanding of the hydrodynamics of sludge is required for the process design and optimization of waste water plants. Slatter (2008) and Spinosa and Lotito (2003) emphasized the importance of predicting the behaviour of sludge as it flows through various treatment processes such as pumping and transportation, chemical conditioning, mixing, storage and dewatering.

Baroutian et al (2013) defined sludge as the solid residue from the municipal waste water treatment process. There are three types of sludge: primary, secondary or activated sludge and digested sludge. Primary sludge has been defined by Bhattacharya (1981) as a flocculated mixture of organic and inorganic matter with gas bubbles trapped within the suspension. It is the product of primary clarification during the waste water treatment process. Bhattacharya (1981) explained that its flow behavior can be altered dramatically due to concentration, composition and temperature and that it is almost impossible to determine the effect of dimension, shape, size distribution and surface nature of the solid particles in the flocs because the solid particles have no fixed structure. The bacteria in primary sludge are said to be held together through nonspecific Lif-shitz van der Waals forces as well as hydrogen and chemical bonds (Cui et al., 2011, Bayoudh et al., 2009).

Secondary sludge, or activated sludge as it is often named, is the product of the secondary treatment process whereby it is removed via flotation and sent to a sludge settler. It is made up of polysaccharide and protein rich bacteria and micro-organisms that form extracellular polymeric substances (EPS). According to Wingender et al (1999), the EPS form a three dimensional gel like structure with a negative surface charge (Jia et al., 1996). Keiding et al (2001) and Sutherland (2001) explained that secondary sludge behaves as a gel when interacting with water and forms flocs, whilst Flemming (1996) stated that the structure is held together by electrostatic and hydrogen bonds.

Another difference between primary and secondary sludge is that they do not have the same density because the density of primary sludge is obtained from settling of coarse particles while the density of secondary sludge is obtained from flotation.

Digested sludge is the product of the anaerobic digestion process. It is a mixture of primary and secondary sludge (Baroutian et al., 2013) that has been stabilized through the anaerobic digestion process. We will not go further with the description of digested sludge as it is not the subject of this paper.

Spinosa and Lotito (2003) explained that rheology can be applied in the design and optimization of various unit operations of the waste water treatment process whereby the rheological properties influence the operating conditions and scale up calculations of various processing units such as tanks, settlers, pumping stations and transport lines as well as heat exchangers. As a result, the current literature on sludge focuses on the rheological characterisation of sludge in the liquid regime (Slatter, 2008, Eshtiaghi et al., 2012b, Eshtiaghi et al., 2012a, Baudez et al., 2011b, Slatter, 1997) whereby viscous forces are dominant.

Few studies have focused on the rheological behavior of primary sludge. The pioneering work of Bhattacharya (1981) and more recently Moeller and Torres (1997) are the only two studies to date that address the rheology of primary sludge. Concentrated primary sludge (3.77 to 7.48% TS) has been described by Bhattacharya (1981) as a shear thinning yield stress fluid whilst Moeller and Torres (1997) modeled the flow properties of dilute primary sludge (1 to 3% TS) using the power law model, suggesting that no yield stress was detected. The inconsistency between the two studies is due to the solids concentration of the characterized sludges which emphasizes that the yield stress depends on the solids concentration. The current literature focuses on the rheological characterization of activated sludge which is usually described as a complex non-Newtonian, viscoelastic (Baudez and Coussot, 2001, Baudez et al., 2013a), shear thinning fluid (Bhattacharya, 1981, Baudez et al., 2011b, Eshtiaghi et al., 2012b) which exhibits temperature dependency (Dieudé-Fauvel et al., 2009, Baudez et al., 2013b). Moreover, Tabuteau et al (2006), Baudez (2008), Tixier et al (2003a) and (2003b) illustrated that secondary sludge is thixotropic and undergoes aging as the solid structure is able to rebuild under shear. Baudez et al (2013a) also studied the viscoelastic behavior of secondary sludge and concluded that the observed behavior resembles gel structure.

The complexity of sludge as well as a lack of uniformity associated with sludge rheometric techniques (Seyssiecq et al., 2003, Ratkovich et al., 2013, Eshtiaghi et al., 2013b) have highlighted that sludge is a highly difficult material to characterise in order to design and optimize waste water treatment plants.

This study focuses on the comparison of the rheological behaviour of primary and secondary sludge as a feed for anaerobic digestion. Anaerobic digestion requires the constant mixing and degradation of the feed sludge. As stated earlier, whilst the current literature focuses on the rheological characterisation of activated sludge, the aim here is to get a better understanding of the sludge entering the digester (i.e. primary and secondary sludge) in order to understand how its rheological properties will influence the anaerobic digestion process instead of characterising sludge once it has been digested (i.e. digested sludge). Hence, the yielding properties, thixotropy and apparent viscosity of primary and secondary sludge will be investigated.

In this paper, we demonstrate that primary sludge behaves as a colloidal suspension while secondary sludge is a more gel-like material.

4.2 Materials and method

4.2.1 Sample Preparation

Samples of primary sludge were obtained from Bessay (Allier, France) and secondary sludge was obtained from Vichy (Allier, France).

Primary sludge with an initial total solids (TS) concentration of 2.8 wt% was thickened to 3.7, 5.5, 6.8 and 8.2% TS using the vacuum filtration technique such that the floc structure would not be altered. Secondary sludge was sampled at the outlet of the dewatered centrifuge step of a waste water treatment plant (its solid concentration was at 22%) and was diluted using tap water to various concentrations (2.8, 4.0, 5.0, 6.5 and 9.2% TS) to reach the usual concentrations entering into a digester.

4.2.2 Rheometric technique

An Anton Paar physica MCR 300 dynamic stress rheometer equipped with the wide gap vane geometry (diameter of the cup, D_c = 39.0 mm; diameter of the vane, D_v = 25.0 mm, height of vane, H = 70.0 mm) was used to investigate the impact of shear history, yielding, impact of time of rest and flow properties of the samples. The vane geometry was employed to avoid artefacts such as the inertia and end effect (Dzuy and Boger, 1985). Tool surfaces were roughened to reduce wall slip (Baudez, 2008).

Table 12: Summary of applied increasing and decreasing steps stress for each sample

Total solids concentration (%)	2.8% PS	3.7% PS	5.5% PS	6.8% PS	8.2% PS	2.8% SS	4.0% SS	5.0% SS	6.5% SS	9.2% SS
Applied stress, (Pa)	0.014	0.73	0.72	2.91	72.76	0.03	1.46	0.72	2.91	101.7
	0.049	1.0	1.4	3.6	80.3	0.1	2.0	1.4	4.4	116.4
	0.064	1.3	2.2	4.4	87.0	0.3	2.6	2.2	5.8	120.8
	0.109	1.5	2.5	5.1	91.5	0.5	3.0	2.9	7.3	123.7
	0.154	1.5	2.9	5.4	94.7	0.6	3.1	3.6	7.7	126.7
	0.192	1.5	3.1	5.8	97.5	0.7	3.0	4.4	8.0	131.0
	0.215	1.5	3.4	6.3	100.4	0.9	3.1	4.8	8.3	135.3
	0.250	1.6	3.6	6.6	101.7	0.9	3.2	4.9	8.7	138.2
	0.263	1.8	3.9	6.8	109.4	1.2	3.5	5.4	10.2	141.1
	0.278	1.9	4.2	7.3	116.4	1.3	3.8	5.8	11.6	145.5
	0.301	2.0	4.4	8.7	123.8	1.4	4.0	6.5	13.1	160.1
	0.327	2.2	4.5	9.4	131.0	1.9	4.4	7.3	14.6	189.2
	0.361	2.5	4.8	10.1	145.4	2.2	4.2	8.8	18.9	218.3
	0.359	2.7	5.1	10.9	176.1	2.5	5.3	10.2	21.8	247.4
	0.429	2.7	5.4	11.6	202.3	2.7	5.5	12.9	24.7	276.5
	0.584	3.0	5.8	13.1	228.5	2.9	5.8	14.6	29.1	305.6

The experimental procedure was conducted as follows: first sludge was presheared (150s) at a shear stress corresponding to a rotational velocity within the range of 3 to 10 rotations per second (rps) depending on the solids concentration of sludge in order to obtain a homogenous material that is always in the same initial state of de-structuration (Baudez, 2008, Coussot, 2005). This preshear was followed by a short period of rest (150s) to let the structure rebuild (Coussot, 2005). Then, successive increasing and decreasing steps of constant stress were applied for duration of 86s; the ranges of applied stress for different concentrations of primary and secondary sludge are listed in Table 12. The comparison of increasing and decreasing steps allowed us to analyse the impact of shear history on the rheological properties of both sludge.

Using a similar procedure, the impact of time of rest was investigated. First, the sludge was presheared (150s), followed by different times of rest. The time of rest was varied between 60s, 120s, 900s, 1800s and 3600s. Next, a constant stress, below the yield stress was applied and the resulting creep response was measured. The experiments were carried out at a temperature of 20°C.

Because the wide gap vane geometry was used, the flow curves were recalculated using the derivatives of the torque – deflection angle curve such that the angular velocity, $\omega = \theta/t$, the shear stress at the surface of the rotating vane, $\tau_{R_i} = M / (2 \pi H R_i^2)$ and the shear rate at the surface of the rotating vane, $\dot{\gamma}_{R_i} = [2 R_o^2 / (R_o^2 - R_i^2)] \omega$, where θ is the deflection angle (rad), t is the time (s), M is the torque (Nm), H is the height of the vane (m), R_i and R_o are the radii of the rotating vane and cup (Mezger, 2006).

4.3 Results and discussion

When a constant stress is applied, the creep curve of both primary and secondary sludge followed a curve for which the asymptotic value can be modelled by:

$$\gamma \sim A t^n + B$$

Where n<1 means that the derivative of the shear strain, the shear rate, is a decreasing function of time and no steady state flow can be reached; the sludge is in its solid regime. When n=1, the derivative of the shear strain is constant and a steady state flow can be reached and the sludge is in its liquid regime. Thus, sludge yields from the solid state to the liquid state when n=1. The creep curve is illustrated in Figure 15 and its derivative – the shear rate as a function of time is illustrated in Figure 16.

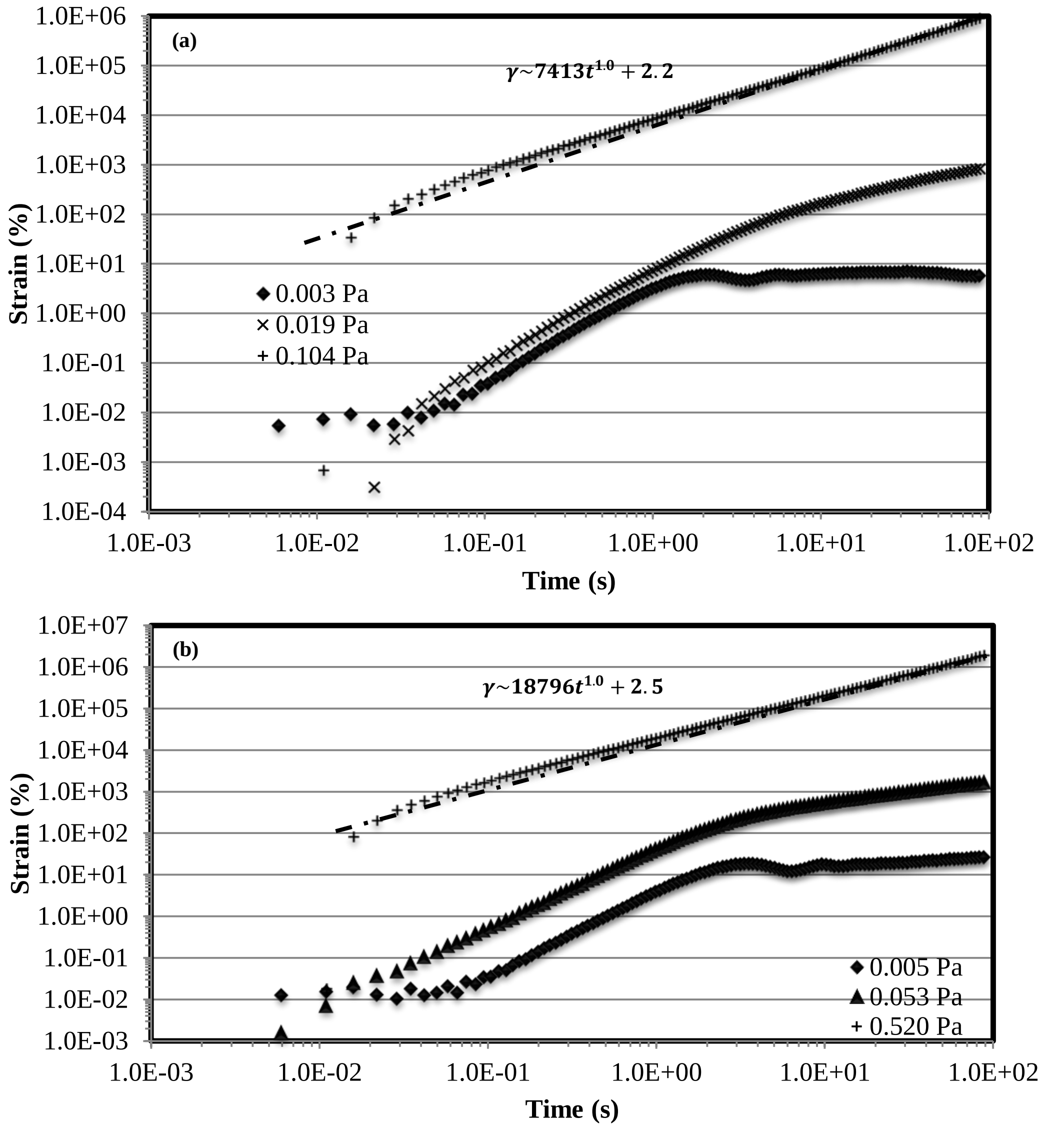

Figure 15: Creep curve for (a) 2.8% primary sludge and (b) 2.8% secondary sludge (the dashed line shows the asymptote of the highest strain where the power law index is equal to 1

This observation is similar to the creep response of digested sludge studied by Baudez et al (2011b) such that below a critical stress, sludge undergoes restructuration and is said to be in the viscoelastic regime and above the critical stress, it is destructured and flows steadily (i.e. liquid regime).

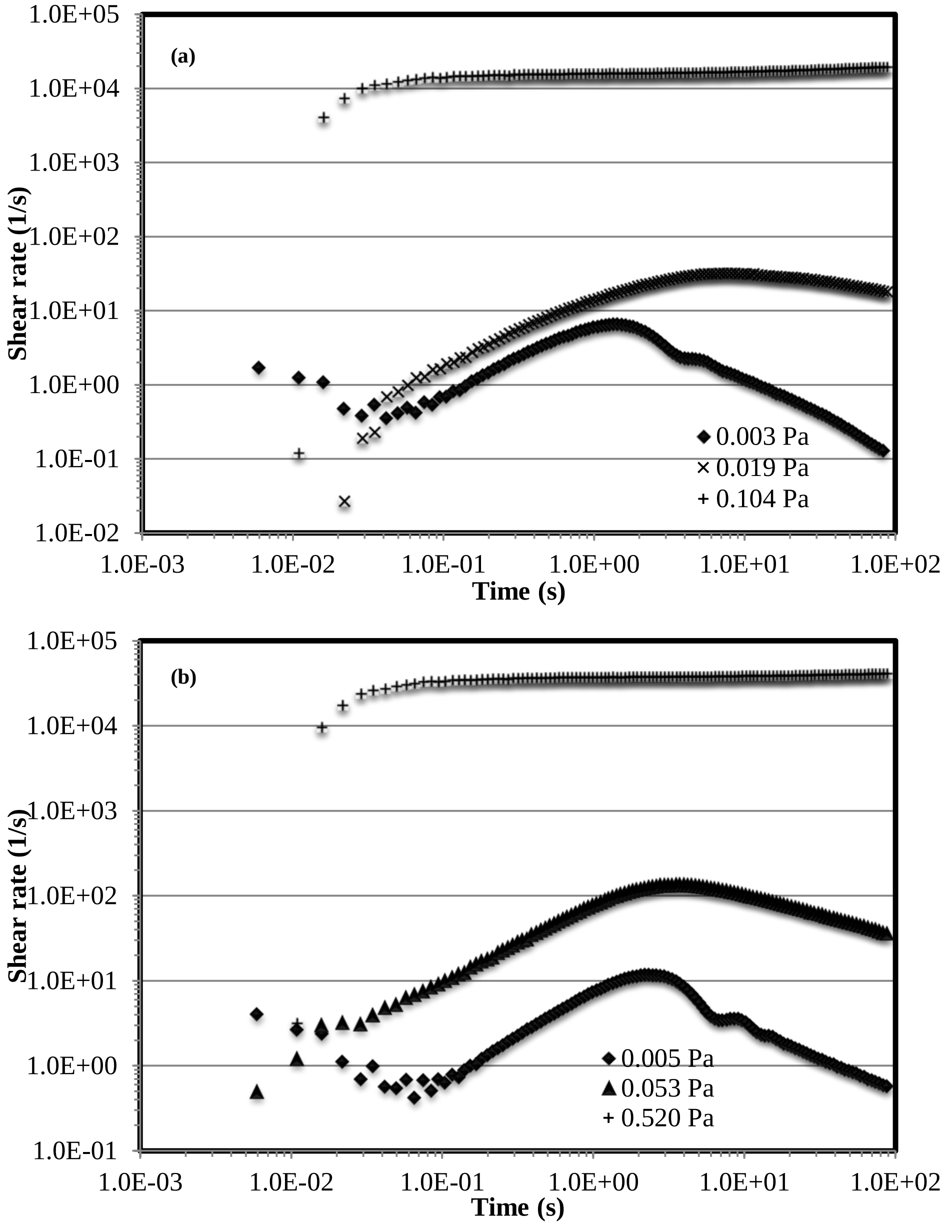

Figure 16: Shear rate (1/s) versus time (s) below, above and equal to the critical shear stress for (a) 2.8% primary sludge and (b) 2.8% secondary sludge

By plotting the evolution of the power law index of the asymptote of the creep curve as a function of applied stress (Figure 17), an overshoot was observed in the power law index of primary sludge as the stress approaches towards the critical stress whilst secondary sludge undergoes a smoother transition from solid to liquid regime similar to gels (Baudez et al., 2013a) (illustrated by the solid line in Figure 17). The overshoot of the power law index of primary sludge indicates that the derivative of the shear strain, the shear rate, accelerates rapidly with time suggesting that the sludge structure collapses rapidly from solid-like to liquid-like; however, no steady state flow can be reached. The flow is disordered suggesting that more time is required to reach steady state flow at the applied stress; as such the yielding of primary sludge is very abrupt and dependent on the shearing time, a characteristic of highly colloidal suspensions (Coussot et al., 2002).

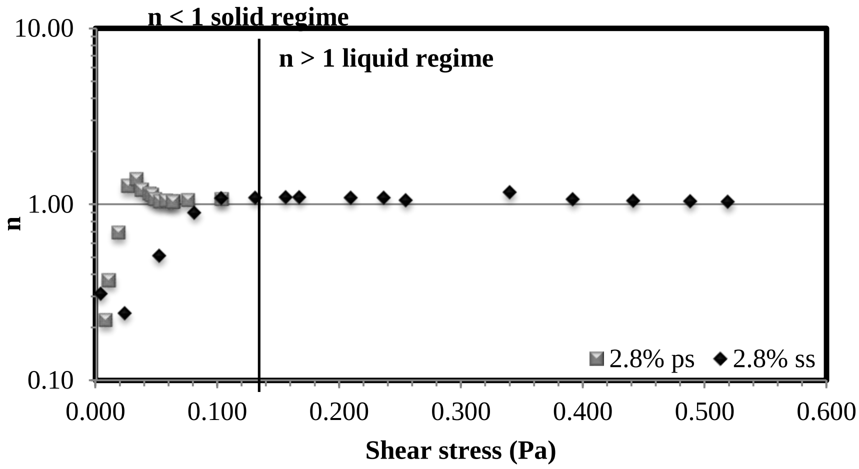

Figure 17: Power law index of sludge (2.8% primary sludge, 2.8% secondary sludge) with the solid line illustrating the yielding of secondary sludge

The solid-like behaviour of sludge was investigated by applying a constant stress (below the yield stress) and varying the time of rest between the preshear and creep; the creep response is illustrated in Figure 18. Figure 18 (a) illustrates that the time of rest had little impact on the yielding of the primary sludge (as the strain increased slowly for the duration of the creep and towards the same final strain) and that it remained in the solid regime. However, Figure 18 (b) illustrates that when secondary sludge subjected to a small time of rest (60 or 120s), the strain increases slowly, followed by a rapid increase indicating that the secondary sludge is initially in the solid regime, however, as the creep progresses, it yields and flows steadily into steady state flow; the structure did not rebuild during short periods of rest. In contrast, when the secondary sludge is subjected to a prolonged time of rest (≥900s and same constant stress that is below the yield stress), the strain increases slowly for the duration of the creep indicating that the sludge remains in the solid regime.

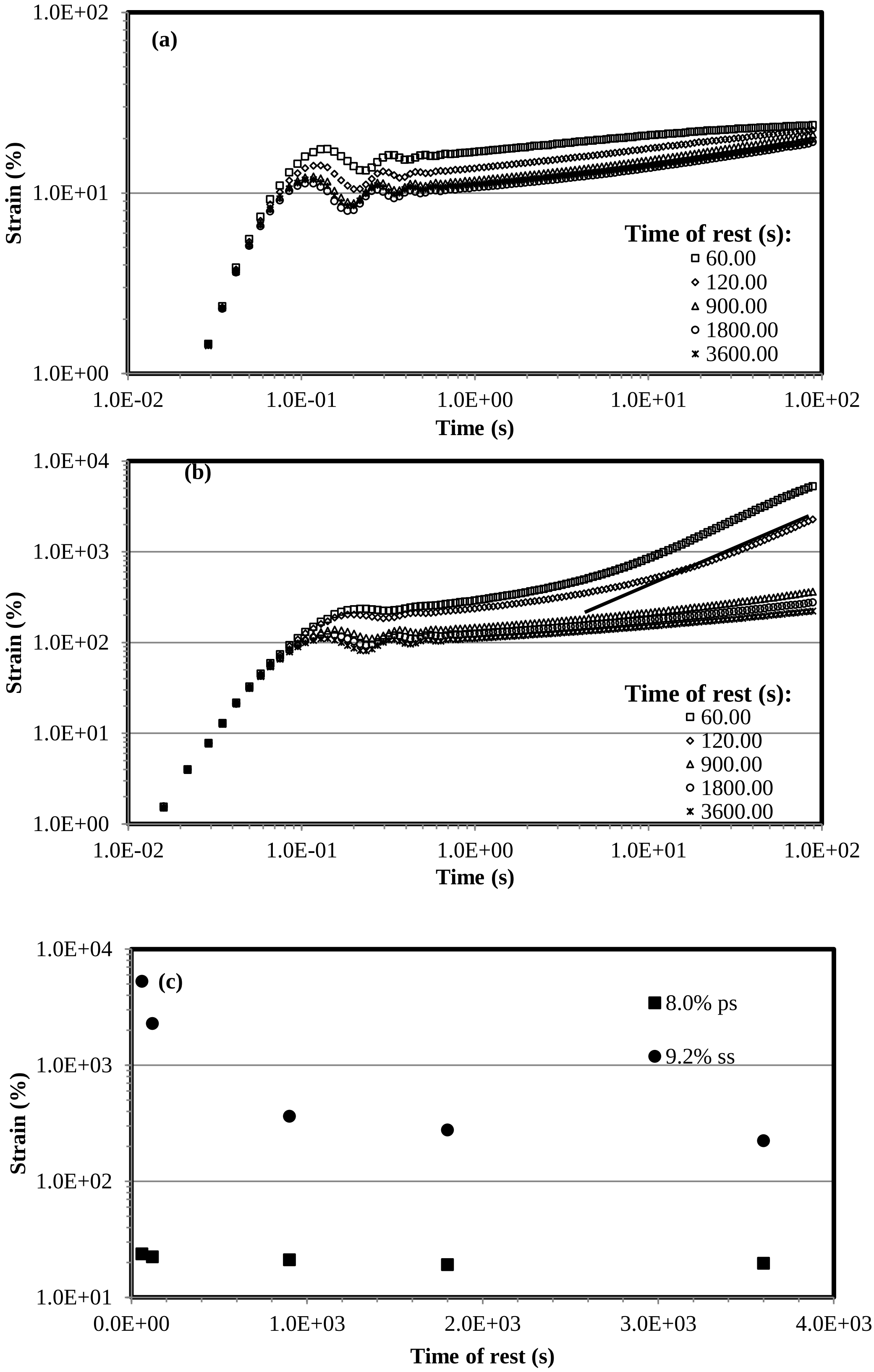

Figure 18: Creep curve for (a) thickened secondary sludge (8.2% TS, 20 °C) at a constant stress, below the yield stress; (b) thickened primary sludge (9.2% TS, 20 °C) at a constant stress, below the yield (the solid line indicates the yielding of secondary sludge) and (c) shear strain versus the time of rest for both primary and secondary sludge

Baudez (2008) explains that the rheological behaviour of sludge is driven by a competition between colloidal forces which tend to rebuild the structure (i.e. physical aging) and hydrodynamic forces which tend to maintain the solid structure broken (i.e. shear rejuvenation). This means that primary sludge undergoes physical aging and its behaviour is dependent on the strength of the network structure (where colloidal forces dominate) as well as the applied stress. In the case of secondary sludge and for a short time of rest, hydrodynamic forces are dominant which keep the structure broken (i.e. sludge exhibits shear rejuvenation) and result in deflocculation. In contrast, at prolonged time of rest, colloidal forces dominate; the secondary sludge undergoes physical aging whereby the network structure becomes stronger which results in flocculation.

Figure 18 (c) illustrates that for both primary and secondary sludge the strain decreases with time of rest (more evident with secondary sludge) indicating that the solid like characteristics were dependent on the time of rest as demonstrated by Baudez (2006) and Tabuteau et al (2006). Buadez (2006) investigated the thixotropic behaviour of sludge and highlighted that the sludge structure was able to rebuild after a period of rest indicating that the solid – like behaviour was influenced by time of rest. In another study conducted by Tabuteau et al (2006), the time dependent behaviour of sludge was investigated under creep experiments. Tabuteau et al (2006) demonstrated that below a critical shear stress, the sludge did not flow steadily and above the critical shear stress, the material flowed, however, it took longer to reach steady state flow as the time of rest increased indicating that the sludge undergoes restructuration and flocculation during prolonged rest times. The observed behaviour is in agreement with the works of Dursun and Dentel (2009) that used the gel approach to model the behaviour of secondary sludge. In contrast, the time of rest had little impact on the strain highlighting that the rheological behaviour of primary sludge is shear dependent rather than time dependent which was in agreement with colloidal suspensions (Coussot et al., 2002).

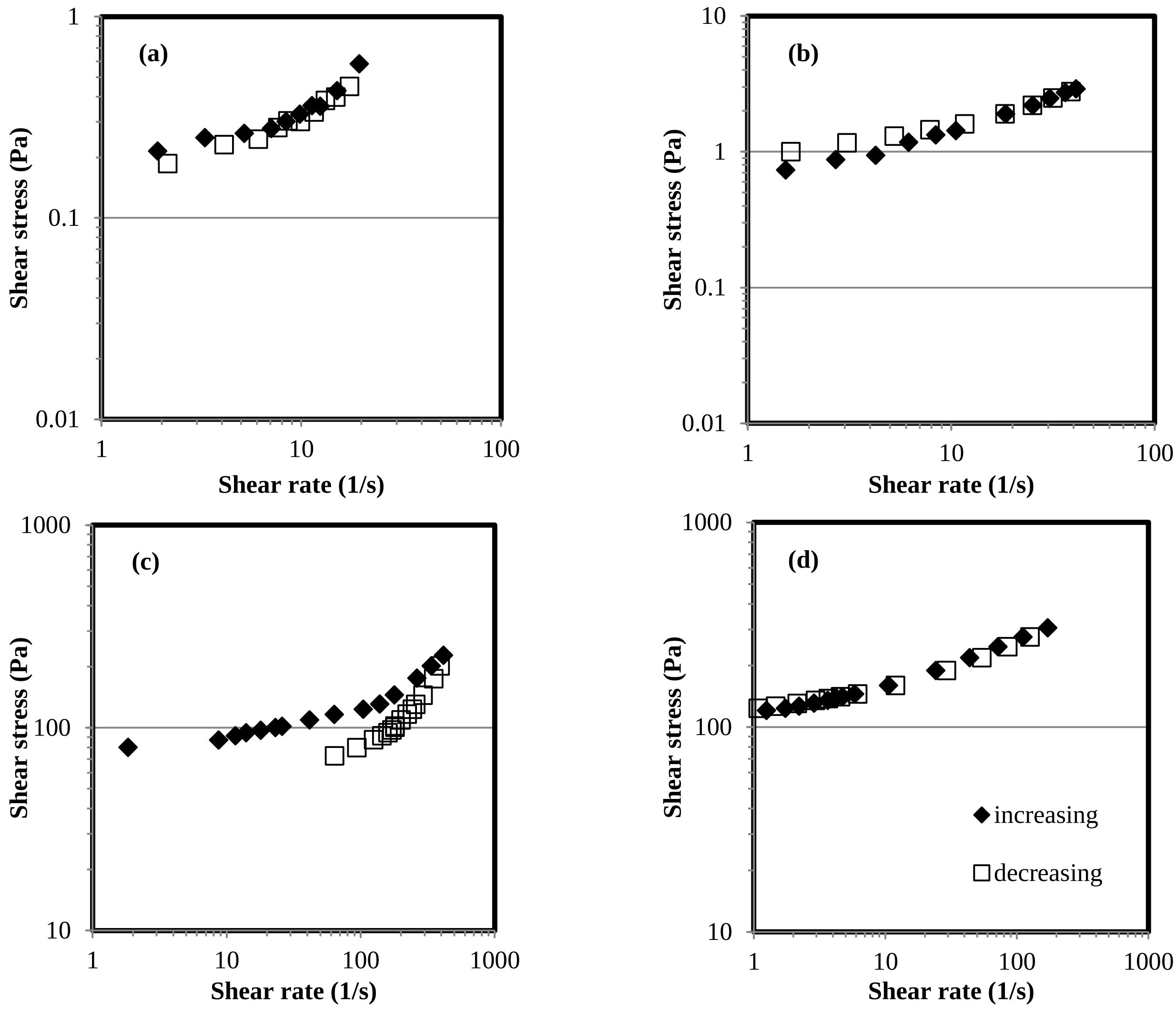

Figure 19: Rheogram of (a) dilute primary sludge (2.8% TS, 20 °C); (b) dilute secondary sludge (2.8% TS, 20 °C); (c) thickened primary sludge (8.2% TS, 20 °C); (d) thickened secondary sludge (9.2% TS, 20 °C)

By developing the increasing and decreasing rheograms and investigating the impact of previous shear on sludge using the hysteresis loop formed inside the rheogram, the abrupt flowage and stoppage of primary sludge was highlighted. Previous shear history had little to no influence on the flow behaviour of diluted primary sludge, diluted secondary sludge and thickened secondary as illustrated in Figure 19 (a), (b) and (d) whereby the increasing and decreasing rheograms are almost superimposed. Figure 19 (c) illustrates that the flow behaviour of thickened primary sludge is influenced by previous shear such that the decreasing rheogram is lower than the increasing rheogram indicating that the corresponding viscosity is lower. Thickened primary sludge also exhibited very abrupt flow stoppage, as shown during the decreasing rheogram (Figure 19c) and not to be able to reach lower shear rate. This peculiar behaviour of primary sludge is similar to what was noticed by Coussot et al (2002) with colloidal suspensions, such as bentonite, which are highly thixotropic materials. This phenomenon was called viscosity bifurcation because the sludge initially flowed as a liquid for stresses below the critical stress then eventually becomes a solid resulting in abrupt flow stoppage. The degree of thixotropy increases with increasing total solids concentration for primary sludge (Baudez, 2008).

Tixier et al (2003a) and (2003b) performed a hysteresis loop by applying an increasing shear rate from 0 to 800 s^{-1} in three minutes, followed by a constant shear rate of 800 s^{-1} in one minute. Then a decreasing shear rate was applied from 800 s^{-1} to 0 s^{-1}. The hysteresis loop varied depending on the nature of sludge (Tixier et al., 2003b, Tixier et al., 2003a). In contrast, Baudez (2006) demonstrated that the hysteresis loop was due to the displacement of the separating line between sheared and unsheared zones in the gap and that an additional stress was required to break the solid structure during the increasing ramp; the decreasing ramp required no additional stress. As a result, the increasing and decreasing ramps were not superimposed. In the above mentioned cases, the experimental protocol is important in determining the shear history and time dependence of sludge, hence, by using the wide gap vane geometry and by applying increasing and decreasing steps of constant stress, and reconstructing the rheograms from the torque – deflection angle curve, artefacts such as wall slip and the end effect were reduced allowing for the accurate measurement of the shear history dependence of sludge.

The behaviour of primary and secondary sludge in the steady state was investigated by plotting the shear stress versus shear rate known as the flow curve as it provides information in the liquid regime where elastic effects do not play a role (Coussot, 2005). The flow curves of primary and secondary sludge at various concentrations are presented in Figure 20 (a) and (b) whereby the shear stress increased non-linearly with shear rate indicating non-Newtonian fluid behaviour whilst the viscosity decreased as a function of shear rate, corresponding to shear thinning behaviour. Primary and secondary sludge exhibit a yield stress. Such behaviour – non-Newtonian, shear thinning, yield stress has been used to describe primary sludge (Bhattacharya, 1981) and also secondary sludge by several authors (Baudez and Coussot, 2001, Seyssiecq et al., 2008).

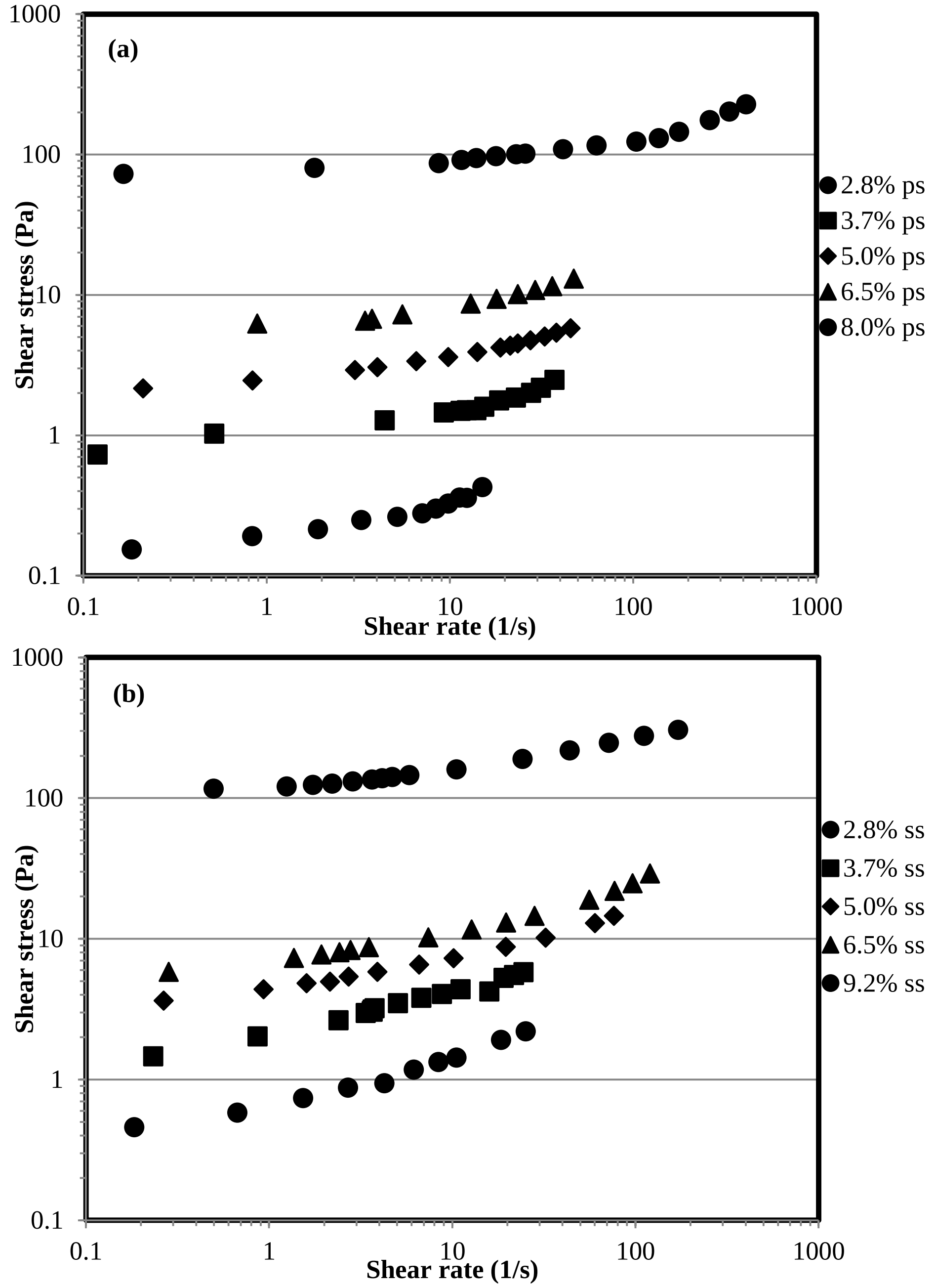

Figure 20: Flow curve of primary sludge at various concentrations (2.8%, 3.7%, 5.5%, 6.8%, and 8.2%) (a) and secondary sludge at various concentrations (2.8%, 4.0%, 5.0%, 6.5% and 9.2%) (b)

Figure 20 (a) and (b) illustrates that the rheological behaviour of sludge increases with increasing concentration which can be attributed to the strengthening of the particle interactions resulting in a stronger network structure. Indeed, in their review on activated sludge rheology, Seyssiecq et al (2003) explained that as the concentration increases, so do the particle interactions resulting in the formation of links between the flocs, which in turn leads to an increase in the rheological properties with concentration. As primary sludge behaves as a colloidal suspension, which is governed by weak attractive forces (i.e. van der Waals forces), weak links are formed between the flocs, such that abrupt flowage and stoppage is observed at low stresses. Secondary sludge on the other hand exhibits gel like behaviour who's EPS rich structure is held together by hydrogen and electrostatic forces resulting in stronger links between flocs allowing for the smooth, clear transition between the solid and liquid regime (Baudez and Coussot, 2001). Other studies which are consistent with our work include Lotito et al (1997)and Slatter (1997). A summary of the major characteristics of primary and secondary sludge are presented in Table 13.

Table 13: Summary of main characteristics of both primary and secondary sludge

	Primary sludge	Secondary sludge
Major interactions	Nonspecific Lif-shitz van der Waals forces as well as hydrogen and chemical bonds weak van der Waals forces*	Hydrogen and electrostatic*
Viscoelastic solid	yes for stresses, below the yield stress	Yes for stresses, below the yield stress
Yielding	abrupt transition from solid to liquid regime	smooth transition from solid to liquid regime
Shear thinning behaviour	Yes	Yes
Ageing	Yes	Yes
Shear rejuvenation	No	Yes
Thixotropy	Shear dependent behaviour	Time dependent behaviour
Temperature dependence	N/A	yes*

The symbol * indicates results coming from the literature and cited in the introduction

The flowing behaviour of primary and secondary sludge can be described by the Herschel-Bulkley model ($\tau = \tau_y + K \gamma^n$) (Figure 21) as it takes into account the low to intermediate shear rate. The Herschel-

Bulkley model has been used to characterise various sludge types because it takes into account the yield pseudo plasticity of sludge (Slatter, 1997, Baudez et al., 2011b).

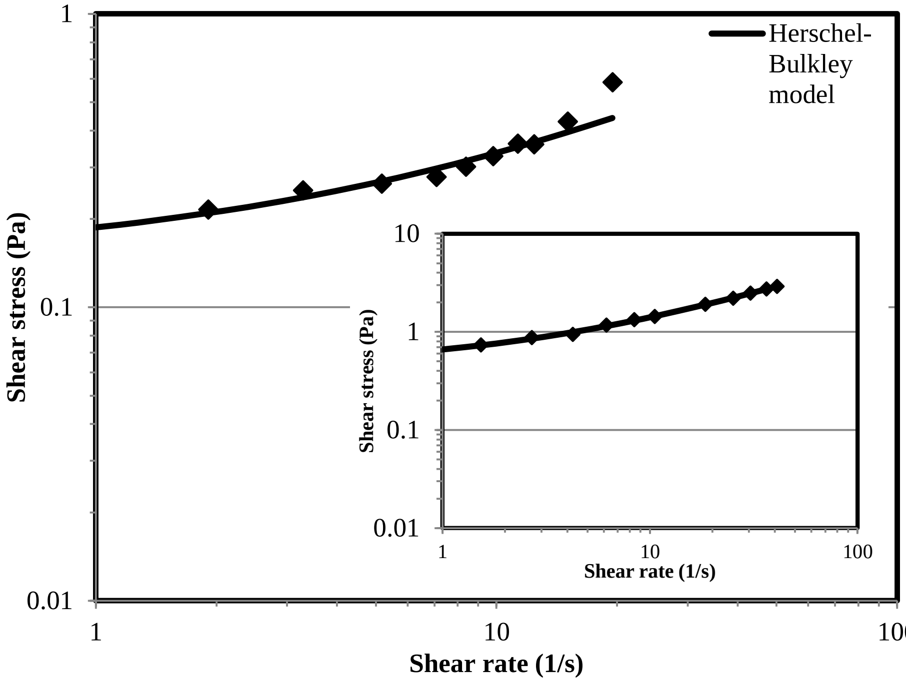

Figure 21: Flow curve of primary sludge (2.8% TS, 20 °C) and secondary sludge (insert, 2.8% TS, 20 °C) modelled using the Herschel-Bulkley model

Whilst several studies (Slatter, 1997, Lotito et al., 1997, Mori et al., 2006) attempted to link the rheological parameters – flow index "n", fluid consistency "K" and yield stress "τ_y" with concentration, we demonstrate that a master curve following a dimensionless form of the Herschel-Bulkley model can be used to determine the true rheological parameters of primary and secondary sludge as shown in Figure 22:

$\tau = \tau_y + K.\dot{\gamma}^n \rightarrow \tau/\tau_c = 1 + (K/\tau_y).\dot{\gamma}^n$

$\tau/\tau_c = 1 + \beta.\Gamma^n$ Eq. 31

, where $\Gamma = (1/\tau_y).\dot{\gamma}$ and $\beta = (K/\tau_y).\tau_y{}^n$

, where τ_y incorrectly symbolizes the yield stress and represents the extrapolated limit, below which there is no steady state flow. K is the fluid consistency, and n is the flow index.

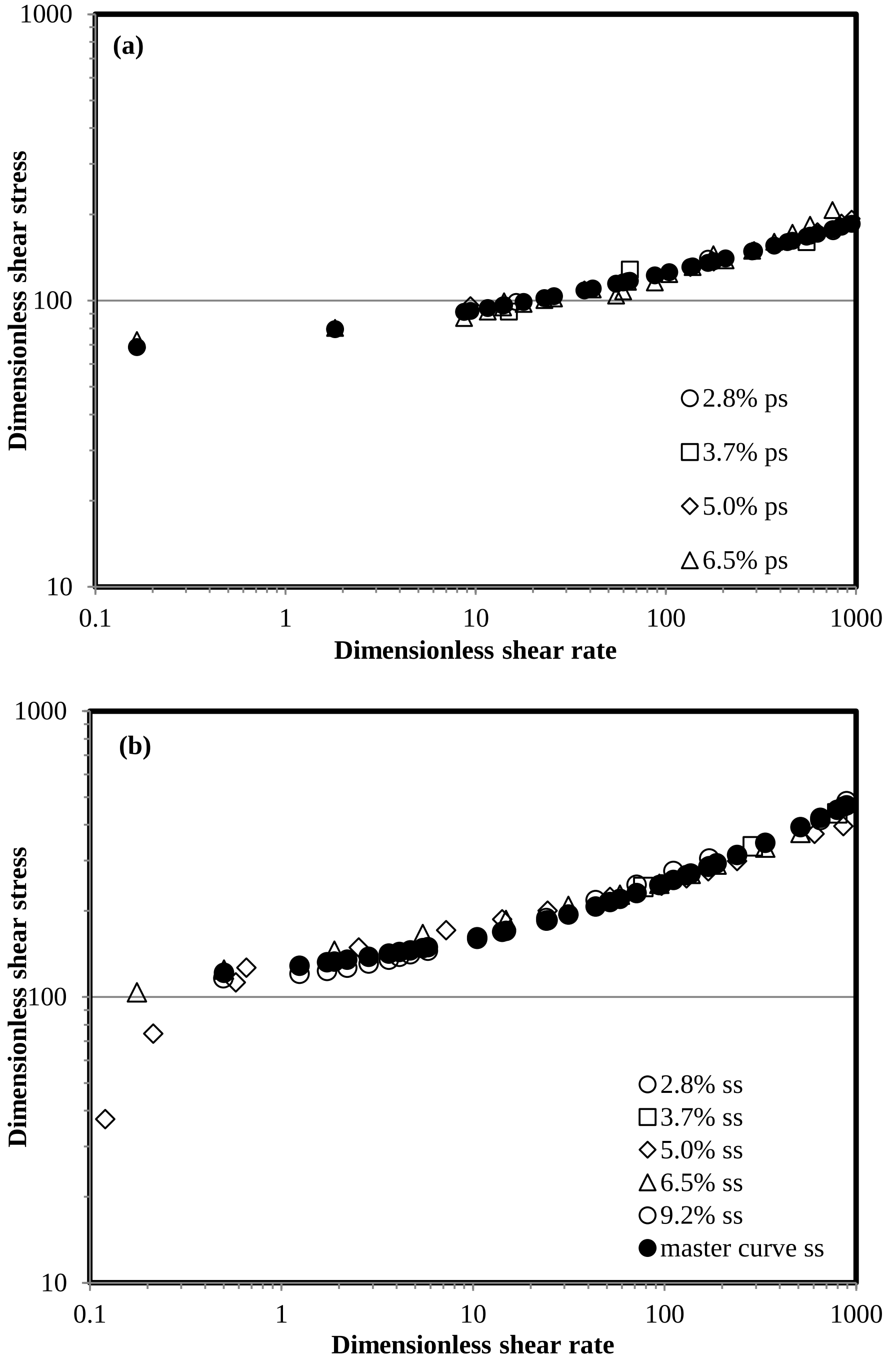

Figure 22: Dimensionless master of (a) primary sludge with the parameters τc= 58.16; K = 17.92; n= 0.29 and (b) secondary sludge with the parameters τy= 106.43; K = 2.05; n= 0.42 at various concentrations

The master curve was able to compare, predict and estimate the rheological properties of the sludge regardless of the solids concentration. First, the shear stress is scaled by the yield stress such that solid interactions, represented by τ_c are smoothed out. The shear rate was then scaled by a dimensionless factor– β taking into account the viscous interactions. This allowed all the curves to go through the same point ($\gamma = 1$, $\tau_y = 1$). From these factors, also known as shift factors (presented in Table 14), the true yield stress and true fluid consistency of sludge at the known solids concentration were recalculated. The flow index was kept constant enabling us to compare the yield stress and fluid consistency. Baudez et al (2011b) and (2013b) developed master curves using a similar approach in order to investigate the flow behaviour of sludge.

Table 14: Summary of shift factors required to scale the flow curves of primary and secondary sludge into the dimensionless form of the Herschel – Bulkley model

Solids concentration (%)	Shift factors	
Primary sludge	**τ_c**	**β**
2.8	0.001	0.001
3.7	0.008	0.008
5.5	0.023	0.023
6.8	0.063	0.063
8	1	1
Secondary sludge	**τ_c**	**β**
2.8	0.0007	0.0007
4	0.003	0.003
5	0.020	0.020
6.5	0.035	0.035
9.2	1	1

Baudez et al (2011b) developed a master curve for digested sludge at various concentrations and explained that in the dimensionless form and from a physical point of view, there is a similarity of the network of interactions within the sludge. Baudez and Coussot (2001) classified the interactions into two groups, hydrodynamic (between solid particles and the surrounding fluid – characterized by the Bingham viscosity) and non-hydrodynamic interactions (between solid particles – characterized by the yield stress). Baudez et al (2011b) states that these interactions do not change with increasing concentration, rather they intensify. In fact, this is true and we demonstrate that the apparent viscosity increases exponentially with concentration whilst the yield stress follows a power law:

$$\eta = \eta_0 \exp(C\beta) \qquad \text{Eq. 32}$$

$$\tau_y = \alpha(C - C_{min})^m \qquad \text{Eq. 33}$$

, where C it the total solids concentration, C_{min} is the lowest concentration below there is no yield stress, m is a parameter related to the fractal dimension of sludge flocs and η_o is the viscosity of the liquid medium. α and β are fitting parameters of the equations. The apparent viscosity of sludge was calculated from the parameters of the dimensionless Herschel-Buckley fluid at a single shear rate of 10 s^{-1}. The evolution of the apparent viscosity and yield stress are presented in Figure 23 (a) and (b) and are in agreement with the current literature on sludge (Baudez et al., 2011b, Baudez et al., 2013b, Baudez, 2008, Sanin, 2002). The evolution of the fluid consistency as a function of concentration (refer to Figure 23c) followed a power law form consistent with Lotito et al (1997):

$$K = aC^b \qquad \text{Eq. 34}$$

, where a and b are fitting parameters. The parameters of Eq. 32, 33 and 34 are presented in Table 15.

Table 15: The parameters of the apparent viscosity (Eq. 32), yield stress (Eq. 33) and fluid consistency models (Eq. 34)

	η_o (Pa.s)	**β**	**α (Pa)**	**C_{min} (%TS)**	**m**	**a**	**b**
Primary sludge	0.19	0.35	0.08	1.85	2.37	0.57	1.47
Secondary sludge	0.07	0.50	0.005	0.96	3.96	0.08	2.30

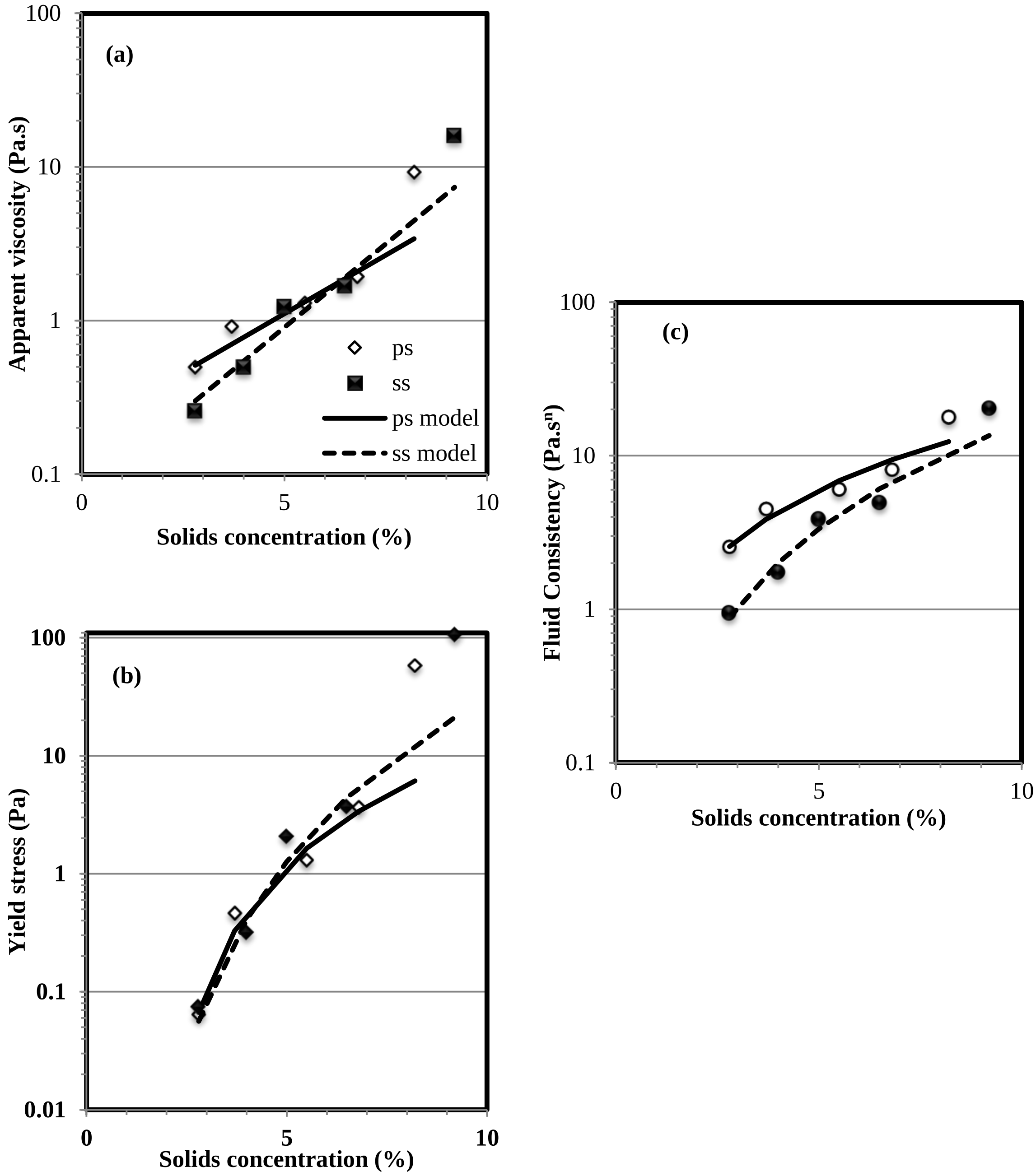

Figure 23: Evolution of the apparent viscosity (a); yield stress (b) and fluid consistency (c) as a function of concentration (%) of sludge; the open points refer to primary sludge and the filled points refer to secondary sludge

The above mentioned correlations can be applied to predict the rheological behaviour of sludge for design and optimization (Baudez et al., 2011b) whereby increasing the solids concentration of sludge leads to an increase in the apparent viscosity and yield stress resulting in an increase in the volume of dead zones if it is mixing tank.

4.4 Conclusion

This paper demonstrates that primary and secondary sludge behave as shear thinning, yield stress fluids. Primary sludge displayed thixotropic behaviour characterised by its shear history dependent behaviour, whilst secondary sludge exhibited time dependent behaviour such that increasing the time of rest resulted in restructuration. At low shear stresses, below the yield stress, the sludge behaved as a viscoelastic solid, however, primary sludge yielded abruptly whilst secondary sludge flowed smoothly into the liquid regime. In the liquid regime, the apparent viscosity, yield stress and fluid consistency of sludge increased with increasing total solids concentration and were modelled using the Herschel-Bulkley model. A master curve was developed using a dimensionless form of the Herschel-Bulkley model allowing for the rheology of sludge at any concentration to be obtained from the master curve.

4.5 Acknowledgement

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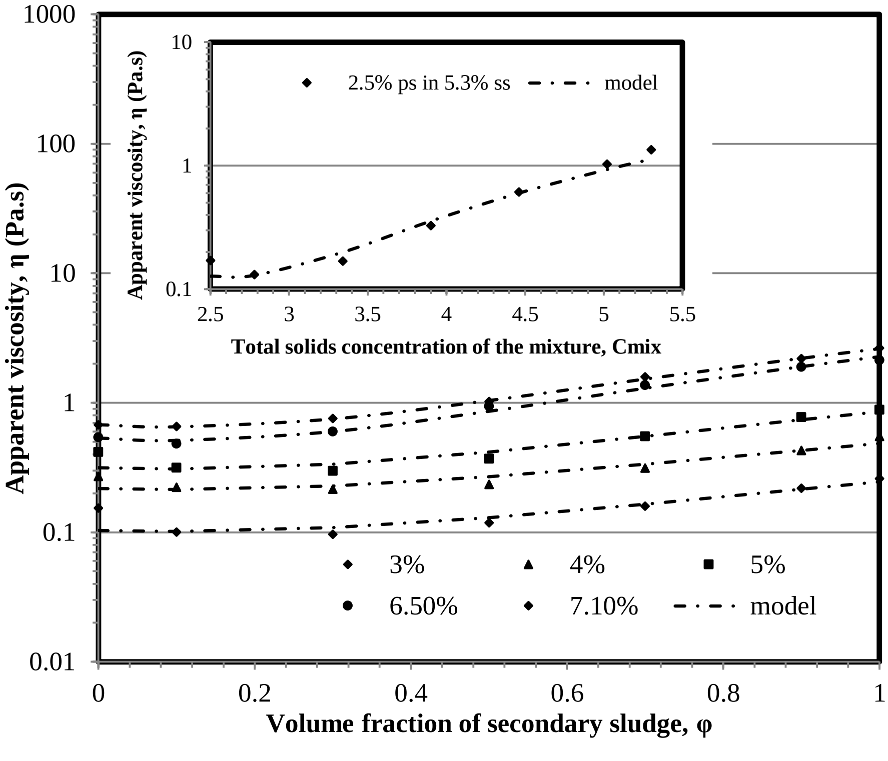
CHAPTER 5

THE APPARENT VISCOSITY AND YIELD STRESS OF PRIMARY AND SECONDARY SLUDGE MIXTURES: IMPACT OF VOLUME FRACTION OF SECONDARY SLUDGE AND TOTAL SOLIDS CONCENTRATION



Chapter 5: The apparent viscosity and yield stress of primary and secondary sludge mixtures: impact of volume fraction of secondary sludge and total solids concentration

Abstract

Sludge rheology plays an important role in the design and optimization of anaerobic digesters. Organic matter such as primary and secondary sludge or a mixture of the two sludges enters the digesters for further digestion and stabilisation. However, there is little information available on how the rheology of the mixed sludge changes. This paper investigates how the rheology of mixed primary and secondary sludge changes when the volume fraction of secondary sludge is altered. This will help predict the rheology of mixed sludge which is required for the design and optimization of pumping and mixing systems.

Mixtures of primary and secondary sludge between 2.5 and 7 %TS behave as non – Newtonian, shear thinning, yield stress materials whereby the apparent viscosity and yield stress of the mixed sludges depends on the volume fraction of secondary sludge and total solids concentration.

The apparent viscosity of primary – secondary sludge mixtures (with same total solids concentration) increases with increasing secondary sludge volume fraction. This suggests that the weak flocs of primary sludge collapse such that the colloidal like particles of primary sludge become trapped and entangled in the gel network structure of secondary sludge. However, when dilute primary sludge is mixed with concentrated secondary sludge (and vice – versa), the apparent viscosity and yield stress of the primary – secondary sludge mixture increases with increasing volume fraction of the concentrated sludge regardless of sludge type. This is due to the strengthening of hydrodynamic and non-hydrodynamic interactions within concentrated sludge.

A master curve was developed to predict the flow behaviour of sludge mixtures. Consequently, correlations were developed to predict the apparent viscosity and a yield stress of sludge mixtures as a function of volume fraction and total solids concentration.

Key words: Primary sludge, secondary sludge, mixtures, blends, rheology, Herschel – Bulkley model

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5.1 Introduction

Renewable energy is one of the key factors in sustainable sludge management. However, the production of biogas from the anaerobic digestion of waste water sludge is challenging because industry is dealing with large quantities of complex material that is not well understood (Baudez et al., 2013b).

Efficient mixing is a key factor influencing the anaerobic digester performance (Karim et al., 2004). Good mixing is required to transfer substrates to microorganisms, to maintain process stability, to maintain a uniform pH and temperature for bacterial growth, to prevent short circuiting and solids deposition in the digester bottom, and also to minimize scum and foam formation (Karim et al., 2004). As such, the processing of large volumes of sludge feed which consists of a mixture of primary and secondary sludge, combined with the fact that the current anaerobic digesters are not designed to process any additional concentrated loads leads to inadequate mixing (Eshtiaghi et al., 2012b, Eshtiaghi et al., 2013b). Inadequate mixing of sludge feed leads to reduced digester efficiency due to the formation of dead zones. The dead zones are made up of the inactive volume within the digester which creates a poor microbial environment for biogas production (Karim et al., 2004).

Consequently, any changes to the sludge feed alters digester performance, as such the importance of predicting the flow behaviour, most importantly, the apparent viscosity and yield stress of mixtures of primary and secondary sludge as the sludge feed to the digester is essential. Any changes in the flow behaviour will have a direct impact on the operating conditions and energy requirements necessary to achieve efficient mixing.

In one of the most recent studies on sludge, Markis et al (2014) investigated the impact of total solids concentration (%TS) on the rheological behavior of individual primary and secondary sludge. Markis et al (2014) demonstrated that at low stresses, below the yield stress, sludge behaved as a viscoelastic solid which was consistent with the literature on activated sludge (Baudez and Coussot, 2001, Baudez, 2008, Baudez et al., 2011b). Primary sludge yielded abruptly, a characteristic of highly thixotropic colloidal suspensions (Coussot et al., 2002) that are governed by weak attractive forces (i.e. Van der Waals forces) (Cui et al., 2011, Bayoudh et al., 2009). The EPS (**E**xtracellular **P**olymeric **S**ubstance) rich structure of secondary sludge, which is held together by hydrogen and electrostatic forces (Flemming, 1996), transitioned smoothly into the liquid regime, characteristic of gels (Baudez et al., 2013a). As such, Markis et al (2014) demonstrated that primary sludge behaved like a colloidal suspension whilst secondary sludge behaved like a gel. In the liquid regime, sludge displayed shear thinning behavior which was consistent with the pioneering works of Bhattacharya (1981) on primary sludge and Slatter (1997), Mori et al (2006) and Seyssiecq et al (2008) on activated sludge. The apparent viscosity increased exponentially with solids concentration whilst the yield stress followed a power law. This was consistent with Baudez (2008), Sanin (2002), Baudez et al (2011b), Lotito and Lotito (2014a) and Jiang et al (2014). However, these studies amongst others, focused on the rheology of one type of sludge – not a mixture.

The latest studies conducted by Baroutian et al (2013) as well as Lotito and Lotito (2014a) focus on the rheological characterization of mixed sludge at a fixed blend ratio. Baroutian et al (2013) studied the impact of solids concentration and temperature on the rheological behavior of a fixed blend ratio of primary and secondary sludge. The mixed sludge consisted of 40% primary and 60% secondary sludge. The mixed sludge was prepared at 4.3, 4.5, 4.9, 7.3 and 9.8%wt solids content. Baroutian et al (2013) found that the yield stress increased with solids concentration. The Herschel – Bulkley model was employed to characterize the flow behavior. Lotito and Lotito (2014a) conducted rheological measurements on different types of sewage sludge including anaerobic digested, raw mixed sludge, return activated sludge for pump design and found that for similar solids content, return activated sludge had the highest yield stress and Bingham viscosity followed by anaerobically digested and primary sludge. The yield stress and fluid consistency coefficient increased following a power law relationship with solids concentration (Lotito and Lotito, 2014a). Lotito and Lotito (2014a) demonstrated that raw mixed sludge was the easiest to pump whilst return activated sludge was the hardest to pump.

In the above mentioned studies, the impact of volume fractions of sludge constituents on the apparent viscosity and yield stress of mixed sludge was not studied. This highlights the lack of research on the rheological characterization of mixed primary and secondary sludge over a wide total solids concentration range and different volume fractions.

To study and understand the rheological characterization of mixed primary and secondary sludge, it is useful to consider the rheology of mixed colloidal suspensions and polymeric gels, which is similar to that of mixed sludge. Studies of Comba and Sethi (2009), Hammadi et al (2014), Abu-Jdayil and Ghannam (2014), Gómez-Díaz and Navaza (2003), Eshtiaghi et al (2013a) and Kelessidis et al (2011) focused on the rheology of mixed colloidal suspensions and polymeric gels. Comba and Sethi (2009) studied the stabilization of highly concentrated suspensions of iron nanoparticles in xanthan gum solution and found that mixing a suspension with a gel led to the formation of a viscous gel with increased stability against aggregation and sedimentation. Comba and Sethi (2009) explained that the suspension of iron nanoparticles was governed by colloidal forces whilst the xanthan gum solution was governed by hydrogen bonding and polymer entanglement leading to the formation of a gel network structure. Moreover, when iron nanoparticles were mixed with xanthan gum solution, the particles were integrated into the gel network structure of the polymer leading to the formation of a more viscous and stable dispersion (Comba and Sethi, 2009). Hammadi et al (2014) demonstrated that when polyethylene oxide (PEO) was added to bentonite clay, the yield stress and fluid consistency index of the mixture increased. Likewise, Hammadi et al (2014) explained that this trend was due to the interactions between clay particles and the viscous effect of the polymer solution.

Abu-Jdayil and Ghannam (2014) studied the change in the rheological properties of a mixture when a low viscosity polymer such as carboxymethylcellulose, CMC (0.02 to 0.5 wt %) solution is added to a highly thixotropic colloidal suspension such as bentonite. They showed that the dispersion viscosity increased significantly when CMC solution was added to bentonite dispersions. This increase was attributed to the adhesion of CMC to the surface of bentonite particles leading to the formation of a network structure within the bentonite suspension (Abu-Jdayil and Ghannam, 2014). Eshtiaghi et al (2013a) studied the impact of adding glass beads suspension to carbopol gel. They prepared suspension-gel mixtures with different volume fractions by blending 0.5, 0.7 and 1% (v/v) glass beads suspension and 0.5, 0.7 and 1.0 % (v/v) carbopol gel. A critical volume fraction was observed corresponding to a volume fraction of 0.2 ($\varphi = 0.2$) of glass bead/ carbopol mixture whereby the elastic and loss moduli changed dramatically. Eshtiaghi et al (2013a) attributed this to the collapse of the gel structure due to a loss in connectivity within the gel structure due to a reduction in polymer – polymer interaction. Kelessidis et al (2011) studied the rheology of mixtures of carbopol gel and bentonite, and demonstrated that at low polymer concentrations less than 0.5g/L the yield stress and flow consistency index decreased due to the liquefying effect caused by the adsorption of polymer to the surface of the bentonite particle, preventing the interaction between the bentonite particles. They also explained that at higher polymer concentrations, not all the polymer was adsorbed on bentonite particles resulting in an increase in the yield stress and fluid consistency index. Gómez-Díaz and Navaza (2003) studied the effect of blending CMC solution with alginate on the apparent viscosity of the mixture. They observed a minimum apparent viscosity (calculated at a shear rate of 165 s^{-1} and 231 s^{-1}) for 40% (v/v) CMC solution. They explained that the minimum apparent viscosity was probably due to a better spatial arrangement of polymer chains within the suspension leading to better packing. This led to a reduced value of the apparent viscosity. The abovementioned studies highlight that the interactions between particles influence the change in the flow behaviour prior to and after mixing, whether it is for a colloidal like suspension or for a polymer like gel.

Furthermore, all of the above mentioned studies have not developed a correlation to describe the evolution of the rheological properties of the suspension when the volume fraction of the gel within the mixture increases. By considering the interaction between primary sludge (with weak van der Waals interactions) and secondary sludge (with hydrogen and electrostatic interactions), this study focuses on the development of correlations for estimating the apparent viscosity and yield stress of mixtures of primary and secondary sludge based on their individual rheological properties and volume fractions.

5.2 Materials and method

5.2.1 Sample preparation

Sludge was sampled from two different locations – in France and Australia. This allowed for the detection of any changes in the rheological behaviour of sludge due to the different treatment processes and environmental conditions the sludge has experienced. Table 5.A.1 contains a summary of the different total solids concentrations which were used to prepare the different mixtures of primary and secondary sludge. Table 5.A.2 contains a summary of the volume of primary and secondary sludge required to make up the mixtures.

5.2.1.1 French sludge

Samples of primary sludge and secondary sludge were obtained from Bessay (Allier, France) and Vichy (Allier, France), respectively. Their initial total solids concentration was 2.8 and 22%, respectively.

The vacuum filtration technique was used to thicken the primary sludge from an initial total solids concentration of 2.8% to 5.0 and 5.4% (w/w) (Markis et al., 2014). Secondary sludge was diluted using tap water to 2.8 and 5.0% (w/w) to obtain the same total solids concentration as those of primary sludge.

First, mixtures of primary and secondary sludge at the same individual total solids concentration were prepared by mixing primary sludge with 5.0% TS with secondary sludge with 5% TS (i.e. PS: SS) at various volume fractions (from 0 to 1 v/v) of secondary sludge. This was followed by preparing mixtures of primary sludge with 5.4% TS and secondary sludge with 2.8% TS, while varying the secondary sludge volume fraction in the mixture from 0 to 1.

5.2.1.2 Australian sludge

Primary sludge was obtained from the Eastern Waste Water Treatment Plant located in Melbourne, Victoria, Australia. Secondary sludge was obtained from the Mount Martha Waste Water Treatment Plant located on the Mornington Peninsula, Victoria, Australia.

Primary sludge and secondary sludge with a low total solids concentration (both at 3.0%TS) were thickened to various total solids concentrations (3.0, 4.0, 5.0, 6.5, 7.1%TS) using the vacuum filtration technique.

Firstly, primary sludge samples with 3, 4, 5, 6.5 and 7.1%TS were mixed with secondary sludge samples at same TS, while varying the secondary sludge volume fraction in the mixture from 0 to 1. Secondly, dilute primary sludge at 2.5% TS was mixed with concentrated secondary sludge at 5.3% TS, while varying the secondary sludge volume fraction in the mixture from 0 to 1.

Both French and Australian sludge samples were stored at 4 °C for 30 days prior to experiments so that any changes to the composition that may alter the rheology were reduced ensuring that the same material is always used throughout in all our experiments (Baudez et al., 2011b).

5.2.2 Rheometric technique

Creep tests were performed on sludge samples from both France and Australia. The experimental procedure consisted of conducting successive creep tests using the following pattern: the sludge was first pre – sheared (150s) at a shear stress corresponding to a high shear rate resulting in a homogeneous material that was always in the same initial state of destructuration (Coussot, 2005, Baudez, 2008, Markis et al., 2014). This was followed by a short period of rest (150s) allowing the structure to rebuild (Coussot, 2005, Baudez, 2008, Markis et al., 2014). Then, a creep corresponding to a constant stress was applied for a specific duration of time (either 120s or 154s). The constant stress that was applied was dependant on the solids concentration.

Successive creep tests were carried out over a wide stress range (in the solid, liquid and transition regimes) to obtain sufficient data for the reconstruction of the flow curve. The flow curve of the sludge mixtures was reconstructed using a procedure detailed elsewhere (Markis et al., 2014).

The creep tests were performed using an Anton Paar physica MCR 300 rheometer in France and a Discovery Hybrid rheometer (HR3) in Australia. Both instruments were equipped with the vane geometry to reduce inertia effects (Dzuy and Boger, 1983, Dzuy and Boger, 1985). The dimensions of the vane geometry for each instrument are summarized in Table 5.A.3. The surfaces of the rheometric tools were roughened to avoid wall slip (Tabuteau et al., 2004).

The experiments were carried out at a temperature of 20°C.

5.3 Results and discussion

5.3.1 The rheological behaviour of primary and secondary sludge and mixtures of primary and secondary sludge

5.3.1.1 Flow behaviour of primary and secondary sludge

The rheological behaviour of sludge in the steady state regime was investigated by reconstructing the shear stress versus shear rate curve (i.e. flow curve). Figure 24 (a) demonstrates that primary (5% TS) and secondary sludge (5% TS) displayed non-Newtonian shear thinning behaviour such that the corresponding apparent viscosity decreased with increasing shear rate (Figure 24c). A yield stress is detected allowing for the flow properties to be estimated using the Herschel – Bulkley model as it takes into account the yield pseudo-plasticity of the sludge (Slatter, 1997). The observed flow behaviour is in agreement with the most recent study conducted by Markis et al (2014) as well as the early work of Bhattacharya (1981), Baudez (2008), Mori et al (2006) and Seyssiecq et al (2008), who described primary and secondary sludges as non-Newtonian shear thinning materials exhibiting a yield stress.

Figure 24 (a) illustrates that secondary sludge requires a greater stress to flow compared to primary sludge at the same total solids concentration of 5%. Seyssiecq et al (2003) explained that the observed behaviour can be attributed to the interactions between particles. The colloid – like structure of primary sludge was governed by Van der Waals forces (Cui et al., 2011, Bayoudh et al., 2009) and the gel like network structure of secondary sludge was governed by hydrogen bonds and electrostatic interactions (Flemming, 1996). As such, weak links are formed between the flocs of primary sludge compared to strong links formed between the flocs of secondary sludge. Secondary sludge breaks down and flows in the steady state regime at higher stresses as compared with primary sludge at the same solids concentration. This may explain the difference in the flow behaviour between primary and secondary sludge at the same total solids concentration.

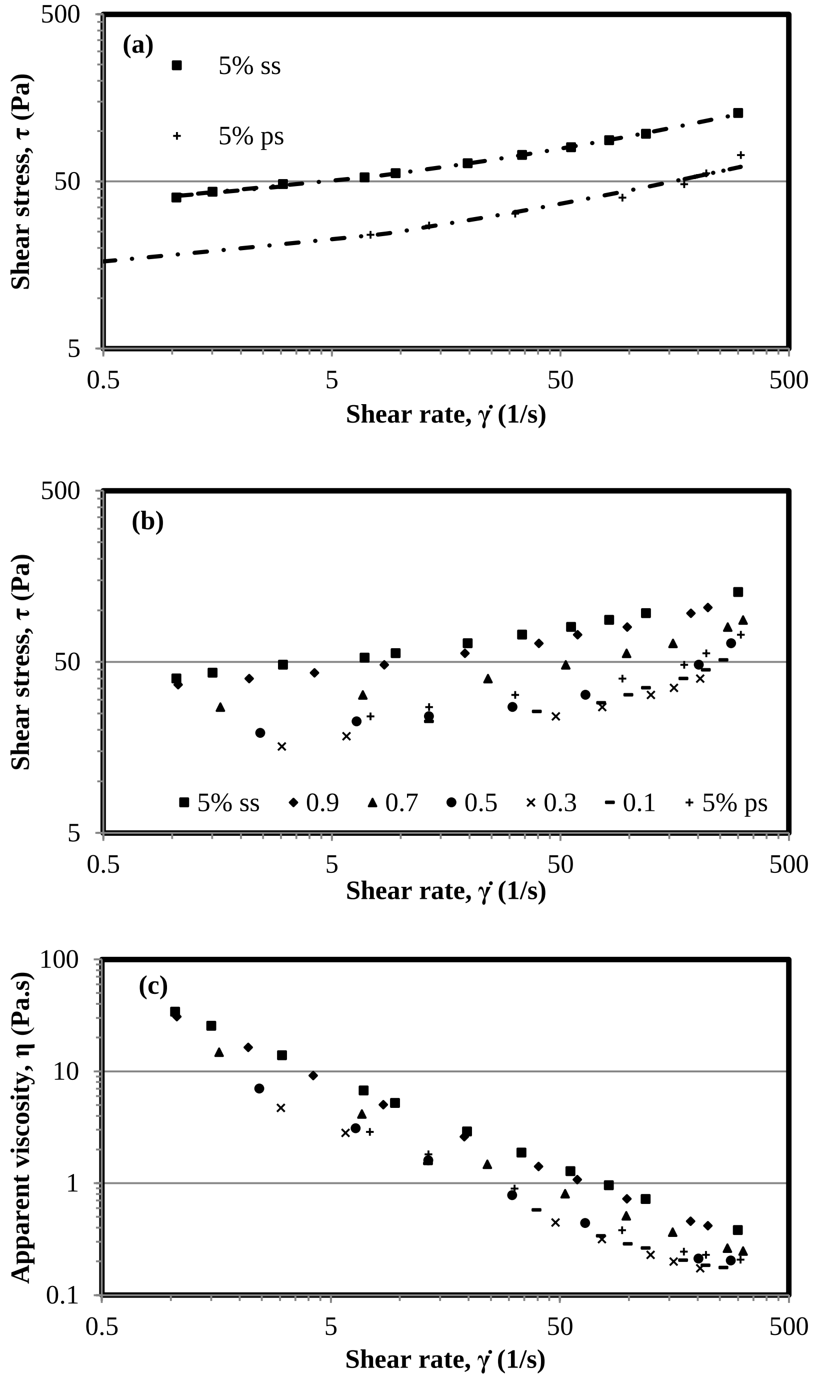

Figure 24: Flow curves of 5% primary sludge and 5% secondary sludge modelled using the Herschel – Bulkley model (b) Flow curves and (c) Apparent viscosity curves of mixtures of 5% primary and 5% secondary sludge – sampled in Australia

Most recently, Markis et al (2014) used the derivative (i.e. shear rate versus time) of the creep curves (i.e. shear strain versus time) to highlight the difference in flow behaviour between primary and secondary sludge. They (Markis et al., 2014) demonstrated that when a constant stress (below the yield stress) is applied to secondary sludge, the derivative of the creep response – the shear rate, decreases as a function of time whereas the shear rate of primary sludge is finitely constant (for the same constant applied stress). This means that secondary sludge remained in the solid regime and required a higher stress to cause flow, whilst primary sludge flowed steadily in the liquid regime. Furthermore, Markis et al (2014) used the power law index of the asymptote $\left(\dot{\gamma} = At^{n} + B\right)$ of the creep curve to demonstrate that primary sludge displayed abrupt yielding similar to shear dependent fluids such as colloidal suspensions (Coussot et al., 2002). However secondary sludge transitioned smoothly into the liquid regime, similar to gels, emphasizing that the interactions governing the structure influence the flow behaviour of sludge. The derivative of the creep curve – the shear rate versus time, is presented in Figure 25 (a) for primary and secondary sludge at the same total solids concentration of 5%. This highlighted the difference in the flow behaviour such that when the same stress was applied, primary sludge flowed whilst secondary sludge remained in the solid regime. Indeed, by comparing the two different responses at a shear rate of 1.0 s^{-1} (for the same applied stress), it is reasonable to assume that primary sludge flows whilst secondary sludge required a higher stress to reach a shear rate of 1.0 s^{-1}; hence, it remained in the solid regime. Furthermore, the colloid like behaviour of primary sludge was highlighted in Figure 25 (b) whereby an undershoot was observed in the power law index of primary sludge suggesting that the flow is disordered and demonstrates that primary sludge yields abruptly. In contrast, secondary sludge transitions smoothly into the liquid regime. This validated what has been presented in literature by Markis et al (2014) and demonstrates that the interactions between the particles of primary and secondary sludge influence the flow behaviour.

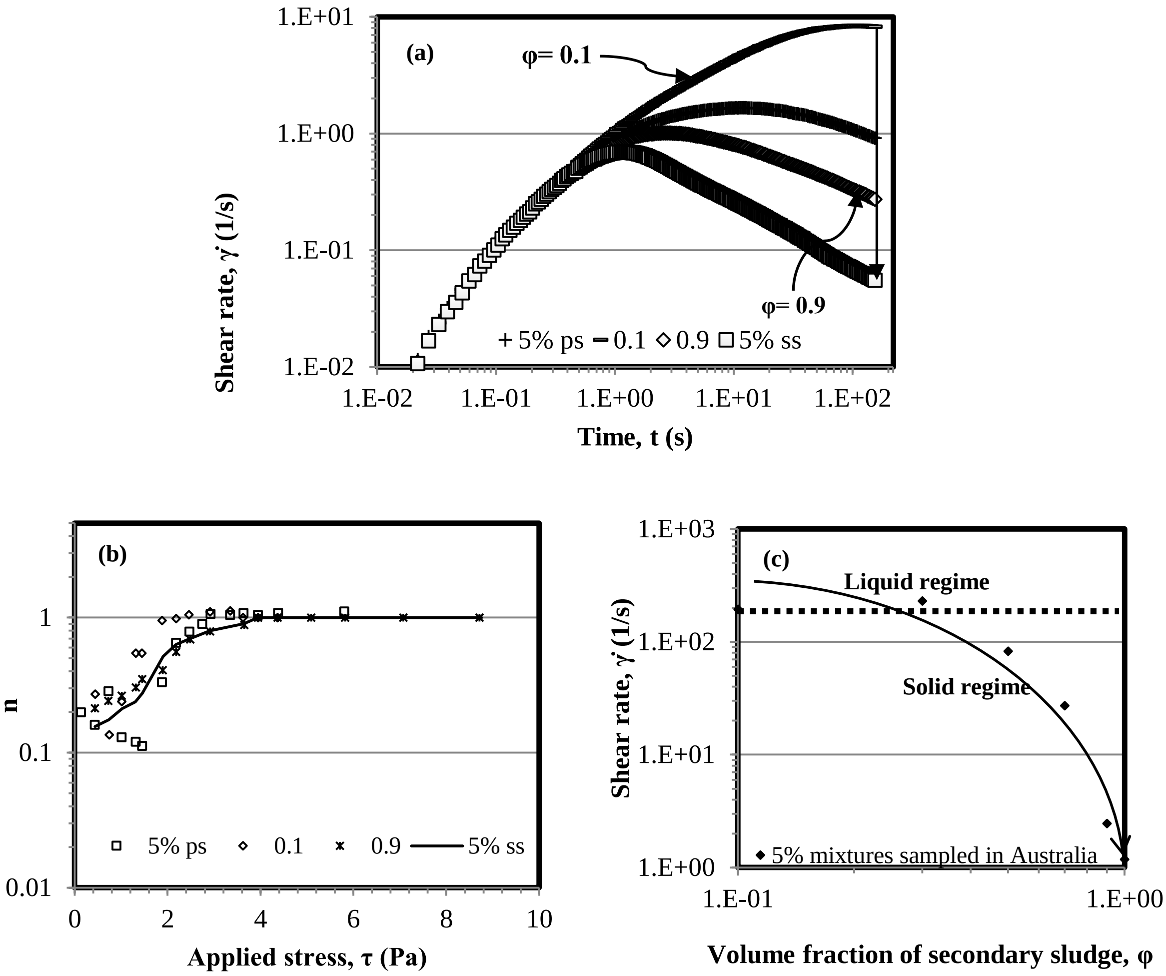

Figure 25: (a) Shear rate versus time curve for mixtures of 5%TS primary sludge and 5%TS secondary sludge (φ = 0.1 and 0.9) sampled in France (τapplied = 2.2 Pa); (b) Power law index of the asymptote of the creep curve for mixtures of 5% primary sludge and 5%secondary sludge sampled in France – the solid line illustrates the yielding; (c) Evolution of the final point of the shear rate versus time curve as a function of volume fraction of secondary sludge for 5% sludge mixtures sampled in France ($\tau_{applied}$ = 2.2 Pa) – the dashed line illustrates the solid – liquid transition

Lotito and Lotito (2014a) observed a difference in the flow behaviour in their study on the rheological characterisation of three sludges (primary, return activated and anaerobically digested sludge) for pumping design purposes. The difference in the flow behaviour was highlighted such that primary sludge flowed at lower stresses (i.e. lowest Bingham parameters and easiest to pump) whilst return activated sludge only flowed at the highest stresses (i.e. highest Bingham parameters and hardest to pump), which is in fact what is shown in this study and explained in terms of particle interactions.

The described difference in flow behaviour between primary and secondary sludge is used to demonstrate that the flow behaviour of mixtures of primary and secondary sludge changes from colloid-like to gel-like as the fraction of secondary sludge added into the mixture increases.

5.3.1.2 Flow behaviour of mixtures of primary and secondary sludge

The rheological behaviour of mixtures primary and secondary sludge (at the same individual total solids concentration) was investigated by plotting the flow curves and corresponding apparent viscosity curves, shown in Figures 24 (b) and (c), respectively. Figure 24 (b) illustrates that the mixtures of 5% primary and 5% secondary sludge sampled in Australia behaved as non-Newtonian fluids whereby the flow curves are non – linear and do not pass through the origin so that a yield stress is detected. Figure 24 (b) demonstrates that the apparent viscosity decreased as a function of shear rate, which is fundamentally shear thinning behaviour. Dilute and concentrated sludge mixtures prepared at the same total solids concentration of primary and secondary sludge and sampled in either France (5% TS) and Australia (3, 4, 6.5, 7.1% TS) displayed the same behaviour (not shown) and were in agreement with the current literature on sludge (Baudez, 2008, Markis et al., 2014, Baroutian et al., 2013, Lotito and Lotito, 2014a). Similarly, sludge mixtures prepared at a two different total solids concentration (i.e. dilute primary sludge mixed with concentrated secondary sludge and vice versa) and sampled in France (5.4% primary in 2.8% secondary) and Australia (2.5% primary in 5.4% secondary) displayed the same non- Newtonian, shear thinning yield stress (not shown). As such, a master curve was developed which compares, predicts and estimates the flow behaviour of mixtures of primary and secondary sludge regardless of the volume fraction and total solids concentration of the sludge mixture. Most importantly, the master curve may be used to obtain the apparent viscosity and yield stress of individual primary and secondary sludge as well as mixtures of these two sludge types.

The master curve was developed by taking the flow curve of 7.1% TS secondary sludge as the reference curve and superimposing the remainder of the flow curves of the sludge mixtures using shift factors.

The shift factors in the x and y axes are presented in Table 16. A dimensionless form of the Herschel – Bulkley model was then fitted following the theoretical form described elsewhere (Markis et al., 2014):

$$\frac{\tau}{\tau_c} = \tau_c + K\dot{\gamma}^n \rightarrow \frac{\tau}{\tau_c} = 1 + \left(\frac{K}{\tau_c}\right)\dot{\gamma}^n \rightarrow \frac{\tau}{\tau_c} = 1 + \beta\Gamma^n \; where \; \Gamma = \left(\frac{\eta_o}{\tau_c}\right)\dot{\gamma} \; and \; \beta = \left(\frac{K}{\tau_c}\right)\left(\frac{\tau}{\eta_o}\right)^n$$

, where τ/τ_c describes the dimensionless shear stress and Γ is the dimensionless shear rate, τ_c is known as the yield stress, below which steady state flow cannot be achieved, η_0 is a measure of the viscosity and equals 1, K is the fluid consistency, and n is the flow index.

Table 16: Summary of the shift factors in the x (S_x) and y (S_y) axes for each mixture

Shift factor in the x axis, S_x								
	France		**Australia**					
Total solids concentration, (%)								
φ	**5 %**	**5.4% in 2.8%**	**3 %**	**4%**	**5%**	**6.5%**	**7.1%**	**2.5% in 5.3%**
0	0.045	0.150	0.067	0.110	0.350	0.700	0.900	0.130
0.1	0.035	0.150	0.050	0.130	0.250	0.700	0.800	0.150
0.3	0.033	0.200	0.100	0.170	0.220	0.550	0.750	0.180
0.5	0.035	0.080	0.135	0.300	0.300	0.700	0.700	0.300
0.7	0.030	0.030	0.170	0.400	0.350	0.850	0.430	0.500
0.9	0.035	0.022	0.170	0.430	0.370	0.900	0.700	0.600
1	0.037	0.025	0.200	0.500	0.380	0.950	1.000	0.900
Shift factor in the y axis, S_y								
Total solids concentration, (%)								
	France		**Australia**					
φ	**5 %**	**5.4% in 2.8%**	**3%**	**4%**	**5 %**	**6.5%**	**7.1%**	**2.5% in 5.3%**
0	0.011	0.042	0.030	0.061	0.125	0.190	0.250	0.040
0.1	0.007	0.040	0.018	0.052	0.087	0.170	0.237	0.032
0.3	0.008	0.035	0.021	0.054	0.080	0.200	0.270	0.043
0.5	0.010	0.018	0.028	0.068	0.107	0.330	0.360	0.095
0.7	0.011	0.007	0.040	0.097	0.165	0.500	0.500	0.200
0.9	0.014	0.003	0.055	0.135	0.235	0.700	0.770	0.350
1	0.017	0.004	0.068	0.180	0.270	0.800	1.000	0.500

The master curve in the dimensionless form of the shear rate (Γ) and shear stress (τ/τ_c) of sludge mixtures, prepared at same total solids concentration of primary and secondary sludge and at two different total solids concentration and sampled in France and Australia is presented in Figure 26. The rheological parameters of the Herschel – Bulkley model (i.e. the fluid consistency, K, and yield stress, τ_c) of each mixture of sludge was recalculated using the shift factors (see table 16) and the rheological parameters of the dimensionless form of Herschel – Bulkley model. The flow index, n, was kept constant. From these parameters, the apparent viscosity at a single shear rate of 100 s^{-1}, similar to the low shear condition within anaerobic digesters as well as the yield stress of each mixture may be calculated. In this way, the impact of volume fraction and total solids concentration on the rheological behaviour of mixtures of primary and secondary sludge can be investigated.

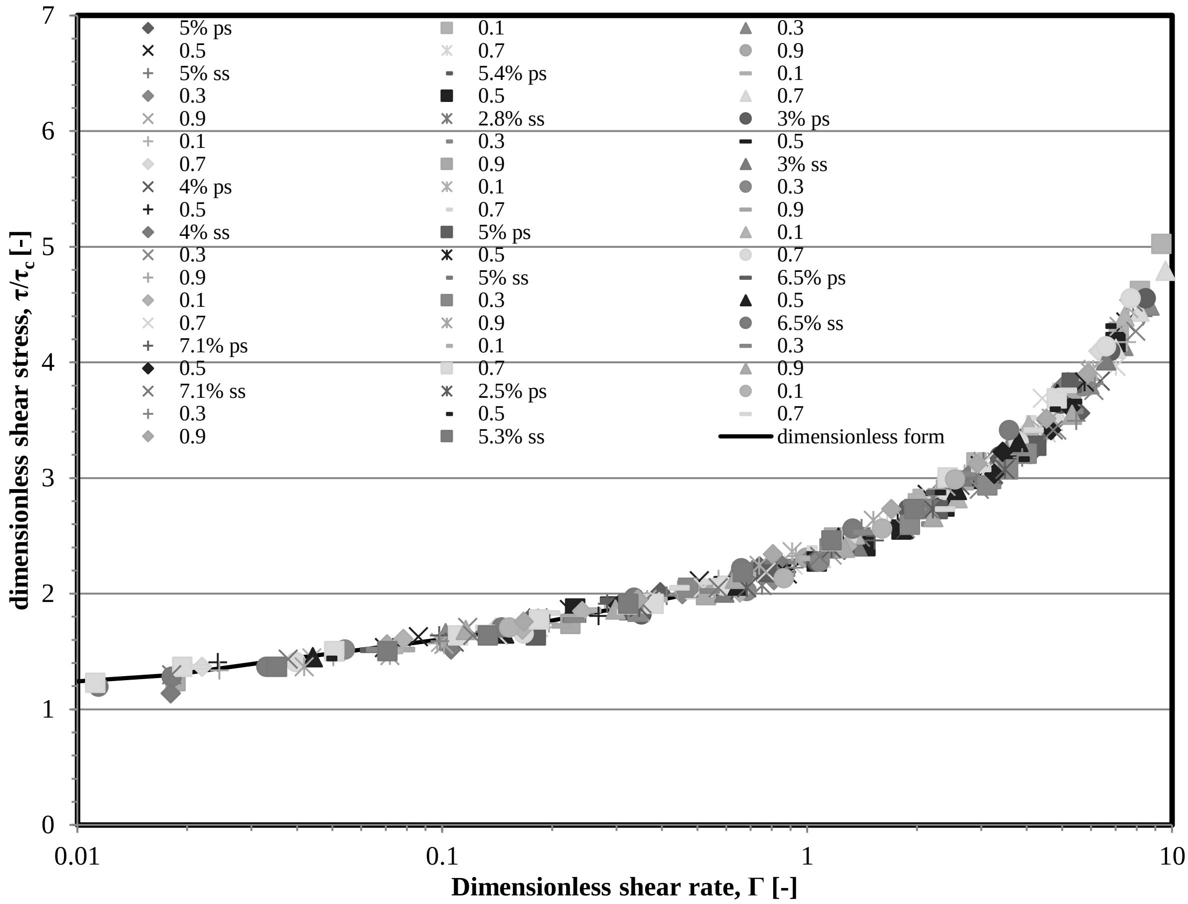

Figure 26: Master curve in the dimensionless form of the Herschel – Bulkley model with the parameters τ_c = 117.13, K = 27.14, n = 0.367, β = 1.33 for mixtures of primary and secondary sludge sampled in France (5% ps in 5% ss; 5.4% ps in 2.8% ss) and Australia (3% ps in 3% ss; 4% ps in 4% ss; 5% ps in 5% ss; 6.5% ps in 6.5% ss; 7.1% ps in 7.1% ss and 2.5% ps in 5.3% ss)

5.3.1.3 Impact of volume fraction of secondary sludge on the apparent viscosity and yield stress of sludge mixtures at a similar total solid concentrations

The evolution of the apparent viscosity (at a constant shear rate of 100 s^{-1}) with volume fraction of secondary sludge (in the range of 0.1, 0.3, 0.5, 0.7 and 0.9 by volume of secondary sludge) is presented in Figure 27 for sludge sampled in France (a) and Australia (b). Figure 27 (a) demonstrates that when secondary sludge is added to primary sludge, the apparent viscosity of the resulting mixture changes from the apparent viscosity of primary sludge. Figure 27 (a) illustrates that the apparent viscosity of the resulting mixture initially decreases (when φ = 0.1), followed by a gradual increase in the apparent viscosity as the volume fraction of secondary sludge increases toward 1 (ie. φ→1) such that secondary sludge (when φ = 1) displays the highest apparent viscosity. Figure 27 (a) shows that the mixture corresponding to a volume fraction of 0.1 (i.e. φ = 0.1) displayed a minimum apparent viscosity (i.e. flows at the lowest shear stress). Figure 27 (b) demonstrates sludge mixtures prepared by mixing secondary sludge to primary sludge at the same total solids concentration and sampled in Australia followed the same trend as French sludge at all range of solid concentrations whereby dilute and concentrated sludge mixtures (3, 4, 5.0, 6.5 and 7.1% TS) displayed a minimum apparent viscosity when φ = 0.1.

Likewise, Figure 27 (c) and (d) demonstrates that the yield stress followed the same trend for sludge mixtures sampled in France (c) and Australia (d). A minimum yield stress was detected when φ = 0.1 for sludge mixtures sampled in France and for sludge mixtures sampled in Australia.

A minimum apparent viscosity or fluid consistency and yield stress was observed by Gómez-Díaz and Navaza (2003) and Kelessidis et al (2011). Gómez-Díaz and Navaza (2003) calculated the apparent viscosity of mixtures of CMC and alginate at two different shear rate values of 164 and 231 s^{-1} and observed a minimum apparent viscosity near 40% (volume %) of CMC. Gómez-Díaz and Navaza (2003) attributed the observed minimum to a better arrangement of the polymer chains within the aqueous solutions leading to a better packing and reduced apparent viscosity. Eshtiaghi et al (2013a) studied the impact of adding (at different volume fractions) glass beads suspension to carbopol gel whereby 0.5, 0.7, and 1% glass beads suspension was added to 0.5, 0.7, and 1.0% carbopol gel, respectively. They observed a critical volume fraction (φ = 0.2) whereby the gel structure collapsed due to a loss in connectivity within the gel structure. As such, the polymer – polymer interactions were reduced. Eshtiaghi et al (2013a) explained that increasing the solids loading interfered with the gel network because particles acted as a barrier which prevented the polymer entanglement of the gel structure. The newly formed particle – gel structure weakened the gel network. Kelessidis et al (2011) showed that when carbopol (up to 1.5% by mass) was mixed with 3 and 4 wt% sodium – bentonite water dispersions, a minimum yield stress and fluid consistency index was detected at low polymer concentrations (so, < 0.5g/L). The yield stress and fluid consistency index then increased with increasing concentrations of polymer. At low polymer concentrations, Kelessidis et al (2011) explained that the yield stress and fluid consistency index decreased because of the liquefying effect. This was caused by the binding of polymers onto the surface of the bentonite particle preventing the edge-to-face interactions of bentonite particles. However, Kelessidis et al (2011) explained that at higher polymer concentrations, not all the polymer was adsorbed resulting in an increase in the yield stress and fluid consistency index.

The minimum apparent viscosity and yield stress is probably due to the liquefying effect caused when secondary sludge is first added to primary sludge, that is corresponding to a low volume fraction of secondary sludge, (φ = 0.1). Cui et al (2011) and Bayoudh et al (2009) explained that the weakly flocculated structure of primary sludge is governed by weak van der Waals forces. It breaks down when secondary sludge is first added. As such, any newly formed links prevent the particle – particle interactions between the particles of primary sludge and result in a reduced apparent viscosity and yield stress value. At higher volume fractions, so when $\varphi > 0.1$, Figure 4 illustrates that the apparent viscosity and yield stress increased.

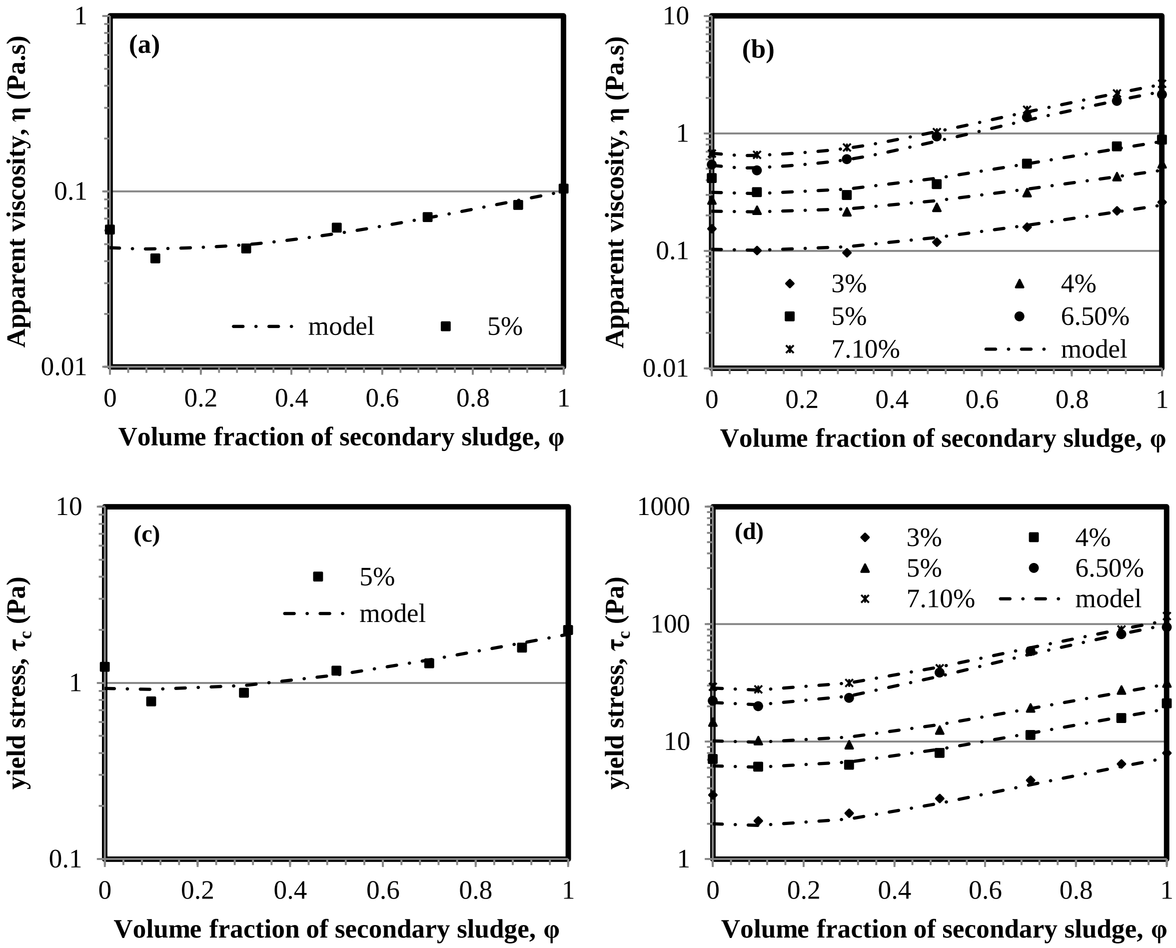

Figure 27: Evolution of the apparent viscosity at a single shear rate of 100s^{-1} as a function of the volume fraction of secondary sludge for sludge mixtures with (a) 5% total solids concentration sampled in France and (b) 3, 4, 5, 6.5 and 7.1% total solids concentration sampled in Australia; evolution of the yield stress as a function of volume fraction of secondary sludge for sludge mixtures with (c) 5% solid concentrations sampled in France (d) 3, 4, 5, 6.5 and 7.1% total solids concentration sampled in Australia

Comparisons were made with the work of Comba and Sethi (2009) and Abu-Jdayil and Ghannam (2014) who demonstrated that a more viscous and stable dispersion was formed resulting in a higher viscosity when colloidal suspensions were added to polymeric gels. Comba and Sethi (2009) explained that colloidal forces governed the flow behaviour within the suspension of iron nanoparticles whilst xanthan gum formed a gel network through hydrogen bonding and polymer entanglement. Comba and Sethi

(2009) states that a gel network structure such as xanthan gum can trap particles and therefore stabilize dispersions. This can be either due to the adsorption of particles into the gel network or the polymer forms a network around the particles. In such cases, a more viscous and stable dispersion is produced. Also, Abu-Jdayil and Ghannam (2014) demonstrated that the apparent viscosity and shear stress, when CMC was added to bentonite, increased and attributed it to inter – particle interactions and ions (or molecules). They explained that when polymers were adsorbed onto the surfaces of particles, it could result in either steric stabilization or bridging flocculation such that the rheological properties and stability of the resulting mixture were altered.

In fact, this is true in the case of the mixtures with a secondary sludge volume fraction higher than 0.1 whereby the flocs within the mixture are bound by both weak interactions as well as electrostatic and hydrogen bonds. As the amount of secondary sludge within the mixture increases (i.e. $\varphi \rightarrow 0.1$), the weak flocs collapse and restructure to form a more gel like network structure. This restructuration may be the entrapment of the primary sludge particles by the network of secondary sludge so that the structure of the resulting sludge mixture is governed by hydrogen and electrostatic interactions. This leads to the formation of strong links between the flocs resulting in flowage at higher shear stresses. Hence, a more viscous mixture is formed that requires higher stresses to achieve steady state flow, which is clearly evident when $\varphi = 0.9$.

Moreover, Comba and Sethi (2009) explained that the higher apparent viscosity they have observed is a direct consequence of network stiffness and rigidity. Indeed, Figure 4 shows that the apparent viscosity and yield stress of mixtures of primary and secondary sludge increases with increasing volume fraction of secondary sludge. This is a clear indication that a more viscous and rigid mixture is formed compared to the primary sludge. Comba and Sethi (2009) went a step further and defined the apparent viscosity as a macroscopic measure of the forces opposed to settling. Since mixtures of primary and secondary sludge exhibited elevated apparent viscosity values compared to primary sludge, one can deduce that the mixture of primary and secondary sludge has a lower settleability compared to primary sludge.

A closer look at the derivative of the creep response presented in Figure 25 (a) – the shear rate versus time curve for mixtures of 5% primary and 5% secondary sludge when $\varphi = 0.1$ and 0.9 may be used to explain the described abovementioned trend.

The shear rate versus time curves which are presented in Figure 25 (a) highlight a change in flow behaviour as the volume fraction of secondary sludge increases. When the same constant stress is applied ($\tau_{applied} = 2.2$ Pa) to a sludge mixture with a volume fraction of 0.1 ($\varphi = 0.1$, denoted 0.1 and depicted as the filled dashed bullet in Figure 25a), the shear rate first increased rapidly as a function of time followed by plateau, indicating that the mixture flows steadily in the liquid regime. In contrast, When $\varphi = 0.9$ (denoted 0.9 and depicted as the open diamond bullet in Figure 25a), the shear rate first increased, followed by a rapid decline, indicating that the mixture remained in the solid regime (for the same applied stress). Using these two volume fractions, it can be stated that when the amount of secondary sludge added to the mixture increases from $\varphi = 0.1$ to $\varphi = 0.9$, its flow behaviour changes from the liquid to solid regime for the same applied stress.

By plotting the evolution of the final point of the shear rate (1/s) versus time curve for each volume fraction of secondary sludge, the observed changes to the flow behaviour are highlighted further. Figure 25 (c) shows that the shear rate declines as the volume fraction increases from 0.1 to 0.9. The decline of the shear rate as the volume fraction of secondary sludge increases from 0.1 to 0.9 suggests that the mixture does not flow (i.e. mixture does not move) and remains in the solid regime for the same applied stress ($\tau_{applied} = 2.2$ Pa). As such, mixtures with a higher fraction of secondary sludge (so, $\varphi > 0.1$) require a higher stress to flow steadily into the liquid regime compared to those with a higher fraction of primary sludge ($\varphi \leq 0.1$).

The observed transition from the liquid regime to the solid regime as the volume fraction increases can be attributed to the strengthening of the interactions between the particles which in turn results in a stronger network structure. Indeed, Hammadi et al (2014) explained that the intensification of the interactions between the clay particles and the polymer led to difficult movement in the dispersion medium resulting in an increased yield stress. Similarly, the interactions between the particles of primary sludge and the gel like network structure of secondary sludge intensified as the volume fraction of secondary sludge increased. As such, the mixture became more difficult to move when the same stress was applied ($\tau_{applied}$ = 2.2 Pa) resulting in a higher yield stress. Hence, mixtures with a higher volume fraction required a higher stress ($\tau_{applied}$ > 2.2 Pa) to flow into the liquid regime.

Moreover, the power law index of the asymptote of the creep curve for mixtures of primary and secondary sludge can be used to link the change from the liquid to solid regime to the inter-particle interactions which influence the flow behaviour of sludge mixtures.

The power law index of different volume fractions of mixtures of 5% primary and 5% secondary sludge is illustrated in Figure 25 (b). When $\varphi = 0.1$ (denoted 0.1 and shown as an open diamond in Figure 25b; represents 10% secondary sludge and 90% primary sludge), an abrupt and distinct undershoot is observed – similar to the power law index of primary sludge described in section 5.3.1.1 and similar to the phenomenon described by Markis et al (2014). The undershoot seen for $\varphi = 0.1$ implies that as n approaches 1 ($n \to 1$), the derivative of the shear strain, which is the shear rate, decelerates then accelerates rapidly with time suggesting that the structure rebuilds then collapses rapidly from solid-like ($n < 1$) to liquid-like ($n > 1$) and vice versa, yet no steady state flow can be reached. As such the flow is disordered demonstrating that the mixture with $\varphi = 0.1$ experiences abrupt yielding. Coussot et al (2002) explained that this is a characteristic of highly colloidal suspensions such as bentonite. However, when $\varphi = 0.9$, the undershoot shown levels such that the mixture transitions smoothly into the liquid regime – as described by Markis et al (2014) for secondary sludge. Markis et al (2014) attributed this smooth transition to the gel like characteristics of secondary sludge. It is also similar to the power law index of secondary sludge, presented in Section 5.3.1.1. By comparing the power law index of the asymptote for the mixture containing mainly primary sludge ($\varphi = 0.1$) and for the mixture containing mainly secondary sludge ($\varphi = 0.9$), we show that the interactions within the mixture may be altered as the volume fraction of secondary sludge approaches 1 and influence the flow behaviour. By making a comparison with the work of Comba and Sethi (2009), it can be deduced that the primary sludge particles may be trapped within the rigid polymer matrix of secondary sludge.

The abrupt yielding and disordered flow described using the undershoot of the power law index when the mixture contains mainly primary sludge (φ ≤ 0.1 in Figure 2b) also implies that at lower shear rates, particle settling may occur. When mixture with φ ≤ 0.1 is subjected to low shear rates (or shear stress), the mixture cannot flow steadily over time in the liquid regime leading to abrupt stoppage after initial flow. This indicates that the sludge mixture initially undergoes deflocculation, followed by flocculation whereby large aggregates are formed. These aggregates result in a less stable mixture that can settle (i.e. φ ≤ 0.1 undergoes particle settling). However, the smooth transition of the power law index when φ = 0.9 (see Figure 25b) suggests that a more stable mixture is formed.

Figure 28 (a) and (b) illustrates that the apparent viscosity and yield stress of mixtures of primary and secondary sludge increases with the increasing total solids concentration of the mixture. This was attributed to the strengthening of the particle interactions within the mixture as the total solids concentration of the mixture increased. In fact, this was also explained by Seyssiecq et al (2003) and Baudez (2008) and Baudez et al (2011b). Seyssiecq et al (2003) explained that when the concentration increases, stronger links are formed between the flocs due to the strengthening of the particle interactions, as such the apparent viscosity and yield stress increases. Similarly, Baudez (2008) and Baudez et al (2011b) explained that increasing the total solids concentration strengthened the hydrodynamic interactions (between the solid particles and surrounding fluid) and non-hydrodynamic interactions (between the solid particles). Baudez (2008) and Baudez et al (2011b) explained that the apparent viscosity and yield stress are a direct measure of these interactions, as such they increase with total solids concentration. Furthermore, Baudez (2008) and Baudez et al (2011b) demonstrated that the apparent viscosity of digested sludge followed an exponential increase with total solids concentration whilst the yield stress followed a power law. More recently, Markis et al (2014) validated this for separate primary and secondary sludge over a wide range of total solids concentration.

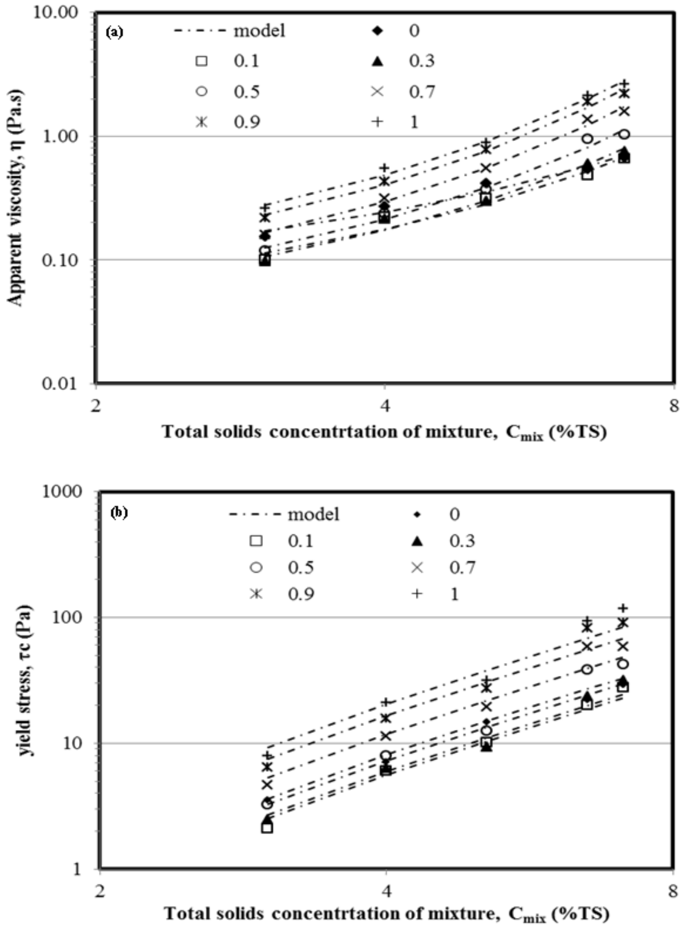

Figure 28: Evolution of the (a) Apparent viscosity at a single shear rate of 100s^{-1} and (b) Yield stress as a function of the total solids concentration of the mixture (at different volume fractions) for sampled in Australia

Since the exponential and power law models were employed by Markis et al (2014) on individual primary and secondary sludge, it is reasonable to assume that the model may be used to predict the apparent viscosity and yield stress of mixtures of primary and secondary sludge as a function of total solids concentration of the mixture. These are presented in Figure 28 for mixtures sampled in Australia. The equations are as of the following.

$$\eta = \eta_0 \exp(C\beta)$$ Eq. 35

$$\tau_y = \alpha(C - C_{min})^m$$ Eq. 36

, where C_{mix} is the total solids concentration of the mixture, η_0 is the viscosity of the liquid (Pa.s), m is a fitting parameter related to the fractal dimension of sludge flocs and equal to 2, α and β are fitting parameters.

α and β followed a polynomial relationship with volume fraction of secondary sludge, and η_0 can be estimated using Figure 5.A.1. A summary of the parameters required for Eq. 35 and Eq. 36 are presented in Table 17.

C_{min} is the lowest concentration below which there is no yield stress. Since we have demonstrated that the yield stress is influenced by the presence of secondary sludge within the mixture, when the mixtures are prepared at a similar total solids concentration, C_{min} was kept at a constant value of 0.96. This was the minimum concentration determined elsewhere (Markis et al., 2014) for secondary sludge, below which no yield stress was detected.

Table 17: Summary of the parameters required to fit Eq. 35 and 36

Apparent viscosity							
France							
φ	0	0.1	0.3	0.5	0.7	0.9	1
η_0	0.70	0.67	0.68	0.68	0.67	0.71	0.75
β	-0.49	-0.56	-0.53	-0.48	-0.45	-0.43	-0.40
Australia							
η_0	0.06	0.03	0.02	0.03	0.03	0.04	0.05
β	0.34	0.43	0.49	0.53	0.57	0.57	0.56
Yield stress							
France							
α	0.08	0.05	0.05	0.07	0.08	0.10	0.12
Australia							
α	0.79	0.61	0.65	0.88	1.28	1.81	2.22

In all cases, when the apparent viscosity and yield stress are plotted as a function of volume fraction of secondary sludge, a minimum occurs when φ = 0.1. This suggested that the evolution of the apparent viscosity and yield stress with volume fraction of secondary sludge should follow a power law function with a minimum volume fraction:

$$\eta_{mix\,(C_{PS}=C_{SS})} = \eta_{SS}\,[\alpha(\varphi - \varphi min)^2 + \beta] \quad \text{Eq. 37}$$

$$\tau_{mix\,(C_{PS}=C_{SS})} = \tau_{SS}\,[\alpha(\varphi - \varphi min)^2 + \beta]$$ Eq. 38

The total solids concentration of the individual sludge is neglected from the correlations because the primary sludge and secondary sludge were mixed at the same individual total solids concentration.

η_{ss} and τ_{ss} are the apparent viscosity and yield stress of secondary sludge, these are selected because it was shown that when the mixtures were prepared by mixing sludge at a similar concentration, the apparent viscosity and yield stress of the mixture are influenced by the presence of secondary sludge.

φ_{min} is the minimum volume fraction, which takes into account the liquefying effect experienced when secondary sludge is first added. $\varphi_{min} = 0.1$ in Eq. 3 and 4. The dashed line in Figure 27 represents the model shown in Eq. 37 and 38.

α and β are fitting parameters. The evolution of α and β as a function of total solid concentrations of the mixture required to calculate the apparent viscosity of the mixture is presented in Figure 5.A.2 (a) and (b). The same parameters (α and β) required to calculate the yield stress of the sludge mixture are presented in Figure 5.A.2 (c) and (d). A summary of the parameters of Eq. 37 and 38 are presented in Table 18.

Table 18: Summary of the parameters required to fit Eq. 37 and 38

Eq. 37					
France					
	5%				
α	0.63				
β	0.45				
Australia					
	3%	**4%**	**5%**	**6.5%**	**7.1%**
α	0.68	0.60	0.76	1.02	0.92
β	0.39	0.39	0.35	0.24	0.25
Eq. 38					
France					
	5%				
α	0.63				
β	0.45				
Australia					
	3%	**4%**	**5%**	**6.5%**	**7.1%**
α	0.82	0.75	0.81	1.03	0.84
β	0.29	0.29	0.31	0.22	0.235

5.3.2 Impact of mixing dilute secondary sludge with thickened primary sludge (and vice – versa) at different volume fractions (of secondary sludge) on the apparent viscosity and yield stress of sludge mixtures

The impact of mixing dilute secondary sludge to thickened primary sludge (and vice – versa) at different volume fractions on the apparent viscosity and yield stress of mixtures of primary and secondary sludge was investigated. The evolution of the apparent viscosity (at a constant shear rate of 100 s^{-1}) and yield stress as a function of the change in total solids concentration of the mixture (at each different volume fraction) are presented in Figure 29 for sludge sampled in France (a and b) and Australia (c and d).

Figure 29 (a) illustrates that when dilute secondary sludge (2.8% TS) is added to concentrated primary sludge (5.4% TS), the apparent viscosity of the mixture decreased towards the apparent viscosity of 2.8% TS secondary sludge. In contrast, Figure 29 (c) illustrates that when 5.3% TS secondary sludge was added to 2.5% TS primary sludge, the apparent viscosity of the mixture increased towards the apparent viscosity of 5.3% secondary sludge. Likewise, the yield stress, presented in Figure 29 (b) and (d) followed the same observed trend. A minimum apparent viscosity and yield stress was observed at a specific total solids concentration of the mixture (see Eq. 39 and Eq. 40) which corresponded to the volume fraction of 0.1 ($\varphi = 0.1$). This minimum is also attributed to the liquefying effect caused when secondary sludge is first added into the mixture.

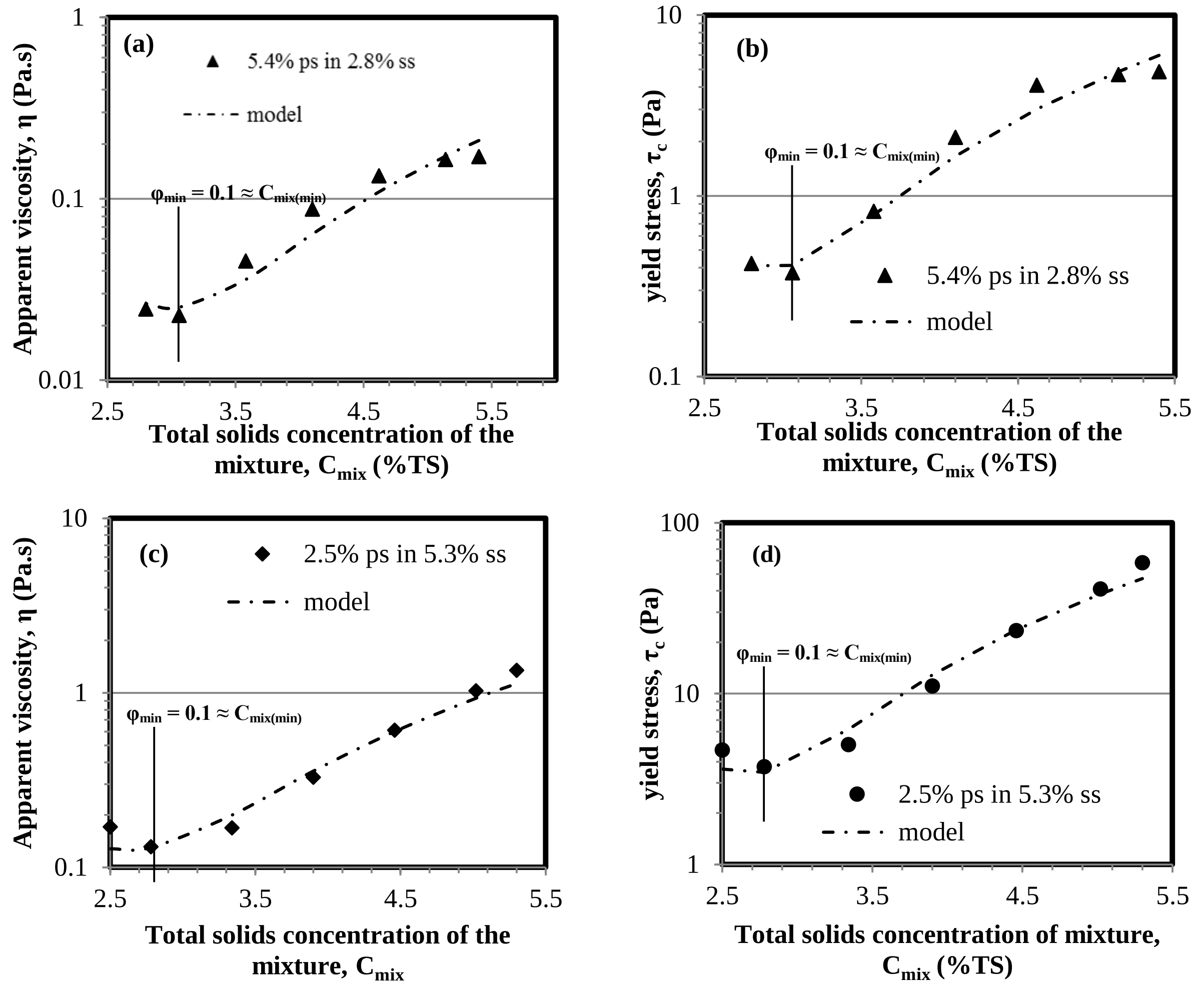

Figure 29: Evolution of (a) the apparent viscosity at a single shear rate of 100s^{-1} and (b) the yield stress as a function of total solids concentration of the mixture for sampled in France and the evolution of the same rheological properties (c and d) sampled in Australia

The observed trend in apparent viscosity and yield stress of the mixture was in fact due to the dilution and thickening affect. In fact, the apparent viscosity and yield stress of mixtures of primary and secondary sludge were influenced by the presence of concentrated sludge, regardless of the sludge type.

The dilution and thickening effect was caused when a more or less concentrated sludge is added and can be explained in terms of the strength of the particle interactions at higher solids concentrations. As explained earlier using the works of Seyssiecq et al (2003) and Baudez (2008) and Baudez et al (2011b), when the concentration increases, stronger links are formed between the flocs of the mixture due to the strengthening of the hydrodynamic interactions (between the solid and surrounding fluid) and non-hydrodynamic interactions (between the solid particles). As such, the apparent viscosity and yield stress of the mixture increase with increasing solids concentration and are influenced less by the structure of the sludge.

The evolution of the apparent viscosity and yield stress can be modelled according to power law type models. When primary and secondary sludge are mixed at two different total solids concentration, the correlations presented in Eq. 37 and Eq. 38 must be modified to take into account the dilution or thickening of the mixture with increasing volume fraction (i.e. change of the total solids concentration at each different volume fraction).

$$\eta_{mix\,(C_{PS} \neq C_{SS})} = \eta_{TS}\,[\alpha\left(C_{mix} - C_{mix(min)}\right)^2 + \beta] \qquad \text{Eq. 39}$$

$$\tau_{mix\,(C_{PS} \neq C_{SS})} = \tau_{TS}\,[\alpha\left(C_{mix} - C_{mix,(\mathrm{min})}\right)^2 + \beta] \qquad \text{Eq. 40}$$

, where η_{TS} and τ_{TS} are the apparent viscosity and yield stress of the thickened sludge, whether it's primary or secondary. C_{mix} is the change in total solids concentration of the mixture at each volume fraction. α and β are fitting parameters. C_{min} is the minimum total solids concentration of the mixture (corresponding to $\varphi = 0.1$) which takes into account the liquefying effect experienced when secondary sludge is first added. A summary of the parameters of Eq. 39 and 40 are presented in Table 19.

Table 19: Summary of the parameters required to fit Eq. 39 and 40

		Total solids concentration, (%)	**α**	**β**	**$C_{mix(min)}$**
Eq. 39	**France**	5.4% ps in 2.8% ss	0.035	0.027	3
Eq. 40			0.0188	0.027	3
Eq. 39	**Australia**	2.5% ps in 5.3% ss	0.103	0.0347	2.78
Eq. 40			0.0109	0.0212	2.78

5.4 Conclusion

In this study, we have shown that mixtures of primary and secondary sludge behaved as non-Newtonian shear thinning, yield stress materials.

When sludge mixtures were prepared by mixing primary sludge to secondary sludge at a similar total solids concentration, the apparent viscosity and yield stress of mixtures of primary and secondary sludge were influenced by the volume fraction of secondary sludge. This implies that the weakly flocculated structure of primary sludge collapsed leading to the entrapment and entanglement of the particles of primary sludge in the gel network structure of secondary sludge. As such, the resulting mixture had a higher apparent viscosity and yield stress.

When dilute secondary sludge was added to thickened primary sludge and vice versa, the apparent viscosity and yield stress of the resulting mixture increased with increasing volume fraction of the concentrated sludge regardless of sludge type. This was attributed to the strengthening of the hydrodynamic interactions (between the solid interactions and surrounding fluid) and non-hydrodynamic interactions (between the solid particles) within the mixture.

A master curve was developed in the dimensionless form of the Herschel – Bulkley model. This was used to predict the apparent viscosity and yield stress of mixtures of primary and secondary sludge, irrespective of the volume fraction and total solids concentration.

Based on the master curve, correlations were developed to predict the apparent viscosity and yield stress of mixtures of primary and secondary sludge as a function of total solids concentration and volume fraction.

5.5 Acknowledgement

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5.7 Appendix

Table 5. A. 1: Summary of the total solids concentration required to prepare the different mixtures of primary and secondary sludge

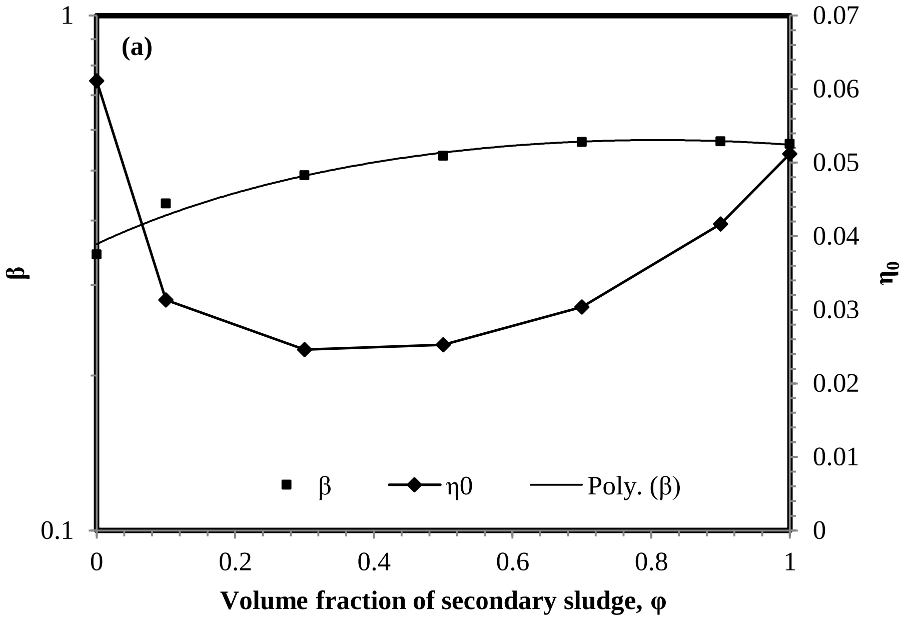
	Primary sludge, (%TS)	Secondary sludge, (%TS)
Australia	5	5
	5.4	2.8
France	3	3
	4	4
	5	5
	6.5	6.5
	7.1	7.1
	2.5	5.3

Table 5. A. 2: Summary of the volume required to mix the different volume fractions of primary and secondary sludge

Primary sludge (mL)	Secondary sludge (mL)	Volume fraction (follow secondary sludge)
100	0	0
90	10	0.1
70	30	0.3
50	50	0.5
30	70	0.7
10	90	0.9
0	100	1.0

Table 5. A. 3: Dimensions of the vane geometry for the two different types of rheometers

Rheometer	Diameter of the bob, D_i (mm)	Diameter of the cup, D_c (mm)	Height, H (mm)
Anton Paar physica MCR 300 rheometer	25	39	70
Discover hybrid rheometer, HR3	15	30	38



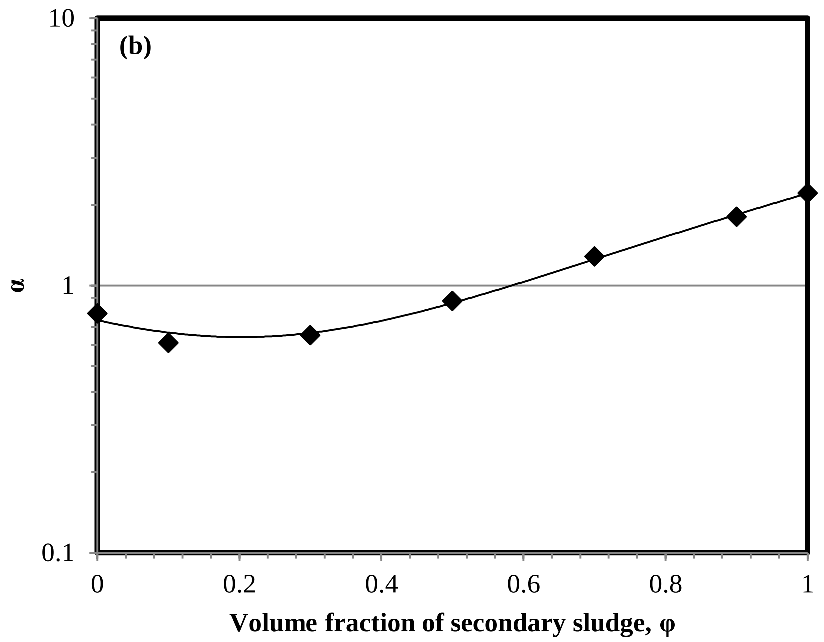


Figure 5. A. 1: Evolution of β and η_0 as a function of volume fraction of secondary sludge required to model Eq. 35; Evolution of α as a function of volume fraction of secondary sludge, required to model Eq. 36, whereby m = 2, C_{min} = 0.96%, $\beta = -0.3253\varphi^2 + 0.5266\varphi + 0.3598$, $\alpha = 2.4652\,\varphi^2 - 0.9956\varphi + 0.7415$

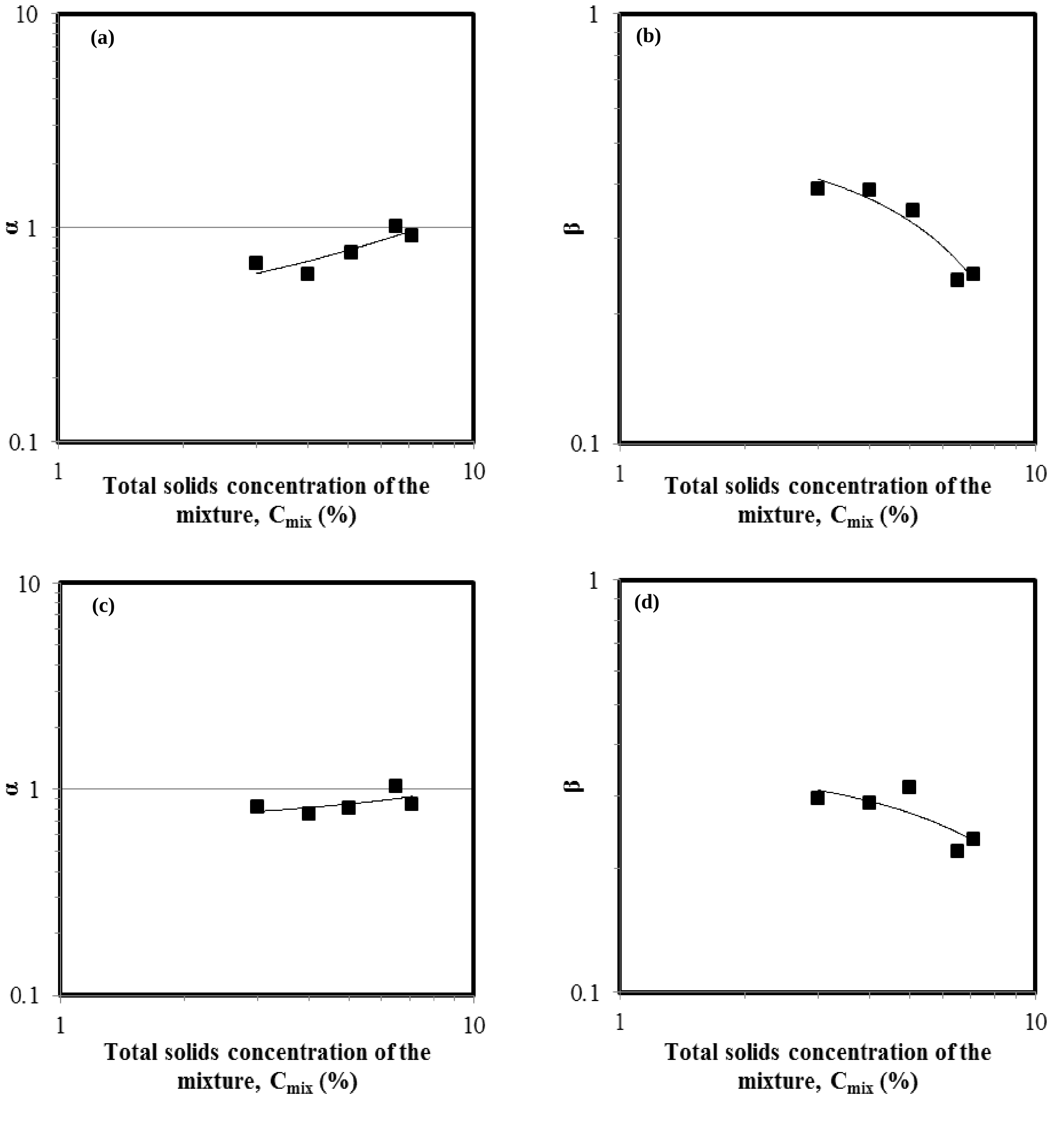
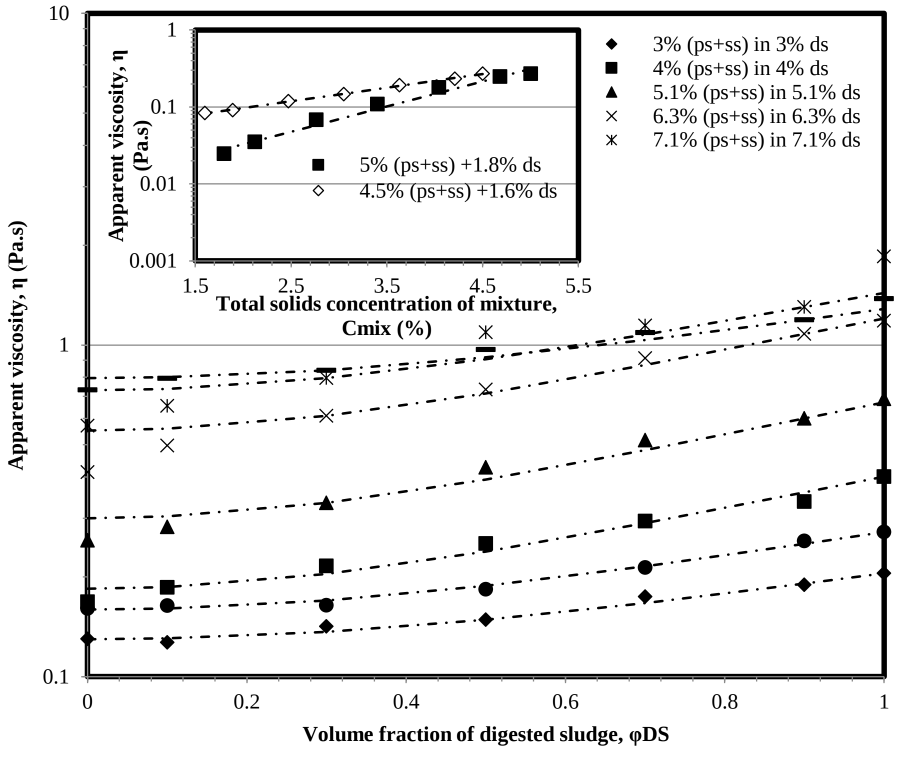


Figure 5. A. 2: (a) and (b) are α and β required to fit the apparent viscosity model of the mixture whereby α = 0.087Cmix +0.35 and β = -0.042Cmix + 0.54; (c) and (d) are the α and β required to fit the yield stress model of the mixture whereby α = 0.035C_{mix} +0.67 and β = -0.0186C_{mix} + 0.365

CHAPTER 6

PREDICTING THE APPARENT VISCOSITY AND YIELD STRESS OF MIXTURES OF PRIMARY, SECONDARY AND DIGESTED SLUDGE: SIMULATING ANAEROBIC DIGESTERS



Chapter 6: Predicting the apparent viscosity and yield stress of mixtures of primary, secondary and digested sludge: simulating anaerobic digesters

Abstract

Predicting the flow behaviour, most notably, the apparent viscosity and yield stress of sludge mixtures inside the anaerobic digester is essential because it helps optimize the mixing system in digesters. This paper investigates the rheology of sludge mixtures as a function of digested sludge volume fraction.

Sludge mixtures exhibited non – Newtonian, shear thinning, yield stress behaviour. The apparent viscosity and yield stress of sludge mixtures prepared at the same total solids concentration was influenced by the interactions within the digested sludge and increased with the volume fraction of digested sludge – highlighted using shear compliance and shear modulus of sludge mixtures. However, when a thickened primary – secondary sludge mixture was mixed with dilute digested sludge, the apparent viscosity and yield stress decreased with increasing the volume fraction of digested sludge. This was caused by the dilution effect leading to a reduction in the hydrodynamic and non-hydrodynamic interactions when dilute digested sludge was added.

Correlations were developed to predict the apparent viscosity and yield stress of the sludge mixtures as a function of the digested sludge volume fraction and total solids concentration of the mixtures. The parameters of correlations can be estimated using pH of sludge mixtures. The shear and complex modulus were also modelled and they followed an exponential relationship with increasing digested sludge volume fraction.

Key words: Primary sludge, secondary sludge, digested sludge, mixtures, viscosity, yield stress

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6.1 Introduction

Municipal sludge is the by – product of the municipal waste water treatment process. It is produced from human and residential waste, as well as industrial waste, farmland and landfill leachates and runoff from streets (Sanin et al., 2011). Sanin et al (2011) describes sludge as an odorous mixture of organic flocs suspended in water whilst Bhhatacharya (1981) and Baudez et al (2013) have defined sludge as a suspension composed of mainly water (more than 95%), mineral particles, dead and alive bacteria (polymeric and dissolved). Two types of sludge are sent to the sludge treatment process – primary and secondary sludge whereby they are treated and stabilized to eliminate odour and remove suspended organic and inorganic matter and reduce pathogens and bacteria (Sanin et al., 2011). Anaerobic digestion is the most commonly used technique to stabilize sludge and reduce its volatile solids by about 40% (Sanin et al., 2011). During anaerobic digestion, the organic matter in primary and secondary sludge or a mixture of the two sludges are degraded in the absence of oxygen with continuous mixing to produce methane gas (CH_4), carbon dioxide (CO_2) and anaerobic digested sludge. The methane gas is used as a source of heat or to generate electricity whilst the anaerobic digested sludge is dewatered (i.e. further treatment) to reduce its volume prior to disposal (Sanin et al., 2011). However, a UNESCO report (Nicklin and Cornwell, 2013) has shown that the amount of sludge generated globally is increasing at an exponential rate due to population growth so that the current sludge treatment plants including anaerobic digesters cannot handle the additional load of sludge without further innovative techniques or optimizing current treatment plants.

Anaerobic digestion requires efficient mixing of the primary and secondary sludge entering the digester to provide an optimum environment for digestion. Karim et al (2004) explains that efficient mixing is necessary to transfer substrates to microorganisms, to maintain process stability, to maintain a uniform pH and temperature for bacterial growth, to prevent short circuiting and solids deposition in the digester bottom as well as to minimize scum and foam formation. However, the exponential production of sludge combined with the fact that anaerobic digesters are inadequately designed, has led to inefficient mixing (Eshtiaghi et al., 2012b, Eshtiaghi et al., 2013b). Karim et al (2004) states that inefficient mixing of sludge leads to the formation of dead zones within the digester and poor microbial environment for biogas production. As a result, the anaerobic digesters fail (Karim et al., 2004).

Any changes to the flow behaviour of sludge entering the digester as well as the recirculated digested sludge through heat exchangers alter the performance of the digesters. As such, predicting the flow behaviour, most notably, the apparent viscosity and yield stress of mixtures of primary, secondary and digested sludge is essential to achieve efficient mixing. This is due to the fact that these two parameters have an impact of on the operating conditions and energy consumption of the digesters.

As mentioned earlier, primary and secondary sludge are fed to the digester where mixing is achieved by means of a constant re-circulation of digested sludge in conjunction with gas injection. Primary sludge, also known as "raw sludge" is the product of the primary treatment process whilst secondary sludge, also known as "waste activated sludge" is the product of the secondary treatment process. Each sludge is biologically different, so that the interactions governing their network structure are also different. This means that primary, secondary and digested sludge flow differently. Bayoudh et al (2009) and Cui et al (2011) explained that the structure of primary sludge was governed by nonspecific Lif-shitz van der Waals forces as well as hydrogen and chemical bonds similar to highly colloidal suspensions such as bentonite (Coussot et al., 2002, Markis et al., 2014). Secondary sludge is composed of polysaccharides and proteins, bacteria and microorganisms which are governed by electrostatic and hydrogen bonds (Flemming, 1996) so that extracellular polymeric substances (i.e. EPS) are formed. Wingender et al (1999) explained that the EPS form a three dimensional gel like negatively charged structure. Forster (1983) found that digested sludge contained proteins and lipopolysaccharides with both hydrophobic and hydrophilic heads. Furthermore, the structure of digested sludge was governed by steric interactions (Forster, 2002) and has been found to behave similar soft glassy materials such as O/W emulsions (Baudez et al., 2013a).

The rheology of individual sludge has been studied extensively over the years (Dick and Ewing, 1967, Bhattacharya, 1981, Battistoni, 1997, Slatter, 1997, Baudez and Coussot, 2001, Seyssiecq et al., 2003, Baudez, 2008, Baudez et al., 2011a, Eshtiaghi et al., 2012b, Eshtiaghi et al., 2013a, Markis et al., 2014, Baroutian et al., 2013), however, there is little to no information on the rheology, notably, on the apparent viscosity and yield stress of mixtures of primary, secondary and digested sludge. Markis et al (2015) is the only study that investigates the impact of volume fraction and total solids concentration on the apparent viscosity and yield stress of mixtures of primary and secondary sludge. Mixtures of primary and secondary sludge as well as the individual sludge displayed non – Newtonian, shear thinning yield stress behavior, consistent with the previous work on individual sludge. Markis et al (2015) demonstrated that when mixtures of primary and secondary sludge are prepared at the same total solids concentration, the apparent viscosity and yield stress increases with increasing volume fraction of secondary sludge. Moreover, Markis et al (2015) demonstrated that when thickened sludge is mixed with dilute sludge (regardless of being primary or secondary), the apparent viscosity and yield stress increased with increasing the volume fraction of the thickened sludge regardless of the sludge type. They explained that this was due to the strengthening of hydrodynamic and non – hydrodynamic interactions within concentrated sludge which was consistent with other studies (Markis et al., 2014, Baudez, 2008, Baudez et al., 2011b).

In the above mentioned studies, the impact of volume fraction of digested sludge on the apparent viscosity and yield stress of mixtures of primary, secondary and digested sludge was not investigated. This highlights the lack of research focusing on the rheological characterization of mixtures of primary, secondary and digested sludge over a wide total solids concentration and different volume fraction. Consequently, this study focuses on the rheology of sludge mixtures which will help understand the flow behavior of sludge inside digesters. Correlations have been developed to estimate the apparent viscosity and yield stress of mixtures of primary, secondary and digested sludge as a function of total solids concentration and volume fraction of digested sludge. Additionally, the parameters of these correlations have been linked to the pH of sludge mixtures. The shear compliance and shear modulus of sludge mixtures are presented to highlight the changes in flow behavior after digested sludge is introduced to the mixture of primary and secondary sludge.

6.2 Materials and method

Sludge was sampled in two different seasons – summer (December 2013 to February 2014) and winter (May to July 2015). It was also sampled from two different treatment plants – the Mount Martha waste water treatment plant (Mornington Peninsula, Australia) and the Eastern treatment plant (Bangholme, Australia). Hence, any changes to the flow behaviour of sludge due to changes in environmental conditions experienced by sludge during different seasons (summer or winter) may be detected. Table 20 contains a summary of the different locations used to sample the sludge over the two different seasons. Table 21 contains a summary of the different total solids concentration required to prepare the different mixtures of sludge. Table 22 contains a summary of the volume required to prepare the different mixtures of primary, secondary and digested sludge.

Table 20: Summary of the type of sludge and its sampling location

Location	Year	Sludge type
Mount Martha Waste Water Treatment Plant	2014	Primary, secondary and digested sludge
Mount Martha Waste Water Treatment Plant	2015	Secondary sludge
Eastern Treatment plant	2015	Primary and digested sludge

6.2.1 Sample preparation

Dilute primary, secondary and digested sludge were thickened to the various total solids concentrations required and shown in Table 21 using the vacuum filtration technique. The samples were stored at 4 °C for 30 days prior to conducting the experiments. This ensured that the same material was always used throughout the experiments by reducing any changes to the composition (Curvers et al., 2009, Baudez et al., 2011b) without affecting the rheology.

Table 21: Summary of the total solids concentration required to prepare the different sludge mixtures

Year	Primary sludge (%TS)	Secondary sludge (%TS)	Digested sludge (%TS)
2014	4.2	4.2	4.2
	7	7	7
	5	5	1.8
	3	3	3
2015	4	4	4
	5.1	5.1	5.1
	6.3	6.3	6.3
	7.1	7.1	7.1
	4.5	4.5	1.6

First, mixtures of primary, secondary and digested sludge at the same total solids concentration were prepared. As such, the total solids concentration of the resulting mixture was equal to the total solids concentration of the individual primary, secondary and digested sludge. A 50:50 (v/v %) mixture of primary and secondary sludge was prepared by mixing an equal volume of primary sludge with an equal volume of secondary sludge. Then different volume fractions of digested sludge were then added to this mixture, summarized in Table 22. For example, (refer to Table 21 and 22), 3% primary sludge first was mixed with 3% secondary sludge; then 3% digested sludge was added to the 3% primary – secondary sludge mixture at different volume fractions ranging from 0 – 1.

Table 22: Summary of the required volume to prepare a sample with different volume fractions of digested sludge

Primary and secondary sludge mixture (mL)	Digested sludge (mL)	Sample volume fraction
100	0	0
90	10	0.1
70	30	0.3
50	50	0.5
30	70	0.7
10	90	0.9
0	100	1.0

Following on from this, concentrated primary – secondary sludge mixtures (5% and 4.5 %TS) were mixed with dilute digested sludge (1.8% and 1.6%TS) at different volume fraction. Two different batches were prepared so that two different seasons were covered – the summer of 2014 (5% primary – secondary sludge mixture in 1.8% digested sludge) and the winter of 2015 (4.5% primary – secondary sludge mixture in 1.6% digested sludge). By preparing the primary – secondary mixture and digested sludge at different solid concentrations, the anaerobic digester conditions – which is mixing a thickened mixture of primary and secondary sludge to dilute digested sludge may also be simulated.

A pH probe (S20 SevenEasyTM pH, Mettler Toledo) was employed to measure the pH of primary, secondary and digested sludge prior to and after these sludges was mixed. First, the probe was placed in the sludge sample for 60s allowing the meter to equilibrate. The pH was then read off the meter. The probe was rinsed with water prior to and after each reading.

6.2.2 Rheometric technique

Creep tests were carried out using a similar procedure detailed elsewhere (Markis et al., 2014) such that the experimental procedure consisted of performing successive creep tests using the following pattern: pre-shear (150s) – rest (150s) – creep (120s). The sludge was pre – sheared at a high shear stress (corresponding to a high shear rate) such that the same initial state of destructuration was achieved (Markis et al., 2014, Baudez, 2008, Coussot, 2005). A short period of rest was provided so that the structure of the sludge could rebuild (Markis et al., 2014, Baudez, 2008, Coussot, 2005). A constant creep (i.e. shear stress), dependent on the total solids concentration of the mixture (Markis et al., 2015) was then applied for a duration of time (Markis et al., 2014, Baudez, 2008, Coussot, 2005).

Similar to a procedure described elsewhere (Markis et al., 2014), successive creep tests were performed over a wide shear stress range (in the solid, liquid and transition regimes) allowing the flow curves to be reconstructed from the torque (M) and the deflection angle (θ) data.

A Discovery Hybrid rheometer (HR – 3) equipped with the wide gap vane geometry (vane diameter, D_v = 15 mm, cup diameter, D_c = 30 mm and vane height, H = 38 mm) was employed to perform the creep tests at 20 °C. The vane geometry was used to reduce inertia effects (Dzuy and Boger, 1983, Dzuy and Boger, 1985) and the surfaces of the geometry were roughened to avoid wall slip (Tabuteau et al., 2004, Tabuteau, 2006, Baudez, 2008).

The mixtures of primary, secondary and digested sludge, prepared at the same total solids concentration (at the range of 4.2 and 7.0%) and sampled in the summer of 2014 exhibited the same flow behaviour to the samples in 2015. As such, the complete set of results for sludge sampled in different seasons was not presented.

6.3 Results and discussion

6.3.1 Flow behaviour of mixtures of primary and secondary sludge (at a fixed volume fraction of 50:50) and digested sludge: impact of total solids concentration

The steady state flow behaviour of a primary – secondary sludge mixture at a fixed volume fraction of 50:50 was investigated by reconstructing the shear stress versus shear rate data described elsewhere (Markis et al., 2014) presented in Figure 30 (a). Figure 30 (a) illustrates that mixtures of primary and secondary sludge behaved as non – Newtonian fluids whereby the flow curves were non – linear and did not pass through the origin so that a yield stress was detected. Moreover, the corresponding apparent viscosity (not presented) decreased as a function of increasing shear rate demonstrating shear thinning behaviour. The observed behaviour is consistent with the experimental data presented by Baroutian et al (2013) on the rheological characterisation of mixtures of primary and secondary sludge at a fixed ratio of 60:40 (v/v) and over a wide total solids concentration (4.3, 4.5, 4.9, 7.3 and 9.8%wt). Furthermore, it was consistent with the study conducted by Markis et al (2014) on individual primary and secondary sludge as well as mixtures of primary and secondary sludge (Markis et al., 2015) and earlier works on sludge by Bhattacharya (1981), Baudez (2008), Mori et al (2006) and Seyssiecq et al (2008).

Figure 30 (a) illustrates that the flow behaviour of mixtures of primary and secondary sludge prepared at a fixed volume fraction of 50:50 increases with increasing total solids concentration of the mixture. Similarly, Figure 30 (b) illustrates that digested sludge is also a shear thinning, yield stress material whereby it's apparent viscosity and yield stress increases with increasing solids concentration. Moreover, this trend was also observed by Markis et al (2015) and they attributed it to the strengthening of the particle interactions within the mixture. Indeed, Seyssiecq et al (2003) explained that particle interactions with stronger links between the flocs are formed when the total solids concentration increases. This results in a higher apparent viscosity and yield stress. Baudez (2008) and Baudez et al (2011b) validated this assumption by demonstrating that the hydrodynamic interactions (between the solid particles and surrounding fluid) and non – hydrodynamic interactions (between the solid particles) increased as the total solids concentration increases. Baudez (2008) and Baudez et al (2011b) explained that the apparent viscosity and yield stress were a measure of the hydrodynamic interactions and non – hydrodynamic interactions, respectively.

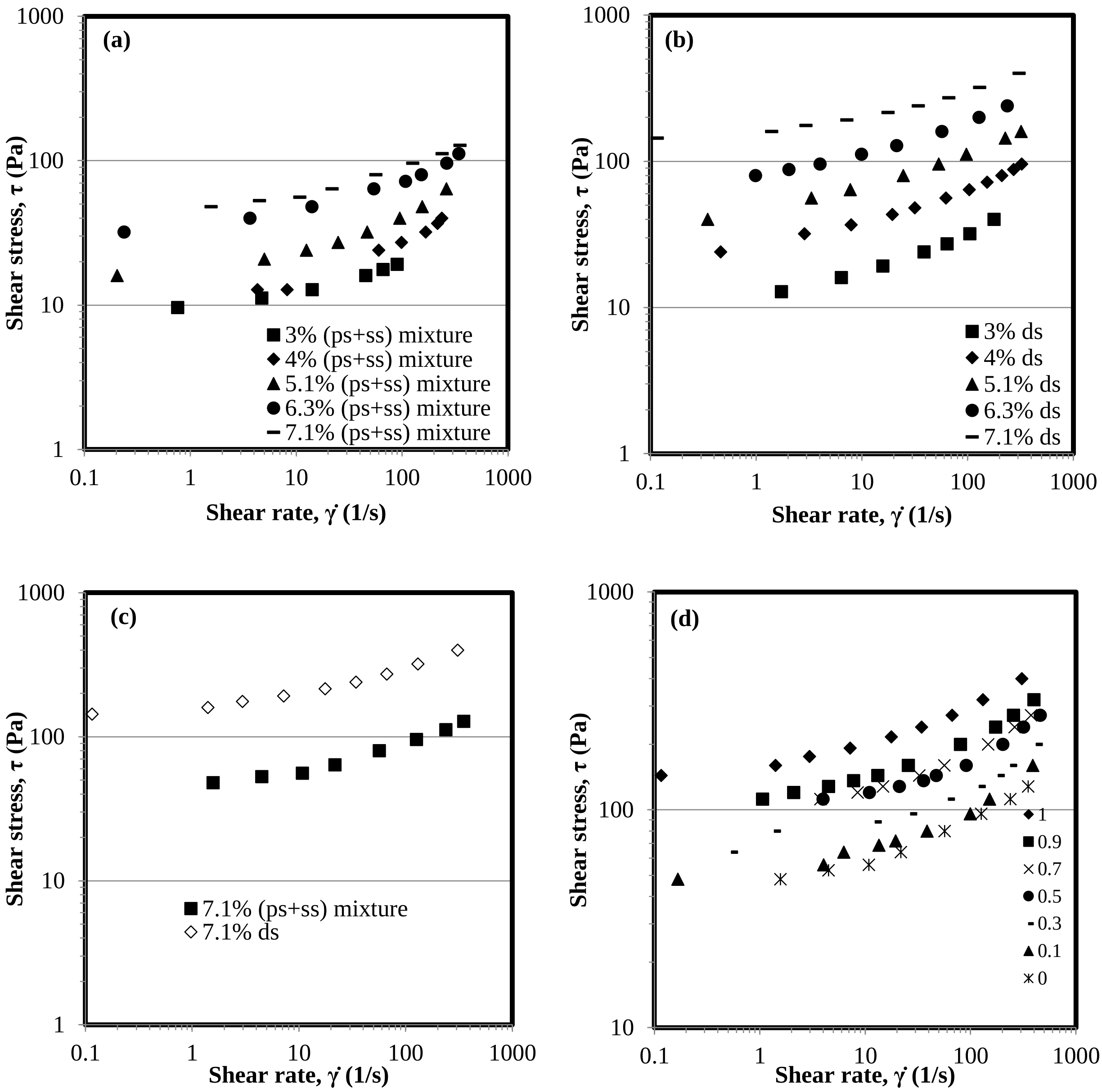

Figure 30: Flow curve of (a) mixtures of primary and secondary sludge at a fixed volume fraction of 50:50 and over a wide total solids concentration (b) digested sludge over a wide total solids concentration (c) 7.1% primary – secondary sludge mixture and 7.1% digested sludge and (d) mixtures of 7.1% primary – secondary sludge mixed with 7.1% digested sludge and at different volume fractions (volume fraction of 1 represents digested sludge and volume fraction of zero represents the mixture of primary and secondary sludge)

Baudez (2008), Baudez et al (2011b) as well as Sanin (2002) demonstrated that the apparent viscosity followed an exponential increase whilst the yield stress followed a power law as a function of total solids concentration. Markis et al (2014) and most recently, Markis et al (2015) validated the correlations presented by Baudez (2008) and Baudez et al (2011b) on individual primary and secondary sludge as well as mixtures of primary and secondary sludge.

Figure 30 (c) also highlights that digested sludge requires higher stresses to flow compared to the mixture of primary and secondary sludge for all concentrations (only shown for 7% sludge concentration). The observed behavior was shown by Markis et al (2015) whom demonstrated that the primary – secondary sludge mixture flowed at lower stresses compared to secondary sludge because weak links are formed by a combination of van der Waals interactions, electrostatic and hydrogen bonds. On the other hand, Digested sludge flowed at higher stresses because its proteins and lipopolysaccharide structure (Forster, 1983) is governed by steric interactions (Forster, 2002).

Mixtures of 7% primary, secondary and digested sludge which were prepared at different volume fractions from 0 – 1 and presented in Figure 30 (d) and displayed the same non – Newtonian, shear thinning, and yield stress behaviour.

Since we have shown that the flow behavior of primary – secondary sludge mixtures, digested sludge and primary – secondary – digested sludge mixtures display the same flow behavior consistent with the literature on sludge mixtures (Baroutian et al., 2013, Markis et al., 2015) and on individual sludge (Markis et al., 2014, Baudez et al., 2011b), it is reasonable to assume that a master curve can be developed to compare and predict the apparent viscosity and yield stress of mixtures of sludge as a function of total solids concentration. The master curve was developed by taking the flow curve of 7.1% TS digested sludge as a reference curve and superimposing the remaining flow curves to the reference curve. Shift factors, in the x and y axes were employed to superimpose any flow curve; the shift factors are summarized in the Appendix (Table 6.A.1). A dimensionless form of the Herschel – Bulkley model was then employed using a procedure described elsewhere (Markis et al., 2014):

$$\frac{\tau}{\tau_c} = \tau_c + K\dot{\gamma}^n \rightarrow \frac{\tau}{\tau_c} = 1 + \left(\frac{K}{\tau_c}\right)\dot{\gamma}^n \rightarrow \frac{\tau}{\tau_c} = 1 + \beta\Gamma^n \ where\ \Gamma = \left(\frac{\eta_o}{\tau_c}\right)\dot{\gamma} \ and\ \beta = \left(\frac{K}{\tau_c}\right)\left(\frac{\tau}{\eta_o}\right)^n$$

, where τ/τ_c is the dimensionless shear stress and Γ is the dimensionless shear rate, τ_c is the yield stress of reference curve – below which steady state flow cannot be achieved, μ_0 is a measure of the infinite shear viscosity which equals 1 (Pa.s), K is the fluid consistency (Pa.s^n) of reference curve, and n is the flow index.

The dimensionless form of the master curve – presented in Figure 31 summarizes the flow curves, for all the samples including individual primary, secondary and digested sludge as well as primary – secondary sludge mixtures at the fixed volume fraction (50:50) and primary – secondary – digested sludge mixtures at two different seasons. Markis et al (2014) explained that the shear stress was scaled by the yield stress, τ_c and the shear rate was scaled by K/τ_c. As such, the non – hydrodynamic and hydrodynamic interactions, which represent the solid and viscous interactions, were smoothed out. In fact, this is true, as Baudez et al (2011b) explained that in its dimensionless form and from a physical point of view, the interactions within sludge, which are characterized by the apparent viscosity and yield stress, are similar. Hence, the apparent viscosity and yield stress of each mixture were calculated using the parameters of the Herschel – Bulkely model of the master curve (K, τ_c and n).

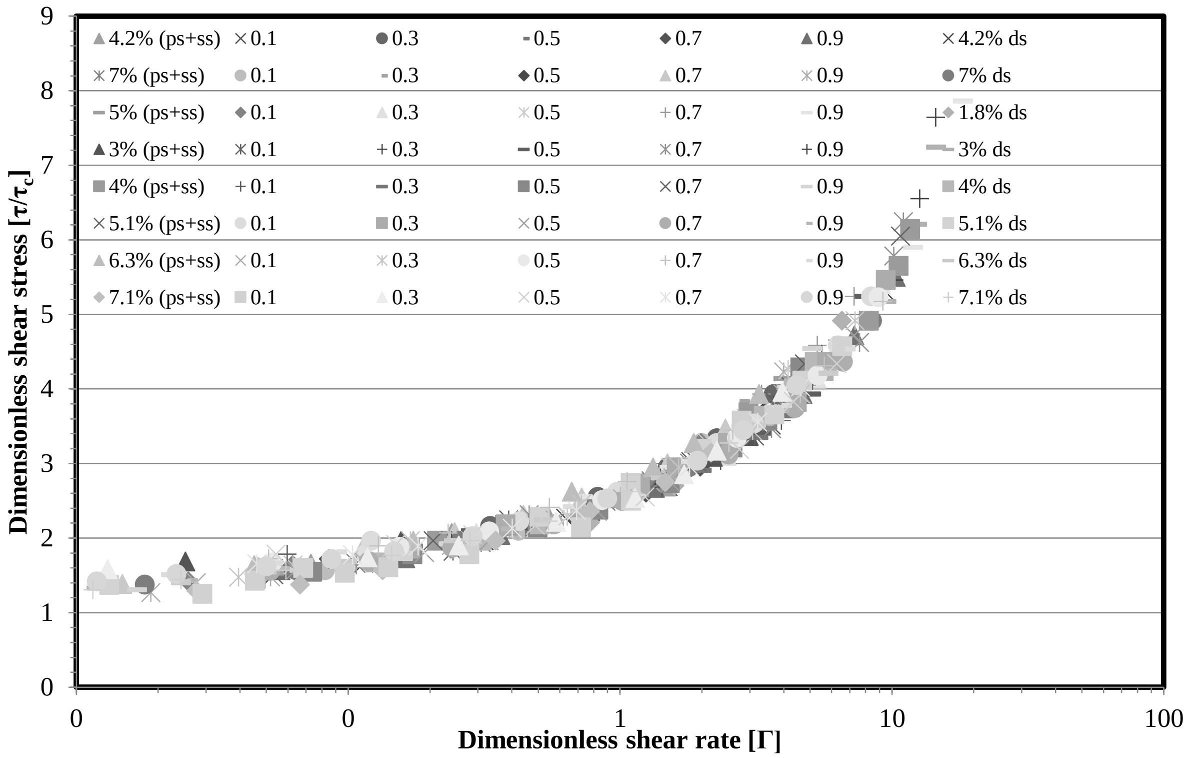

Figure 31: Master curve, in the dimensionless form of the Herschel - Bulkley model for all sludge mixtures sampled in summer (4.2 and 7% sludge mixtures; 5% primary and secondary sludge mixed with 1.8% digested sludge) and winter (3, 4, 5.1, 6.3, and 7.1% sludge mixtures; 4.5% primary and secondary sludge mixed with 1.6% digested sludge); the parameters of the dimensionless curve are τ_c = 81.38 Pa, K = 23.09 Pa.s^n, n = 0.4; β = 1.0016

Correlations were then applied to predict the apparent viscosity and the yield stress of mixtures of primary and secondary sludge and digested sludge as a function of total solids concentration for each different volume fraction (shown in Figure 32). In Figure 32, the primary – secondary sludge mixtures are denoted 0 and shown as the closed diamond whilst digested sludge is denoted 1 and shown as the strikethrough cross; the model, presented in Eq. 41 and Eq. 42 is shown as the dashed line.

Figure 32 (a) and (b) illustrates that apparent viscosity and yield stress of mixtures of primary, secondary and digested sludge, at different volume fractions ranging from 0 and 1, increased with increasing total solids concentration of the mixture. This further validated the work of Baudez (2008), Baudez et al (2011b) and most recently, the work of Markis et al (2014) and Markis et al (2015) on individual primary and secondary sludge as well as mixtures of primary and secondary sludge. As such, the apparent viscosity followed an exponential growth whilst the yield stress followed a power law model:

$$\eta = \eta_0 \exp(C\beta) \quad \text{Eq. 41}$$

$$\tau_y = \alpha(C_{mix} - C_{min})^m \quad \text{Eq. 42}$$

, where $C_{mix} = C_{(PS+SS)}(1 - \varphi_{DS}) + C_{DS}\varphi_{DS}$

, where φ_{DS} is the volume fraction of digested sludge; η_{mix}, τ_{mix} and C_{mix} are the apparent viscosity, yield stress and total solids concentration of the mixture; $C_{(PS+SS)}$ and $C_{(DS)}$ are the total solids concentration of the primary – secondary sludge mixture and the total solids concentration of digested sludge. α, β, m and η_0 are fitting parameters. η_0 is the viscosity of the liquid (Pa.s), m is related to the fractal dimension of sludge flocs (Baudez, 2008, Baudez et al., 2011b). C_{min} is the minimum concentration, below which there is no yield stress (Baudez, 2008, Baudez et al., 2011b). The parameters of Eq. 41 and 42 are summarized for different volume fraction of digested sludge in Table 6.A.2 of the appendix.

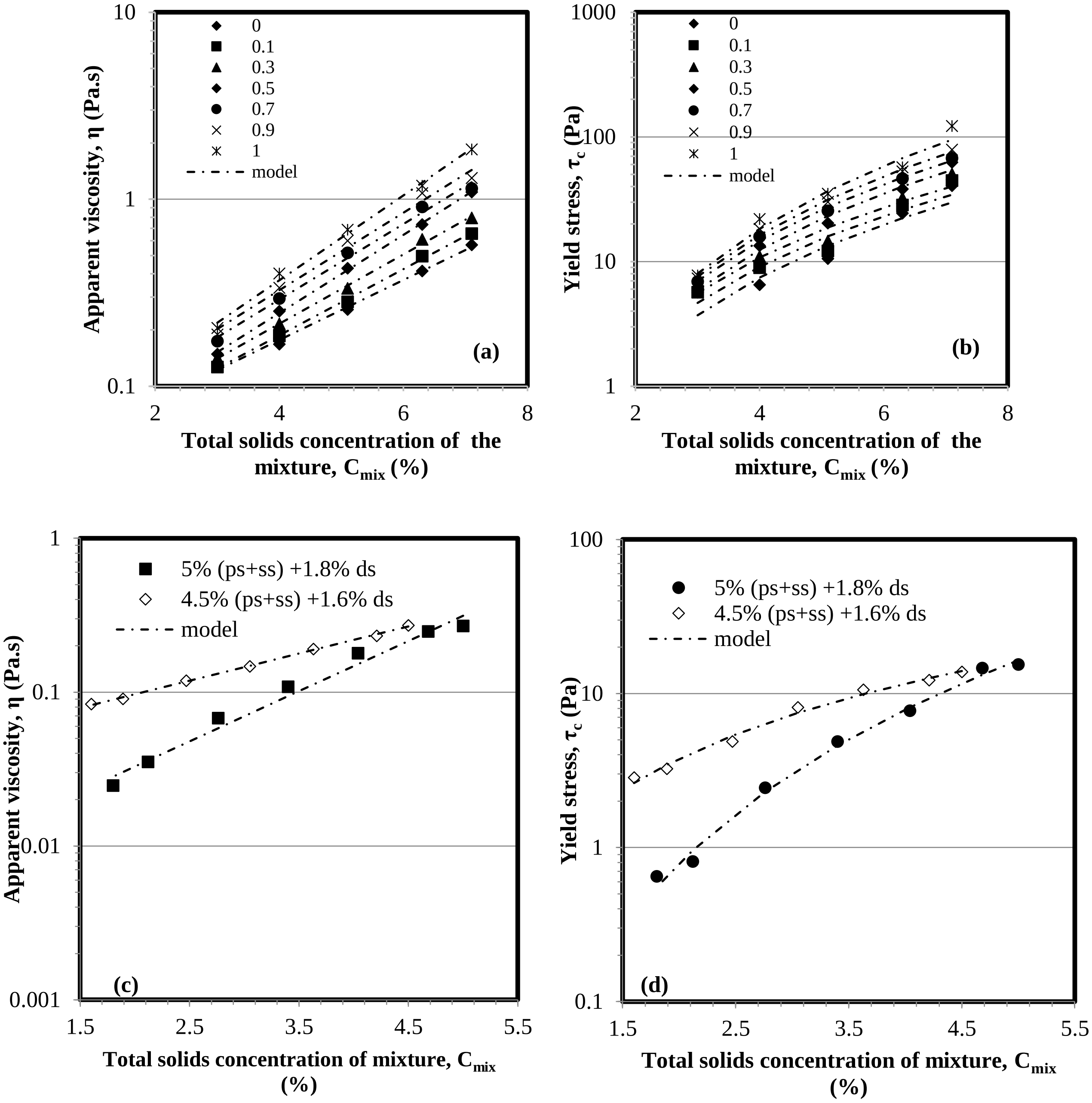

Figure 32: (a) Apparent viscosity (at a constant shear rate of 100 s^{-1}) and (b) yield stress for mixtures of sludge sampled in summer and prepared at the same total solids concentration; as well as (c) apparent viscosity (at a constant shear rate of 100 s^{-1}) and (d) yield stress for mixtures of sludge prepared at different total solids concentration of the mixture

Figure 32 (c) and (d) shows the apparent viscosity and yield stress of mixtures of a concentrated (i.e. thickened) primary – secondary sludge mixture mixed with dilute digested sludge at different volume fractions ranging from 0 to 1. The apparent viscosity and yield stress was modelled using Eq. 41 and 42. The parameters of Eq. 41 and 42 are summarized in Table 6.A.3 of the appendix for mixtures of thickened primary – secondary sludge mixed with dilute digested sludge for sludge sampled in the summer of 2014 and winter of 2015.

Figure 32 (c) demonstrates that when a thickened primary – secondary sludge mixture (5.0%TS) is mixed with dilute digested sludge (1.8%TS), the apparent viscosity of the resulting sludge mixture decreases towards the apparent viscosity of 1.8% TS digested sludge. This is attributed to the dilution effect. Likewise, the yield stress, presented in Figure 32 (d) followed the same trend. Moreover, thickened primary – secondary sludge mixture (4.5%TS) is mixed with dilute digested sludge (1.6%TS) and sampled in the winter of 2015 followed the same trend as the summer samples.

Indeed, Markis et al (2015) observed the same dilution effect when they mixed thickened primary sludge to dilute secondary sludge. This work also confirmed earlier works of Seyssiecq et al (2003), Baudez (2008) and Baudez et al (2011b) whom explained that decreasing the concentration due to dilution effect leads to the formation of weaker links between the flocs within the mixture. So, when a thickened primary – secondary sludge mixture is added to dilute digested sludge, the hydrodynamic and non – hydrodynamic interactions are reduced. This was reflected through the reduced values of the apparent viscosity and yield stress of the mixtures.

The parameters of the presented correlations were plotted against a measurable sludge characteristic such as pH for mixtures of primary – secondary and digested sludge. Figure 33 shows that the pH is independent of the total solids concentration of the mixture, allowing for the parameters of the apparent viscosity and yield stress of any sludge mixture to be estimated for any total solids concentration and known volume fraction.

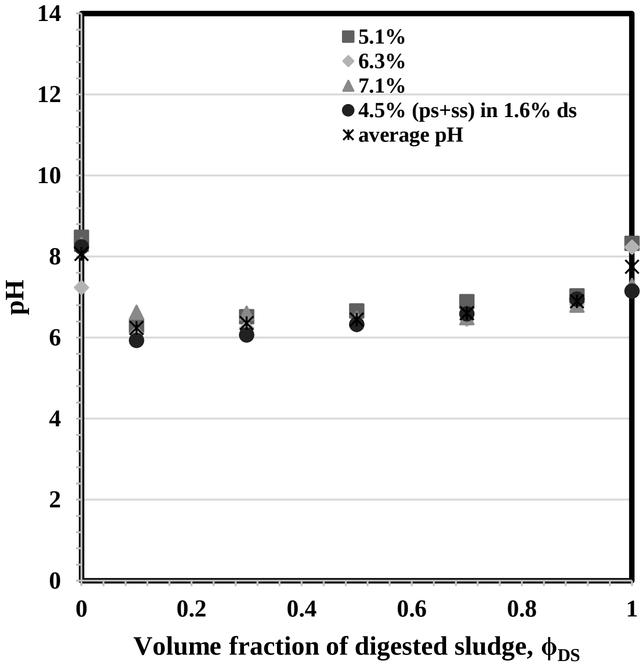

Figure 33: Evolution of pH with volume fraction of digested sludge for sludge mixtures prepared at the same and at different total solids concentration

As such, the parameters of the apparent viscosity and yield stress model, presented in Eq. 41 and 42 were plotted with the pH of the primary-secondary-digested sludge mixtures corresponding to each volume fractions ranging from 0 – 1, shown in Figure 34.

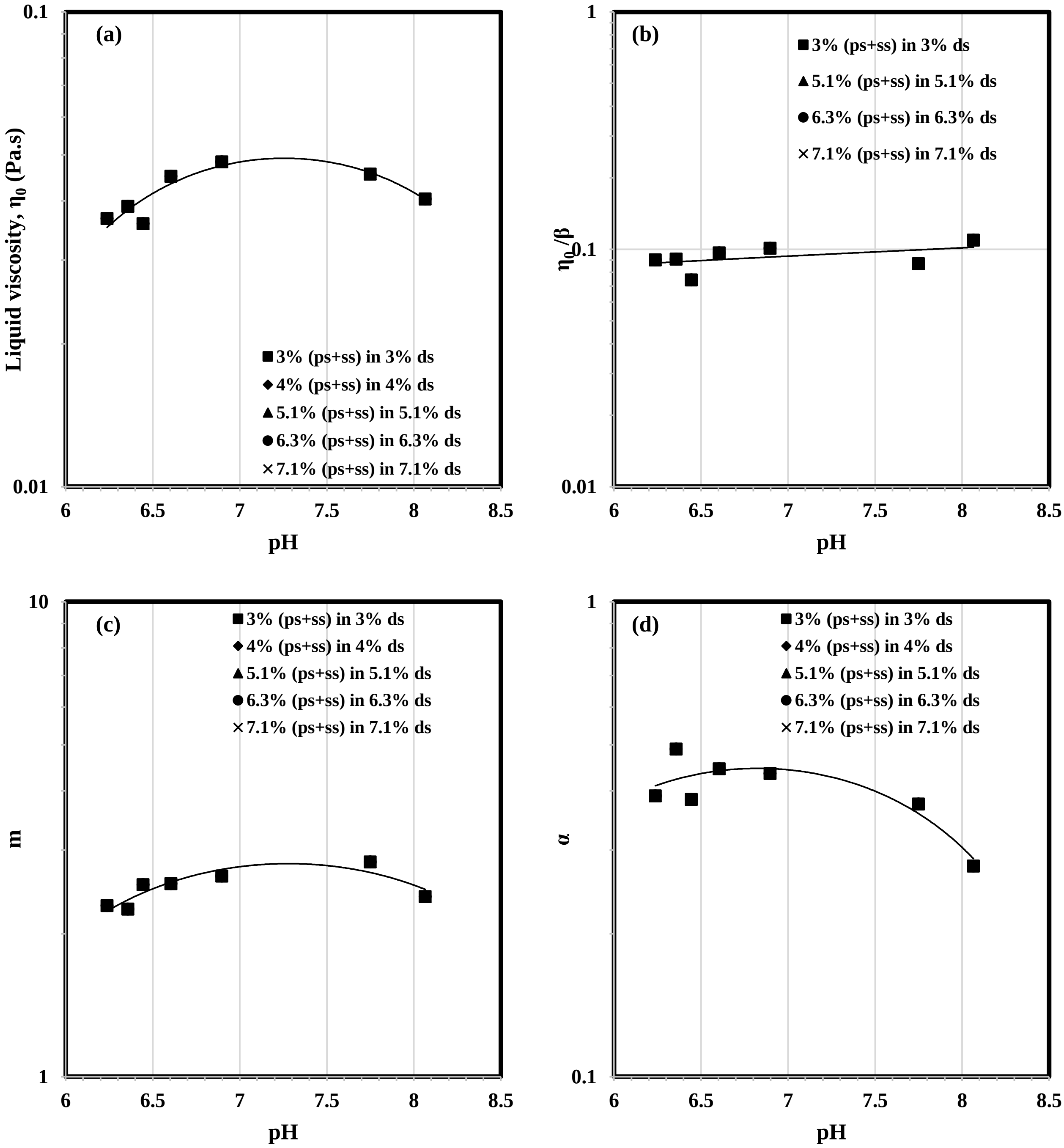

Figure 34: Evolution of the (a) the liquid viscosity, η_0 and (b) the ratio between η_0/β required for Eq. 1 whereby $\eta_0 = -0.0135pH^2+0.1965pH - 0.667$ and $\beta/\eta_0 = 0.0079pH + 0.0385$; the evolution of (c) m and (d) α required to model Eq. 2 whereby $m = -0.5329pH^2+7.7574pH - 25.423$ and $\alpha = -0.1033pH^2+1.4103pH - 4.369$ of the mixtures of primary, secondary and digested sludge as a function of pH for sludge mixtures

6.3.2 Flow behaviour of mixtures of primary and secondary sludge (at a fixed volume fraction of 50:50) and digested sludge: impact of volume fraction of digested sludge

The apparent viscosity was calculated at a single shear rate of 100 s^{-1} so that the shear conditions within the anaerobic digesters may be simulated. The evolution of the apparent viscosity (at a constant shear rate of 100 s^{-1}) with volume fraction of digested sludge ranging from 0 – 1 is presented in Figure 35 (a) for sludge mixtures sampled in the summer of 2014 and in the winter of 2015. Figure 35 (a) illustrates that when digested sludge is added to the mixture of primary and secondary sludge, the apparent viscosity of the resulting mixture increases as the volume fraction of digested sludge increases. Likewise, the yield stress, presented in Figure 35 (b) for sludge mixtures sampled in the summer of 2014 and winter of 2015 followed the same trend. Hence, this trend was observed for all sludge mixtures sampled in the summer of 2014 and the winter of 2015 suggesting that seasonal changes had little impact on the flow behaviour of sludge mixtures. Therefore, both the apparent viscosity at a single shear rate (100s^{-1}) and yield stress can be modelled as a function of digested sludge volume fraction. The apparent viscosity and yield stress evolved following a power law type function. The correlations are presented as the dashed line in Figure 35.

$$\eta_{mix}(\,C_{PS+SS} = C_{DS}) = \,\eta_{DS}\,[\alpha(\varphi)^2 + \beta] \qquad \text{Eq. 43}$$

$$\tau_{mix}(\,C_{PS+SS} = C_{DS}) = \,\tau_{DS}\,[\alpha(\varphi)^2 + \beta] \qquad \text{Eq. 44}$$

, where η_{DS} and τ_{DS} are the apparent viscosity and yield stress of digested sludge because it has been shown that the flow behaviour of digested sludge has a dominant effect. α and β are fitting parameters. A summary of the parameters of Eq. 43 and 44 are presented in Table 6.A.4 of the appendix.

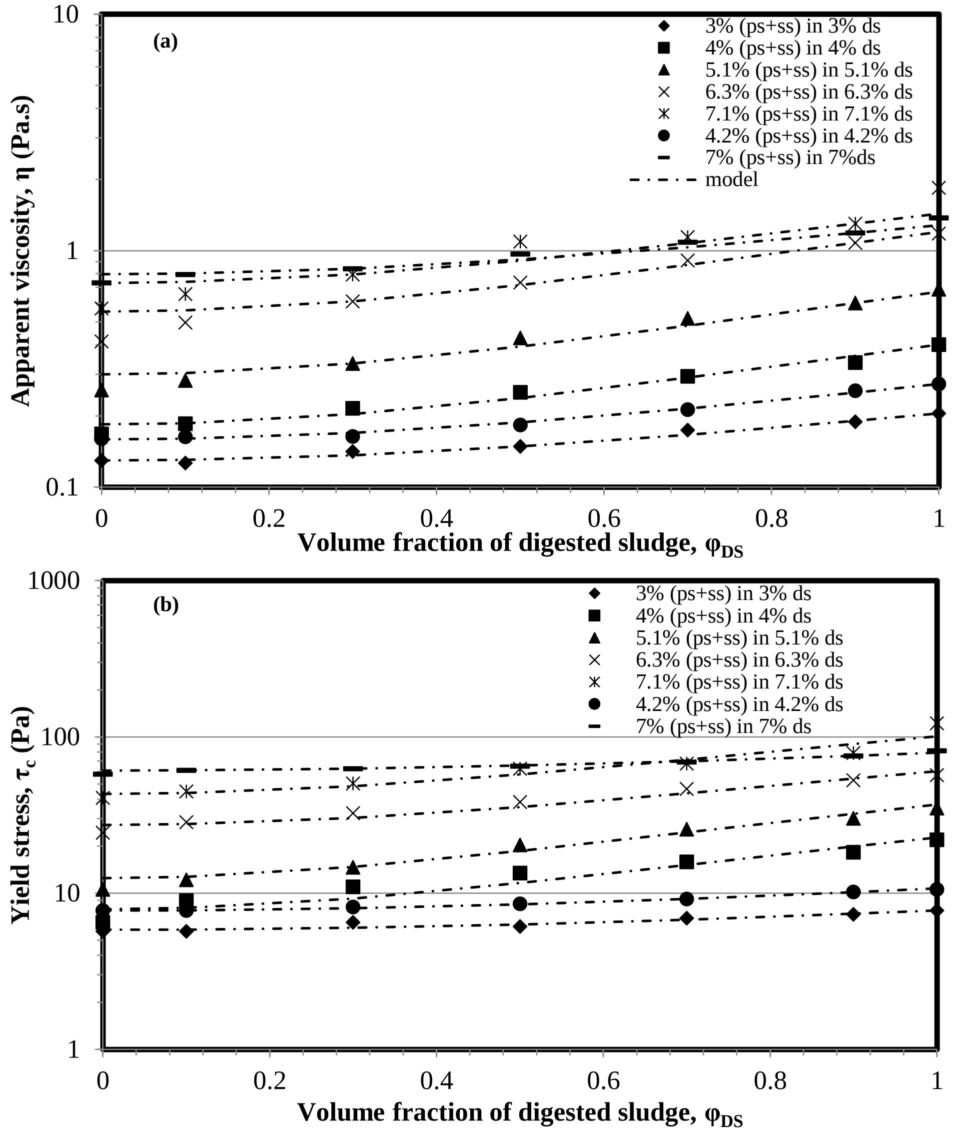

Figure 35: Evolution of the (a) apparent viscosity at a single shear rate of $100 s^{-1}$ and (b) yield stress as a function of the volume fraction of digested sludge for sludge mixtures sampled in the summer (4.2, and 7%) and winter (3, 4, 5.1, 6.3 and 7.1%)

The total solids concentration of the individual sludge is neglected from the correlations because the primary – secondary sludge were mixed at the same individual total solids concentration to digested sludge.

6.3.3 Impact of volume fraction of digested sludge on the shear compliance and shear modulus of sludge mixtures

Except when the primary – secondary sludge mixture is mixed with dilute digested sludge, the apparent viscosity and yield stress of the mixture increases as digested sludge volume fraction increases. This implies that the interactions governing the flow behavior of the mixture are influenced by the presence of digested sludge.

The shear compliance and shear modulus of the sludge mixtures is presented in Figure 36 to demonstrate that the solid interactions are altered as the volume fraction of digested sludge increases. The shear compliance is defined as the ratio between the shear strain and shear stress, $J(t) = \gamma / \tau$ and may be used to measure the strength of the material (Tabuteau, 2006) or to describe the strength of the interactions within the sludge mixtures.

Figure 36 (a) illustrates that when mixtures of 7.1% TS primary and 7.1% TS secondary sludge are subjected to a constant stress, below the yield stress, the resulting shear compliance accelerates, then plateaus as a function of time, indicating that the 7.1% primary – secondary sludge mixture is in the solid regime (i.e. behaves as a viscoelastic solid). When the sludge mixture is subjected to a constant stress, above the yield stress, the resulting shear compliance accelerates as a function of time, indicating that the sludge mixture is flowing steadily in the liquid regime. Likewise, Figure 36 (b) shows that 7.1% digested sludge follows the same trend. This behaviour is a characteristic of soft glassy materials such as suspensions, gels and emulsions (Coussot, 2005, Coussot et al., 2006, Coussot, 2007) and in fact any yield stress material (Baudez et al., 2013a, Eshtiaghi et al., 2013a). However, Figure 36 (b) shown that digested sludge exhibits both shear and time dependent so that when the applied stress equals the yield stress, the shear compliance first accelerates followed by a plateau then eventually accelerates as the creep progresses. This indicates that when the stress is first applied, the digested sludge remained in the solid regime, and then as the creep progresses as a function of time, it transitions into the liquid regime.

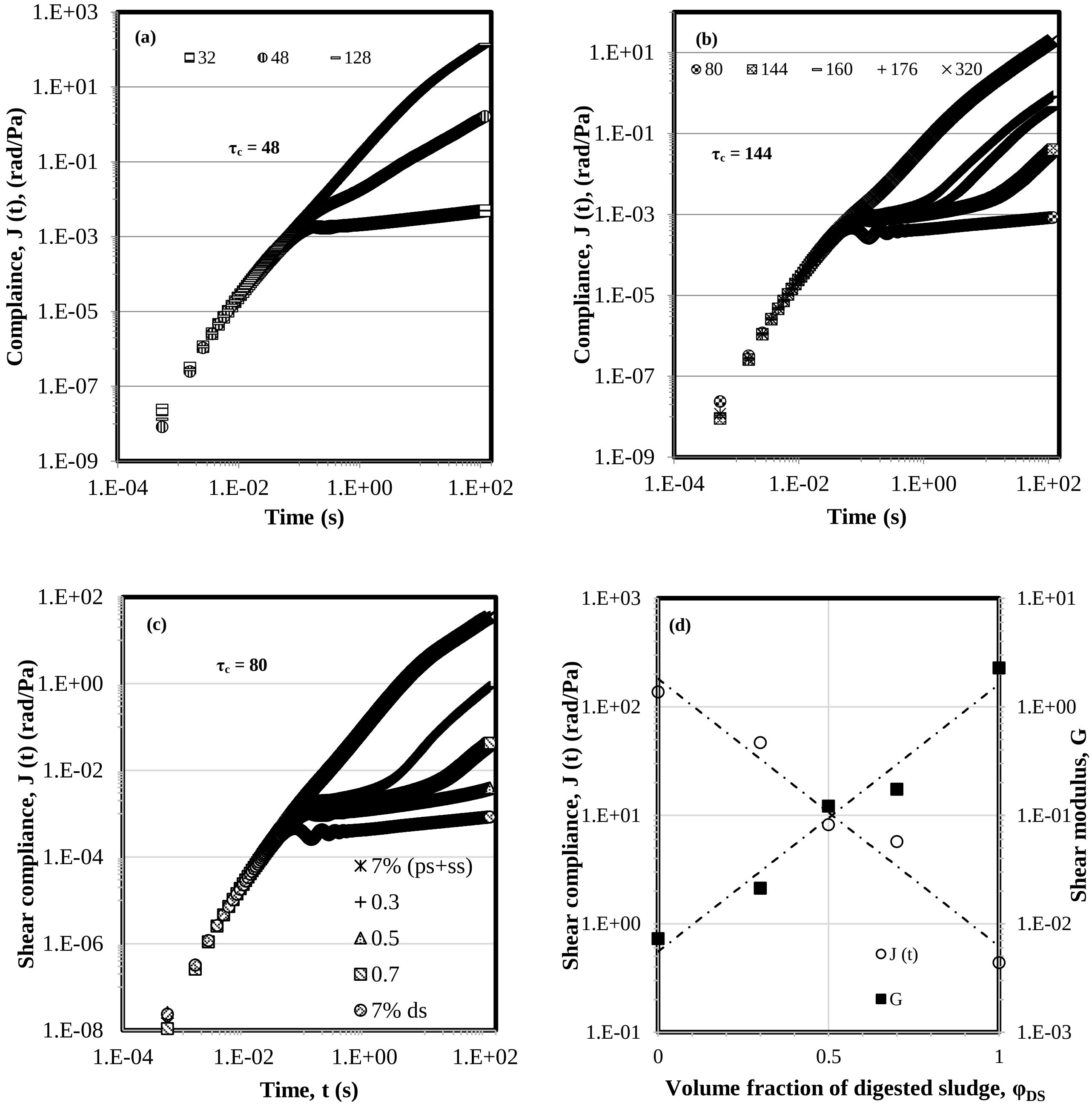

Figure 36: Shear compliance, J (t) versus time for (a) 7.1%primary and secondary sludge mixtures (b) 7.1% digested sludge, (c) 7.1% primary – secondary – digested sludge mixtures at different volume fractions (0 – 1), and (d) Shear compliance, J (t) and shear modulus, G for 7.1% mixtures of primary, secondary and digested sludge as a function of digested sludge volume fraction

The 7.1% primary – secondary sludge mixture exhibits only shear dependent behaviour such that the creep time had little impact on the solid – liquid transition. Baudez (2008) explained that the shear and time dependent behaviour are due to the thixotropic behaviour of sludge whereby sludge undergoes either shear rejuvenation or physical aging over time leading to either a smooth or an abrupt transition from the solid to liquid regime. In fact, the primary – secondary sludge mixture undergoes shear rejuvenation whereby its flow behavior is dependent only on the applied shear (or corresponding stress). This means that it only transitions into the liquid regime when the applied stress equals the yield stress, regardless of the duration of creep (i.e. time). Digested sludge undergoes both shear rejuvenation and physical aging so that when a constant stress is applied for a short period of time, hydrodynamic forces dominate keeping the structure broken (i.e. sludge exhibits shear rejuvenation) leading to deflocculation. However, for prolonged time, non – hydrodynamic interactions dominate so that the digested sludge undergoes physical aging leading to flocculation so that the digested sludge remains in the solid regime. Hence, the solid – liquid transition of digested sludge not only depends on the applied stress but also the duration of creep (i.e. time). Furthermore, Figure 36 (c) demonstrates that the shear compliance of primary – secondary – digested sludge mixtures (for example when $\varphi = 0.5$ or 0.7) first increased, followed by a plateau then increased as the creep progressed. This suggests that the addition of digested sludge to the mixture of primary and secondary sludge changed the flow behavior. As a result, the mixtures of primary – secondary and digested sludge are both shear and time dependent materials undergoing both shear rejuvenation and physical aging.

Indeed, Markis et al (2014) demonstrated that the flow behaviour of primary sludge was a shear dependent, because time of rest (between the pre – shear and creep) had little impact on the final strain. Secondary sludge was both shear and time dependent so that the increasing the time of rest between pre – shear and creep resulted in reduced strain values such that it remained in the solid regime. This means that the shear compliance of primary sludge did not change as a function of time of rest, whilst the shear compliance of secondary sludge decreased with increasing time of rest. As such, primary sludge experienced shear rejuvenation similar to highly thixotropic colloidal suspensions (Coussot et al., 2002) whilst secondary sludge experienced both shear rejuvenation and physical aging similar to gels, emulsions or soft glassy materials (Baudez et al., 2013a). In this study, the primary – secondary sludge mixture undergoes shear rejuvenation similar to primary sludge (Markis et al., 2014) indicating that its network structure behaves similar to colloidal suspensions. However, when digested sludge is added, the behaviour of the resulting sludge mixture changes again so that it undergoes both shear rejuvenation and physical aging similar to gels or emulsions. Tabuteau (2006) highlighted that the compliance of sludge decreased with increasing time of rest indicating that the structure of sludge strengthened over time similar to glass aging.

In the case of primary – secondary – digested sludge mixtures, its shear and time dependent behaviour increases as the volume fraction of digested sludge increases. This indicates that the network structure of these mixtures become stronger as evidenced by plateau of the shear compliance due to physical aging leading to flocculation. Therefore, the primary – secondary – digested sludge mixtures remained in the solid regime for the same applied stress as the primary – secondary sludge mixtures. Furthermore, primary – secondary – digested sludge mixtures transitioned from colloidal like behavior, which undergo shear rejuvenation to more soft glassy behavior similar to gels and emulsions undergoing physical aging and shear rejuvenation. Coussot (2007) explained that colloidal gels or in our case, colloidal suspensions, are governed by short interactions such as van der Waals forces. This means that at low volume fractions of digested sludge, the short interactions of primary – secondary – digested sludge mixture become negligible and the sludge mixture deflocculates (when subjected to a constant stress) and undergoes shear rejuvenation so that it flows steadily in the liquid regime. However, at high volume fractions of digested sludge (for example, $\varphi = 0.7$), the steric interactions within digested sludge (Forster, 2002) lead to physical aging. Hence, higher stresses are required for the sludge mixtures to flow. In fact, the shear compliance of the mixture of primary – secondary – digested sludge decays exponentially with increasing the volume fraction of digested sludge (refer to Figure 36d).

$$J\,(t) = 180.8e^{(-5.7\varphi)} \qquad \text{Eq. 45}$$

Moreover, Figure 36 (d) illustrates that the shear modulus, defined as the measure of rigidity and calculated as the reciprocal of the shear compliance, $G = 1/\,J\,(t)$, increased exponentially with digested sludge volume fraction:

$$G = 0.0055e^{(5.7\varphi)} \qquad \text{Eq. 46}$$

The observed trend emphasises that the solid interactions within the mixture do in fact alter the flow behaviour such that the mixture becomes more rigid and remains in the solid regime as the volume fraction of digested sludge increases.

6.4 Conclusion

This paper demonstrates that mixtures of primary, secondary and digested sludge behave as complex, non – Newtonian, shear thinning materials exhibiting a yield stress similar to soft glassy materials such as colloidal suspensions, gels and emulsions.

The apparent viscosity and yield stress of sludge mixtures were altered dramatically with increasing digested sludge volume fraction. When the sludge mixtures were prepared by mixing a primary – secondary sludge mixture (50: 50 v/v) with digested sludge (prepared at the same total solids concentration), the apparent viscosity and yield stress values were elevated as the volume fraction of digested sludge increased. This trend was attributed to the enhancement of the solid interactions within the sludge mixture so that the shear compliance followed an exponential decay and shear modulus followed an exponential growth as a function of digested sludge volume fraction. However, when a thickened primary – secondary sludge mixture (50: 50 v/v) was added to dilute digested sludge, the network structure of the digested sludge had little influence on the apparent viscosity and yield stress. Instead, the total solids concentration governed the flow behaviour so that the apparent viscosity and yield stress of the final mixture were reduced with increasing volume fraction of digested sludge. This was due to the dilution effect which leads a reduction of the hydrodynamic interactions and non-hydrodynamic interactions.

Finally, correlations were developed to predict the apparent viscosity and yield stress of the three types of sludge mixtures as a function of volume fraction of digested sludge and total solids concentration of the mixture. Furthermore, it was shown that the parameters required to predict the apparent viscosity and yield stress of sludge mixtures may be estimated using the pH of the sludge mixtures. This makes it easier to estimate the energy requirements of the digesters, scale up or optimize the mixing system within anaerobic digesters.

6.5 Acknowledgements

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6.6 References

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6.7 Appendix

Table 6. A. 1: Summary of shift factors in the x (S_x) and y (S_y) axes

Shift factor in the x axis, S_x									
	Total solids concentration, (%)								
	Summer of 2014			Winter of 2015					
φ	**4.2%**	**7%**	**5.0 in 1.8%**	**3%**	**4%**	**5.1%**	**6.3%**	**7.1%**	**4.5 in 1.6%**
0	0.5	3	0.9	0.37	0.25	0.3	1	2	0.6
0.1	0.47	2.7	1	0.4	0.5	0.35	0.9	1.7	0.67
0.3	0.55	2.4	0.35	0.43	0.6	0.37	0.7	1.3	0.8
0.5	0.45	1.6	0.4	0.3	0.7	0.48	0.65	0.9	0.8
0.7	0.35	1.3	0.2	0.27	0.73	0.55	0.6	1	0.3
0.9	0.27	1.3	0.055	0.25	0.75	0.57	0.53	1.1	0.2
1	0.25	1	0.08	0.23	0.77	0.6	0.5	1.5	0.17

Shift factor in the y axis, S_y									
	Total solids concentration, (%)								
	Summer of 2014			Winter of 2015					
φ	**4.2%**	**7%**	**5.0 in 1.8%**	**3%**	**4%**	**5.1%**	**6.3%**	**7.1%**	**4.5 in 1.6%**
0	0.095	0.71	0.19	0.07	0.08	0.13	0.3	0.5	0.17
0.1	0.095	0.75	0.18	0.07	0.11	0.15	0.35	0.55	0.15
0.3	0.1	0.77	0.095	0.08	0.135	0.18	0.4	0.62	0.13
0.5	0.105	0.8	0.06	0.075	0.165	0.25	0.47	0.77	0.1
0.7	0.113	0.85	0.03	0.085	0.195	0.315	0.57	0.83	0.06
0.9	0.125	0.93	0.01	0.09	0.225	0.37	0.65	0.97	0.04
1	0.13	1	0.008	0.095	0.27	0.43	0.7	1.5	0.035

Table 6. A. 2: Summary of parameters required for Eq. 41 and 42

		η_0 (Pa.s)	β	α	m
	0	0.02	0.54	1.03	2.49
	0.1	0.02	0.56	0.97	2.56
	0.3	0.01	0.58	1.05	2.53
Summer of 2014	0.5	0.02	0.59	1.11	2.51
	0.7	0.02	0.58	1.21	2.50
	0.9	0.03	0.55	1.36	2.48
	1	0.02	0.58	1.37	2.52
	0	0.04	0.37	0.28	2.39
	0.1	0.04	0.41	0.39	2.29
	0.3	0.04	0.43	0.49	2.25
Winter of 2015	0.5	0.04	0.48	0.38	2.54
	0.7	0.05	0.47	0.44	2.55
	0.9	0.05	0.48	0.43	2.64
	1	0.05	0.52	0.38	2.83

C_{min} = 1.95% for sludge sampled in summer and C_{min} = 0.05% for sludge sampled in winter

Table 6. A. 3: Summary of parameters for Eq. 41 and 42 required to model sludge mixtures prepared at different total solids concentration

	η_0	β	α	m	C_{min}
Summer of 2014	0.007	0.752	0.089	3.262	0.05
Winter of 2015	0.043	0.407	1.310	1.586	0.05

Table 6. A. 4: Summary of parameters for Eq. 43 and 44 required to model sludge mixtures prepared at the same total solids concentration

	Total solids concentration of mixture, C_{mix} (%)	α	β	η_{DS} (Pa.s)	α	β	τ_{DS} (Pa)
Summer 2014	4.2	0.42	0.58	0.27	0.28	0.73	10.58
	7	0.35	0.58	1.38	0.23	0.75	81.38
Winter 2015	3	0.37	0.63	0.21	0.25	0.75	7.73
	4	0.54	0.46	0.40	0.67	0.36	21.97
	5.1	0.54	0.44	0.69	0.70	0.36	34.99
	6.3	0.55	0.47	1.18	0.58	0.48	56.97
	7.1	0.38	0.40	1.85	0.47	0.35	122.07

CHAPTER 7

INDUSTRIAL IMPLICATIONS

Chapter 7: Industrial implications

7.1 Introduction

Optimization of the unit operations within the sludge treatment process is essential as it affects the overall efficiency of the wastewater treatment process. Anaerobic digestion is one of the key processes employed to stabilize the feed sludge by reducing its solids content by 40% to produce anaerobic digested sludge and biogas (Sanin et al., 2011). The inlet sludge is a hazardous material that is biologically active (Sanin et al., 2011). The organic matter in the sludge feed (primary and secondary sludge or a mixture of the two) is stabilised after entering the digesters. The anaerobic digested sludge is more stable compared to the sludge feed (primary and secondary sludge). As such, its flow behaviour is different compared to the sludge feed. Hence, predicting the flow behaviour of sludge mixtures prior to and after mixing in digesters may be used to estimate and simulate the flow behaviour within the digesters.

Over the years, Slatter (1997, 2001, 2008) has consistently shown the flow behaviour, most notably, the apparent viscosity and yield stress influence the performance of unit operations such as digesters, pipes, heat exchangers and settling tanks within the wastewater treatment process. In this chapter, we outline how the developed correlations for predicting the viscosity and yield stress of sludge mixtures can be utilized in industry.

7.2 Master curve

The master curves which were developed in this study to predict the flow behaviour of sludge mixtures may be employed to obtain the flow curve of any sludge type regardless of being primary, secondary, digested or a mixture of these three types of sludges within the studied total solids concentration and volume fraction (Markis et al., 2015). To demonstrate how the master curve may be used to predict the flow curve of a sludge sample, a hypothetical sludge mixture, 3.7% primary – secondary sludge mixture with a volume fraction of 0.5 will be used as an example.

First, the parameters of the master curve are obtained from Chapter 5: τ_c = 117.13 Pa, K = 27.14 Pa.s^n and n = 0.37.

The table presented below contains the shift factors in the x and y axes for different concentrations and volume fractions of primary and secondary sludge mixtures. This table was extracted from Chapter 5 and will be used to estimate the flow curves.

Table 23: Shift factors in the x and y axes for primary - secondary sludge mixtures

Shift factor in the x axis, S_x								
	Total solids concentration, (%)							
	France		**Australia**					
φ	**5%**	**5.4% in 2.8%**	**3%**	**4%**	**5%**	**6.5%**	**7.1%**	**2.5% in 5.3%**
0	0.045	0.150	0.067	0.110	0.350	0.700	0.900	0.130
0.1	0.035	0.150	0.050	0.130	0.250	0.700	0.800	0.150
0.3	0.033	0.200	0.100	0.170	0.220	0.550	0.750	0.180
0.5	0.035	0.080	0.135	0.300	0.300	0.700	0.700	0.300
0.7	0.030	0.030	0.170	0.400	0.350	0.850	0.430	0.500
0.9	0.035	0.022	0.170	0.430	0.370	0.900	0.700	0.600
1	0.037	0.025	0.200	0.500	0.380	0.950	1.000	0.900
Shift factor in the y axis, S_y								
	Total solids concentration, (%)							
	France		**Australia**					
φ	**5%**	**5.4% in 2.8%**	**3%**	**4%**	**5%**	**6.5%**	**7.1%**	**2.5% in 5.3%**
0	0.011	0.042	0.030	0.061	0.125	0.190	0.250	0.040
0.1	0.007	0.040	0.018	0.052	0.087	0.170	0.237	0.032
0.3	0.008	0.035	0.021	0.054	0.080	0.200	0.270	0.043
0.5	0.010	0.018	0.028	0.068	0.107	0.330	0.360	0.095
0.7	0.011	0.007	0.040	0.097	0.165	0.500	0.500	0.200
0.9	0.014	0.003	0.055	0.135	0.235	0.700	0.770	0.350
1	0.017	0.004	0.068	0.180	0.270	0.800	1.000	0.500

The known data is presented:

%TS = 3.7%, φ = 0.5, τ_c = 117.13 Pa, K = 27.14 Pa.s^n and n = 0.37

The shift factors, presented in Table 23 for 4 %TS were used to estimate the Herschel – Bulkley parameters of a hypothetical 3.7% primary – secondary sludge mixture. These shift factors followed a polynomial relationship as a function of volume fraction ranging from 0 – 1 for 4 %TS primary – secondary sludge mixtures:

$$S_x = 0.041\varphi^2 + 0.3601\varphi + 0.0959 \qquad \text{Eq. 47}$$

$$S_y = 0.2036\varphi^2 - 0.0907\varphi + 0.0607 \qquad \text{Eq. 48}$$

So, by substituting φ =0.5 and solving Eq. 47 and 48, the shift factors were calculated and equal S_x = 0.286 and S_y = 0.066 for a 3.7% mixture at a volume fraction of 0.5.

These shift factors may then be used to determine the flow properties of the 3.7% sludge mixture at a volume fraction of 0.5 using the following equations:

$$K = k_{(master)}.(S_y/{S_x}^n) \qquad \text{Eq. 49}$$

$$\tau_y = \tau_{y(master)}.S_y \qquad \text{Eq. 50}$$

, where K is the fluid consistency (Pa.s^n) and τ_c is the yield stress (Pa) of the hypothetical sludge mixture; S_x and S_y are the shift factors in the x and y axes and n is the flow index and equals 0.37.

As such, K = 2.855 Pa.s^n and τ_c = 7.76 Pa and n = 0.37.

The Herschel – Bulkley model for a 3.7% primary – secondary – digested sludge mixture at a volume fraction of 0.5 is $\tau = 7.76 + 2.855\dot{\gamma}^{0.37}$.

The master curve or the calculated Herschel – Bulkley model may then be used in CFD software to simulate the mixing patterns in anaerobic digesters or critical velocity calculation in pipe lines.

7.3 Apparent viscosity and yield stress correlations

The apparent viscosity and yield stress of sludge or sludge mixtures may be estimated using the developed correlations presented in Chapter 4, 5 and 6. In this chapter, the correlations presented in Chapter 4 and 5 are used as an example for either individual primary or secondary sludge or for primary – secondary sludge mixtures.

7.3.1 Individual primary or secondary sludge

The general form of the correlations used in Chapter 4 to calculate the apparent viscosity, yield stress and fluid consistency of either primary or secondary sludge for a known total solids concentration are presented below:

$$\eta = \eta_0 \, e^{(C\beta)} \qquad \text{Eq. 51}$$

$$\tau_y = \alpha \, (C - C_{min})^m \qquad \text{Eq. 52}$$

$$K = aC^b \qquad \text{Eq. 53}$$

The parameters required for each of the above equations is presented in Table 24 for primary and secondary sludge at a single shear rate of $10s^{-1}$.

Table 24: Parameters required to predict for Eq. 51, 52 and 53 for primary and secondary sludge

	η_o (Pa.s)	β	α (Pa)	C_{min} (%)	m	a	b
Primary sludge	0.19	0.35	0.08	1.85	2.37	0.57	1.47
Secondary sludge	0.07	0.50	0.005	0.96	3.96	0.08	2.30

For example, to find the apparent viscosity, yield stress and fluid consistency of 3.7% primary sludge at a single shear rate of $10s^{-1}$: first, the parameters from Table 24 for primary sludge are substituted into Eq. 51, 52 and 53 to obtain the following correlations for primary sludge:

$\eta \; = \; 0.19 \; exp \; (0.35. C)$

$\tau_y \; = \; 0.08 \; (C - 1.85)^{2.37}$

$K = \; 0.57 \; C^{1.47}$

Next, C = 3.7 is substituted into the above equations and solved. The apparent viscosity, yield stress and fluid consistency become:

η = 0.69 Pa.s, τ_c = 0.34 Pa, K = 3.9 Pa.s^n

7.3.2 Mixtures of primary and secondary sludge

The general form of the correlations presented in Chapter 5 to predict the apparent viscosity and yield stress for primary – secondary sludge mixtures prepared by mixing primary sludge to secondary sludge at the same total solids concentration are presented below:

$$\eta_{mix\,(C_{PS}=C_{SS})} \; = \; \eta_{SS} \, [\alpha(\varphi - \varphi_{min})^2 + \beta] \qquad \text{Eq. 54}$$

$$\tau_{mix\,(C_{PS}=C_{SS})} \; = \; \tau_{SS} \, [\alpha(\varphi - \varphi_{min})^2 + \beta] \qquad \text{Eq. 55}$$

The parameters of Eq. 54 for the apparent viscosity calculation may be estimated using the equations below:

$$\alpha = 0.087 C_{mix} + 0.35 \qquad \text{Eq. 56}$$

$$\alpha = 0.042 C_{mix} + 0.54 \qquad \text{Eq. 57}$$

The parameters of Eq. 55 for the yield stress calculation may be estimated using the equations below:

$$\alpha = 0.035 C_{mix} + 0.67 \qquad \text{Eq. 58}$$

$$\alpha = -0.0186 C_{mix} + 0.365 \qquad \text{Eq. 59}$$

For example, the procedure required to predict the apparent viscosity of a 3.7% primary – secondary sludge mixture with a volume fraction of 0.5 (φ = 0.5) follows:

First, α = 0.67 and β = 0.38 are calculated using Eq. 56 and 57 by substituting C_{mix} = 3.7 because both primary and secondary sludge are mixed at the same solid concentration. α and β are then substituted into Eq. 54 whereby $\eta_{ss\,(3.7\%)}$ = 0.55 Pa.s and φ_{min} = 0.1.

So, $\eta_{mix(C_{PS}=C_{SS})} = 0.55\,[0.67.(0.5-0.1)^2+0.38] = 0.26\,Pa.s$

Therefore the apparent viscosity of a 3.7% primary – secondary sludge mixture with a volume fraction of 0.5 is 0.26 Pa.s.

The yield stress of the mixture can be calculated using the same procedure.

7.4 Sludge pumping

In this section, a procedure is presented to show how the developed correlations for predicting apparent viscosity of 7.1% primary – secondary – digested sludge mixtures at different volume fractions between 0 and 1 can be used to optimise the volume fraction of digested sludge in the mixture to minimise the pumping power and energy costs. The data required to predict the pumping power was extracted from various sources (Sanin et al., 2011, Slatter, 1997, Slatter, 2001, Slatter, 2008) as well as (Eshtiaghi et al, 2012); this is presented in Table 25:

Table 25: Specification of pipeline

Diameter, D	Flow rate, Q	Velocity, v	Length, L
m	m^3/s	m/s	m
0.30	0.14	1.96	100

In the first step, the apparent viscosity of this hypothetical 7.1% primary – secondary – digested sludge mixtures may be calculated following the same procedure explained in Section 7.3.2 for different volume fractions (presented in Table 26).

The pumping power was calculated using a procedure described elsewhere (Slatter, 1997, Slatter, 2001, Slatter, 2008). The data presented in Table 25 was substituted into the various equations, presented below and following the steps for each volume fraction ranging from 0 – 1 for the 7.1% sludge mixture:

For example, for 7.1% primary – secondary – digested sludge mixture with a digested sludge volume fraction zero (φ = 0 which means that there is no digested sludge in the mixture), the wall shear stress can be calculated using Eq. 60 a higher value than the yield stress is calculated.

The yield stress of the primary – secondary sludge mixture was calculated using the same procedure explained in Section 7.3.2 for an equal volume of primary and secondary sludge:

$\tau_y = 40.69$Pa for 7.1%.

$$\tau = 1.5\tau_y \qquad \text{Eq. 60}$$

$$\tau = 1.5.(40.69) = 61\ Pa$$

The friction factor, *f* was then calculated for known calculated wall stresses (τ_0) in Eq. 60:

$$f = \frac{2\tau_0}{\rho v^2} \qquad \text{Eq. 61}$$

$$f = \left[\frac{2(61)}{(1000)(1.96)^2}\right] = 0.032$$

Next, the head loss, H_{loss} (m), was calculated by substituting the friction factor, *f*, velocity, v (m/s), diameter, D (m), length, L (m) obtained from Table 25 and g = 9.81 m/s^2 into Eq. 62:

$$H_{loss} = \frac{4fL}{D}\frac{v^2}{2g} \qquad \text{Eq. 62}$$

$$H_{loss} = \left[\frac{4(0.032)(100)}{(0.3)}\right]\left[\frac{(1.96)^2}{2(9.81)}\right] = 8\ m$$

The pumping power, P (W), was then calculated by substituting the flow rate, Q (m^3/s), the Head loss, H_{loss} (m), the apparent viscosity, η (Pa.s) and density, ρ = 1000 m^3/kg into Eq. 63:

$$P = \frac{\rho g Q H_{loss}}{\eta}$$ Eq. 63

$$P = \frac{(1000)(9.81)(0.14)(8)}{(0.34)} = 32825\ W = 33kW$$

The energy cost was calculated by multiplying the pumping power with the electrical cost - $\$0.14/kw$. This value was obtained from online data for electrical cost.

So, $\frac{\$}{hr} = (33)(0.14) = \$4.6/hr$

This was repeated for each volume fraction for the 7.1% primary – secondary – digested sludge mixtures. The pumping power and energy cost calculations are summarized in Table 26 for different volume fractions of digested sludge from 0 – 1 for sludge mixtures at total solids concentration of 7.1%.

Table 26: Summary of pumping power and energy cost calculations for a 7.1% primary – secondary – digested sludge mixture at different volume fractions (0 – 1)

φ	τ_y Pa	η Pa.s	f	H_{loss} m	P W	P kW	Pumping cost per hr $/hr
0.00	40.69	0.34	0.032	8	32825	33	4.6
0.10	44.76	0.40	0.035	9	31335	31	4.4
0.30	50.46	0.48	0.039	10	28968	29	4.1
0.50	62.66	0.67	0.049	13	25917	26	3.6
0.70	67.55	0.70	0.052	14	26767	27	3.7
0.90	78.94	0.80	0.061	16	27553	28	3.9
1.00	122.07	1.12	0.095	25	30216	30	4.2

To determine the optimum conditions required to achieve minimum pumping power and energy requirements, the pumping power and energy costs are plotted against the volume fraction of digested sludge as presented in Figure 37.

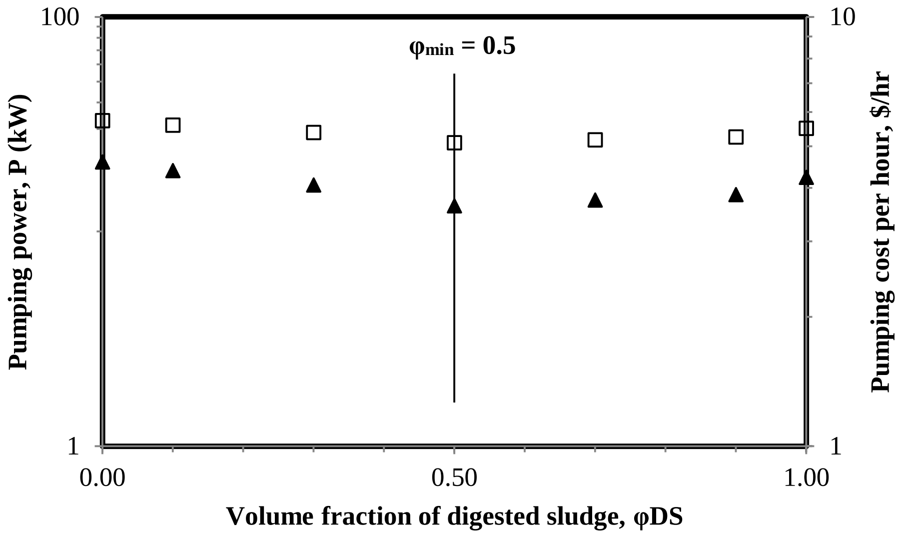

Figure 37: Impact of increasing volume fraction of digested sludge on pumping power and energy cost for 7.1% primary – secondary – digested sludge mixtures

Figure 37 shows that minimum pumping power and therefore energy costs may be achieved for a 7.1% primary – secondary – digested sludge mixture at a digested sludge volume fraction of 0.5 when it is mixed with a mixture of primary and secondary sludge (50:50 v/v). It is worth noting that those predictive correlations in Chapter 6 were developed for primary – secondary – digested sludge mixtures prepared by mixing an equal volume (50:50 v/v) of primary sludge to secondary sludge.

The above presented procedure demonstrates how developing predictive correlations to calculate the apparent viscosity of the primary – secondary – digested sludge mixtures can help determine the optimum amount of digested sludge required to mix with mixtures of primary – secondary sludge (50:50 v/v) to minimise power consumption and pumping cost of mixtures of primary – secondary and digested sludge.

7.5 Sludge mixing

Since it has been demonstrated that the apparent viscosity and yield stress of primary, secondary and digested sludge as well as mixtures of these three sludges are different, it is reasonable to assume that these changes influence the efficiency of mixing. To show how an optimum sludge mixture may be selected using the correlations that were developed in Chapter 6, a mechanically mixed anaerobic digester was selected. To achieve good mixing to prevent short circuiting and solid deposition at the bottom of digesters and maximize biogas production, a specific power input of 5 W/m^3 was recommended by USA EPA guideline (1987). The required specification of a mechanically mixed digester was obtained from elsewhere (Yu et al., 2011) for 5 and 10% sludge; these are presented in Table 27.

Table 27: Specification of a mechanically mixed digester (Yu et al., 2011)

Digester diameter, D_D	**Impeller diameter, D_i**	**Volume of digester, V_D**	**Height of digester, H_D**	**Impeller speed, N**	**Impeller speed, N**
m	**m**	**m^3**	**m**	**RPM**	**RPS**
0.150	0.050	0.500	0.370	1.000	0.017

First, the power input, P (W), was calculated using the specific power input, P_s and the volume of the digester, V_D (m^3):

$$P = P_S \, . V_D \qquad \text{Eq. 65}$$

$$P = (5)(0.37) = 2.5\, W$$

The power number, was then calculated using the Power input, P (W), the impeller speed, N, diameter of the impeller ($D_i = D_D/3$) and the density ($1000 kg/m^3$):

$$N_P = \frac{P}{N^5 {D_i}^3 \rho} \qquad \text{Eq. 66}$$

$$N_P = \frac{2.5}{(0.017)^5 (0.15)^3 (1000)} = 10$$

The mixing Reynolds number was estimated using Figure 38 extracted from McCabe et al (2005).

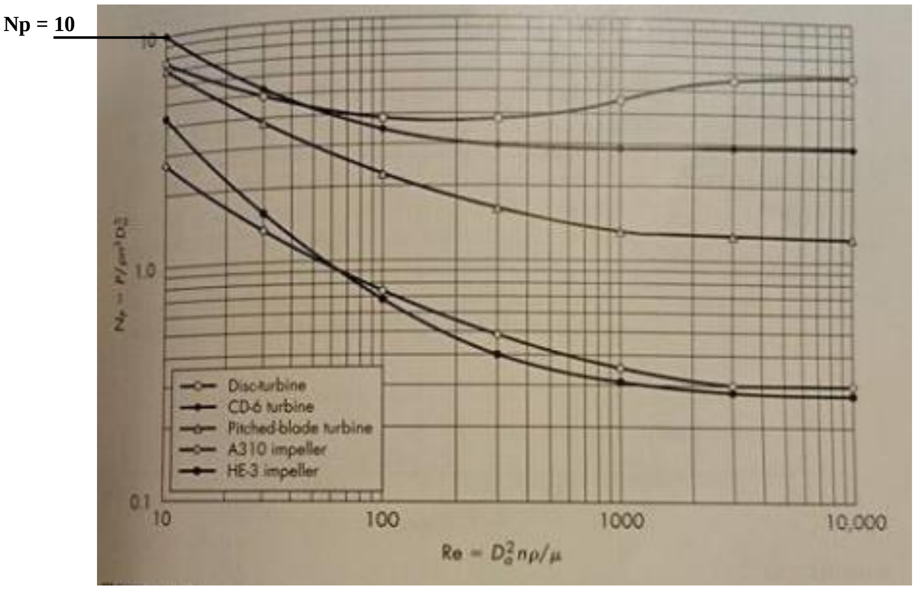

Figure 38: Reynolds number versus power number for various impellers (McCabe et al, 2005)

Therefore, for CD-6 turbine $Re_m = 10$

The apparent viscosity was then calculated substituting the mixing Reynolds number, Re_m, N, diameter of the impeller, Di, and density into mixing Reynolds number equation:

$$Re_m = \frac{ND_i{}^2\rho}{\eta} \qquad \text{Eq. 67}$$

$$10 = \frac{(8)(0.05)^2(1000)}{\eta} \rightarrow \eta = 0.042\ Pa.s$$

If it is assumed that the primary – secondary sludge mixture enters the anaerobic digester with an inlet solids concentration higher than the concentration of digested sludge within the digester, then the apparent viscosity correlation presented in Chapter 6 for a sludge mixture prepared by mixing a thickened primary – secondary sludge mixture to dilute digested sludge may be used to find optimum solids concentration within the digester.

The total solids concentration of the mixture corresponding to the specific power input of 5 W/m^3 required to achieve efficient mixing as recommended by USA EPA guideline (1987) may then be calculated. This was accomplished by using the following equations, extracted from Chapter 6 whereby $\eta_0 = 0.007$ Pa.s and $\beta = 0.752$.

From Eq. 67, an apparent of viscosity of 0.042 Pa.s is required for a good mixing:

$$\eta = \eta_0 \exp(C_{mix}) \rightarrow 0.042 = (0.007 \exp(0.752 C_{mix}) \rightarrow C_{mix} = 4.40\%$$

This suggests that a 4.4% TS sludge mixture is required to achieve efficient mixing using the specific power of 5 W/m^3 recommended by USA EPA guideline (1987). Furthermore, by using Eq. 68, extracted from Chapter 6, it is possible to calculate the volume fraction of the primary – secondary sludge mixture as well as the volume fraction of digested sludge required to achieve efficient mixing at the specific power of 5 W/m^3.

For example, for a hypothetical mixture with a concentration of 4.5% primary – secondary sludge mixture is mixed with 1.6% digested sludge, in a digester. So, by substituting this data into Eq. 68, the volume fraction of primary – secondary sludge is 0.95 and the volume fraction of digested sludge is 0.05. These are the required volume fractions to achieve efficient mixing using the recommended specific power of 5 W/m^3 by the USA EPA guideline (1987).

$$C_{mix} = C_{(PS+SS)}(1 - \varphi_{DS}) + C_{DS}\varphi_{DS} \quad \text{Eq. 68}$$

, where $C_{(PS+SS)} = 4.5\%$, $C_{DS} = 1.6\%$ and $C_{mix} = 4.4\%$

$$4.4 = (4.5)(1 - \varphi_{DS}) + (1.6)\varphi_{DS} \rightarrow \varphi_{DS} = 0.05, \varphi_{(PS+SS)} = 0.95$$

This outcome seems unrealistic because the required data such as the inlet solids concentration, the volume ratio of primary to secondary sludge as well as the solids concentration of digested sludge were assumed values. However, this procedure was aimed at showing how it is possible to predict the required volume fraction to achieve good mixing.

7.6 Conclusion

This chapter shows how the developed knowledge can be implemented by industry to design and optimize unit operations within the wastewater treatment process. A procedure is outlined to demonstrate how the developed master curves as well as developed apparent viscosity and yield stress models may be used to estimate the parameters of a sludge mixture of known total solids concentration and volume fraction within the investigated range. Additionally, this chapter provides a procedure to explain how to use the developed models to optimize the power requirements of unit operations such as pumps and to obtain the required sludge volume fraction to be mixed so that efficient mixing is achieved.

7.7 References

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CHAPTER 8

CONCLUSIONS AND RECOMMENDATIONS

Chapter 8: Conclusions and recommendations

8.1 Introduction

Sludge is produced from human and residential waste, industrial waste and hospital waste, runoff from streets, farmlands and landfill leachates. It undergoes treatment in the municipal wastewater treatment process so that a more stabilized, less hazardous and more useful product is produced.

The wastewater treatment process produces three types of sludge – primary, secondary and digested sludge. Each type of sludge is produced from a different treatment process so that its flow behaviour is different depending on the treatment process. Primary sludge is the product of the primary clarification process and is very difficult to handle whilst secondary sludge is the product of the secondary treatment process. The primary and secondary sludge or a mixture of the two sludges enters the digesters for further treatment whereby it is mixed using gas injection combined with a recirculation of digested sludge. The products of anaerobic digestion are digested sludge and biogas. The digested sludge has a lower pathogen level and is more stable compared to primary and secondary sludge.

The anaerobic digestion process is the most commonly used method to stabilize sludge, however, the additional sludge loads means that a more concentrated sludge requires treatment. Furthermore, the current anaerobic digesters are not designed to handle additional sludge loads. Hence, anaerobic digestion is becoming an inefficient method to treat and stabilize sludge. The first step in the optimizing and improving the efficiency of the anaerobic digestion process is to understand the how and why flow behaviour, most importantly, the how and why apparent viscosity and yield stress changes before and after sludge is mixed. These two flow parameters are studied because ultimately, they influence the efficiency of the digestion process.

In this study, the impact of volume fraction and total solids concentration of the sludge mixture on the flow behaviour of different types of sludge mixtures was investigated through extensive rheometric experimentation. This study focused on how and why the main flow parameters, mainly the apparent viscosity and yield stress of the sludge mixture, changed after the different types of sludge were mixed, depending on the volume fraction and total solids concentration.

One of the aims was to investigate the rheology (solid, transitional and liquid regime) of individual primary and secondary sludge over a wide range of total solids concentration so that any significant differences in their rheology may be detected. The next objective was to investigate the rheology of mixtures of primary and secondary sludge. This aimed at understanding how and why the volume fraction of secondary sludge influenced the apparent viscosity and yield stress of mixtures of primary and secondary sludge. In addition, the impact of increasing the volume fraction of digested sludge on the apparent viscosity and yield stress of the primary – secondary – digested sludge mixtures was also investigated. In this way, the flow behaviour of sludge in anaerobic digesters may be predicted.

The intention of this chapter is to summarize the major outcomes achieved in this thesis and initiate ideas for future work.

8.2 Conclusions

The key finding from this study which have been attained for the first time in the field of sludge rheology are as of follows:

- Primary sludge, secondary sludge, digested sludge, primary – secondary sludge mixture and primary – secondary – digested sludge mixtures behaved as non – Newtonian, shear thinning materials exhibiting a yield stress (Chapter 4, 5, 6).
- At low shear stresses, below the yield stress, sludge or a sludge mixture, behaved as a visco – elastic solid similar to soft glassy materials or yield stress materials such as colloidal suspensions, gels and emulsions (Chapter 4, 5, 6).
- Primary sludge yielded abruptly similar to highly thixotropic colloidal suspensions experiencing viscosity bifurcation. Secondary sludge transitioned smoothly into the liquid regime, similar to gels (Chapter 4).

- In the steady state, sludge or a sludge mixture followed a dimensionless form of the Herschel – Bulkely model and it was possible to develop a master curve for different sludge mixtures and concentrations. Based on the master curve, correlations were developed to predict the apparent viscosity and yield stress of different sludge mixtures based on the flow parameters of the individual sludge as well as their volume fraction (Chapter 4, 5, 6).
- The apparent viscosity of primary, secondary and digested sludge as well as any sludge mixture increased exponentially with increasing total solids concentration whilst the yield stress increased following a power law model as a function of total solids concentration. These observations were attributed to the strengthening of the hydrodynamic and non-hydrodynamic interactions within the sludge (Chapter 4).
- The apparent viscosity and yield stress of primary – secondary sludge mixtures, prepared by mixing primary sludge to secondary sludge at a similar total solids concentration were influenced by the volume fraction of secondary sludge. The apparent viscosity and yield stress of primary – secondary sludge mixtures increased following a power law with volume fraction of secondary sludge (Chapter 5).
- The apparent viscosity and yield stress of a primary – secondary sludge mixtures prepared by mixing dilute secondary sludge to thickened primary sludge (and vice versa) was influenced by the dilution effect. As such, the apparent viscosity and yield stress of the resulting mixture increased with increasing volume fraction of the concentrated sludge regardless of sludge type following a power type function (Chapter 5).
- A minimum apparent viscosity and yield stress was detected for primary – secondary sludge mixtures corresponding to a volume fraction of 0.1. This was attributed to the liquefying effect experienced when secondary sludge is first added (Chapter 5).
- The apparent viscosity and yield stress of primary – secondary – digested sludge mixtures, prepared by mixing a primary – secondary sludge mixture (50: 50 v/v) with digested sludge (at the same total solids concentration) increased with increasing volume fraction of digested sludge. This was attributed to the enhancement of the solid interactions within the sludge

mixture. The apparent viscosity and yield stress followed a power law type model as a function of volume fraction of digested sludge (Chapter 6).

- The shear compliance and shear modulus of primary – secondary – digested sludge mixtures increased exponentially as a function of digested sludge volume fraction validating the assumption that the solid interactions within the mixture were strengthened as the volume fraction of digested sludge increased (Chapter 6).
- The apparent viscosity and yield stress of primary – secondary – digested sludge mixtures, prepared by mixing a thickened primary – secondary sludge mixture (50: 50 v/v) with dilute digested sludge was influenced by the dilution effect so that the hydrodynamic interactions and non-hydrodynamic interactions were reduced. The apparent viscosity and yield stress of the final primary – secondary – digested sludge mixture was reduced with increasing volume fraction of digested sludge. This trend was similar to the thickened primary – dilute secondary sludge mixture. The apparent viscosity followed an exponential model whilst the yield stress followed a power law type model with total solids concentration of the primary – secondary – digested sludge mixtures. Finally, the parameters required to predict the apparent viscosity and yield stress of sludge mixtures were estimated from the pH of the sludge mixtures (Chapter 6).
- The developed master curves as well as the developed apparent viscosity and yield stress correlations for different types of sludge mixtures may be used to estimate the Herschel – Bulkely model. Moreover, the correlations may be used to predict the optimum power and energy costs of unit operations such as pumps and mixing systems. (Chapter 7).

8.3 Recommendations

- Investigate mixing thickened primary –secondary sludge at a different volume fraction (30:70 and 70:30 (v/v) with digested sludge at different volume fractions (0 – 1).
- This study may be extended to investigate the impact of temperature on different sludge mixtures rheology, most notably, the apparent viscosity and yield stress.

- This study may be extended to investigate the impact of temperature in conjunction with air injection on the rheology of sludge mixtures. Most notably, the study should focus on how the apparent viscosity and yield stress changes so that anaerobic digester conditions may be simulated. Any correlations that are developed will ultimately be used to estimate the apparent viscosity and yield stress within anaerobic digesters.