

# **Digestion Processes and Their Effect on Dewatering and Bound Water Content of Sludge**

**Sangeetha Subramanian**

Thesis submitted to the faculty of  
the Virginia Polytechnic Institute and State University  
in partial fulfillment of the requirements for the degree of

**Master of Science  
in  
Environmental Engineering**

John T. Novak, Ph.D., Chair  
Gregory D. Boardman, Ph.D.  
Clifford W. Randall, Ph.D.

February 4<sup>th</sup>, 2005  
Blacksburg, Virginia

Keywords: J-vap process, sludge dewatering, bound water, water distribution in sludge, dilatometer, digestion

Copyright 2004, Sangeetha Subramanian

# Digestion Processes and Their Effect on Dewatering and Bound Water Content of Sludge

Sangeetha Subramanian

## **Abstract**

Solids handling can contribute to a significant portion of the operational costs of a wastewater treatment plant, contributing up to 50% of the total expenses in certain instances. Sludge dewatering and drying therefore become necessary not only from the operational perspective, but also from the economical viewpoint. The J-Vap process combines the above-mentioned processes, by pressure filtration of sludge followed by application of vacuum and heat. However, when cationic polymer conditioned sludge is dewatered in the J-Vap, the polymer is suspected to interact with the filter media at high temperatures, resulting in the formation of a skin layer that hinders efficient dewatering. The first part of the study has looked at various digestion processes and how they affect the skin formation phenomenon. The results showed that temperature played a significant role in determining the amount of polymer that adhered to the filter media.

The second part of the study focused on different kinds of digestion processes and their effects on extracellular polymeric substances, bound water content and dewatering. Bound water tests were used to determine the maximum achievable solids concentration on dewatering. Bound water content of solids obtained from field centrifuges run at different torques and g values were evaluated and fitted on a standard graph obtained from lab pressed sludge with different solids concentration. The bound water was seen to decrease with increasing solids content till 20%, after which a nearly constant 1.0 g of bound water was present for every gram of dry solids seen. The results indicate that nearly 50% solids concentration could be achieved on mechanical dewatering. In reality, only 30 to 35% solids concentration was attained both in the lab and on the field. It was determined that dilatometry attributed the increase in cake solids to the decrease in bound water. However, the use of bound water as a predictive tool for determining cake solids was not practical since the bound water calculations use the solids content in the calculations.

## **Acknowledgements**

I gratefully acknowledge the guidance I have received from Dr John Novak over the period of my masters education at Virginia Tech. He has given the research group freedom of thought and allowed our growth in various directions while being readily accessible for any doubts and clarifications we have had. He has been especially kind to me in keeping me funded and focused on research. I thank my other committee members – Dr. Clifford Randall and Dr. Gregory Boardman for their inputs and timely guidance.

I acknowledge the monetary support and sound advice that I have received for the completion of this project from Dr. Abu-Orf Mohammad (US Filter) and Dr. Sudhir Murthy (DCWASA) over the course of a year and a half.

I would also like to acknowledge and thank the timely help and advice I have had regarding lab practices and experimental methods from Julie Petruska and Jody Smiley. Betty Wingate and Sherry Burke have been most co-operative and supportive in administrative issues and I duly acknowledge their help. I thank Christopher Muller, David Inman and Nitin Kumar for having helped me out with a ready supply of sludge whenever I needed it and sharing their knowledge with me. I also thank Heather Rectanus for her comments and suggestions on this thesis.

Last but not the least, I would like to express my appreciation to my professors, friends and colleagues at Virginia Tech with a special mention to Dr. Nancy Love, Nitin Verma and Sheetal Bhatt for just being there and making the world of a difference in my life. I would also like to acknowledge Ayesha, Baskar, Divya, Monique, Nanda, Rahul and Tasneem for having been there at various points of my life.

## **Dedication**

### ***To my family...***

My father for encouraging me to pursue my dream

My mother for inspiring in me, her love of all life, truth and education

My brother, grandparents and uncle for their unquestioned love and affection towards me

# Table of Contents

Abstract.....	ii
Acknowledgements.....	iii
Dedication.....	iv
Table of Contents.....	v
List of Figures.....	vi
List of Tables.....	viii
I Literature Review.....	1
Introduction.....	1
Digestion effects on sludge.....	2
Bound water content and its relationship to mechanical dewatering.....	5
Solids content and its relationship to bound water content.....	9
Bound water content and thermodynamics.....	9
The J-Vap Process and filter skin formation.....	11
Engineering significance.....	14
Reference.....	15
II Manuscript 1.....	20
Digestion Processes and their Effect on Filter skin formation.....	20
Abstract.....	20
Introduction.....	21
Research Objectives.....	24
Materials and Methods.....	24
Results and Discussion.....	26
Conclusions.....	32
Acknowledgement.....	33
Reference.....	34
III Manuscript 2.....	54
Effect of Digestion Processes on Bound Water Content of Sludge.....	54
Abstract.....	54
Introduction.....	54
Research objectives.....	56
Materials and methods.....	57
Results and Discussion.....	59
Conclusions.....	64
Acknowledgement.....	65
Reference.....	81
Vita.....	84

# List of Figures

## Mauscript 1

Figure II-1: Evolution of cake height and liquid pressure during constant pressure dewatering of sludge.....	37
Figure II-2: Dewatering profiles of Pepper’s Ferry sludge conditioned with different polymers.....	38
Figure II-3: CST at optimal dose for Pepper’s Ferry sludge using different polymers ....	39
Figure II-4: Optimal dose polymer requirements for Pepper’s Ferry sludge .....	40
Figure II-5: Polymer requirements versus minimum CST at optimal dosage for Pepper’s Ferry sludge.....	41
Figure II-6: SRF of Pepper's Ferry sludge conditioned with different polymers .....	42
Figure II-7: Analysis of SRF and compression time required to dewater to 4 psi liquid pressure .....	43
Figure II-8: Cake solids content obtained on compression dewatering of sludge conditioned with different polymers to 4, 6, 8 and 10 psi liquid pressures versus time taken.....	44
Figure II-9: Minimum CST for different sludges conditioned with polymer SH 650 .....	45
Figure II-10: Polymer requirements of SH 650 at optimal dosage for different sludges ..	46
Figure II-11: SRF of different kinds of digested sludge after conditioning at optimal dosage .....	47
Figure II-12: Dewatering profiles of different kinds of digested sludge conditioned with SH 650 .....	48
Figure II-13: Polymer requirements versus CST at optimal dosage for different sludges	49
Figure II-14: SRF and polymer residual of thermophilic digested sludge at different temperatures .....	50
Figure II-15: SRF and polymer residual of Thermo – aerobically digested sludge at different temperatures .....	51
Figure II-16: SRF and polymer residual of mesophilic digested sludge at different temperatures .....	52
Figure II-17: SRF and polymer residual of Meso - aerobically digested sludge at different temperatures .....	53

## Mauscript 2

Figure III-1: Comparison of polymer requirements at optimal dosage between anaerobically digested and sequentially digested sludges .....	70
Figure III-2: Comparison of minimum CST of anaerobically digested and sequentially digested sludges.....	71
Figure III-3: Soluble protein concentration of anaerobically digested and sequqntially digested sludges.....	72

Figure III-4: Polysaccharide concentration of different sludges subjected to anaerobic and sequential digestion .....	73
Figure III-5: Figure showing additional VS destruction on sequential digestion.....	74
Figure III-6: Relationship between sludge solids and cake solids obtained on centrifugation .....	75
Figure III-7: Comparison of sequentially and anaerobically digested sludges compression dewatering profiles. ....	76
Figure III-8: Cake solids content obtained on centrifugation at 10,000g for 15 minutes and its relation to bound water content measured by dilatometry.....	77
Figure III-9: Relationship between sludge solids and bound water content, showing the presence of an unremovable water fraction associated with the sludge .....	78
Figure III-10: Relationship between cake solids obtained on centrifugation and compression cell loading rate.....	79
Figure III-11: Solids content predicted using bound water calculations versus observed solids concentration .....	80

# List of Tables

## Literature Review

Table I-1 Different techniques used to measure physical distribution of water in sludge ..7

## Mauscript 1

Table II-1: Digestion techniques and SRT maintained .....36

## Mauscript 2

Table III-1: Classification of water distribution according to different authors..... 66

Table III-2: Description of the digestion processes adopted in this study ..... 67

Table III-3: Description of centrifuges from which field samples were taken..... 68

Table III-4: Solids content and bound water content of field centrifuged samples ..... 69

# I Literature Review

## ***Introduction***

Wastewater can be defined as a combination of liquid and any solid materials generated by the use of water for various domestic and industrial purposes. This water is generally characterized by the presence of organic matter, nutrients such as nitrates and phosphates, colloidal and particulate matter and microbes including pathogens. The presence of biologically available sources of carbon and nutrients results in the growth of microbes, planktonic and plant life, which exert an oxygen demand on the system if the water is released to streams, lakes and estuaries without treatment.

The basic objectives of wastewater treatment include:

1. removal of toxic pollutants present,
2. reduction of biochemical oxygen demand, and
3. reduction of pathogens to a safe level before releasing the water into the environment (Metcalf and Eddy, 2003).

In the process, separation of solids from the liquid phase plays an important role. Up to 60% of the solids present in typical municipal wastewater is settleable and removed through primary and secondary clarification processes. Soluble organic matter is converted into biological floc which also allows it to be removed by settling. The solids so obtained are high in organic content and need to be stabilized before suitable disposal methods can be considered. Stabilization is essential in order to avoid post – disposal organic degradation, which can result in vector and odor problems. As a side consequence of stabilization, the solid mass is also reduced. Aerobic or anaerobic digestion techniques are typically used to stabilize solids produced in wastewater treatment. In anaerobic digestion, pathogen reduction is also achieved and a useful side product – methane gas is produced (Inman, 2004).

The kind of stabilization/ digestion process adopted, namely - aerobic or anaerobic digestion is decided depending on the size of the plant, concentration of the wastes being

treated and the Biochemical Oxygen Demand (BOD) to Chemical Oxygen Demand (COD) ratio. Large-scale plants often select anaerobic digestion processes, as they can be energy self-sufficient. Anaerobic digestion, however is more prone to process upsets, results in a poorer quality effluent with respect to dewatering properties and requires greater operator care, capital investment and machinery. Aerobic digestion is often suggested for small and medium scale plants with excess blower capacity (Grady *et al.*, 1999).

### ***Digestion effects on sludge***

Digestion processes have been known to cause significant changes in sludge constituents such as particle size distribution (Jin *et al.*; 2004), solids composition, protein and polysaccharide content (Novak *et al.*; 2003, Novak *et al.*; 2004, Park *et al.*; 2004) and water distribution between the different phases (Jin *et al.* 2004). The extent and intensity of the changes in composition depend on various parameters such as SRT, temperature of digestion, oxygen concentration and mixing intensity. These changes have corresponding impacts on various aspects of sludge including dewatering and bound water content. The forthcoming sections discuss the effects in detail of each of these parameters in relation to the dewaterability.

### **Effect of floc and particle size on dewatering**

Barbusiniski and Koscielniak (1997) studied the changes in floc size and sludge specific area during aerobic digestion. They found that the particle size distribution changed significantly with increasing periods of digestion. An initial analysis of the sludge revealed maximum frequency of occurrence of particles in the size range between 75 to 100  $\mu\text{m}$ . After aerobic digestion for a period of 24 days, the highest frequency of occurrence was for particles in the size range of 15 to 35  $\mu\text{m}$ . On further digestion, the most frequent particle size range fell to the 10 to 15  $\mu\text{m}$  size range. They also observed that the SVI increased till day 22 and fell sharply afterwards, signaling the formation of pin-point floc.

Jin *et al.* (2004) measured the bound water content of activated sludge from seven different full scale activated sludge wastewater treatment plants in Australia by

centrifugal dewatering and correlated it to their Capillary Suction Time (CST). Meeten and Smeulders (1995) define capillary suction time as the time taken for the free water in sludge to permeate through a fixed distance on Whatman 17 type filter paper. Jin *et al.* (2004) found a linear relationship between the bound water content and CST. Further, they also found a good correlation ( $R^2 = 0.83$ ) between the floc size and CST.

Guan *et al.* (2003) measured the floc size and fractal dimension of the floc and correlated it with the CST. They found that an increase in floc size and a decrease in fractal dimension corresponded to a decrease in the CST. They suggest that upon addition of polymer, the loose flocs coalesced into a network of similar porosity. They also indicated that the correlation might hold only for low-pressure filtration devices or vacuum belt filter units, where compaction of cake does not occur to a significant extent. In the case of pressure filtration devices, they expect that the correlation would be lost between individual floc characteristics and sludge dewaterability.

Liss *et al.* (2002) studied the effects of Solids Retention Time (SRT) on microbial floc structure using environmental scanning electron microscopy. The study focused on the effect of SRT in Sequential Batch Reactors (SBR) on microbial community composition in activated sludge and settleability. They found that larger sized flocs were formed at lower SRTs. The stability of flocs exhibited to different environmental conditions like pH, ionic strength, cations and the presence of chelating agents like EDTA, however increased with the SRT.

### **EPS effects on dewaterability**

Different studies have shown that extra cellular protein concentrations affect the dewaterability of sludge to a significant extent. A review of literature by Liao *et al.* (2001) revealed that EPS had been shown to have both positive and negative correlations with dewaterability and compressibility of sludge as measured by Sludge Volume Index (SVI). They also report that some studies have revealed no correlation between EPS and SVI. In their study on the effects of SRT on the production of EPS in SBRs, they found that the ratio of proteins to polysaccharides increased with SRT, while the total amount of

EPS present in solution was not found to vary significantly. They further showed that the EPS concentration and composition affected the sludge settleability.

Novak et al (2001) studied the effects of aerobic and anaerobic digestion on the release of soluble EPS and its effects on dewatering measured by SRF, CST and polymer requirements. Their study showed that anaerobic digestion released more proteins than did aerobic digestion, and the concentration of proteins increased with SRT till day 10, after which, it stabilized. The increase in protein concentration was simultaneously accompanied by a deterioration of dewatering characteristics shown by increasing CST. A later study by Novak et al. (2003) focused on the characterization and quantification of biopolymers released during aerobic and anaerobic digestion over a range of temperatures. The results showed that the rate of release of biopolymers was increased at elevated temperatures, whereas the quantity of EPS remained nearly the same.

As a part of their digestion studies, Novak et al. (2003) identified the existence of two distinct fractions of polymers in sludge – namely a divalent cation associated fraction and an iron – bound fraction. The authors found that aerobic and anaerobic digestion released different portions of polymers from the sludge and neither of the two processes could separately be accountable for total degradation of the biopolymers. Novak et al. (2004) conducted experiments in which they aerobically and anaerobically digested sludge samples and related soluble and colloidal protein concentrations to dewatering parameters – SRF and CST. Particle size distribution study of the biopolymers revealed that the colloidal fraction between 1.5  $\mu$ m and 30 Kilo-daltons was responsible for the decrease in dewaterability, in cases where high protein concentration was observed. They also noted that aerobically digested sludges had a larger fraction of polysaccharides in them, while the main fraction of EPS in anaerobically digested sludges was composed of proteins.

A study by Park et al. (2004) showed that the digestion process altered the protein to polysaccharide ratio. Anaerobic digestion released a considerable amount of proteins into solution, which negatively impacted dewatering as measured by CST. Aerobic

digestion increased the concentration of soluble polysaccharides and resulted in better dewaterability as compared to sludge digested anaerobically. Martinez et al. (2004) studied the effect of proteins and polysaccharide concentrations on Sludge Volume Index (SVI). Their study suggested that the molecular weight of the EPS played a more significant role in determining the SVI than the concentrations themselves. The study by Jin et al (2004) showed that dewatering properties were correlated with the concentration of extractable proteins present in the sludge. They measured extractable EPS by the cation exchange resin method and correlated this to the CST of sludge obtained from seven different activated sludge wastewater treatment plants in Australia. The study confirmed that EPS contributed to the water binding ability of sludge in two distinct phases – the bound water phases and the free water phase. The dewatering parameter – CST showed a negative correlation with the extractable EPS.

### ***Bound water content and its relationship to mechanical dewatering***

Water distribution in sludge can be broadly classified based on the ease of its removal from the system. One of the more popular classifications of the water distribution as defined by Smith and Vesilind (1995) is:

- Free water: the water that is not associated with the solid particles and is free to move between individual sludge particles,
- Interstitial water: the water trapped in the crevices and interstitial spaces of flocs and microorganisms,
- Surface or vicinal water: the fraction of water held to particle surfaces by adsorption and adhesion, and
- Water of hydration or bound water: water that is chemically bound within the particle structure and can be removed only by the application of heat.

Traditionally, bound water has been operationally defined to predict the fraction of water that will remain associated with the solids after mechanical dewatering. Therefore, the method of bound water determination has a significant impact on the numerical results.

Some of the more popular methods of bound water determination are addressed in the following discussion.

### **Determination of bound water**

Vaxelaire and Cezac (2004) reviewed the available techniques for determination of the moisture distribution in sludge. A brief overview of the drying curve technique, Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC), dilatometry, and centrifugal tests is discussed in the forthcoming paragraphs in terms of techniques and their respective advantages and disadvantages.

The drying test involves monitoring the evaporation flux and predicts the fractions of different waters present on the basis of the type of the bond between the water and the solids. A typical curve shows four distinct phases, a short period of increasing temperature, a period of constant rate of drying, a falling rate period, where the drying boundary progresses into the material, and a final slow falling rate period due to the slow evaporation of the bound water. The point of transition between the constant rate and the falling rate period is used to quantify the amount of bound water as it signifies the transition of the drying process from being controlled by external conditions to properties intrinsic to the material being dried. Kopp and Dichtl (2000) calibrated the thermogravimetric method (drying curve method) to predict the maximum possible dewatering results for different kinds of polymer-conditioned sludge.

Dilatometry measures the amount of bound water present in sludge, where bound water is defined as the fraction of water that does not freeze at  $-20\text{ }^{\circ}\text{C}$ . This is calculated as the difference between the amount of total water present (obtained from drying at  $105\text{ }^{\circ}\text{C}$ ) and the amount of free water present in sludge.

DTA measures the difference in temperature between a thermally inert reference standard and the sample in relation to imposed temperatures. Changes in the energy levels of the sample (both exothermic and endothermic) can be determined by plotting the temperature difference against the imposed temperatures. The temperature data can be transformed into the amount of heat required and/ or into amount of heat required for transformation of unfrozen water.

DSC measures the variation of heat absorbed/ released by a sample placed in a controlled atmosphere and subjected to a temperature scan. At a given threshold temperature defined for determining bound water content, the amount of heat released can be directly attributed to the amount of free water present in the sample. The bound water content can be determined by finding the difference between the total and the free water present.

Centrifugal tests have also been suggested to determine bound water content. Colin and Gazbar (1995) have suggested centrifugation at 60,000 g for 120 minutes to determine the maximum solids content achievable by centrifugation. The water still remaining in the solid matrix is termed bound water.

Kopp and Dichtl (2000) surveyed the literature and tabulated different tests that were performed to determine bound water content and the respective definitions of bound water according to the different studies undertaken. Some of the more pertinent data are given in the table below.

**Table I-1 Different techniques used to measure physical distribution of water in sludge**

<b>Author</b>	<b>Method</b>	<b>Definition of bound water</b>
Jones and Gortner (1932)	Dilatometry	Water content that does not freeze at 0°C
Hukelekian <i>et al</i> (1952)	Dilatometry	Water content that does not freeze at -20 °C
Lee <i>et al</i> (1995)	DSC	Sum of interstitial, surface and intracellular water
Coackley <i>et al</i> (1957)	Drying curve method	Bound water refers to intracellular water separated at first critical point. Surface water refers gets separated at second critical point
Robinson <i>et al</i> (1992)	Dilatometry	Bound water is the sum of interstitial, surface and chemically bound water.
Herwijn (1996)	Drying curve method	Bound water has a binding enthalpy of greater than 1Kj/ Kg
Smollen (1987 & 1990)	Drying at 30 and 105 °C	Interstitial water evaporates at 30 °C, while bound water evaporates at 105 °C

Source: Kopp and Dichtl (2000)

Relative advantages and disadvantages of the methods are discussed in the following material. The drying curve method should follow both the sample size and the sample mass during the test, since the method is based on flux data. For shrinkable materials like biological sludge, it becomes all the more important to keep track of both these parameters. The use of dilatometric tests has been argued by many from both favorable and unfavorable points of view. Robinson and Knocke (1995) reported the dilatometry method to be more accurate and repeatable than the drying curve tests. However, Smith and Vesilind (1995) report a number of difficulties in the implementation of the test including the high sensitivity of the system to the presence of air and gases in sludge, the formation of bubbles as the temperature is lowered and the sensitivity of the test to ambient temperature and pressure.

DTA and DSC are more sensitive tests than the dilatometric tests, but the choice of the threshold temperature plays a very important role and the heterogeneity of sludge samples can cause large error margins to creep in to these tests. The centrifugal test is an operationally defined test and according to the method suggested by Colin and Gazbar, centrifuge speeds of up to 60,000 g are needed to determine the fraction of water that remains associated with the solid fraction on maximal mechanical dewatering. Attaining such high speeds in laboratory settings may be a problem. Further, Yen and Lee (2001) point out that the Centrifugal Settling Method (CSM) for determination of bound water depends on the assumption that the sludge cake matrix formed on centrifugation is plastic, and the sludge cake once deformed by stress will not rebound on release of the stress. They disproved this assumption and therefore, the use of CSM as a method for determining the bound water content has been questioned.

On comparing all the above-mentioned methods of bound water determination, the choice of the dilatometric method for this study can be justified for the following reasons:

- Good reproducibility,
- Minimal equipment and capital costs,
- Bound water determination based on thermodynamic principles that can be manually controlled over a wide temperature range, and

- Errors associated with the method can be visually checked instantaneously and immediate rectifying measures taken or tests repeated.

### ***Solids content and its relationship to bound water content***

Early studies by Robinson (1989) showed that the bound water content decreased exponentially as the solids content increased. The author showed that a nearly linear relationship existed between cake solids content and bound water content when solids concentrations were in the range of 15 to 60%. For solids concentrations below 15 %, the bound water was not seen to vary much with an increase in solids concentration. This can be attributed to the fact that bound water content is measured in terms of grams of bound water per gram of dry solids present. When the solids concentration fell below 15%, the amount of free water present was large enough to contribute significant error/interference in the measurement of bound water. This was explained in the study by Smith and Vesilind (1995), where very low solids concentrations were used to determine bound water concentrations. However, in their study, it was seen that the error was much greater at a solids concentration of below 1% and rapidly decreasing at higher solids concentrations.

### ***Bound water content and thermodynamics***

Dilatometry and other cooling/ drying techniques like DSC and DTA were developed using a thermodynamic basis. Dilatometry determines the volume of water that remains unfrozen at a temperature below the freezing point of free water. According to Wu *et al.* (1998), choosing a freezing temperature for water is equivalent to setting the binding strength used to divide the bound fraction from the unbound fraction. Lee and Lee (1995) gave a detailed thermodynamic approach to these tests. They assumed bound water to be an ideal solution containing  $n_B$  mols of solute and  $n_A$  mols of water. The chemical potential of bound water was thus reduced by a binding strength equivalent to  $E_B$  (Kj/ kg dry solids). They said that, in the dilatometric tests, unfrozen water could be equated to the amount of moisture showing a freezing temperature less than the selected freezing temperature ( $T_f$ ). The bonding energy in a dilatometric experiment could then be expressed as

$$E_B = \frac{T_f - T_{f0}}{T_{f0}} \Delta H_f$$

where

$E_B$  is the bonding energy associated with the bound water fraction,

$T_f - T_{f0}$  refers to the difference in temperature between the starting and the threshold temperature and

$\Delta H_f$  refers to the enthalpy change during the process.

Since pressure remains a constant during the experiment and changes in volume are recorded against a change in temperature, the change in enthalpy can be calculated as a function of the above-mentioned variables. Lee (1996) found that the binding energy of water to the sludge particles was approximately 25 kJ/ Kg when the freezing temperature was taken to be  $-20^{\circ}\text{C}$ . Based on the calculations, Lee suggested that both strongly bound and weakly bound water constituted about 5 kg/ kg DS. He also suggested that, the fraction of moisture getting frozen in the dilatometric tests represented the interstitial and surface water fractions as the binding strength was approximately the same as for a normal physisorption process.

Kieding et al. (2001) analyzed dilatometry from the thermodynamic perspective of freezing point depression of a pure solvent (water) in the presence of ions, microbial particles and macromolecules like EPS acting as solutes. They re-evaluated data from Smith and Vesilind (1995) taking into account the colligative properties of the solutes and found that results represented a continuous freezing of liquid water in a solution containing a substance of an average molecular weight of 179 g/ mol and formed super cooled water of 5.1 g water per gram of solids present. They concluded that there was no argument in favor of the theory presented by Smith and Vesilind for the existence of definitive pools of water. They also studied vapor pressure reduction and re-evaluated data from drying curve experiments performed by Kopp and Dichtl (2000) and found that it fit a model where the average molecular weight of the solute was found to be 89 g/ mol. Based on the above-mentioned fits of data to thermodynamic approaches, Keiding et al. (2001) suggested that description of water properties in sludge should be

approached from a more basic thermodynamic point of view. Bound water content, soluble EPS, conditioning of sludge are all factors that have been discussed above and shown to affect the dewaterability of sludge.

### ***The J-Vap Process and filter skin formation***

Chamber filter presses have undergone evolution over the last few decades, with the changes in the material of construction; mode of operation, and the introduction of mechanized plate movement and programmable logistics control making the unit more robust, versatile and operator friendly. The material of construction was changed from cast iron to polypropylene and rubber in the 1970s making the equipment lighter and easier to operate. The change in material from cast iron to rubber and polypropylene also led to the development of diaphragm filter plates capable of squeezing out more water from the cake after pressure dewatering of sludge in the filter press. The J-Vap is one of the latest modifications in the evolution of the filter press (Price and Shaw, 2001).

The J-Vap process was developed by the dewatering systems group of US Filter and patented in the year 2000. The process involves initial dewatering of sludge by pressure filtration as adopted in any normal chamber filter press followed by the application of vacuum and heated water in the temperature range of 65 to 80 oC that causes energy efficient drying of sludge after dewatering. The application of a vacuum results in a lowering of vapor pressure in the system and thereby, enabling the water present in the sludge matrix to evaporate at a much lower temperature supplied (by the heated water) than at atmospheric pressure. Since the cake density is not controlled by the pressure filtration step, faster cycle times can be adopted. The technique has been advertised to achieve up to 99% solids concentration. Some of the important advantages that the system has over the conventional chamber filter press are its capacity to achieve increased solids concentration, reduced feed cycle times, the production of class-A biosolids due to the application of elevated temperature and odor control (US filter, 2000; Price and Shaw, 2001).

It is known that a sludge subjected to different kinds of digestion processes will yield products with differing dewaterabilities (Novak et al., 2001; Mikkelsen and Keiding,

2002; Park et al., 2004). Conditioning has been shown to affect the rate of water release more than the total amount of free water present in the matrix (Kopp and Dichtl, 2001). The J-Vap process has been observed to suffer from its inability to achieve a high solids concentration upon conditioning the sludge with organic high molecular weight polymers. Filter skin formation by the polymer adjacent to the filter media on application of pressure and high temperature has been suspected to be the cause behind the problem (Mohammad A.M, 2003). The filter skin can be defined as a layer of highly compressed sludge particles adjacent to the filter media that resists the passage of water through the filter media, thereby reducing the attainable solids content on dewatering (Novak et al., 1999).

Sorensen et al. (1996) developed a numerical model that combined both the filtration and expression steps of pressure dewatering. Their model predicted that only a thin skin layer of compressed flocs formed at the filter cake/ medium interface was responsible for the resistance to dewatering and not the whole porous cake structure itself. Novak et al. (1999) studied the effects of skin formation by sludge particles during compression dewatering using the compression-permeation cell. They found that polymer addition resulted in the formation of highly compressible flocs and consequently in a lower dewatering rate. They also concluded that the increase in resistance to dewatering was due to the formation of the skin layer mentioned above. They suggested that conditioning affected only the rate of water removal and not the total removable water content itself. Further work on the presence of the filter skin has since been carried out by Zhao et al. (1998), Chang et al. (2002) and by Zhao and Bache (2002).

Zhao et al. (1998) studied the polymer residual concentrations in conditioned sludge supernatants and filtrates by means of size exclusion chromatography. The emergence of the residual coincided with the increase in specific resistance, when the polymer dose was increased beyond the optimal dose. Further, Zhao and co-workers also noted that the polymer residual in the supernatant was more than that present in the filtrate and therefore concluded that the excess polymer that was trapped on the filter media was responsible for an increase in the filter media resistance, which has traditionally been

considered to be negligible for SRF calculations. In later work on filter blinding, Zhao and Bache (2002) acknowledge that they have considered cake resistance and filter resistance as quasi-dependent entities and their results stating filter blinding to be dominated by colloid-polymer interaction is dependent on this assumption.

Chang et al (2002) proposed the use of NMR spectroscopy for the determination of polymer residual concentrations in wastewater matrices, as the NMR utilized unique properties of the polymer and offered sufficient accuracy and recovery of the polymer from the complex wastewater matrix. Polymer residuals in conditioned wastewater sludge have been measured by colloid and charge titration methods (Gher and Kalluri, 1983, Lee and Liu, 2000, Chang et al., 2002). Keenan et al. (1998) also suggested the use of size exclusion chromatography as a means of studying cationic, anionic and non-ionic polymer concentrations in conditioned supernatants of sludge. They found that the measured polymer residual increased sharply once the adsorption requirements by the colloids in sludge was satisfied. They also suggested that this method of determining polymer residuals could be used to accurately optimize polymer dosing for conditioning sludge.

Thus, it has been established that polymer residuals in conditioned supernatants can be used as an index for monitoring optimal polymer dosages and entrapment of polymer on filter media in the form of a polymeric filter skin layer. Charge titration with the potassium salt of Poly Vinyl Sulfuric Acid (PVSA) as the standardizing solution and ortho toluidine blue as the indicator has been used by different research groups to determine polymer residuals in sludge (Sakurada et al., 2004; Mikkelsen, 2003; Chang et al. 2002; and Kam and Gregory, 1999). The colloid titration method has been shown to have the second best recovery rate of sample after NMR in a comparative study of four different techniques to determine polymer residuals by Chang et al. (2002). Dentel (2004) found size exclusion chromatography to be unsuitable for wastewater sludge matrices due to the heterogeneous nature of compounds present. The charge titration method was found to be a simple, reproducible method for the determination of polymer residuals in sludge.

### ***Engineering significance***

The J-Vap process is capable of delivering very high solids content if the filter skin phenomenon is prevented from occurring. The skin is hypothesized to consist of highly compressed sludge particles and a layer of polymer that makes it difficult for further removal of water from the sludge. In the first part of this study, the efficiency of different polymers and the skin formation potential of different kinds of digested sludge were investigated. By studying the effect of temperature on skin formation and polymer entrapment, the J-Vap process can be optimized for each combination of sludge and polymer being used. This can result in a net improvement of the throughput being achieved by the equipment.

The second part of the study focused on the water distribution in sludge and the effect of digestion processes on the bound water content. The dewaterability of sludge by centrifuges was studied in relation to the bound water content and a relationship sought between the bound water content measured by dilatometry and dewatering achievable by centrifugation.

## **Reference**

- Barbusinski K. and H Koscielniak (1997) Activated Sludge Floc Structure during Aerobic Digestion. *Wat. Sci. Tech.* **36:11**, 107 – 114.
- Chang L., M.D. Burch, N.J. Griskowitz and S.K. Dentel (2002) NMR spectroscopy for determination of cationic polymer concentrations *Wat Res.* **36**, 2255 – 2264.
- Colin F. and S. Gazbar (1995) Distribution of Water in Sludges in Relation to their Mechanical Dewatering *Wat. Res.* **29**, 2000 – 2005.
- Dentel S.K. (2004) Personal communications.
- Erdinçler A., S. Koseogly and T. Onay (2001) The Role of blending in Polymer Conditioning of Waste Activated Sludge *Wat. Sci. Tech.* **44:6**, 63- 66.
- Gher R. and Kalluri R. (1983) The effects of short term storage and elevated temperatures on the flocculation activity of aqueous polymer solutions. *Wat. Poll. Res. Can.* **18**, 23 – 30.
- Guan J., R. Amal and T. D. Waite (2003) Effect of Floc Size on Biosolids Capillary Suction Time *Wat. Sci. Tech.* **47:12**, 255 – 260.
- Houghton J. I., and T. Stephenson (2002) Effect of Influent Organic Content on Digested Sludge Extracellular Polymer Content and Dewaterability. *Wat. Res.* **36**, 3620 – 3628.
- <http://www.usfilter.com/NR/rdonlyres/A7C538E2-E4F8-46E2-ACC7-3CA08F907EB5/0/jvap.pdf>
- Inman D.C. (2004) Comparative Studies of Alternative Anaerobic Digestion Techniques. Master's Thesis at Virginia Polytechnic Institute and State University.

- Jin B., B. Wilen and P. Lant (2003) A Comprehensive Insight into Floc Characteristics and their Impact on Compressibility and Settleability of Activated Sludge. *Chem. Engg. Journal* **95**, 221 – 234.
- Jin B., B. Wilen and P. Lant (2004) Impacts of Morphological, Physical and Chemical Properties of Sludge Flocs on Dewaterability of Activated Sludge. *Chem. Engg. Journal* **98**, 115 - 126.
- Kam S.K. and J. Gregory (1999) Charge Determination of Synthetic Cationic Polyelectrolytes by Colloid Titration. *Coll and Surf A : Physicochem Eng Aspects* **159**, 165 – 179.
- Keenan H.E., E.N. Papavasiliopoulos and D. H. Bache (1998) Measurement of Polymer Residuals in an Alum Sludge. *Wat. Res.* **32**, 3173 – 3176.
- Keiding K., L. Wybrandt and P.H. Nielsen (2001) Remember the water – a comment on EPS colligative properties. *Wat. Sci. Tech.* **43:6**, 17 – 23.
- Kopp J. and N. Dichtl (2000) Prediction of Full-scale Dewatering Results by Determining the Water Distribution of Sewage Sludges. *Wat. Sci. Tech.* **42:9**, 141 – 149.
- Kopp J. and N. Dichtl (2001) Influence of the Free Water Content on the Dewaterability of Sewage Sludges *Wat. Sci. Tech.* **44:10**, 177 – 183.
- Kopp J. and N. Dichtl (2001) Prediction of full-scale dewatering results of sewage sludges by the physical water distribution *Wat. Sci. Tech.* **43:11**, 135 – 143.
- Lee C.H. and J.C. Liu (2000) Enhanced sludge dewatering by dual polyelectrolytes conditioning. *Wat Res.* **34**, 4430 – 4436.
- Lee D.J. (1996) Interpretation of Bound Water Data Measured via Dilatometric Technique. *Wat. Res.* **30**, 2230 – 2232.

- Liao B.Q., D. G. Allen, I. G. Droppo, G. G. Leppard and S. N. Liss (2001) Surface Properties of Sludge and their Role in Bioflocculation and Settleability *Wat Res.* **35**, 339 – 350.
- Liss S. N., B.Q. Liao, I. G. Droppo, D. G. Allen and G G. Leppard (2002) Effect of Solids Retention Time on Floc Structure. *Wat. Sci. Tech.* **46: 1-2**, 431 – 438.
- Martinez F.O., Lema J. B., Mendez R., Lopez F. C., Gomez J. (2004) The Role of Exopolymeric Protein on the Settleability of Nitrifying Sludge. *Biores. Technol.* **94**, 43- 48.
- Metcalf and Eddy Inc. (2003) Wastewater engineering: Treatment and reuse. 4th Edition. McGraw-Hill, New York, NY.
- Mikkelsen L.H. (2003) Applications and Limitations of the Colloid Titration Method for Measuring Activated Sludge Surface Charges. *Wat. Res.* **37**, 2458 – 2466.
- Mohammad A.M. (2003) Personal communications.
- Novak J.T., C. D. Muller and S. N. Murthy (2001) Floc Structure and the Role of Cations *Wat. Sci. Tech.* **44:10**, 209 – 213.
- Novak J.T., C. Park and M.M. Abu-Orf (2004) Conditioning and Dewatering of Digested Waste Activated Sludges. *Jour Res. Sci. Tech.* **1**, 47 – 53.
- Novak J.T., M.L. Agerbaek, B.L. Sorensen, J.Aa. Hansen (1999) Conditioning, Filtering and Expressing Waste Activated Sludge. *Jour. Env. Engg.* **125**, 816 – 824.
- Novak, J.T., Sadler, M.E., and Murthy, S.N. (2003) Mechanisms of Floc Destruction During Anaerobic and Aerobic Digestion and The Effect on Conditioning and Dewatering of Biosolids. *Water Res.*, **37**, 3136 - 3144.

- Park C., Mohammad M. A., Novak J.T. (2004) Predicting the Digestibility of Waste Activated Sludges. *Wat Environ Res.* **(in Press)**.
- Price M. and Shaw D. (2001) How the Filter Press is Meeting Today's Demands. *Filt and Sep.* **38**, 26 – 28.
- Robinson J.K. (1989) The Role of Bound Water Content in Defining Sludge Dewatering Characteristics. Master's Thesis at Virginia Polytechnic Institute and State University.
- Sakurada O., Y. Kato, N. Kitto, K. Kameyama, T. Hattori and M. Hashiba (2004) Colloidal Titration of Aqueous Zirconium Solutions with Poly (Vinyl Sulfate) by Potentiometric Endpoint Detection Using a Toluidine Blue Selective Electrode. *Anal Sci.* **20**, 311 – 314.
- Smith J.K. and P. A. Vesilind (1995) Dilatometric Measurement of Bound Water in Wastewater Sludge. *Wat. Res.* **29**, 2621 – 2626.
- Sorensen P. B., P. Moldrup and J. AA. Hansen (1996) Filtration and Expression of Compressible Cakes. *Chem. Engg. Sci.* **51**, 967 – 979.
- Vaxelaire J. and P. Cezac (2004) Moisture Distribution in Sludge : a Review. *Wat. Res.* **38**, 2215 – 2230.
- Vesilind P. A. and C.C. Hsu (1997) Limits of Sludge Dewaterability *Wat. Sci. Tech.* **36:11**, 87 – 91.
- Wu C.C., C. Huang and D. J. Lee (1998) Bound Water Content and Water Binding Strength of Sludge Floccs. *Wat. Res.* **32**, 900 – 904.
- Yen P.S. and D.J. Lee (2001). Errors in Bound Water Measurement using Centrifugal Settling Method. *Wat. Res.* **35**, 4004 – 4009.

Zhao Y.Q. and D.H. Bache (2002) Polymer Impact on Filter Blinding during Alum Sludge Filtration. *Wat Res.* **36**, 3691 – 3698.

Zhao Y.Q., E.N. Papavasiliopoulos and D.H. Bache (1998) Clogging of Filter Medium by Excess Polymer During Alum Sludge Filtration. *Filt and Sep.* 947 – 950.

## II Manuscript 1

### ***Digestion Processes and their Effect on Filter skin formation***

Sangeetha Subramanian, Nitin Kumar, M. Abu-Orf Mohammad, John T. Novak

#### ***Abstract***

The J-Vap Process is designed to combine the dewatering and sludge drying operations into a single unit operation by modifying the chamber filter press to achieve high cake solids. The process involves the application of vacuum and circulation of heated water through chambers surrounding the partially dewatered cake. The reduced vapor pressure due to the vacuum increases the evaporation rate of water at a lower temperature than at atmospheric pressure. The interaction of the polymer used to condition the sludge with the filter media at the temperatures provided was suspected to result in the formation of a filter skin layer which impeded dewatering. This study was undertaken to determine if the resistance to filtration resulting from filter skin formation could be overcome by proper polymer selection. The results showed that polymer selection was important in determining the rate of dewatering and the time of pressure application played a more critical role in determining the extent of dewatering. Different kinds of digestion processes also resulted in end products with distinct dewatering properties. The polymer residual in the filtrate followed an inverse trend with the Specific Resistance to Filtration (SRF) and the maximum concentration of residual in the filtrate (corresponding with the lowest SRF) was measured at 50 °C after which the SRF increased with a corresponding decrease in the polymer residual measured in the filtrate. This could be an indication of the polymer adhering to the filter media, when the temperature increased beyond a threshold. An alternate hypothesis suggests the release of proteins and polysaccharides due to deflocculation with increase in temperature.

Key words: Filter skin formation, Cationic polymer, Colloid titration, Polymer residual

## ***Introduction***

It is known that solids handling comprises a significant fraction of operational costs for wastewater treatment plants. Operational costs in the range of 30 to 50% of the total expenses have been found to result from solids handling (Mikkelsen and Keiding, 2002). A decrease in the sludge volume by increased water removal may result in significant savings, independent of the final mode of disposal. In instances where land filling is adopted, increased cake solids will result in reduction of hauling costs and required landfill space. If incineration of solids is adopted, reducing the moisture content of the cake will increase the calorific value of the solids. If land disposal is used, transportation costs will come down by reducing the volume of sludge (La Heij et al., 1996; Mihoubi et al., 2003).

The J-Vap process combines two unit operations – filtration and drying into one unit, thereby reducing the number of units needed and associated capital costs, space and personnel requirements. This process has been advertised to be capable of achieving up to 99% solids, which would result in significant savings by reducing hauling and handling costs. The process involves a patented reduction chamber incorporated into the diaphragm filter press, where a vacuum can be applied along with the circulation of water heated to about 80 °C. The vacuum reduces the vapor pressure and consequently, the water in the sludge is capable of evaporating at a lower temperature. The vacuum removes the moisture in the vapor state and a sludge cake with very high solids content is achieved. Other benefits of the system include pathogen reduction due to the application of high temperatures, ability to select the desired solids content and a totally enclosed structure to reduce odor emissions (U.S. Filter, 2000).

It has been recognized that chemical conditioning and mechanical dewatering are two important, interdependent factors in the dewatering of wastewater sludge. Inorganic compounds such as lime, alum and ferric salts and synthetic organic polymers have been used to aid coagulation and flocculation of the biological particles found in wastewater sludge prior to dewatering (Lotito and Spinosa, 1990; Novak *et al.*, 1999; Zhao, 2003; Jin

*et al.*, 2004). Chemical conditioning of biological sludge has been found to significantly improve the dewaterability of the sludge. However, overdosing of conditioning agents has been shown to have a detrimental effect on dewatering (Zhao *et al.*, 1998; Wu *et al.*, 1997). Novak *et al.* (1998) determined that conditioning with iron led to the formation of more porous structured cake that could undergo a higher degree of compression dewatering. They suggested that iron conditioning resulted in reduced skin formation and the choice of polymers would play an important role in determining the extent of dewatering.

In this study, the pressure filtration mechanism and associated filter skin formation are investigated. The theoretical presence of such a filter skin or resistive layer responsible for most of the pressure drop was promulgated by the filtration model proposed by Sorenson *et al.* (1996). Novak *et al.* (1998) showed that a thin resistive layer comprised of compressed sludge flocs was formed by compression dewatering of sludge. Further work on the presence of the filter skin has since been carried out by Chang *et al.* (2002) and by Zhao and Bache (2002). There have been suggestions that skin formation can occur when the polymer dosages exceed the optimal dose. Both Sorensen *et al.* (1993) and Novak *et al.* (1999) worked with compression of waste activated sludge and reported the existence of higher than expected liquid pressures after prolonged dewatering due to the formation of the skin layer.

Figure II-1 explains the concept behind the measurements made by the compression dewatering cell. The liquid pressure transducer measures the pressure exerted by the liquid phase of the sludge. As long as free water is present in the sludge matrix, the liquid pressure remains a constant, representing the filtration phase of dewatering. The figure shows an increase in cake height and a decrease of the liquid suspension height with time due to consolidation of sludge particles as the liquid is filtered. Once the free water has been removed and the water fractions associated with the solids are expressed, and the liquid pressure decreases. This pressure decrease is accompanied by the compression of the sludge cake, resulting in a decrease of cake height seen inside the compression cell.

Though the relative liquid pressure is shown to approach zero with time, it may plateau at a slightly higher pressure, as all the liquid cannot be removed by application of pressure. Further work by Couturier *et al.* (2003) on liquid pressure showed that hydraulic pressure measured by the traditional compression cells might not be a true representation of the liquid pressure, since air trapped between the piston and the sludge could permeate the cakes, resulting in an incorrect value of liquid pressure profiles. They suggested the application of a low vacuum pressure to remove the air trapped before starting data acquisition.

The contribution of polymers towards filter skin formation was promulgated by Zhao *et al.* (1998). Zhao *et al.* (1998) studied the polymer residual concentrations in conditioned sludge supernatants and filtrates by means of size exclusion chromatography. The emergence of residual polymer coincided with an increase in specific resistance as the polymer dose increased beyond the optimal dose. The optimal dose for conditioning agents has been defined as either the amount of chemicals that needs to be added to yield a distinct maximum dewatering rate or as the lowest amount of chemicals that needs to be added to result in an acceptable dewatering performance (Christensen *et al.*, 1993). Further, Zhao and co-workers also noted that the polymer residual concentration in the supernatant was greater than that present in the filtrate and therefore concluded that excess polymer trapped on the filter media was responsible for an increase in the filter media resistance, which has traditionally been considered negligible for SRF calculations. In a later work on filter blinding, Zhao and Bache (2002) acknowledge that they considered cake resistance and filter resistance as quasi-dependent entities and their results, stating that filter blinding is dominated by colloid-polymer interaction, are dependent on this assumption.

Polymer residuals in conditioned wastewater sludge have been measured by colloid and charge titration methods (Gher and Kalluri, 1983, Lee and Liu, 2000, Chang *et al.*, 2002). Chang *et al.* (2002) proposed the use of NMR spectroscopy for the determination of polymer residual concentrations in wastewater matrices, as the NMR method utilized

unique properties of the polymer and offered sufficient accuracy and recovery of the polymer from the complex wastewater matrix. Zhao and coworkers used size exclusion chromatography to determine polymer residual concentrations in conditioned alum sludge. Since NMR and SEC are comparatively more complicated techniques than colloid titration, it was decided to adopt the colloid titration method for the purpose of this study.

### ***Research Objectives***

Though a considerable amount of research has been conducted separately on filter blinding during compression dewatering and conditioning with cationic polymers of sludge, data need to be obtained on the combined effects of polymer dosing and pressure dewatering, which is more representative of actual field processes. The research objectives of this study were:

1. examination of different polymers to determine if they affect the rate of dewatering or the extent of dewatering,
2. examination of dewatering properties of different kinds of sludge with regard to the fastest dewatering polymer,
3. investigation of polymer residuals in optimally dosed samples and filtrates to determine if polymer adheres to the filter media, and
4. to determine the relationship between temperature and the amount of polymer trapped in the sludge cake for each kind of sludge.

### ***Materials and Methods***

Primary and waste activated sludges were obtained from the Pepper's Ferry wastewater treatment facility and stored at 4 °C until used. The two sludges were mixed in a 1:1 ratio by volume and the optimal dose of polymer was determined using the Capillary Suction Time (CST) test according to method 2710G of Standard Methods (APHA, 1995). Total and volatile solids were determined for the sample according to Standard Methods (APHA, 1995). Six different polymers (five liquid phase high molecular weight cationic polymers - CPB 1220, CP 2235, CP 2555, CP 2580, CP 3100 and a solid based polyacrylamide polymer - SH 650) obtained from Clear Water Inc. were used in the study. A one percentage solution of the polymer was added to 100 ml of sludge and

sheared in a low speed blender for 10 seconds to ensure uniform dispersion when determining dose response using CST.

Dewatering properties of the different polymers were also estimated by compression cell dewatering. The compression cell has been used by many investigators to determine dewaterability and compressibility of sludge (La Heij *et al.* 1996; Novak *et al.* 1998; Couturier *et al.* 2003). Novak *et al.* (1999) and Zhao and Bache (2002) showed that the volume of sludge dewatered played an important role in determining the final cake solids content. In this study, 200 ml of sludge was treated at the optimal dose of each polymer and dewatered in the compression cell to characterize dewatering under pressure. In the first phase of the study determining the response of the sludge to different conditioning agents, the sample was conditioned at the respective optimal dosage for each kind of polymer and dewatered in the compression cell starting at a liquid pressure of 20 psi to a final liquid pressure of 4 psi. In the second phase of the study investigating the effect of conditioning agents on the rate of dewatering and the extent of dewatering, the cell was operated at an initial liquid pressure of 20 psi to final liquid pressures of 10, 8, 6 or 4 psi. The SRF of the sludge conditioned with different polymers was also monitored and correlated to the compression cell tests. The time taken for compression dewatering was studied in relation to the liquid pressure

### **Effects of Digestion Processes on Dewaterability**

In this phase of the investigation, digestion effects on dewatering were studied. Primary and waste activated sludge from the Blue Plains facility, DC Water and Sewer Authority (DC WASA) were digested in lab pilot scale reactors. Table II-1 describes the digestion methods and conditions followed in the study.

The above-mentioned sludges were tested with the polymer that provided the fastest rate of dewatering to determine the effects of digestion on dewatering. Polymer requirements and CST of the different sludges at optimal dosage was compared. The SRF of each sludge conditioned at the optimal dose was determined, as was the compression test response starting at 20 psi liquid pressure to a final liquid pressure of 2 psi.

## **Effect of Temperature on Dewatering and Polymer Residual**

Since the J-Vap process involves the application of vacuum and high temperature, the effect of temperature on polymer interaction with the filter media was studied. In order to determine the presence of the filter skin, polymer residuals were monitored in the supernatants of the optimally conditioned samples as well the filtrate obtained from the SRF tests. The difference between the supernatant and the filtrate concentrations was assumed to represent the quantity of polymer being retained on or within the filter media along with the sludge.

Optimally conditioned sludge was heated in a temperature controlled water bath and SRF performed at 10 °C increments of temperature from 20 to 70 °C. The polymer residuals were monitored in the optimally dosed sample, as well as in the filtrate of each of the SRF tests over the temperature range to determine the effect of temperature on the polymer residual. The charge titration method suggested by Gher and Kalluri (1982) was adopted to monitor the polymer residual concentrations in the supernatant and the filtrate. A standard solution of Poly Vinyl Sulfuric Acid Potassium (PVSAK) was used to neutralize the positive charges present in solution due to the cationic polymer. Toluidine Blue O (TBO) was used as the indicator solution, with the end point being the color change from blue to light purple.

## ***Results and Discussion***

Of the six polymers tested, the efficiency (lowest compression time to achieve a liquid pressure of 4 psi) of the polymers decreased in the order SH 650, CP 2580, CPB 1220, CP 2555, CP 3100 and CP 2235 (Figure II-2). The dose curve tests showed that both the CST and polymer requirement at the optimal dose were also in the order mentioned above for the sludge tested. The CST and the polymer requirements are shown in Figure II-3 and Figure II-4 respectively. Figure II-3 shows the CST of the sludge after conditioning with the different polymers at their optimal dosage. It is evident that the polymer SH 650 produced a sludge with a much lower final CST than the rest of the polymers tested.

Figure II-4 shows the polymer requirements at optimal dosage in terms of kg of polymer required per ton of Dry Solids (kg/ ton DS) processed. This determination becomes important from a cost perspective because the polymer requirement is much lower for the polymer SH 650 as compared to the others. Dentel (2004) notes that solid phase polymers can contain 90 to 100% active polymer by weight while liquid phase emulsion polymers generally contain 30 to 45% active compounds by weight. Since SH 650 is a solid phase polymer, it is possible that it has a greater active fraction as compared to other liquid phase polymers. However, even if the dose were diluted to make it equivalent to the liquid polymers, it would still require the lowest dose.

Figure II-5 shows the linear positive correlation between the CST and polymer requirements at optimal dosage. A corresponding increase in the polymer requirements was seen with increase in the minimum CST at optimal dosage. This might reflect the differential binding capacity of the polymers to the colloidal fraction present in the sludge. Figure II-6 shows the SRF of the sludge conditioned at the optimal dose with different polymers. The trend observed is nearly the same as that observed for CST versus polymer shown in Figure II-2. Figure II-7 provides a comparison of the SRF and the time taken by different polymers to dewater the sludge to 4 psi liquid pressure in the compression cell. A nearly linear correlation is observed and as the time taken to dewater to 4 psi increases, the SRF at optimal dose also increases.

In their review of literature on CST measurements representing filterability, Meeten and Smeulders (1995) define the CST as a measure of the time taken by free water in the sludge to permeate through a fixed distance on Whatman 17 filter paper. Since the compression cell test is a profile of the liquid pressure against time, and represents the removal of the interstitial and surface water, it was not known if the CST would accurately predict the outcome of this test. On analysis, it was seen that the CST had a good correlation with the compression cell tests. Further, the SRF was also correlated to the compression cell tests and is shown in Figure II-7. Though CST data is not shown in Figure II-7, as the time taken to dewater to 4 psi liquid pressure increases, both CST and SRF increase. This suggests that the distribution of water in the free phase, intra-floc and

intra-cellular phases might be proportional and a good estimate of the compressibility of sludge can be obtained from a test measuring any of these phases of water.

For the sake of this study, the efficiency of compression dewatering has been described by the time taken for the different samples to reach 4 psi liquid pressure and the final cake solid content obtained. Figure II-8 shows the time taken and the solids content obtained from the sample conditioned with different polymers and dewatered to liquid pressures of 10, 8, 6 and 4 psi. It should be noted that at 4 psi, the cake is at its driest. It can be seen that the solids content increases linearly with increasing time and then appears to plateau after reaching about 25% solids in each case. The data point corresponding to the polymer CP 2235 in the 8 psi experiment showed very low solids concentration despite being dewatered for the longest time in the series. This is thought to be an erroneous data point and has been circled as an outlier. The order in which the different polymers yielded cakes dewatered to specific liquid pressures was conserved in the four experiments performed. We can therefore conclude that the time of compression pressure application plays a more important role in determining the final cake solids content than the polymer used. If a fast water releasing polymer like SH 650 is used, the time taken to reach a designated liquid pressure is shortened, but this results in lower cake solids content. The results clearly show that the dewatering function is more dependent on the kind of polymer used and the compression time rather than on the liquid pressure at which compression is terminated. Thus, a faster dewatering polymer need not necessarily be the polymer of choice if the final cake solids is of importance. Further, it is also observed that the sequence of polymers has been conserved in nearly all the experiments performed. We can deduce that the polymers affect the rate of dewatering more than the extent of dewatering, which is dependent on the time of pressure application.

Mihoubi *et al.* (2003) proved the existence of an empirical relationship between SRF and the consolidation ratio during compression dewatering and solids concentration of sludge used. La Heij *et al.* (1994) and Novak *et al.* (1999) showed that increasing the mechanical pressure during compression dewatering caused only a minor decrease in the

void ratio of the sludge. Zhao and Bache (2002) analyzed the effects of applied pressure, time and polymer doses on compression dewatering of alum sludge. Their results showed that the solids content increased with time, while the polymer dose enhanced water release, but not the total solids concentration achieved. They also found that the loading rate had a significant impact on increasing the final solids content. All the above-mentioned experiments and results concur with this study in that the time of pressure application is an important factor in determining the final cake solids concentration as compared to the polymer selected for conditioning.

### **Results from digestion experiments**

Digested sludges from anaerobic thermophilic, anaerobic mesophilic, thermophilic followed by aerobic digestion and mesophilic followed by aerobic digestion were conditioned with SH 650, as it was required in the smallest quantities and gave a lower CST, SRF and a fast dewatering profile as compared to the other polymers. The sludge in the thermo-aero reactors as mentioned in Table II-1 was digested anaerobically at 55 °C for 20 days followed by aerobic digestion at 31 °C for 6 days. It was observed that this sludge exhibited the best dewatering characteristics in terms of CST, polymer requirements, SRF and time required to dewater to 2 psi liquid pressure. Figure II-9 and Figure II-10 show the minimum CST and amount of SH 650 polymer required at optimal dosage respectively for each of these sludges. Thermo-aero and Meso-aero sludges have about the same polymer requirements. Since the CST of the Thermo-aero sludge is less than that of the Meso-aero sludge, it appears that the best overall dewatering characteristics are exhibited by the Thermo-aero sludge. Meso sludge has high polymer requirements and a longer CST and is therefore categorized as the poorly dewatering sludge of those tested.

The SRF of the digested sludges conditioned with polymer at the optimal dose is shown in Figure II-11. The SRF tests are in concordance with the CST test and suggest that the Thermo-aero sludge is the best dewatering sludge and Meso sludge the worst dewatering one amongst those studied. Figure II-12 shows the data collected from the compression dewatering of the different sludges conditioned at their optimal doses with polymer SH

650. The Meso sludge took the longest time to dewater to 4 psi, while the Thermo-aero sludge dewatered the fastest. The sequentially digested sludges dewatered faster than the single stage anaerobically digested sludges. The data from figures 9, 10, 11 and 12 establish that different digestion procedures result in producing sludge with distinct dewatering characteristics. In this study, it is seen that the thermo – aero sludge was the best in terms of dewatering and compression followed by meso – aero, thermo and meso sludge.

A comparison of Figure II-5 and Figure II-13 show a reversed trend in polymer requirements and the CST at optimal dosage. In Figure II-5, the CST was seen to increase linearly with increasing polymer requirements and it was hypothesized that it might be due to the differential binding capacity of each of the polymers to the colloidal fraction present in sludge. In the case of digested sludges depicted in Figure II-13, the CST is seen to decrease as the optimal polymer dosage requirements increase, although the correlation is not strong. Since only one polymer, SH 650 is used, this suggests that different amounts of biocolloids are released into solution upon subjecting the sludge to different digestion conditions. Novak et al. (2004) suggest that biocolloids in different kinds of digested sludge might be different from one another and could play a vital role in determining polymer requirements. In this study, it is suggested that the increased polymer requirements might be a reflection of the amount of biopolymer present in solution phase. More research on water distribution and biopolymer estimation in the presence of cationic polymers are needed to clarify these results.

Since the J-Vap process is operated at temperatures at nearly 80 oC, the interaction of the polymer with the sludge and the filter media was studied over a range of temperatures from 20 degrees to 70 degrees. In order to evaluate the effect of temperature on filter skin formation, colloid titrations were performed to determine polymer residuals present in optimally dosed sludge supernatants and filtrates obtained by performing SRF. Zhao et al. (2003) have shown that an increase in SRF was accompanied by a decrease in polymer residuals coming out with the filtrate. They showed the possible existence of a relationship between polymer dosage and an increase in SRF. A similar approach was

undertaken here and the amount of polymer that was getting trapped on the filter media was calculated using a simple mass balance between the amounts of polymer residual measured in the sludge supernatant versus the amount of polymer that was in the filtrate.

On performing the experiment at different temperatures, it was seen that the SRF initially decreased with temperature and then increased above 50 °C. The trend was for all the sludges tested. Figure II-14 through Figure II-17 show the variation of SRF and polymer residuals observed in the filtrates of different digested sludges with temperature. A comparison between the different figures reveals that the thermo-aero sludge had the lowest SRF and highest polymer residuals coming through. Thermophilic digested sludge had a higher SRF as compared to sludge subjected to a subsequent aerobic phase of digestion. The mesophilic digested sludge started off with a higher SRF than the Meso-aero digested sludge, but with increasing temperature, the meso-aero sludge was observed to have greater SRF than the mesophilic digested sludge.

A study of polymer residual concentrations in the filtrate and the variation of SRF over a temperature range of 20 to 70 °C revealed that the SRF initially fell with temperature and then increased again. This corresponded to an increase of polymer residuals being observed in the filtrate followed by a decrease as soon as the SRF started to climb up. These results correspond well with those observed by Zhao et al. (2003) regarding the entrapment of polymer relative to an increase in SRF.

A different perspective to the interpretation of the result can show that EPS release increases at higher temperatures and might be responsible for both the increase in SRF and the corresponding decrease in polymer residuals seen in the filtrate. Investigations by Novak et al. (2003) and Skidmore and Reyes (2004) have shown EPS concentrations increase linearly with temperature after approximately 45 °C. In this investigation, the SRF was seen to increase beyond 50 °C. This seems to be a more likely explanation for the increase in SRF and the decrease of polymer residuals in the filtrate, as the released bio-polymers will exert a demand on the polymer residual present in the conditioned sludge. However, since a method to determine protein concentration in the presence of

cationic polymers has not yet been devised, this hypothesis has not been tested as a part of the study. Alternatively, tagging of polymers with a dye to determine its presence in the sludge matrix or on the filter media can also be used to establish if the polymer is interacting with the filter media or with biocolloids present in the sludge.

It appears that the operators of the J-Vap system may want to consider operating at 50 °C to maximize dewatering. Once maximum filtration and expression of water has taken place under the least resistance conditions, a drier cake can be attained, by raising the temperature further, thereby increasing evaporation of the moisture held within the cake.

## **Conclusions**

The main conclusions that can be draw from this study are

1. The kind of polymer chosen to treat a particular sludge will influence the rate of dewatering rather than the extent of dewatering. Further, the polymer's efficiency on dewatering different kinds of digested sludge will vary, as digestion processes affect the concentration of the biopolymer and bio-colloids in solution. Specifically, it was seen that SH 650 performed the best amongst the six polymers tested and the thermo-aerobically digested sludge showed the best dewatering characteristics among the digested sludges.
2. Though, CST, SRF and compression cell dewatering measure rates of dewatering of different fractions of water present in the sludge, a good correlation was seen to exist between them. Sludges that dewatered well were characterized by a low CST and SRF and a rapidly falling dewatering profile, and vice-versa.
3. An analysis of filter skin formation showed that the polymer residuals coming through the filter media on vacuum filtration increased with increase in temperature up to 50 °C and then declined. This was accompanied by a simultaneous decrease in SRF till the point where the residual concentration peaked (50 °C), followed by an increase in SRF. The lowest SRF and highest polymer residual concentrations were observed at 50 °C for the sludges experimented in this study.
4. Digestion processes significantly affect the dewaterability of sludge and the strength of polymer-sludge interactions. It is important that the polymer

interaction with the filter media be studied for each kind of digested sludge before any calls are made about the polymer's efficacy for dewatering/ drying by the J-VAP process.

Further research needs to be done in terms of establishing the mass balance between the polymer residuals in the conditioned sludge and filtrate. Also, the effect of pressure on polymeric filter skin formation needs to be studied in conjunction with the effect of temperature to give a realistic solution to the problem under consideration. The effect of cellular disruption on increase of temperature needs to be studied more carefully. For processes employing elevated temperatures such as the J-Vap, polymer demand may need to be optimized for operational temperatures and not at ambient temperature.

### **Acknowledgement**

The work was sponsored by US filter dewatering systems research group.

## Reference

<http://www.usfilter.com/NR/rdonlyres/A7C538E2-E4F8-46E2-ACC7-3CA08F907EB5/0/jvap.pdf>

Chang, Lin-Li, Martha D. Burch, Nancy J. Griskowitz and Steven K. Dentel (2002) NMR spectroscopy for determination of cationic polymer concentrations *Wat. Research* **36**, 2255 – 2264.

Christensen J.R., Sorensen P.B., Christensen G.L. and Hansen J.A. (1993) Mechanisms for overdosing in sludge conditioning *Jour. Environ. Engg* **119**, 159 – 171.

American Public Health Association; American Water Works Association; and Water Environment Federation (1995) Standard Methods for the Examination of Water and Wastewater. 19th Ed., Washington, D.C.

Dentel S.K. (2004) Personal communications.

Gher R. and Kalluri R. (1983) The effects of short term storage and elevated temperatures on the flocculation activity of aqueous polymer solutions *Wat. Poll. Res. Can.* **18**, 23 – 30.

Jin B., Britt-Marie Wilen and Paul Lant (2004) Impacts of morphological, physical and chemical properties of sludge flocs on dewaterability of activated sludge *Chem. Engg. Jour.* **98**, 115 – 126.

Keenan H.E., Eleftheios N. Papavasilopoulos and David H. Bache (1998) Measurement of polymer residuals in an alum sludge *Wat. Residues* **32**, 3173 – 3176.

La Heij E.J., P.J. A. M. Kerkhof, A.J.M. Herwijn, W.J.Coumans (1996) Fundamental aspects of sludge filtration and expression *Wat Residues* **3**, 697 – 703.

Lee C.H. and J.C. Liu (2000) Enhanced sludge dewatering by dual polyelectrolytes conditioning *Wat Residues* **34**, 4430 – 4436.

Lotito V. and L. Spinosa (1990) Sewage sludge conditioning by polyelectrolytes *Filt. and Sep.* **27**, 122 – 125.

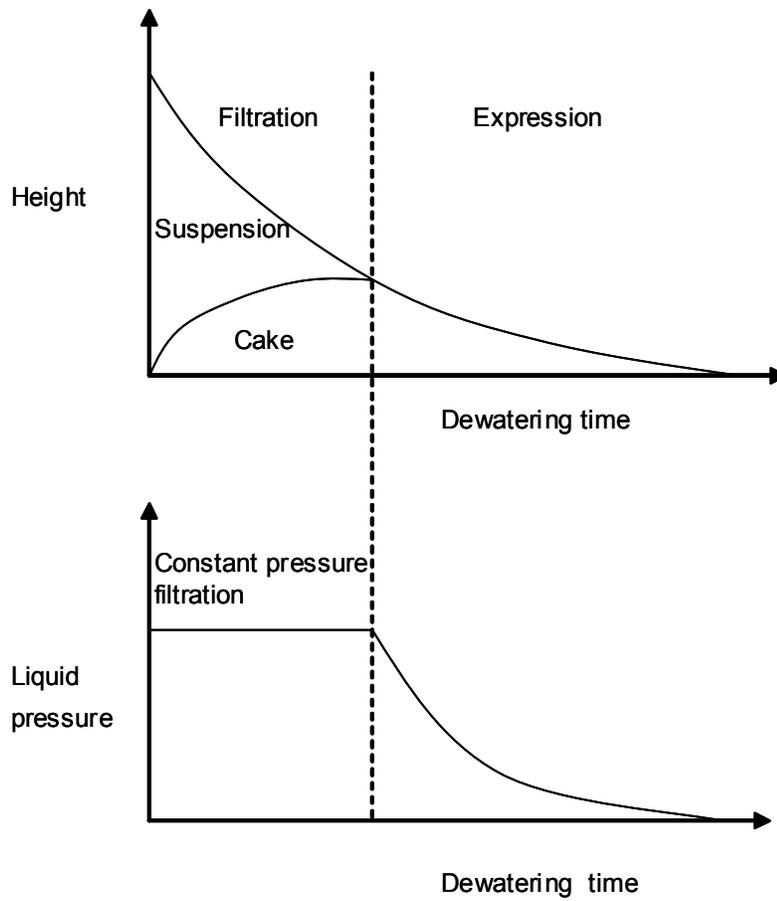
Meeten G.H. and J.B.A.F. Smeulders (1995) Interpretation of Filterability measured by the Capillary Suction Time Method *Chem. Eng. Sci.* **50**, 1273 – 1279.

Mihoubi D., J. Vaxelaire, F. Zagrouba and A. Bellagi (2003) Mechanical dewatering of suspension *Desal.* **158**, 259 – 265.

- Mikkelsen L.H. and K. Keiding (2002), Physico-chemical characteristics of full scale sewage sludges with implications to dewatering. *Wat. Research* **36**, 2451–2462
- Novak J.T., Mikkell L. Agerbaek, Birgette L. Sorensen and Jens Aa. Hansen (1999) Conditioning, filtering and expressing waste activated sludge *Jour. of Environ. Engg* **125**, 816 – 824.
- Sorensen P.B., Per Moldrup and Jens AA. Hansen (1996) Filtration and expression of compressible cakes *Chem. Engg. Sci.* **51**, 967 – 979.
- Tchobanoglous, G., Franklin L. Burton and David H. Stensel (2003). Wastewater Engineering – Treatment and reuse. 4<sup>th</sup> Ed. Tata McGraw-Hill, New Delhi.
- Wu C.C., Chihpin Huang and D.J.Lee (1997) Effects of polymer dosage on alum sludge dewatering characteristics and physical properties *Coll and Surf A: Physicochem and Engg Asp.* **122**, 89 – 96.
- Zhao Y.Q. (2003) Correlations between floc physical properties and optimum polymer dosage in alum sludge conditioning and dewatering *Chem Engg Jour* **92**, 227 – 235.
- Zhao Y.Q. and D.H. Bache (2002) Integrated effects of applied pressure, time, and polymer doses on alum sludge dewatering behavior *Waste Management* **22**, 813 – 819.
- Zhao Y.Q., E.N. Papavasiliopoulos and D.H. Bache (1998) Clogging of filter medium by excess polymer during alum sludge filtration *Filt and Sep.* **35**, 947 – 950.

**Table II-1: Digestion techniques and SRT maintained**

<b>Digestion technique</b>	<b>Temperature (°C)</b>	<b>SRT (days)</b>	<b>Referred to as</b>
Mesophilic anaerobic digestion	35	20	Meso
Mesophilic anaerobic – aerobic digestion	35+31	20+6	Meso aero
Thermophilic anaerobic digestion	55	10	Thermo
Thermophilic anaerobic – aerobic digestion	55+31	10+6	Thermo aero



**Figure II-1: Evolution of cake height and liquid pressure during constant pressure dewatering of sludge**

Ref: Novak *et al.* (1999)

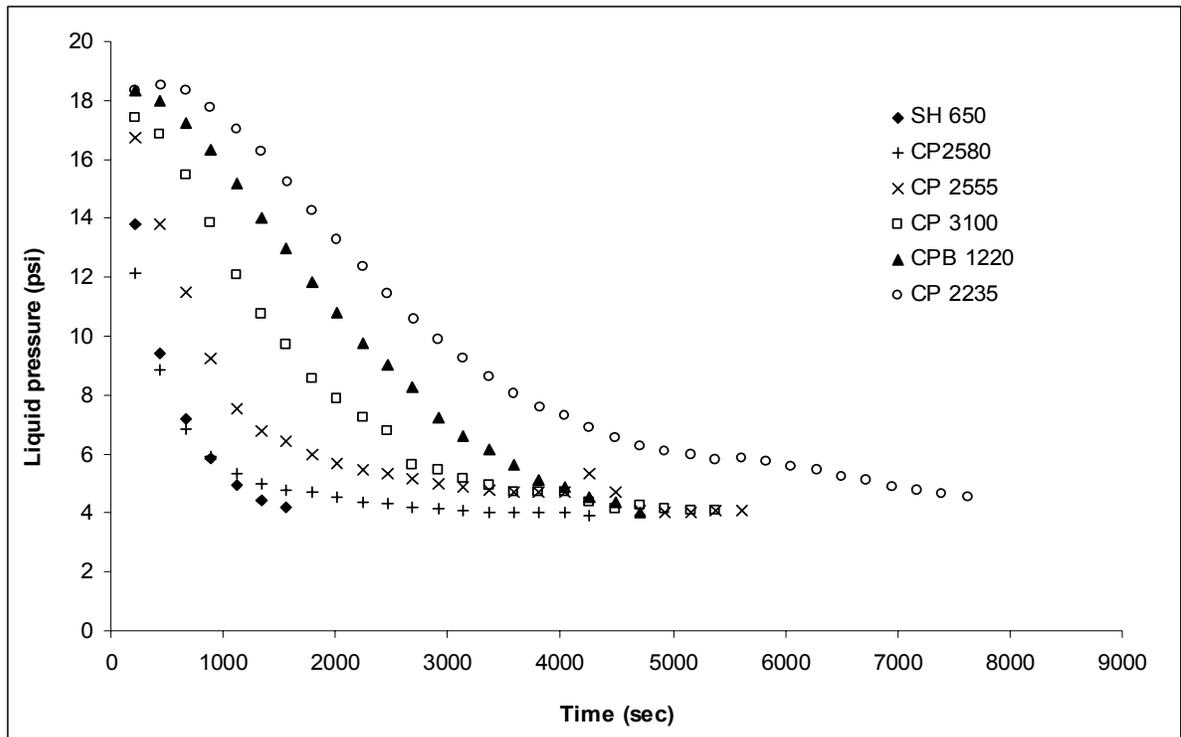
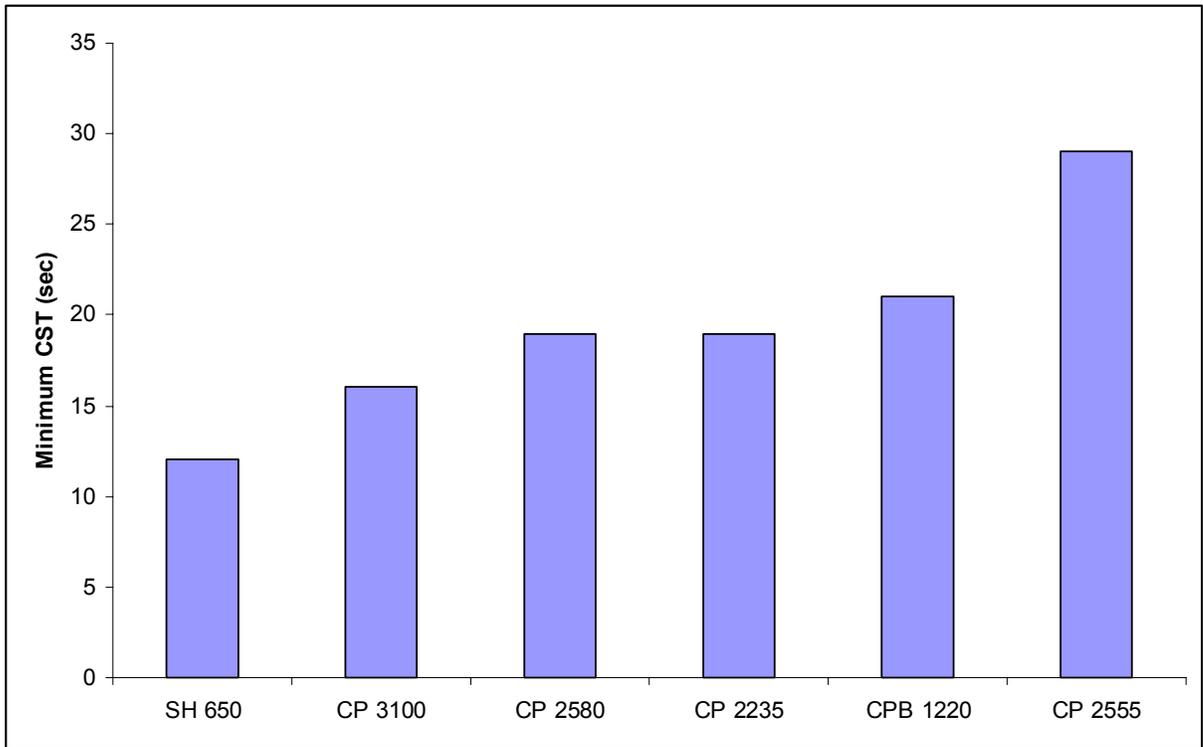
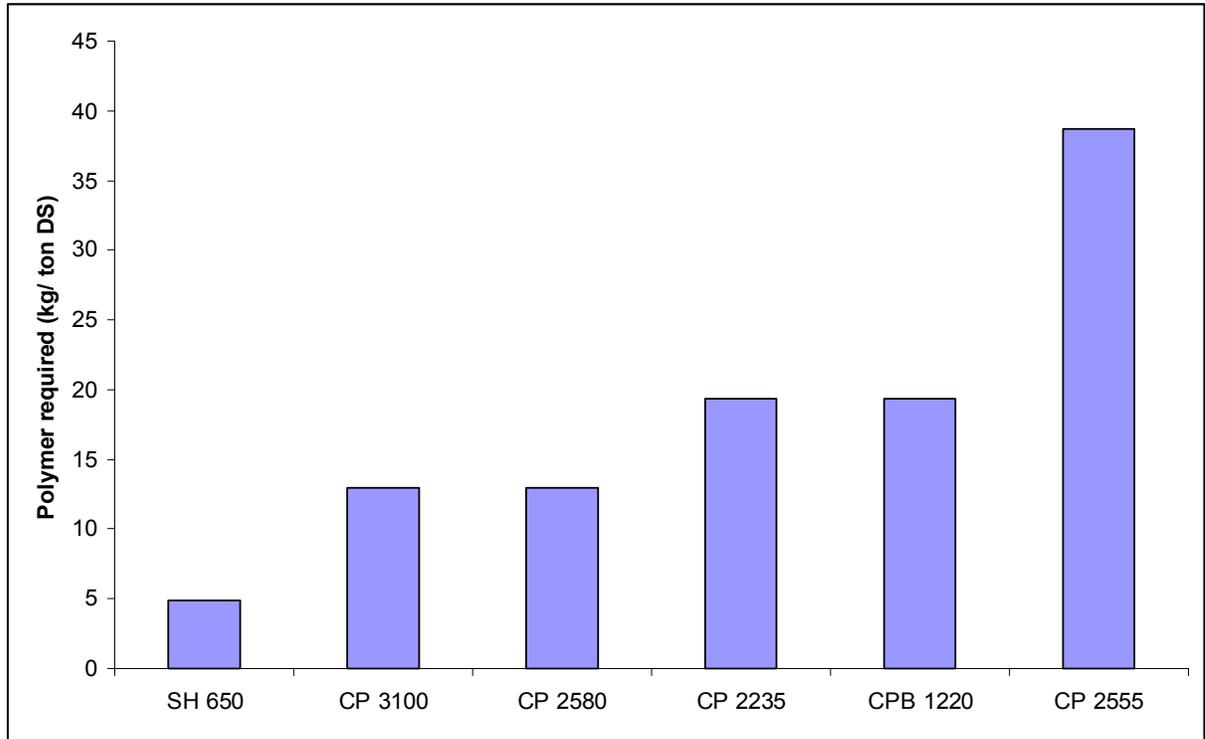


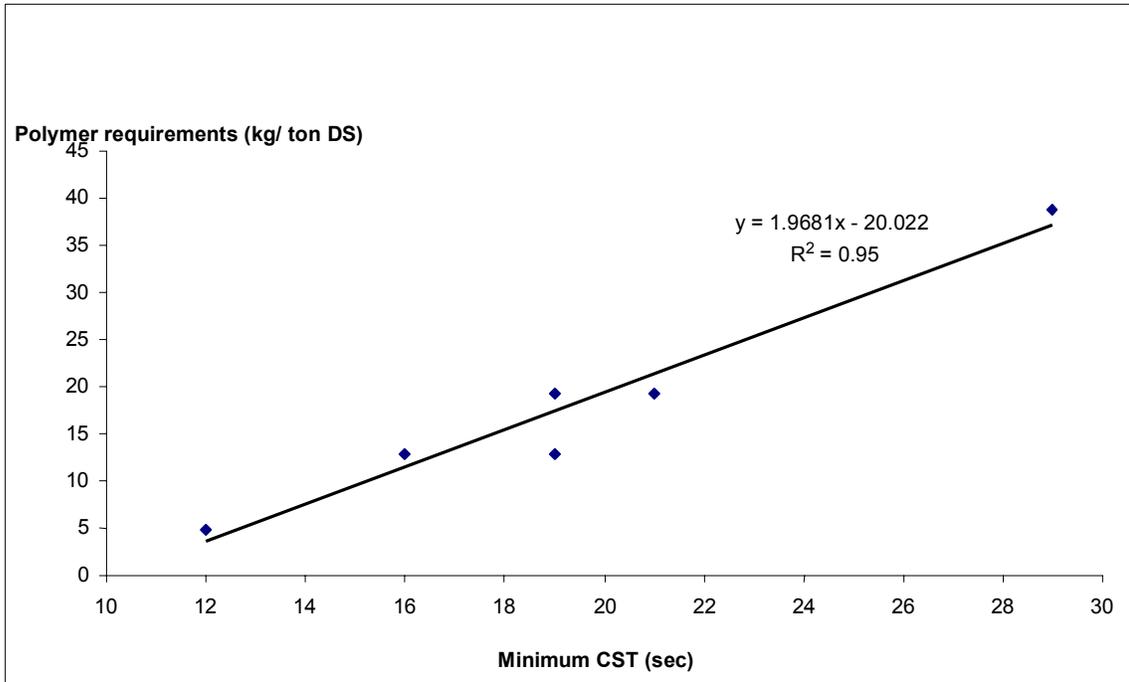
Figure II-2: Dewatering profiles of Pepper's Ferry sludge conditioned with different polymers



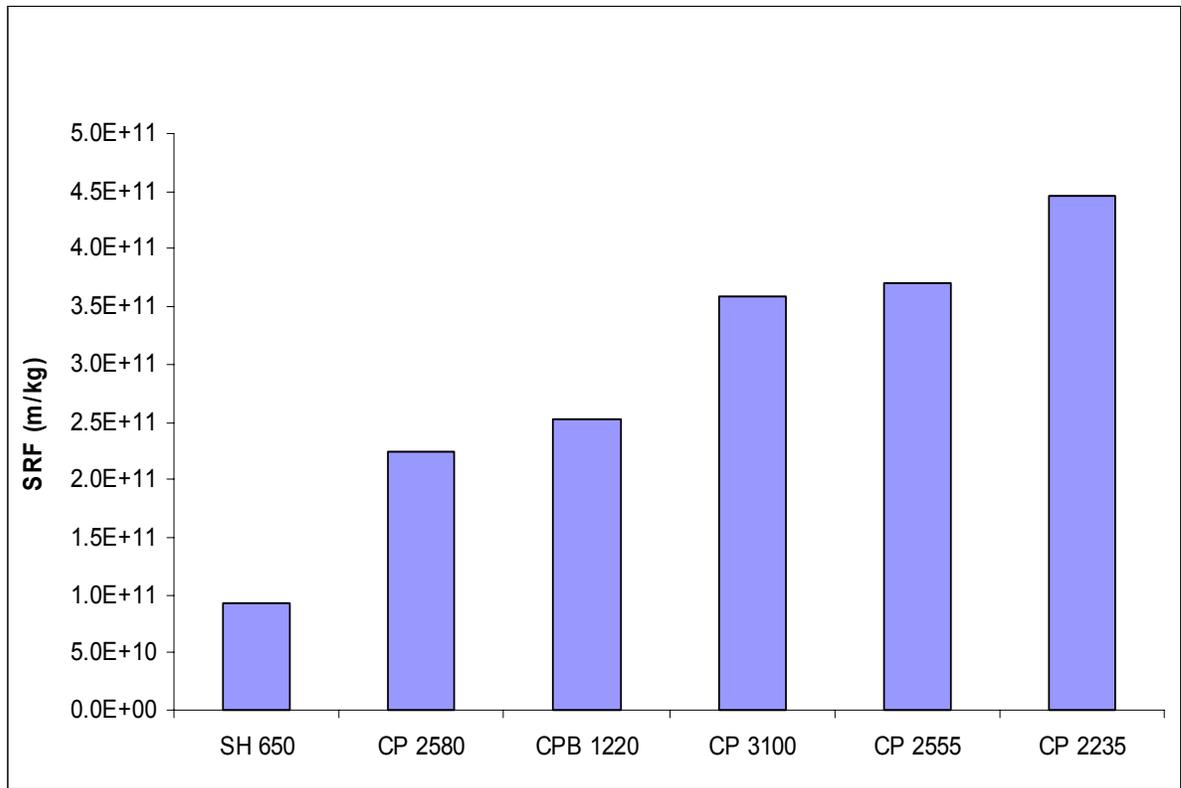
**Figure II-3: CST at optimal dose for Pepper’s Ferry sludge using different polymers**



**Figure II-4: Optimal dose polymer requirements for Pepper's Ferry sludge**



**Figure II-5: Polymer requirements versus minimum CST at optimal dosage for Pepper's Ferry sludge**



**Figure II-6: SRF of Pepper's Ferry sludge conditioned with different polymers**

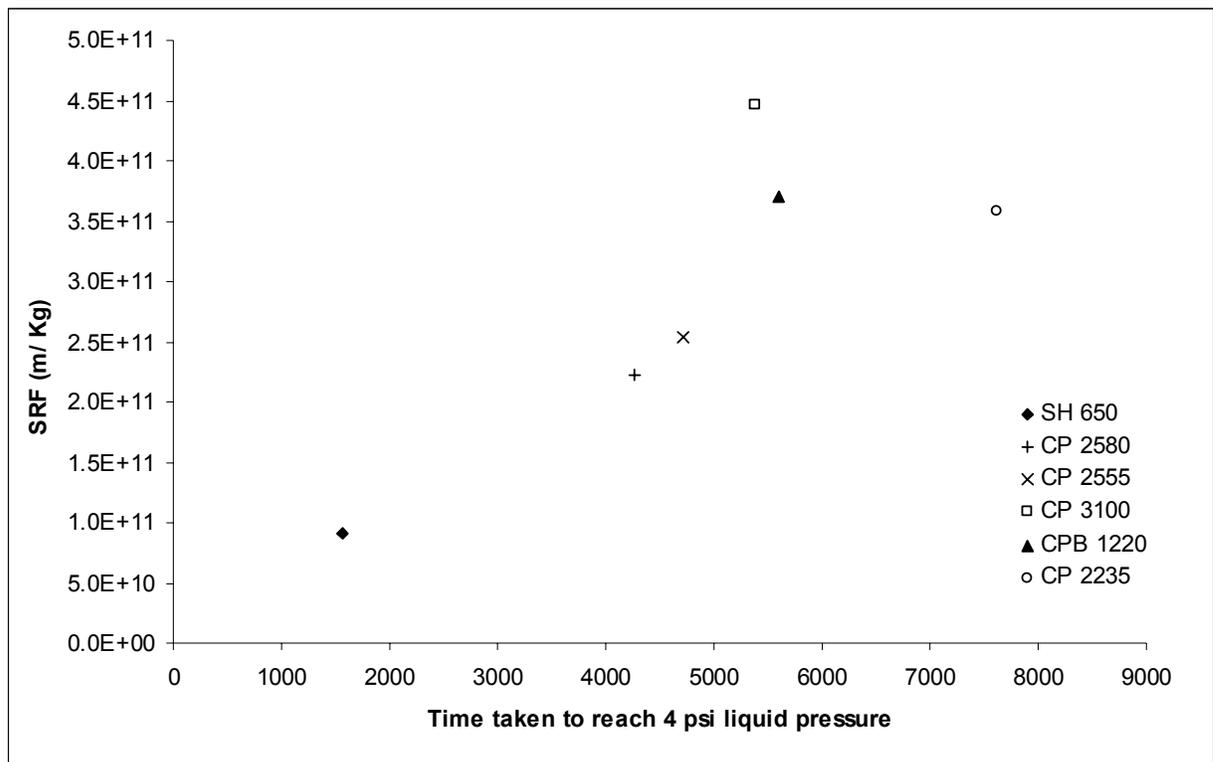


Figure II-7: Analysis of SRF and compression time required to dewater to 4 psi liquid pressure

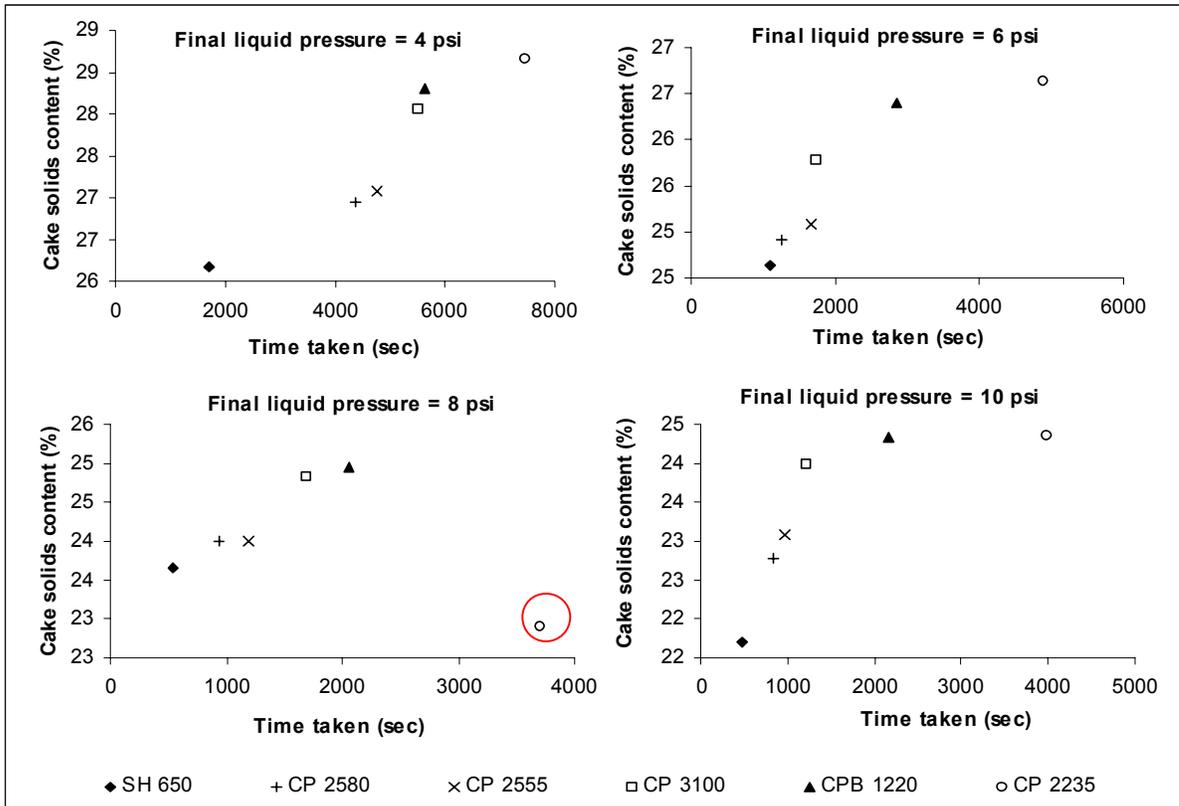
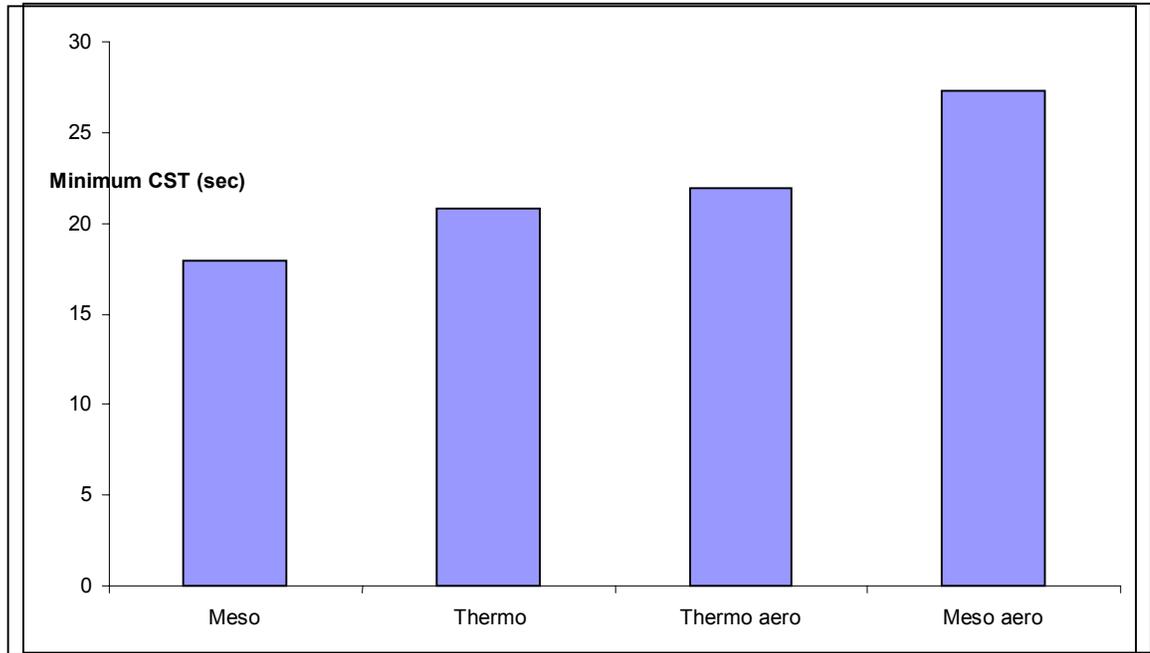
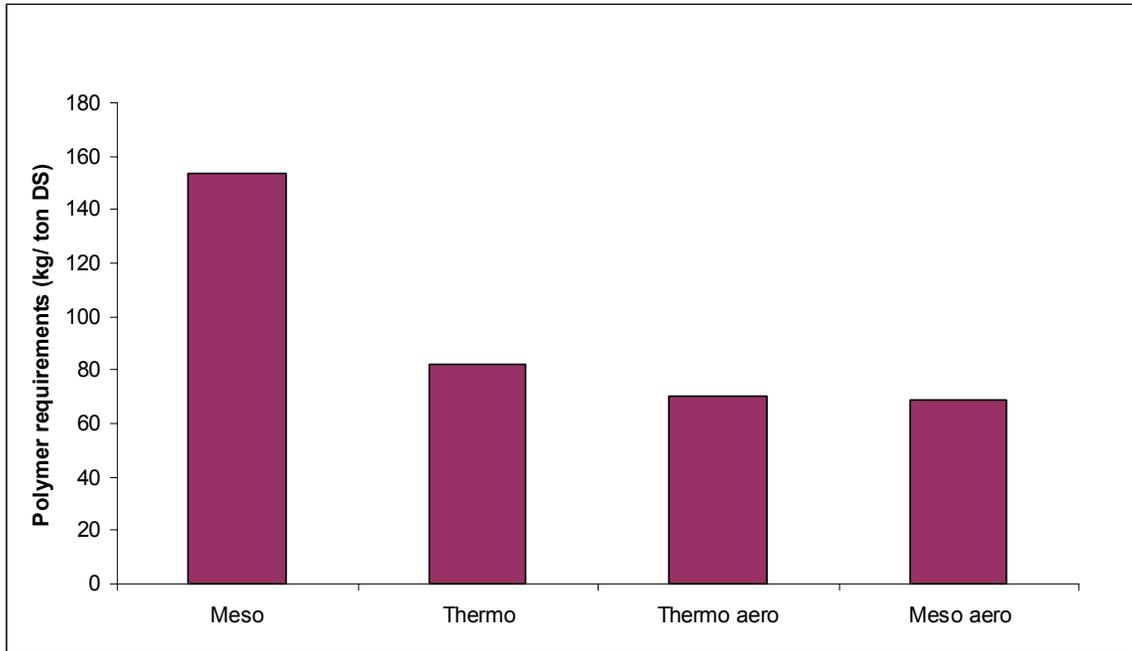


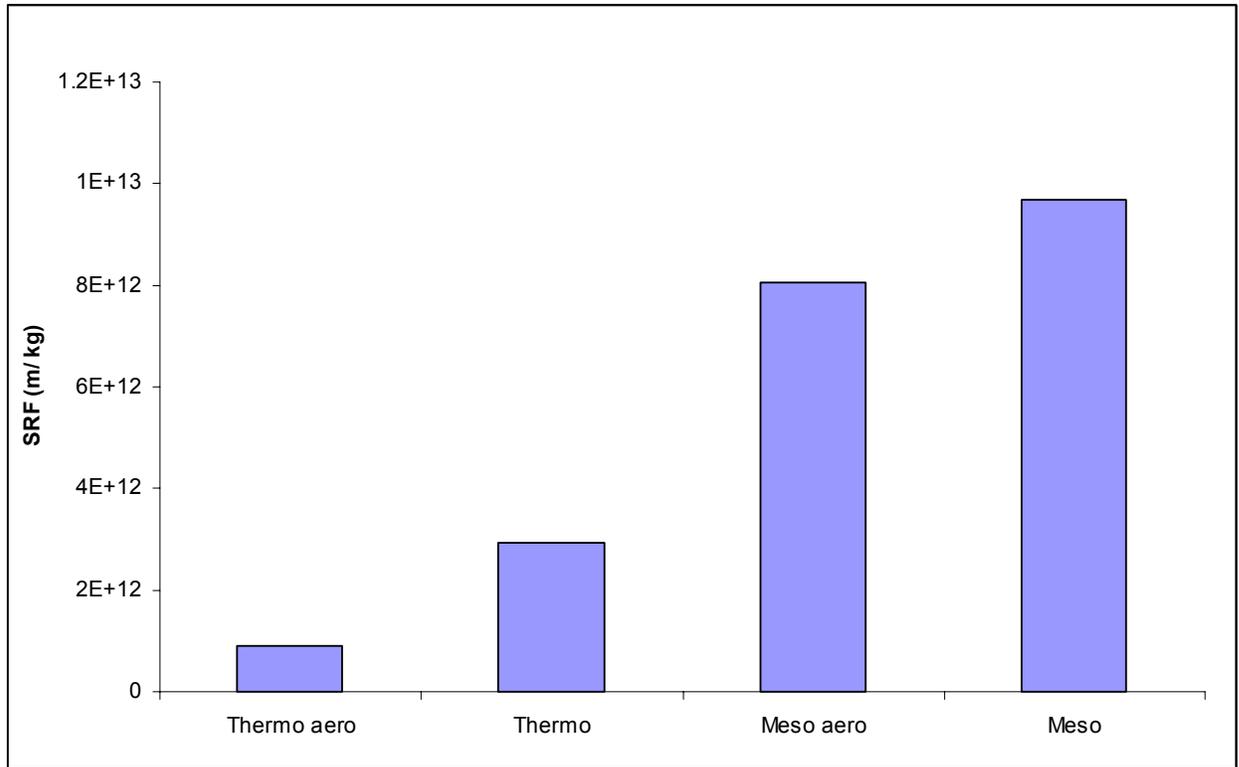
Figure II-8: Cake solids content obtained on compression dewatering of sludge conditioned with different polymers to 4, 6, 8 and 10 psi liquid pressures versus time taken



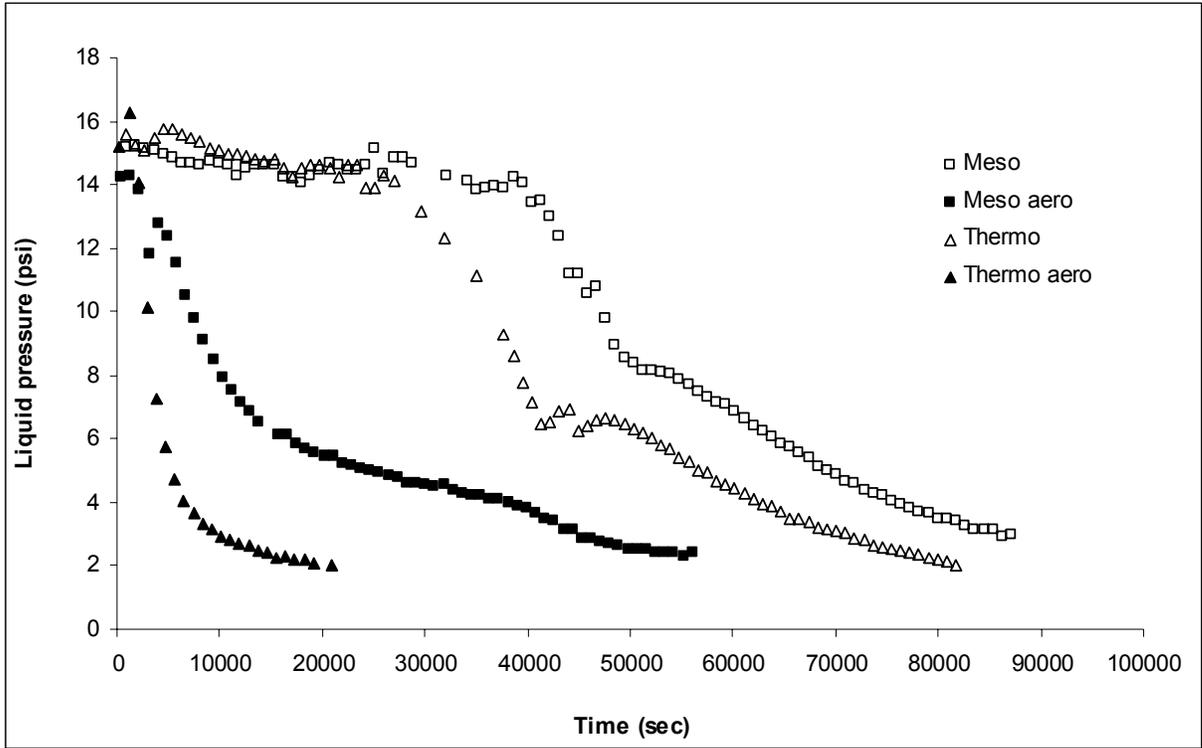
**Figure II-9: Minimum CST for different sludges conditioned with polymer SH 650**



**Figure II-10: Polymer requirements of SH 650 at optimal dosage for different sludges**



**Figure II-11: SRF of different kinds of digested sludge after conditioning at optimal dosage**



**Figure II-12: Dewatering profiles of different kinds of digested sludge conditioned with SH 650**

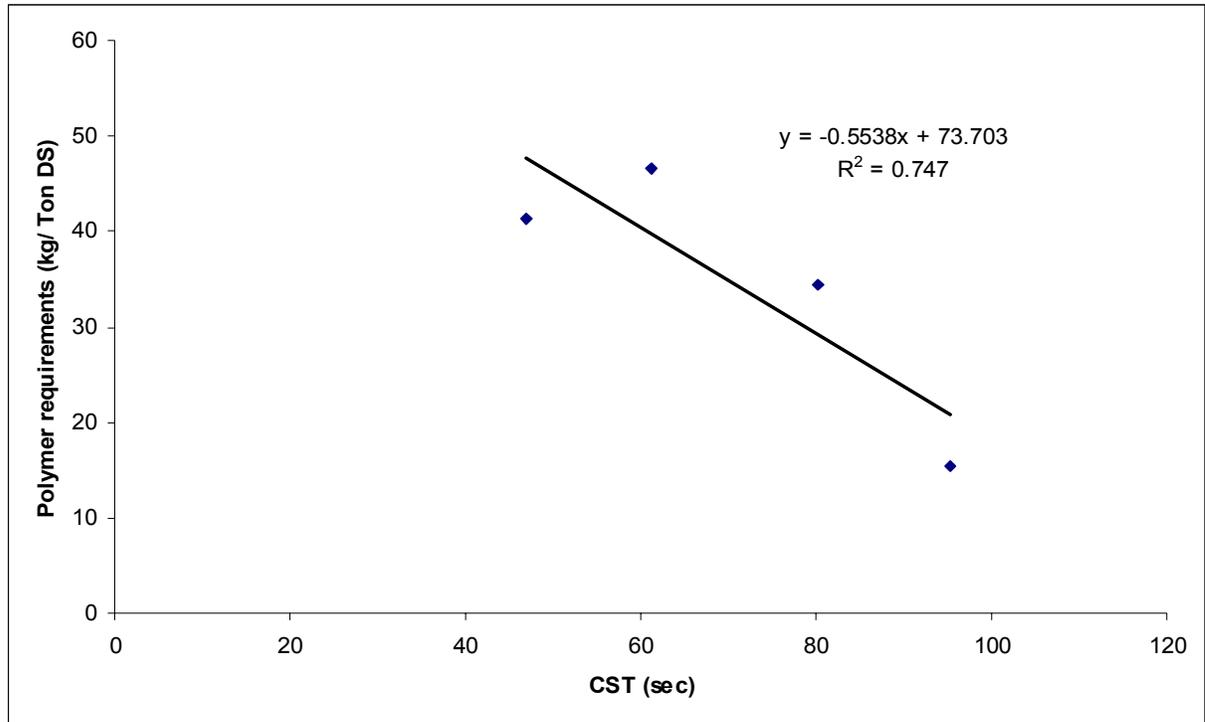


Figure II-13: Polymer requirements versus CST at optimal dosage for different sludges

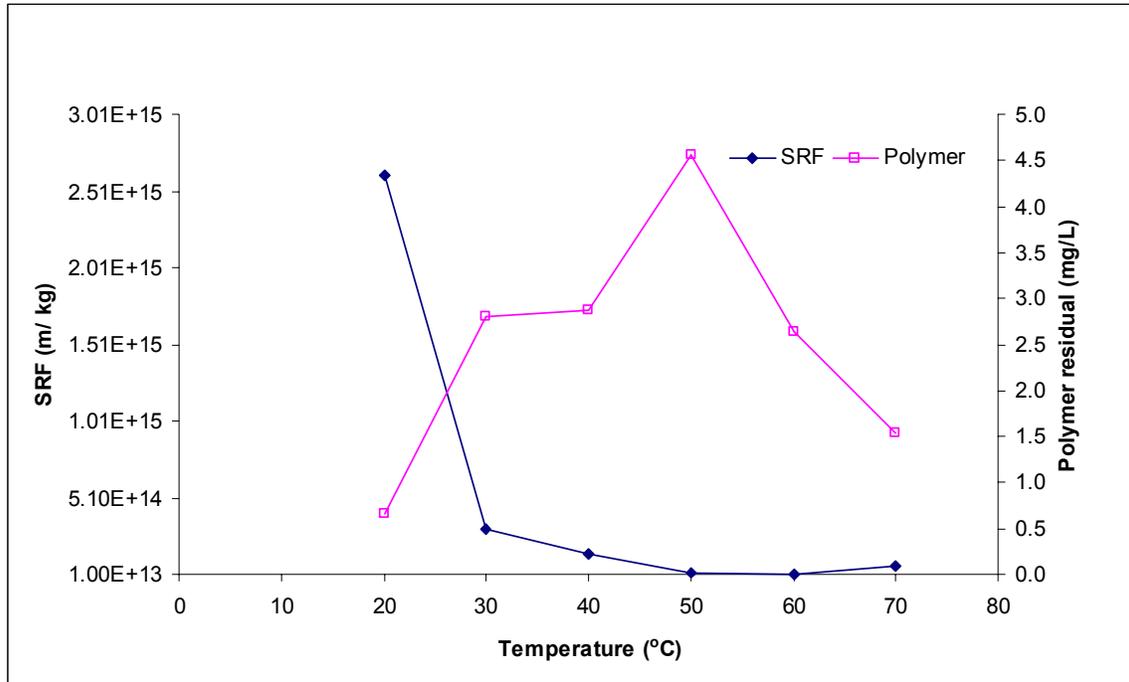
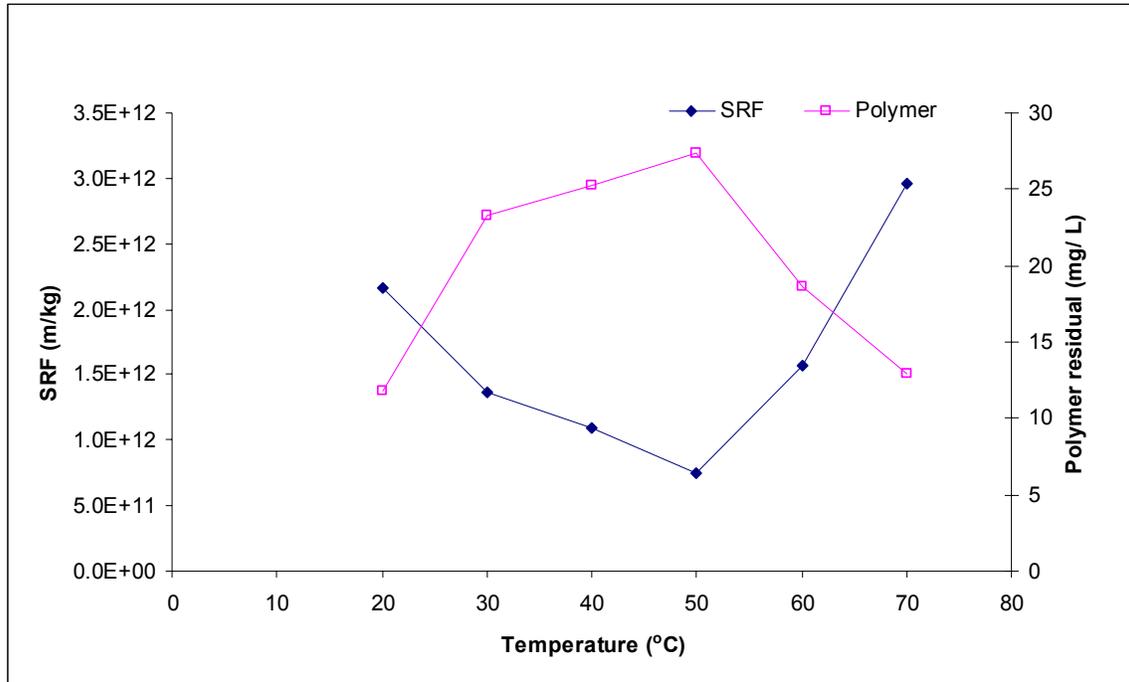


Figure II-14: SRF and polymer residual of thermophilic digested sludge at different temperatures



**Figure II-15: SRF and polymer residual of Thermo – aerobically digested sludge at different temperatures**

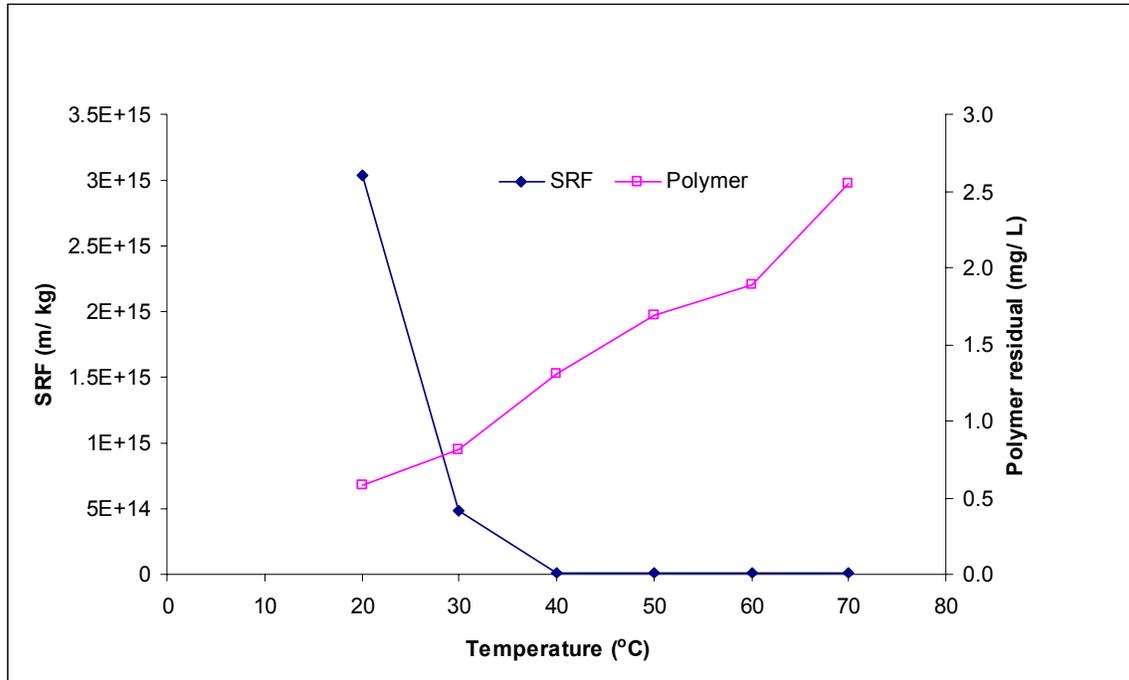
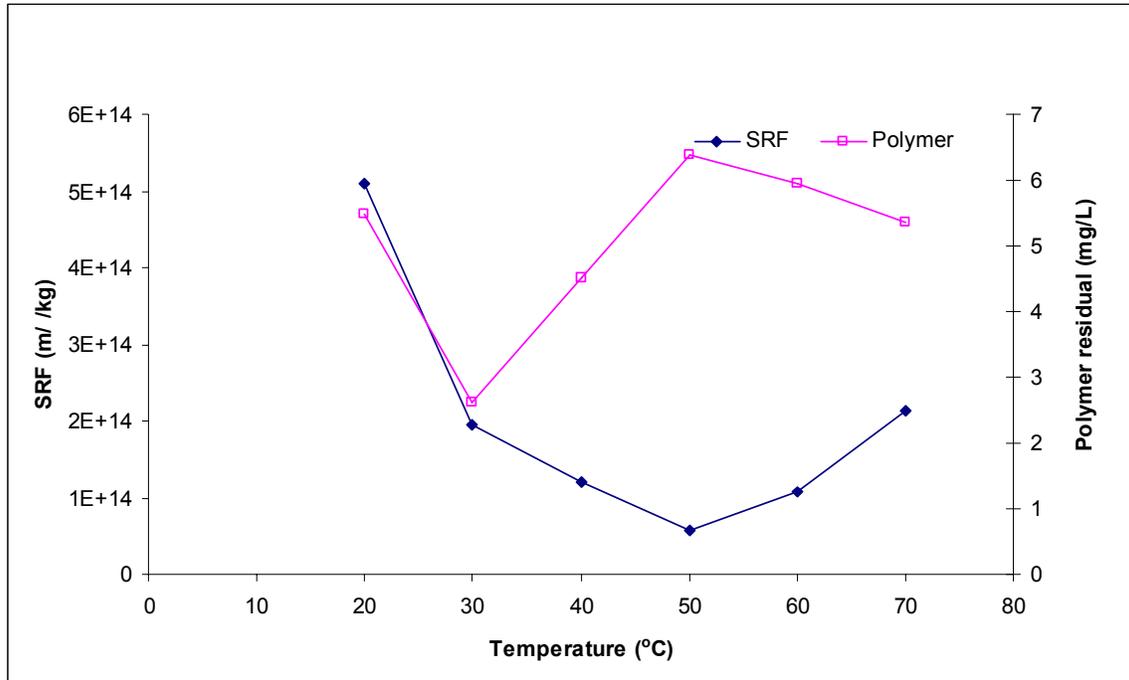


Figure II-16: SRF and polymer residual of mesophilic digested sludge at different temperatures



**Figure II-17: SRF and polymer residual of Meso - aerobically digested sludge at different temperatures**

### **III Manuscript 2**

#### ***Effect of Digestion Processes on Bound Water Content of Sludge***

Sangeetha Subramanian, Nitin Kumar, Sudhir Murthy, John T. Novak

#### ***Abstract***

Water distribution in sludge has been studied by dilatometry for over 5 decades and used to predict the maximum achievable solids concentration on dewatering. This study looks at different digestion processes and their effects on water distribution in sludge. It was seen that when an anaerobic digestion phase was followed by an aerobic digestion phase, the bound water content decreased and resulted in a higher cake solids concentration by mechanical dewatering. The study also established the importance of incorporating the effects of temperature and density differences of free water used in the calculations. Further, correlation between the bound water content and achieved solids content of field samples was examined to see if the bound water was solely dependent on the solids content or was influenced by other dewatering factors like torque and g values.

Keywords : Dilatometry, Water distribution in sludge, Digestion techniques, Bound water content

#### ***Introduction***

It has been long suggested that water distribution in sludge plays a key role in affecting the dewaterability. Pramanik (1994) conducted a literature survey in which he describes the different kinds of classifications of water distribution. A classification of water distribution according to different authors is provided in Table III-1.

The four phase water distribution suggested by Smith and Vesilind (1995) consisting of bound water, surface water, interstitial water and free water is one of the most widely used classifications of water distribution in sludge. Smith and Vesilind (1995) used dilatometry to investigate the distribution of water in activated sludge. Two notable conclusions of the above study were that the precision of the dilatometric technique decreased with decreasing solids concentration and the accuracy of the method could not

be verified without an independent knowledge of the quantity of bound water. Vesilind and Hsu (1997) conducted further experiments with dilatometry and tried to determine the temperature at which all the free water froze. They found that a greater fraction of water froze at  $-35\text{ }^{\circ}\text{C}$  than at  $-8\text{ }^{\circ}\text{C}$ . They also suggested that almost all of the significant portion of free water froze at  $-8\text{ }^{\circ}\text{C}$ . Most dilatometric studies have however used the temperature range between  $+20\text{ }^{\circ}\text{C}$  and  $-20\text{ }^{\circ}\text{C}$  to determine bound water content.

Bound water is measured in dilatometry by determining the amount of water that remains unfrozen at  $-20\text{ }^{\circ}\text{C}$ . Other methods of determining bound water content include Differential Scanning Calorimetry (DSC), Differential Thermal Analysis (DTA), drying curve method and centrifugal determination of bound water (Vaxelaire and Cezac, 2004). Smith and Vesilind (1995) note that the various problems in using dilatometry include its high sensitivity to ambient temperature, gases present in the sample matrix and the formation of bubbles due to degassing upon cooling. Despite some of the shortcomings, dilatometry remains a standard method to determine bound water content due to its good repeatability (Robinson and Knocke, 1992) and low costs compared to other methods.

Digestion processes significantly affect the water distribution and dewaterability of sludge. Digestion aims at reducing the solids content of sludge by a minimum of 45% while also reducing the pathogens in the sludge, odor potential, vector problems associated with land disposal and post-disposal organic matter degradation. Studies have shown contradictory effects on the dewaterability of sludge after digestion. Houghton and Stephenson (2002) cite numerous studies where different opinions have been expressed about the dewaterability of sludge on digestion. They consider that the sludge dewaterability might be related to the influent sludge characteristics and extent of digestion. Digestion basically refers to the endocytosis phase of cell growth, where the cells start to consume themselves for food. This results in the rupture of biomass, and the release of proteins, polysaccharides and water held inside the cell (Grady, Diagger and Lim, 1999).

Studies by Novak et al (2004), Park et al. (2004), and Jin et al. (2004) have shown that the concentration and composition of extra cellular polymeric substances plays an important role in determining the dewaterability of sludge. These studies state that the dewaterability of sludge measured by different methods including CST, SRF and SVI show poorer dewatering when the amount of proteins present as EPS increases in solution. Novak et al. (2003) showed that EPS in sludge was associated with cations. Aerobic digestion resulted in the release of calcium and magnesium associated biopolymers while anaerobic digestion resulted in the release of iron and aluminum associated proteins. Thus, they suggested the existence of two distinct fractions of EPS. They also showed that the ratio of proteins to polysaccharide in solution decreased when aerobically digested, while it increased on anaerobic digestion. Park et al. (2004) also showed similar results in terms of solution proteins to polysaccharide ratios with different types of digestion processes.

### ***Research objectives***

This study focuses on how different digestion processes affect dewatering of sludge. Anaerobic digestion is the method of choice adopted by many large-scale water treatment plants due to the added attraction of methane generation and good pathogen reduction. However, the effluent quality in most cases is poor with regard to sludge handling properties. In order to investigate the possibility of enhancing the sludge quality including dewatering characteristics, the sludge subjected to anaerobic digestion was compared to sequentially (anaerobic/ aerobic) digested sludge. More specifically, the study sought to establish a relationship between the bound water content of digested sludges and the solids content obtained by mechanical dewatering. In addition, the release of biopolymers during digestion was also monitored. The dewaterability of sludge was characterized by compression cell dewatering profiles, CST, polymer requirements and bound water content.

The other objectives of the study were to determine if solids content achieved by using laboratory centrifuges could be used to predict bound water content and maximum achievable solids from field centrifuges. Further, Robinson (1989) had suggested that the

bound water content tended to plateau above a particular solids level. The evaluation of the existence of the plateau and the determination of the solids concentration at which this might occur were also goals of the study. The influence of different digestion processes was studied and the universal nature of the relationship between cake solids and bound water investigated. The correlation between lab-centrifuged cake solids and bound water content was expected to establish a relationship with which actual field data could be predicted.

### ***Materials and methods***

Anaerobically digested sludge was obtained from different wastewater treatment plants including the Pepper's Ferry wastewater treatment plant (VA), the Christiansburg wastewater treatment plant (VA) and the Philadelphia wastewater treatment plant (PA). Also, a mixture of primary and waste activated sludge from DC Water And Sewage Authority was anaerobically digested by various enhanced digestion techniques in the lab. All samples were stored at 4 °C till the experiments were performed. A portion of each of the anaerobically digested samples was subjected to batch aerobic digestion for 6 to 10 days in the lab, to monitor the effect of the aeration phase on the digested sludge. A description of the digestion processes adopted in this study is provided in Table III-2.

Total and volatile solids were determined in accordance with Standard Methods (APHA, 1995). Solids, soluble EPS concentration and composition and bound water content were monitored in all the anaerobically digested samples before and after the aerobic digestion phase. The samples were centrifuged in the lab at 10,000 g for 15 minutes and the solids content of the resulting cakes was determined. These cakes were further dewatered on a compression cell at 40 psi and a compression time of 10 minutes using loading rates of 75, 50, 25 and 15 grams to obtain cakes with different solids concentration.

Pramanik (1994) and Kolda (1995) suggested that dilatometers with a bigger volume produced more accurate results of bound water content as compared to the smaller units. Kolda experimented with different volumes of sludge in the dilatometers and determined 25 to 30 g of sludge to be the best range of sample weight for dilatometers of

approximately 90 ml volume. Experimental units and sample volumes were chosen accordingly. The specific gravity and bulk density of the sludge was measured carefully using a 30 ml pycnometer. The dilatometric units were immersed in an ethanol bath and the temperature was controlled with dry ice. Sufficient time was given between the additions of each batch of dry ice to allow for temperature within the bath to equilibrate. Hydraulic oil (Coastal hydraulic jack Oil – Grade AW 2) was chosen as the dilatometric fluid for all the experiments. Each set of samples was run with a hydraulic oil control, water control and temperature control units.

Total soluble proteins and polysaccharides present in the centrate were determined in the sludge supernatant obtained from the above centrifugation by the Frølund *et al.* (1996) method and Dubois *et al.* (1956) method respectively. The centrate was filtered through a 1.5 µm membrane filter and the concentration of EPS obtained from such a sample was termed total soluble EPS for the context of this study.

Field centrifuged samples from Philadelphia were taken and their bound water content evaluated. The samples are described in Table III-3.

Dewatering was tested by CST, polymer requirements and by studying compression cell dewatering profiles. Optimal dose curves were generated using a 1% dilution of a solid phase cationic polymer – Stockhausen 650 supplied by Clear water Inc. The polymer solution was added to 100 ml of the sludge and blended with a low-speed shearing device to ensure uniform dispersion of polymer in each sample. The optimal dose was the polymer needed to reach the lowest CST.

Couturier *et al.* (2004) pointed out to erroneous liquid pressure measurements being recorded on the compression cell due to entrapment of air between the pressure transducer and the liquid surface. In order to overcome this, they suggested placing another plate in front of the piston carrying the pressure transducer and filling the space in between with the liquid. In the experiments conducted as a part of this study, the

problem was solved by allowing the air inside the cylinder to escape by means of a vent on the piston surface and closing it once water started to seep out of the vent.

The variation of bound water with solids content was monitored. Robinson (1989) had suggested that the bound water content plateaued beyond a threshold solids concentration. The solids concentration at which this occurred was studied. Field centrifuged samples obtained from Philadelphia were analyzed for solids and bound water content and related to data obtained from lab centrifuged and dewatered samples.

### ***Results and Discussion***

An analysis of dewatering parameters will first be discussed with respect to anaerobic and anaerobic – aerobic digestion (hence referred to as sequential digestion). Figure III-1 shows the polymer requirements of the anaerobically digested sludges as compared to the sequentially digested sludges. It can be seen that the sequentially digested samples have a lower optimal polymer dose requirement than the anaerobically digested samples. All cases except for the Christiansburg sludge followed the trend mentioned above. Studies by Novak *et al.* (2003) have shown that anaerobic digestion results in increased conditioner requirements to attain optimal dosage while aerobic digestion reduced the demand. Polymer requirements form an important fraction of chemical costs associated with operation of a treatment plant. So, a reduction in polymer could result in significant cost savings.

Meeten and Smeulders (1995) note that CST tests measure the time taken for the free water in the sludge to permeate through a fixed distance on Whatman 17 chromatographic grade paper. A better dewatering sludge has a lower CST while a poorer dewatering sludge is characterized by a higher CST. Park *et al.* (2004) showed that anaerobic digestion of waste activated sludge resulted in the increase of CST, while further aerobic digestion of sludge resulted in an increase of CST up to 10 days followed by a decrease of CST on prolonged aerobic digestion. They suggest that prolonged aerobic digestion as a follow up procedure to anaerobic digestion results in the degradation of the soluble polysaccharide initially released into solution, resulting in a net improvement in dewatering. The results observed in this study are shown in Figure

III-2. As mentioned in Table III-2, batch aerobic digestion was carried on for periods ranging from 6 to 10 days SRT. The results show a consistent increase in the minimum CST for all the samples after sequential digestion. Further aerobic digestion after anaerobic digestion might cause a shift in the particle size distribution of sludge towards a smaller size range resulting in the increase in resistance towards the permeation of water through the sludge particles.

Digestion processes result in the destruction of solids and in the release of intracellular and inter-floc water, proteins and polysaccharides. The increase in polymer requirements has been shown by Novak *et al.* (2003) to be due to the increase in the soluble protein and polysaccharide concentration in solution following digestion. Figure III-3 and Figure III-4 show the protein and polysaccharide concentration following anaerobic and sequential digestion. The study shows a decrease in protein concentration and an increase in the concentration of polysaccharides by aerobic digestion. This is in agreement with previous studies conducted by Novak *et al.* (2003), Park *et al.* (2004) and Novak *et al.* (2004). Novak *et al.* (2004) suggest that anaerobic and aerobic digestion release different kinds of biopolymers into solution and the charge distribution of these colloids might play an important role in determining the conditioning requirements of the sludge.

Park *et al.* (2004) showed that sequential digestion process could result in up to 10% additional VS destruction. In this study, it was found that sequential digestion resulted in additional VS destruction in the range of 7.5 to 40% depending on the process and the sample being digested. Digestion of DC WASA sludge by sequential mesophilic / aerobic digestion resulted in an additional VS destruction of about 7.5%, while sequential thermophilic/ aerobic digestion of the same sludge yielded up to 41% additional VS destruction. In the case of Pepper's Ferry, Philadelphia and Christiansburg, sequential digestion resulted in additional VS destruction in the range of 25 to 30%. Volatile solids seen in anaerobically digested sludge and sequentially digested sludges are shown in Figure III-5.

In Figure III-6, the relationship between total solids of the sludge and cake solids by lab centrifugation is shown. Despite the scatter of data, it appears that a negative correlation exists between the solids content seen in the sludge and the cake solids attainable on centrifugation. Sequentially digested thermo sludge represented by the top-most point in the graph, has the maximum VS destruction and also provides maximum solids concentration on centrifugation.

Figure III-7 shows the compression dewatering profiles of anaerobically and sequentially digested thermo and meso sludges. It is evident that sequentially digested sludges undergo better dewatering and compression than anaerobically digested sludges. Though the data are not shown for the rest of the samples, the same trend was followed for all the five samples tested as a part of this study with sequentially digested sludges showing consistently better dewatering properties than anaerobically digested sludges.

Bound water content was determined by dilatometry using sludge cakes obtained by centrifugation at 10,000 g for 15 minutes. The parameter is measured in terms of grams of bound water present per gram of Dry Solids (Robinson, 1989; Pramanik, 1994; Smith and Vesilind, 1995; Kolda, 1995). Digestion results in the destruction of solids. So, it was expected that different digestion processes would impact the bound water content of sludge. Figure III-8 shows the cake solids content obtained on centrifugation in relation to the bound water content observed. The bound water content of sequentially digested samples is consistently lower than that of the anaerobically digested samples, suggesting that a second phase of aerobic digestion results in the break up of flocs and the consequent release of intra-floc water as free water. The increase in cake solids obtained on centrifugation is seen to correspond with the decrease in bound water content of the samples. The results suggest that dilatometry measures the bound water content represented by intra-floc and intra-cellular water and that sequential digestion techniques result in a final effluent with better dewatering characteristics as compared to anaerobically digested sludge.

The second phase of the study involved the construction of a graph suggested by Robinson (1989). In the study conducted by Robinson, bound water content was plotted as the independent variable and the solids content as the dependent variable. Since the solids content is used as a part of the calculations needed for evaluating the bound water content of the samples, the solids content was designated as the independent variable and the bound water content as the dependent variable in this study. Robinson found that the bound water content decreased linearly with increasing solids content of above 10%. Vesilind and Hsu (1997) experimented with very low solids content in the range of 0.4 to 2.0 % and found that the bound water content decreased with decreasing solids concentration till about 0.8%, where it leveled off. They attributed this to the non-consolidation of particles at very low solids concentration with a consequent greater surface area being exposed to the surrounding water resulting in a greater fraction of water being present as bound water. In this study, the bound water content was calculated for different solids contents obtained from different sludges, and the results were consolidated on a single graph shown in Figure III-9. The bound water content was dependent on the solids content of the sludge used for the experiment. The bound water content falls as the solids content increases, stabilizing at about 1.0 to 0.75 g of bound water present per gram of dry solids present. Beyond 20% cake solids concentration, bound water content varied little with increasing solids content. The presence of a nearly constant fraction of bound water associated with the sludge beyond 20% cake solids, suggests the presence of a mechanically unremovable fraction of water present in the sludge matrix.

Pressure dewatering characteristics of the different sludges were investigated at different solids loading levels of 75, 50, 25 and 15 g on the compression cell maintained at 40 psi pressure applied for a duration of 10 minutes. Figure III-10 shows the relationship between different centrifuged solids and solids obtained by pressing the cakes on the compression cell at 25 g and 50 g loading. A good fit of the data is seen with higher pressed solids being obtained from centrifuged cakes with higher solids content. Another interesting observation is that as the solids loading increased in the compression phase, the final solids obtained decreased. Similar trends have been reported by Novak *et al.*

(1999) and Zhao and Bache (2002) with regard to the significance of loading rate in determining the final solids content attained by compression.

Field data were analyzed for solids and bound water content. Table III-4 shows the solids and bound water content of the different field samples obtained from Philadelphia. The samples that were centrifuged at the same g values (samples 1 to 3), but differing torques, and the samples obtained by running the different centrifuges at standard operating practices (samples 7 to 12) conform with the trend observed in the lab samples, exhibiting a decrease in bound water content as the solids content of the cake increased. However, the samples run at the same torque and differing g values (samples 4 to 6) show an inverse trend with the bound water content increasing with an increase in cake solids. The total set of field data fit well with the general trend observed between solids content and bound water content. However, the sample size of the field experiments is not sufficient to establish the individual trends. More samples will have to be analyzed to obtain a better idea of the real trend in the field experiments and the effect of centrifuge operation on cake solids and bound water.

The solids content achievable by mechanical dewatering was calculated from the bound water values and compared to the actual solids content of the sludge used for the experiments. As the solids concentration increased, the difference between the expected solids content by bound water calculations and the actual observed solids increased. Thus, the difference between the solids content actually observed and that predicted by using the bound water content keeps increasing with increasing solids concentration. This is shown in Figure III-11. It is hypothesized that the bound water calculations incorporate an error associated with the solids content that increases exponentially, resulting in the discrepancies observed.

Bound water content cannot be used as a prediction parameter in determining the maximum dewaterability of sludge due to the fact that the solids concentration needs to be known before hand to calculate the bound water content and also due to the error involved. Smith and Vesilind (1995) have pointed out that bound water content

measured by dilatometry needs to be verified with an independently determined bound water content. Skidmore and Reyes (2004) compared the bound water content calculated by dilatometry with the solids content obtained on centrifugation in the lab and noted that absence of any significant trend between them. In this study, significant correlation has been established between the solids obtained on lab centrifugation, compression dewatering, field centrifuge dewatering and bound water content calculated by dilatometry. However, the fit of field data is scattered and the accuracy of data needs to be improved by increasing the sample size.

### ***Conclusions***

Bound water content is an interesting parameter that can be used in the description of sludge dewaterability. However, since it is described as a function of the solids content, it cannot be used to predict the solids content.

All sludges tested showed that the sludge dewatering properties improved after being subjected to an aerobic digestion phase following anaerobic digestion. The dewaterability was characterized by three parameters: – polymer requirements, CST and compression dewatering.

It was observed that digestion processes significantly affected the solids concentration achieved by mechanical dewatering with the highest solids concentration obtained on thermo-aero digestion, and the least solids concentration on single-phase, mesophilic anaerobic digestion. The bound water content was found to correlate well with the solids content and field-centrifuged samples also fit well into the curve generated using lab samples ( $R^2$  for the curve including lab and field samples = 0.91).

The bound water content decreased with increasing solids concentration, to a solids content of 20% after which, it plateaued at about 0.75 to 1.0 g of bound water per gram of DS present. This suggests that a maximum solids content of 50 to 57 % can be achieved by mechanical dewatering.

## ***Acknowledgement***

This part of the study was funded in part by DC Water and Sewer Authority.

**Table III-1: Classification of water distribution according to different authors**

<b>Number of phases</b>	<b>Description of water distribution</b>	<b>Author(s)</b>
Two	Free and Capillary water	Novak <i>et al.</i> (1982)
Two	Internal (intra-cellular) and External (extra-cellular) water	
Two	Free and Bound water	Pramanik (1994)
Three	Free, floc associated and bound water	
Four	Free, floc, capillary and bound water	Smith and Vesilind (1994)
Four	Free, capillary held, floc or particle held water and chemically bound water	Coackley and Allos (1962)
Four	Inter-space, interstitial capillary, adsorption and adhesion water and internal waters	Moeller (1983)
Six	Free, inter-floc, floc-adsorbed, inter-particle, particle-adsorbed and particle water	Huang <i>et al.</i> (1979)

Source: Pramanik (1994)

**Table III-2: Description of the digestion processes adopted in this study**

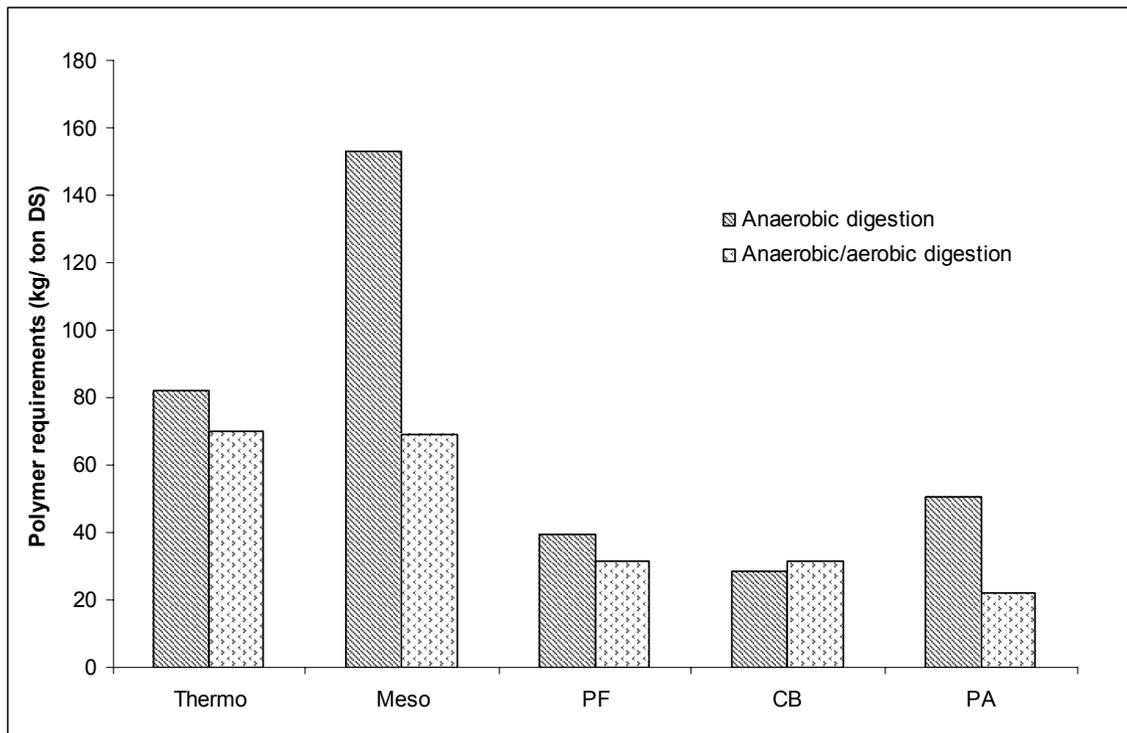
<b>Sample source</b>	<b>Digestion process</b>	<b>Temperature (°C)</b>	<b>SRT (days)</b>
Primary and waste activated sludge from DCWASA	Two phase anaerobic digestion	55	20
Primary and waste activated sludge from DCWASA	Two phase anaerobic aerobic digestion	55 + 31	20+6
Primary and waste activated sludge from DCWASA	Two phase anaerobic digestion	35	10
Primary and waste activated sludge from DCWASA	Two phase anaerobic aerobic digestion	35 + 31	10+6
Pepper's Ferry	Conventional anaerobic digestion	35	45
Pepper's Ferry	Conventional anaerobic digestion followed by aerobic digestion	35 + 25	45+10
Christiansburg	Conventional anaerobic digestion	25	30
Christiansburg	Conventional anaerobic digestion followed by aerobic digestion	25 + 25	30+10
Philadelphia	Conventional anaerobic digestion	35	30
Philadelphia	Conventional anaerobic digestion followed by aerobic digestion	35 + 25	30+10

**Table III-3: Description of centrifuges from which field samples were taken**

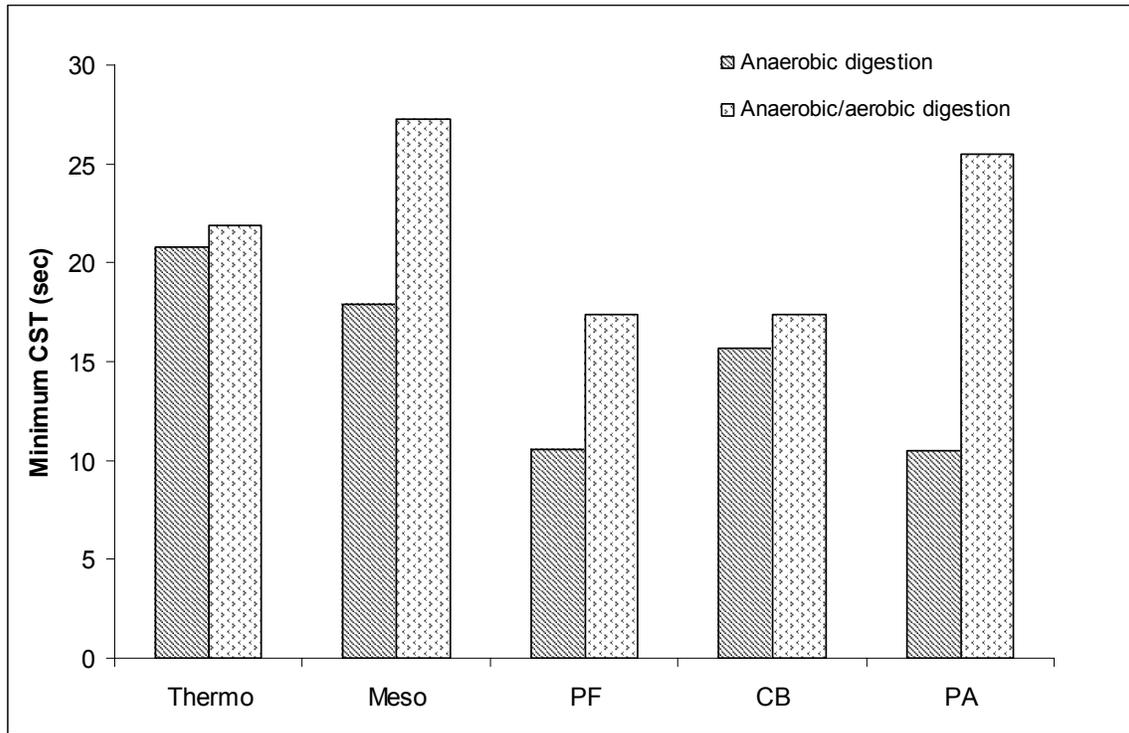
<b>Sample</b>	<b>Machine</b>	<b>g</b>	<b>Torque</b>
Cake 1	Humboldt #2	2460	70
Cake 2	Humboldt #2	2200	70
Cake 3	Humboldt #2	2000	70
Cake 4	Humboldt #2	2460	70
Cake 5	Humboldt #2	2460	63
Cake 6	Humboldt #2	2460	56
Cake 7	Centrysis Mod	2460	70
Cake 8	Bird 10		
Cake 9	Bird 9		
Cake 10	Humboldt 5030	2200	
Cake 11	Humboldt 5029	2080	
Cake 12		1983	

**Table III-4: Solids content and bound water content of field centrifuged samples**

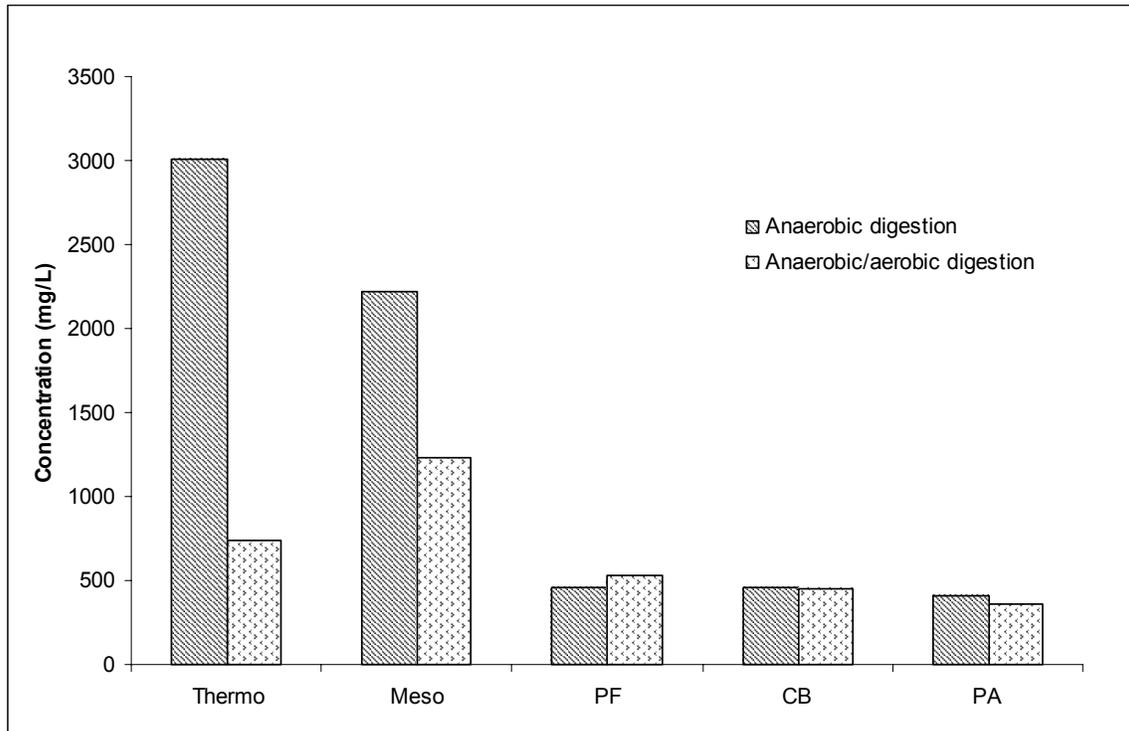
<b>Sample</b>	<b>g</b>	<b>Torque</b>	<b>Solids (%)</b>	<b>Bound water (g/ g DS)</b>
Cake 1	2460	70	32.124	1.218
Cake 2	2200	70	32.727	1.784
Cake 3	2000	70	33.210	1.725
Cake 4	2460	70	28.065	2.221
Cake 5	2460	63	26.665	1.253
Cake 6	2460	56	29.919	0.717
Cake 7	2460	SOP	30.173	1.286
Cake 8		SOP	27.779	2.076
Cake 9		SOP	29.411	0.845
Cake 10	2200	SOP	32.424	0.958
Cake 11	2080	SOP	31.339	0.703
Cake 12	1983	SOP	32.057	1.684



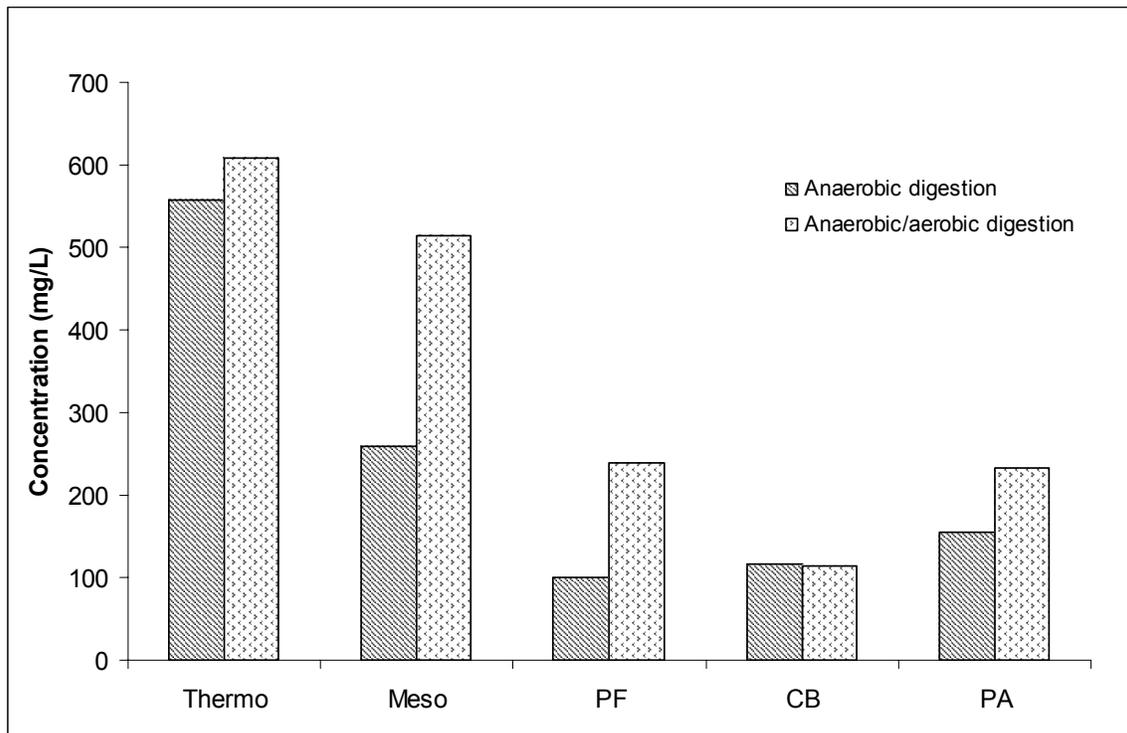
**Figure III-1: Comparison of polymer requirements at optimal dosage between anaerobically digested and sequentially digested sludges**



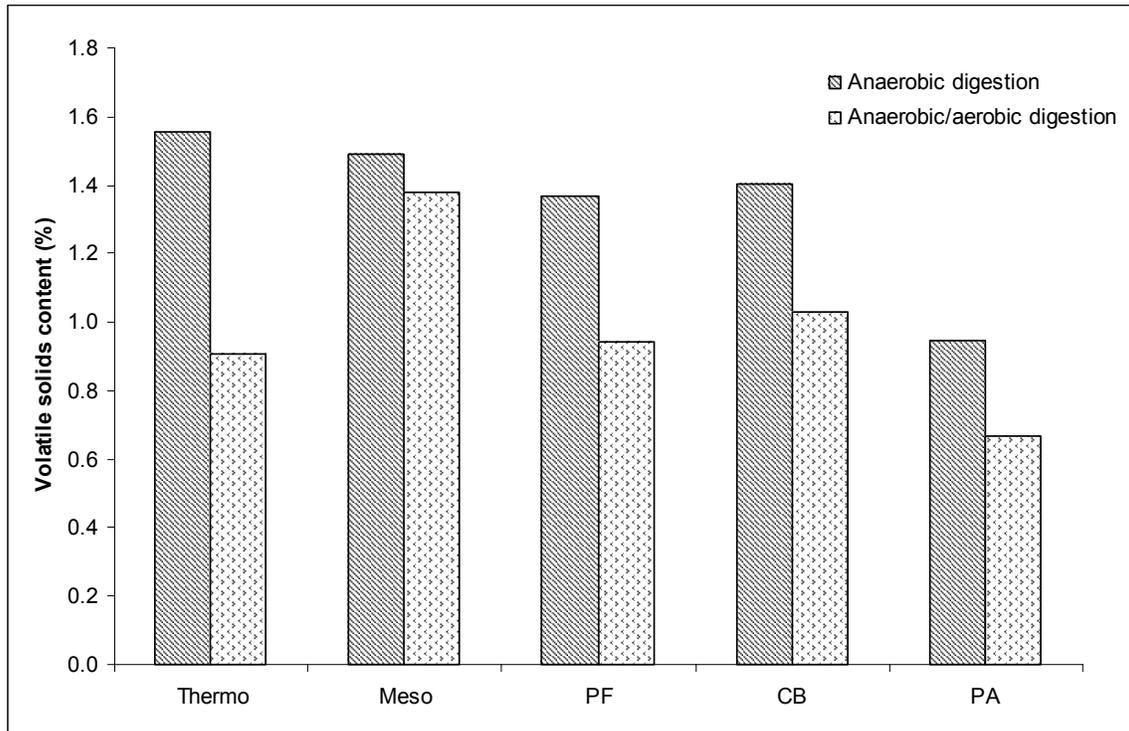
**Figure III-2: Comparison of minimum CST of anaerobically digested and sequentially digested sludges**



**Figure III-3: Soluble protein concentration of anaerobically digested and sequentially digested sludges**



**Figure III-4: Polysaccharide concentration of different sludges subjected to anaerobic and sequential digestion**



**Figure III-5: Figure showing additional VS destruction on sequential digestion**

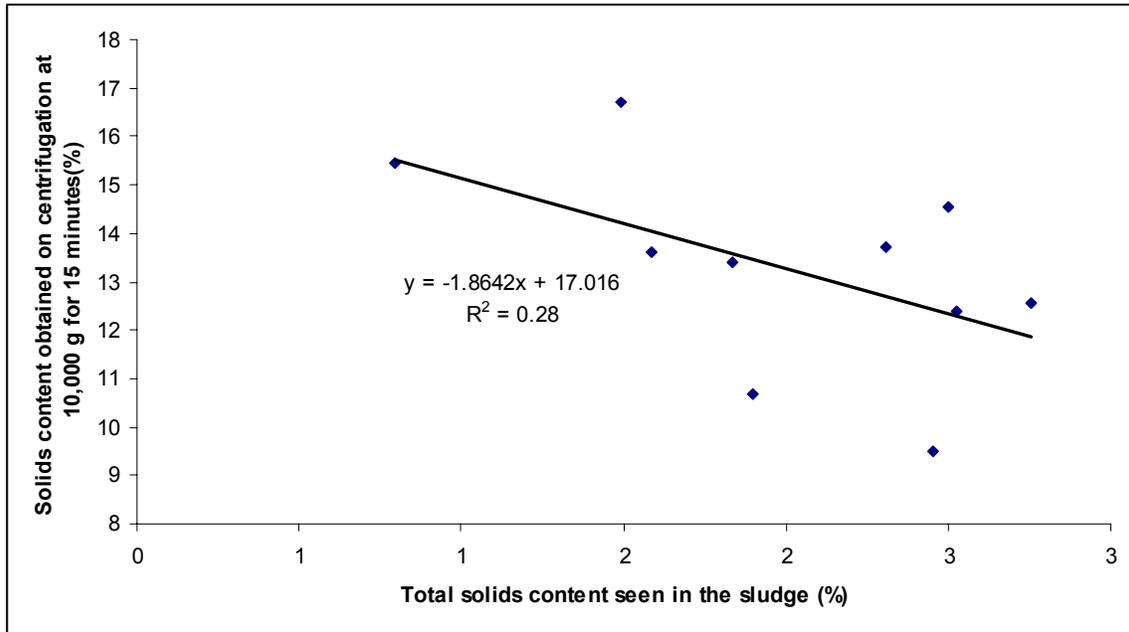
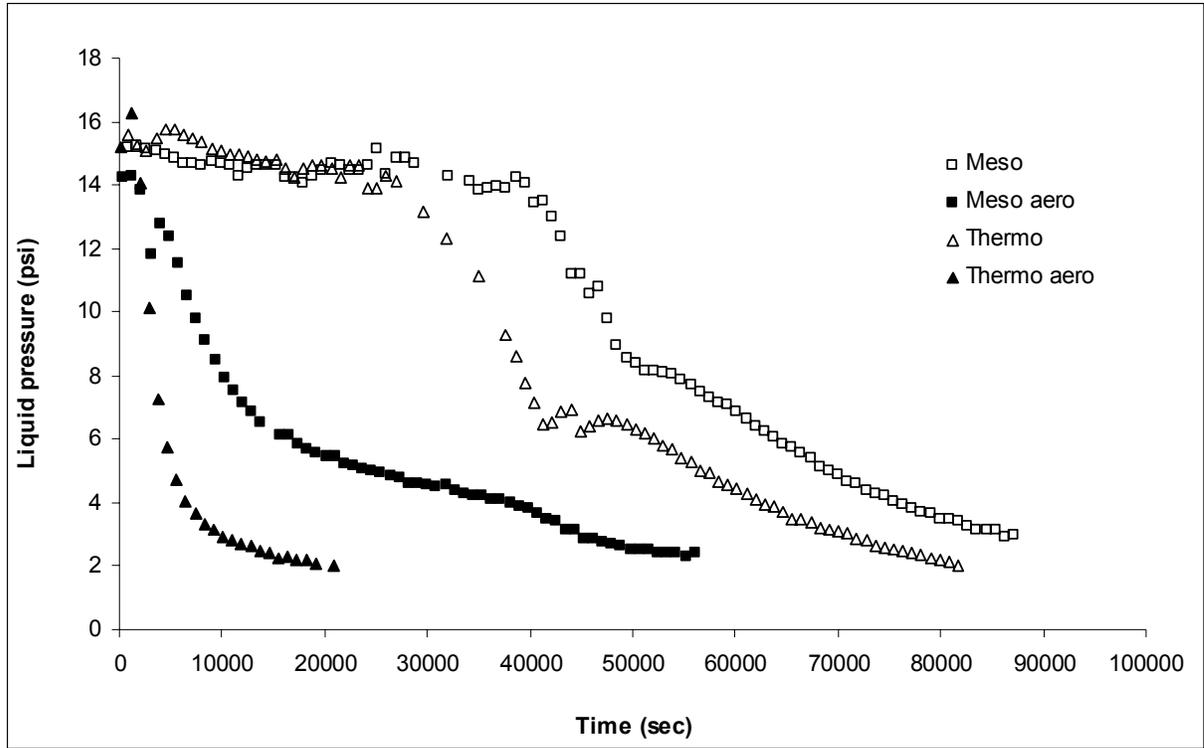
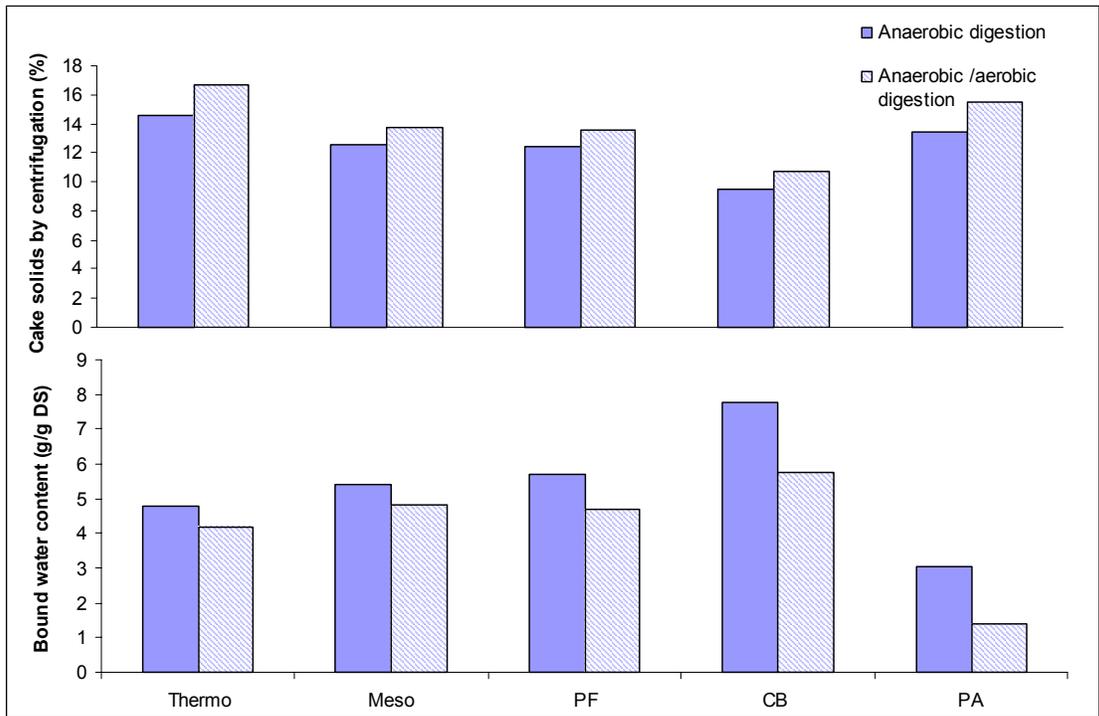


Figure III-6: Relationship between sludge solids and cake solids obtained on centrifugation



**Figure III-7: Comparison of sequentially and anaerobically digested sludges compression dewatering profiles.**



**Figure III-8: Cake solids content obtained on centrifugation at 10,000g for 15 minutes and its relation to bound water content measured by dilatometry**

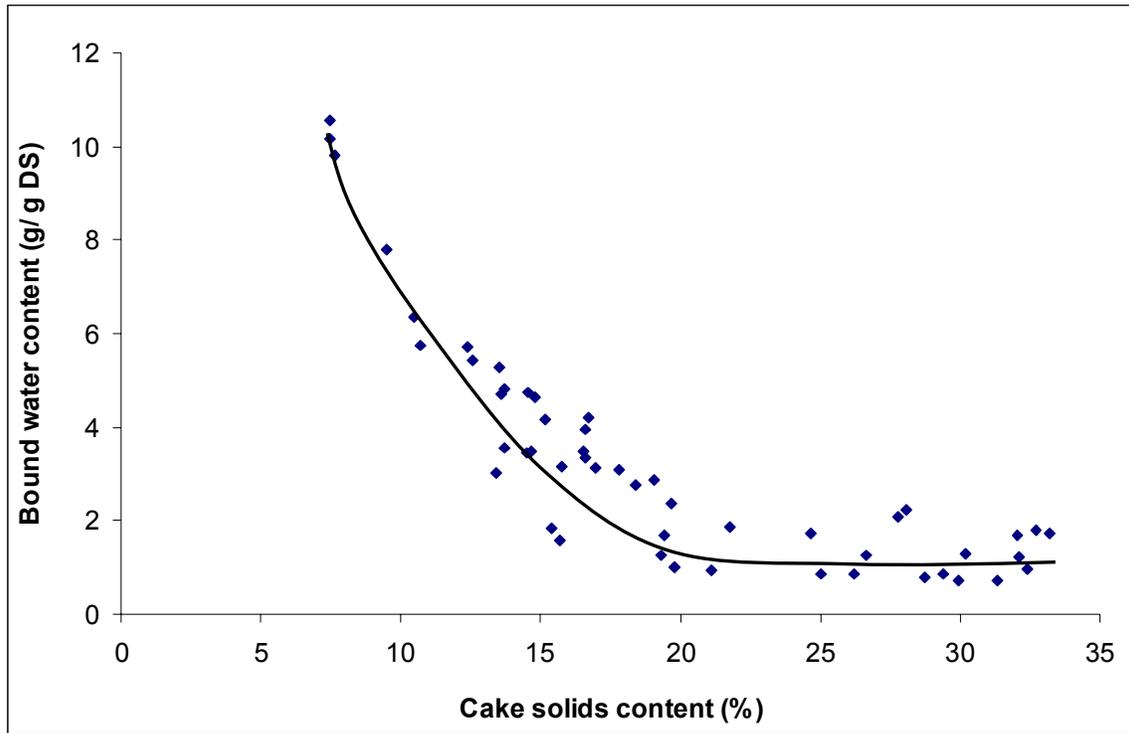
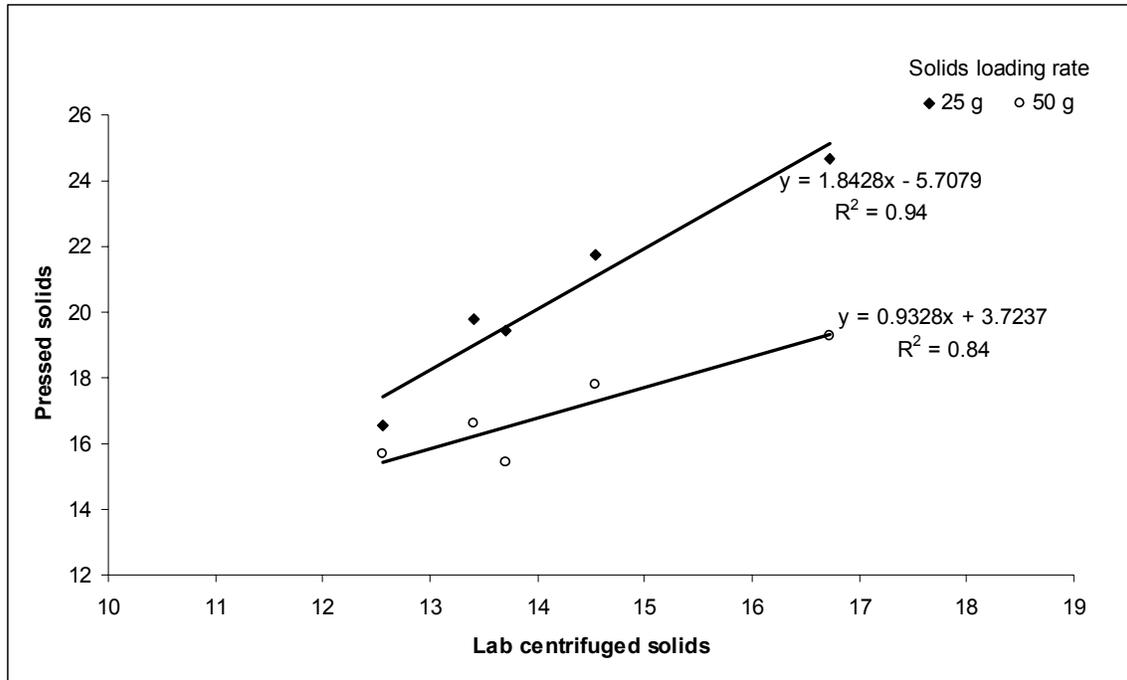
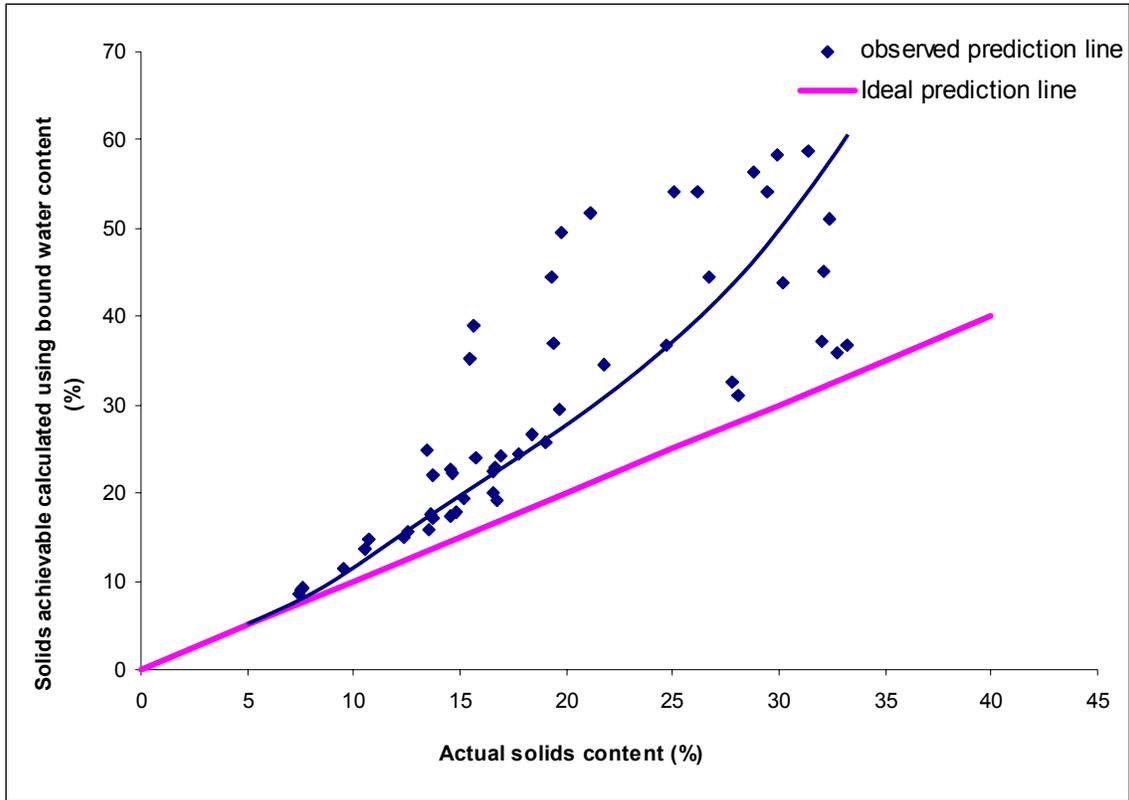


Figure III-9: Relationship between sludge solids and bound water content, showing the presence of an unremovable water fraction associated with the sludge



**Figure III-10: Relationship between cake solids obtained on centrifugation and compression cell loading rate**



**Figure III-11: Solids content predicted using bound water calculations versus observed solids concentration**

## **Reference**

- American Public Health Association; American Water Works Association; and Water Environment Federation (1995) Standard Methods for the Examination of Water and Wastewater. 19th Ed., Washington, D.C.
- Couturier S., M. Valat, J. Vaxelaire and J. R. Puiggali (2003) Liquid Pressure Measurement in Filtration-Compression Cell. *Sep. Sci. Tech.* **38**, 1051 – 1068.
- Dubois, M., Gilles, K.A., Hamilton, J.K., Rebers, P.A., and Smith, F. (1956) Colorimetric Methods for Determination of Sugars and Related Substances. *Analytical Chem.*, **28**, 350.
- Frølund, B., Palmgren, R., Keiding, K., and Nielsen, P.H. (1996) Extraction of Extracellular Polymers from Activated Sludge Using a Cation Exchange Resin. *Water Res.*, **30**, 1749.
- Grady L, Diagger G and Lim (1999) Biological Wastewater Treatment, 2nd Edition. Publishers – Marcel Dekker, USA.
- Houghton J. I. And T. Stephenson (2002) Effect of Influent Organic Content on Digested Sludge Extracellular Polymeric Content and Dewaterability. *Wat. Res.* **36**, 3620 – 3628.
- Jin B., B. Wilen and P. Lant (2004) Impacts of Morphological, Physical and Chemical Properties of Sludge Floccs on Dewaterability of Activated Sludge. *Chem. Engg. Journal* **98**, 115 - 126.
- Meeten G.H. and J.B.A.F. Smeulders (1995) Interpretation of Filterability measured by the Capillary Suction Time Method *Chem. Eng. Sci.* **50**, 1273 – 1279.
- Novak J.T., C. Park and M.M. Abu-Orf (2004) Conditioning and Dewatering of Digested Waste Activated Sludges. *Jour Res. Sci. Tech.* **1**, 47 – 53.

- Novak J.T., Mikkil L. Agerbaek, Birgette L. Sorensen and Jens Aa. Hansen (1999) Conditioning, filtering and expressing waste activated sludge *Jour. of Environ. Engg* **125**, 816 – 824.
- Novak, J.T., Sadler, M.E., and Murthy, S.N. (2003) Mechanisms of Floc Destruction During Anaerobic and Aerobic Digestion and The Effect on Conditioning and Dewatering of Biosolids. *Water Res.*, **37**, 3136 - 3144.
- Park C., M.M.A. Mohammad, J.T. Novak (2004) Predicting the Digestibility of Waste Activated Sludge. *Wat. Environ.Res.* **(In Press)**.
- Park C., Mohammad M. A., Novak J.T. (2004) Predicting the Digestibility of Waste Activated Sludges. *Wat. Environ.Res.* **(in Press)**.
- Pramanik A. (1994) Characterization of water distribution in sludges. Doctral Thesis at Virginia Polytechnic Institute and State University.
- Kolda B. (1995) Impact of Polymer Type, Dosage, and Mixing Regime and Sludge Type on Sludge Floc Properties. Master's thesis at Virginia Polytechnic Institute and State University.
- Robinson J.K. (1989) The Role of Bound Water Content in Defining Sludge Dewatering Characteristics. Master's Thesis at Virginia Polytechnic Institute and State University.
- Skidmore M. and F.D.L. Reyes III (2004) Heat Treatment Combined with Cation Addition to Improve the Dewaterability of Waste Activated Sludge. *WEFTEC 2004 proceedings*.
- Smith J.K. and Vesilind P.A. (1995) Dilatometric Measurement of Bound Water in Wastewater Sludge. *Wat. Res.* **29**, 2621 – 2626.
- Vaxelaire J. and P. Cezac (2004) Moisture Distribution in Sludge : a Review. *Wat. Res.* **38**, 2215 – 2230.

Vesilind P.A. and C.C. Hsu (1997) Limits of Sludge Dewaterability. *Wat. Sci. Tech.* **36** :  
**11**, 87 – 91.

Zhao Y.Q. and D.H. Bache (2002) Integrated effects of applied pressure, time, and  
polymer doses on alum sludge dewatering behavior *Waste Management* 22, 813 –  
819.

## **Vita**

Sangeetha Subramanian was born on 3<sup>rd</sup> May 1979 in Madras, India. She completed her schooling at the Hindu Senior Secondary School, graduating in 1996 standing first in English and Biology. She did her Bachelors of Science in Madras University graduating in 1999 majoring in Zoology. Her interests oriented her towards pursuing a master's degree in Environmental Science in Bharathiar University, one of the few institutions in India where the course was being offered. She graduated with top honors and was the valedictorian of the program for the year 2001. She worked briefly with the Environmental Technology Division of the Central Leather Research Institute under the guidance of Dr. S. Rajamani and decided to pursue a more active engineering career. She was employed with Fluid Control Equipments, India – a company involved in the manufacture of filter presses before coming to the Virginia Tech. to pursue a second master's degree in Environmental Engineering. She is now looking forward to actively starting her professional life with Malcolm Pirnie, Inc. (Newport News, VA.) specializing in water and wastewater treatment design.