LABORATORY-SCALE BIOSOLIDS DEWATERABILITY ASSESSMENT FOR MUNICIPAL WATER RESOURCE RECOVERY FACILITIES

by

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A thesis submitted to the Faculty and Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Masters of Science (Civil and Environmental Engineering).

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ABSTRACT

Dewatering is an important operational component in water resource recovery facilities (WRRFs) to reduce conveyance costs by volume reduction of sludge (wet solid residuals). Cake solids are the agglomerated solids product of dewatering. Cationic polymers are the typical chemical conditioner used to destabilize solids during dewatering to increase the rate and extent of water release from a solids slurry. Dewatering is a costly endeavor for Metro Wastewater Reclamation District, and process upgrades are expected to affect dewaterability. Laboratory-scale dewatering methods that provide cake solids and optimal polymer demand (OPD) would be highly valuable to utilities to estimate cake solids hauling cost, inform required mechanical upgrades, and associated polymer requirements. These data could be used to inform pre-design assessments for process upgrades.

The effect of solids pre-pretreatment impacts on anaerobic digestion was investigated using four mechanistically different methods that provide data on cake solids: Drying Rate, Modified Centrifuge Technique (MCT), Pneumatic Press (Press), and Limit Dryness Test (LDT). Three different methods were used in conjunction with solids conditioning to determine the OPD: Charge Demand, CST, and LDT. The laboratory dewatering methods were used to evaluate cake solids and OPD from Test and Control anaerobic digesters. The Wilcoxon rank sum test was used to examine differences between methods and lab used.

The combined cake solids results from all methods for individual phases were similar (10 to 23% standard deviation). All methods did show an increase in cake solids by a factor of 1.5 to 2.5 when thermal hydrolysis was used as a solids pretreatment process. OPD was at least 1.5 times higher for the test than the control when thermal hydrolysis pretreatment was applied to the

secondary solids; however, the low number of samples analyzed did not meet the Power criteria for the Wilcoxon rank sum test to statistically evaluate differences.

All methods tested in-house provided similar cake solids and polymer results as external laboratories. In addition, simpler methods provided similar outcomes as more time-consuming, involved tests. The laboratory-scale dewatering assessments showed differences in solids pretreatment processes only when cake solids increased by at least a factor of 1.5. Extending the laboratory-scale testing results a full-scale scenario analysis suggests that thermal hydrolysis pretreatment of secondary solids could reduce the annual cake solids hauled by 64,000 wet tonnes but at the expense of a 1.75 times increase in polymer demand. Thus, laboratory assessment of cake solids and polymer demand provides valuable information for the evaluation of potential process modifications.

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CHAPTER 1: INTRODUCTION

Annually, 8 million tons of biosolids (USEPA, 1999) are treated and disposed of at the 16,583 municipal water resource recovery facilities (WRRFs) (LeBlanc et al., 2008) in the United States alone. While not all WRRFs dewater their biosolids, dewatering is an important operational component in both water and wastewater treatment, with the main goal being volume reduction. The volume of biosolids is reduced drastically with every percentage gained in dry solids content. Any process improvements that can improve dewaterability are attractive to utilities because of the potential cost savings. Biosolids treatment and conveyance is a high cost center, and can account for up to 50 to 60% of the operation and maintenance (O&M) costs a WRRF incurs (Wei et al., 2003).

Metro Wastewater Reclamation District (MWRD) owns and operates the Robert W. Hite Treatment Facility (RWHTF) located in Denver, Colorado. The RWHTF treats an average of 530 million liters of wastewater per day for a population equivalent of two million people. The RWHTF has two liquid treatment trains and a centralized solids treatment train (Figure 1.1). In Solids Processing, solids are stabilized through an anaerobic digestion process, and are dewatered with centrifuges. Dewatering is a vital volume reduction process that benefits solids conveyance, and typically produces two streams: cake solids and filtrate. Polymer demand is a measure of the amount of chemical conditioner required to dewater the biosolids efficiently. Currently MWRD manages 29,000 dry metric tons (tonnes) of biosolids annually at the RWHTF. Cake solids are hauled and land applied for beneficial reuse. Optimizing the dewatering process is an important consideration for MWRD because the degree of cake dryness achieved affects related O&M costs (Table 1.1).

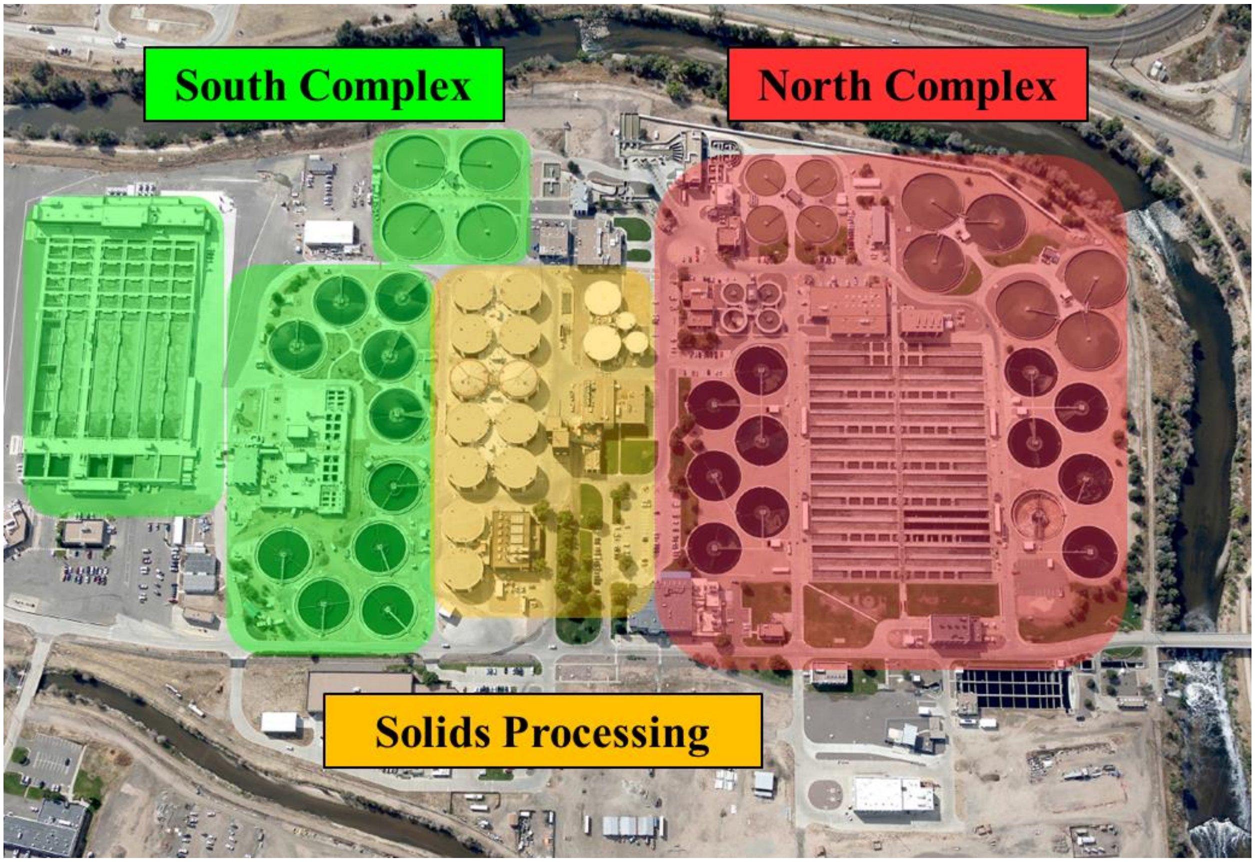

Figure 1.1 Plan view of Metro Wastewater Reclamation District's Robert W. Hite Treatment Facility located in Denver, Colorado.

Table 1.1 Estimated impact of cake solids improvements on annual treatment, hauling, and land application costs for MWRD's Robert W. Hite Treatment Facility.

Cake TS (%)	18	20	22	24
Hauling/Land App	$ 4,031,977	$ 3,549,763	$ 3,316,589	$ 3,226,443
Polymer	$ 2,244,565	$ 2,694,699	$ 3,144,832	$ 3,594,965
Total	**$ 6,276,543**	**$ 6,244,462**	**$6,461,421**	**$ 6,821,408**

The RWHTF is nearing its buildout capacity in Solids Processing, and within the next ten years, upgrades and expansion to anaerobic digestion are required to address the increased loading associated with a growing population. Additionally, stricter nutrient removal regulations pushed MWRD to upgrade biological nutrient removal processes in both the North and South

Complex to include biological phosphorus removal (Bio P). However, full-scale evaluations of Bio P demonstrated that this process upgrade had observed impacts on dewaterability, increasing chemical costs by approximately $1 million to maintain a 21% targeted cake solids (Figure 1.2). Another consequence of Bio P was the acceleration of nuisance struvite accumulation throughout Solids Processing, causing serious impacts on O&M (Figure 1.3).

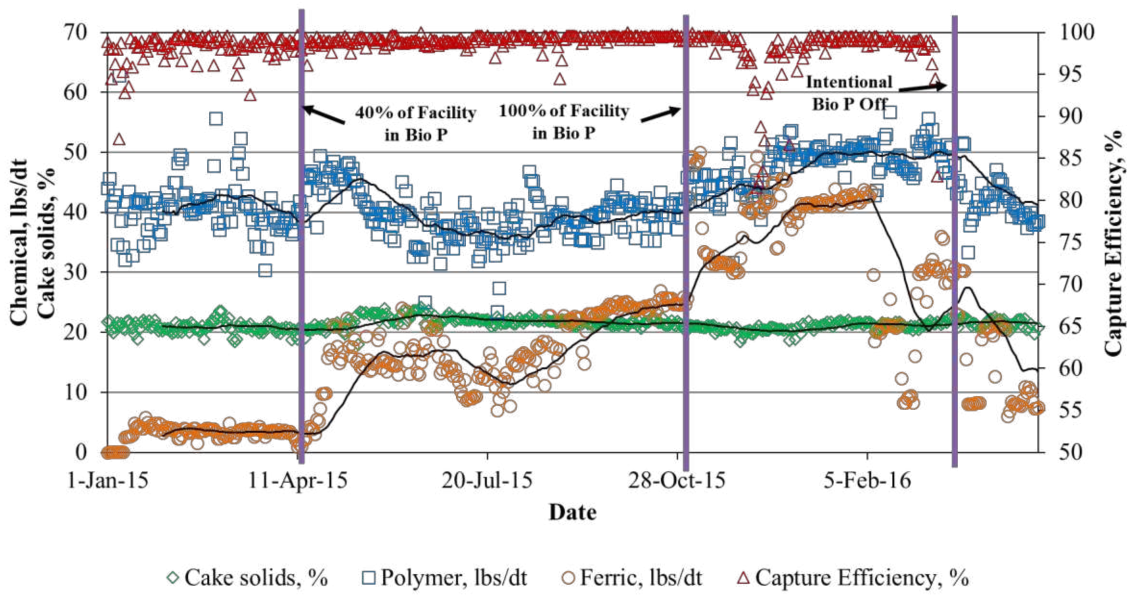

Figure 1.2 Chemical use impacts at the RWHTF during full-scale evaluation of Bio P.

Figure 1.3 Struvite precipitation that has clogged a digester effluent line at the RWHTF.

A current focus of MWRD is evaluating process upgrades to address capacity issues with Solids Processing at the RWHTF, and to reduce nuisance struvite accumulation occurring within solids processing. However, given the experience MWRD has with the impacts process changes can have on dewaterability, utilizing laboratory methods to assess dewaterability was an important component to include in process upgrade evaluations. The research described in this thesis was part of the larger study performed by MWRD investigating the impacts of Secondary Solids Release and Thermal Hydrolysis solids pre-treatments. Secondary Solids Release was investigated by MWRD to assess whether nuisance struvite accumulation occurring within anaerobic digestion was reduced, and Thermal Hydrolysis was examined to determine impacts on anaerobic digester capacity.

The ability to utilize laboratory methods to assess the impacts of process changes on cake solids and polymer demand has the potential to inform MWRD planning and decision-making efforts, as well as process optimization efforts around dewaterability. Typical assessment methods for dewaterability are capillary suction time (CST) and specific resistance to filtration (SRF) (Feng et al., 2008; Fontmorin & Sillanpää, 2015; Murugesan et al., 2014; Yu et al., 2009). However, these rate-based methods of water release do not measure cake solids content (Ngwenya et. al, 2017). Laboratory-scale dewaterability assessment methods that can provide data on cake solids and polymer demand would be valuable to a WRRF. Given the complex nature of biosolids and unique properties of water and solids interactions, finding and developing these methods have been a challenge for several decades. However, methods such as Modified Centrifuge Technique (MCT), Drying Rate, Pneumatic Press (Press), and Limit Dryness Test (LDT) have been proposed because they provide cake solids data. Lab-based methods to determine an optimal polymer demand (OPD) typically are the previously mentioned CST and

SRF. Other methods that can quantify OPD are charge demand and other water release rate methods such as LDT. For this research, OPD methods CST, charge demand, and LDT were used in conjunction with cake solids methods Drying Rate, MCT, Press, and LDT to test two hypotheses: (1) lab-based methods that provide cake solids and OPD data can capture differences stemming from process changes, and (2) cake solids and OPD will not differ across methods if the same solids source and polymer are used. The lab-based dewaterability assessments analyzed in this thesis could provide WRRFs recommendations on the applicability of these methods and their usefulness for infrastructure planning and process optimization.

CHAPTER 2: BACKGROUND

2.1. Wastewater Solids

Water Resource Recovery Facilities (WRRFs) manage solids of varying types from different unit treatment processes: primary solids from primary treatment, secondary solids from activated sludge processes, and stabilized solids that typically come from anaerobic or aerobic digestion processes. The physicochemical characteristics of each type of solids differ, and these differences have been reported to have varying impacts on the extent of dewaterability. The concentration of solids recovered from primary and secondary clarifiers are typically within the 1-2% total solids (TS) range, and are composed of varying constituents including, but not limited to, nutrients, organics, inorganics, metals, proteins, humics, and polysaccharides. Stabilized solids concentrations vary because they are dependent on the feed concentration, the type of treatment applied, and the degree of solids destruction that occurs. To minimize the amount of land area required to convey and equalize these solids streams, WRRFs reduce the volume through mechanical thickening or dewatering. Chemical conditioning of solids can increase the amount of water that can be removed. The dynamic nature of wastewater can make it challenging for WRRFs to maintain target solids concentrations because the amount of chemical conditioner required to achieve these targets is constantly fluctuating.

2.2. Solids Characteristics

Wastewater solids are typically biological in nature, making them complex and challenging to manage. The characteristics of these biosolids can contribute to the extent to which they can be dewatered mechanically. The following sections provide background into the composition and nature of wastewater solids.

2.2.1. Extracellular Polymeric Substances

Microbiological cells in wastewater treatment systems are typically aggregated together to form larger flocculated particles (bio-floc or floc). The floc is held together by microbially produced polymers known as extracellular polymeric substances (EPS). EPS is a complex heterogeneous mixture of microbial secretions, the internal cellular matrix of microbes stemming from cellular lysis and hydrolysis, and other compounds such as bound water and ions, and adsorbed organics and macromolecules (Nielsen & Jahn, 1999). The composition of EPS is highly variable and is dependent on many complex interactions between microbial metabolism, treatment process type, operating conditions, and wastewater characteristics. Additional variability in EPS measurements stems from differences in the extraction methods themselves (Nielsen & Jahn, 1999). EPS composition and presence can affect the physicochemical properties of biosolids (Nielsen & Jahn, 1999; Sheng et al., 2010). The magnitude of EPS in biosolids can account for up to 80% of the total mass (Neyens et al., 2004). EPS is hydrophilic and its water-loving characteristics have been reported to be associated with the protein and polysaccharide components of the EPS. The water binding mechanisms of the protein and polysaccharide components are electrostatic interactions and hydrogen bonding (Neyens et al., 2004), and these interactions of EPS and water greatly influence the dewaterability properties of solids. EPS is typically fractionated into free or soluble EPS and bound EPS, which is further fractionated into loosely bound EPS and tightly bound EPS (Figure 2.1). An increase in the soluble EPS component has been reported to be correlated with an increase in polymer demand of solids (Novak et al., 2003).

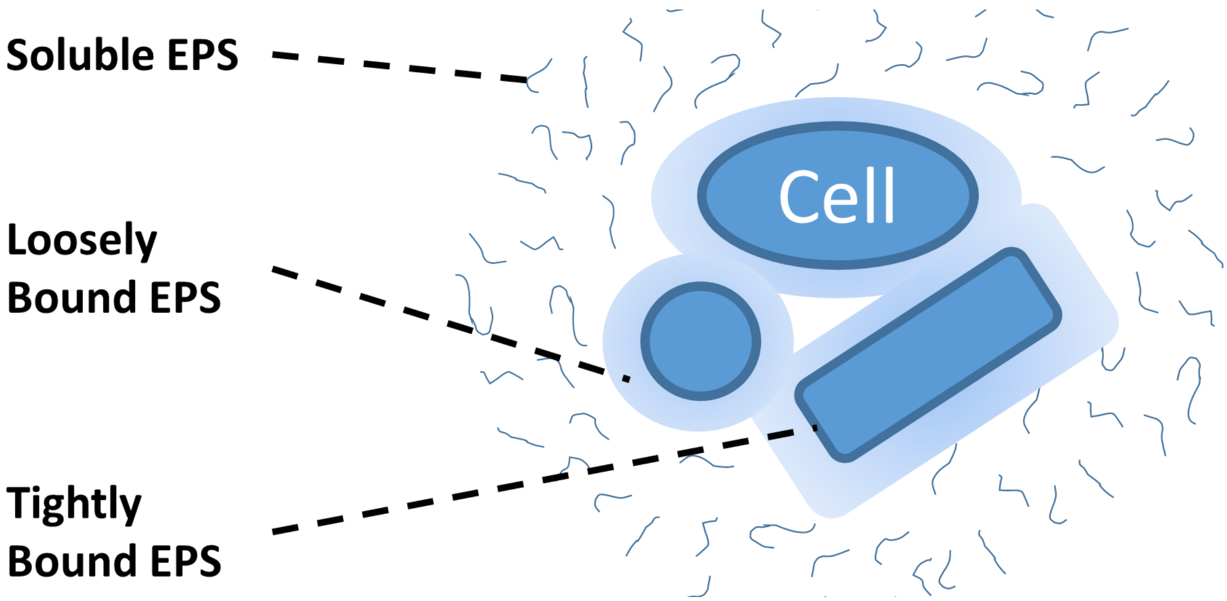

Figure 2.1 EPS fractions as defined by Nielsen & Jahn (1999). Note that EPS as defined here is a mix of what is produced by the microorganisms, the products of lysis and hydrolysis, and other organics present in the wastewater.

2.2.2. Water Fractions and Distribution in Digested Solids

Water unequivocally plays a key role in dewatering. The ultimate goal of dewatering is to both recover water and reduce the volume of the solids. Separating water from biologically and chemically complex slurries continues to be a challenging operational task for WRRFs due to water's interactions with solubilized chemical compounds and ions, microorganisms, colloids, and both organic and inert solids. Depending on its physical state or location on or within biological solids, water has been characterized in the following categories: *free water, interstitial water, vicinal water, intracellular water, and waters of hydration*.

Free water (or bulk water) is defined as the water in suspension that does not interact with suspended solids. Interstitial water has been broadly defined as the water that can get trapped in small "pockets" between aggregated biological cells (Vesilind, 1994). *Intracellular water* is water located on the interior of microbial cells (Despa, 2005; Erdincler & Vesilind, 2000; Kopp & Dichtl, 2001; Lee, 1996). *Vicinal water* is defined as the water that is tightly bound to surfaces, which can include water trapped inside the cells (Erdincler & Vesilind, 2000; Vaxelaire & Cezac, 2004). Waters of hydration refer to water that has been chemically bound such as magnesium ammonium phosphate hexahydrate ($MgNH_4PO_4 \cdot 6H_2O$, MAP, or struvite), which is a mineral that is commonly found in anaerobic digester effluent and was a major focus of the pilot study from which this research stemmed (Wisdom et al., 2018). The term bound water is an operationally defined term dependent on the method of measurement, but has also been used to widely characterize interstitial water, vicinal water, and waters of hydration (Vaxelaire & Cezac, 2004; Vesilind, 1994).

Free water, also referred to as drainable or bulk water, is essentially water that would freely separate from a solids slurry if gravity filtered with no additional force other than gravity applied, and assuming no evaporation occurred. The water that remains in the gravity separated solids is the remaining fractions of water that can and cannot be removed mechanically. Mechanically removable water includes water that through cationic conditioning and subsequent mechanical expression can be removed. This suggests that the fraction of water that cannot be removed mechanically are waters of hydration, intracellular water, vicinal water, interstitial water, and other types that have not been quantified (Figure 2.2).

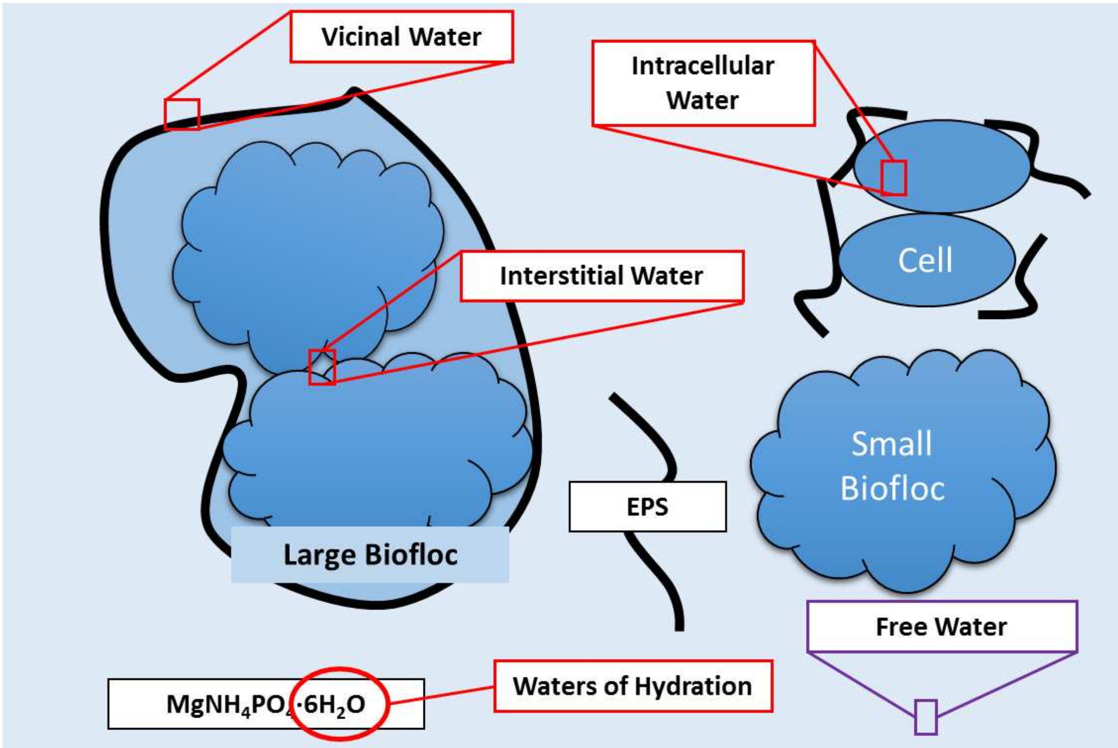

Figure 2.2 Distribution of water in a solids slurry.

2.2.3. Cation Content of Biosolids

The cation content of solids can be changed by biological treatment processes such as biological phosphorus removal (Bio P) and anaerobic digestion, and cations have been reported to influence the dewatering characteristics of solids. Bio P is a treatment process used at WRRFs to remove phosphorus from the liquid stream. Bio P is achieved by creating anaerobic and aerobic environments in activated solids processes to leverage the metabolism of phosphorus accumulating organisms (PAO) to release intercellular phosphorus in the anaerobic environment, followed by luxury phosphorus uptake under aerobic conditions (Oehmen et al., 2007). The phosphorus is removed from the process through secondary solids wasting. The wasted solids are typically sent for further treatment in anaerobic digestion processes. Cations such as magnesium, and potassium have been reported to be co-transported in and out of the PAO cellular mass during PAO release and uptake metabolism to maintain charge balance (Arvin & Kristensen, 1985). During the anaerobic digestion process, biological release of phosphorus and cations occurs, with additional release occurring from cellular lysis. Both monovalent and divalent cations, such as magnesium and potassium, have been observed to influence the dewaterability of secondary solids. Divalent cations are theorized to form bridges between floc particles, cells, and EPS, thereby releasing some of the water associated with the floc particles, thus increasing the cake solids concentration (Figure 2.3) (Higgins & Novak, 1997; Xing et al., 2014). A monovalent:divalent cation ratio of 2 or less was observed to be associated with higher cake solids and reduced polymer consumption in the dewatering of secondary solids (Higgins & Novak, 1997; Higgins et al., 2004).

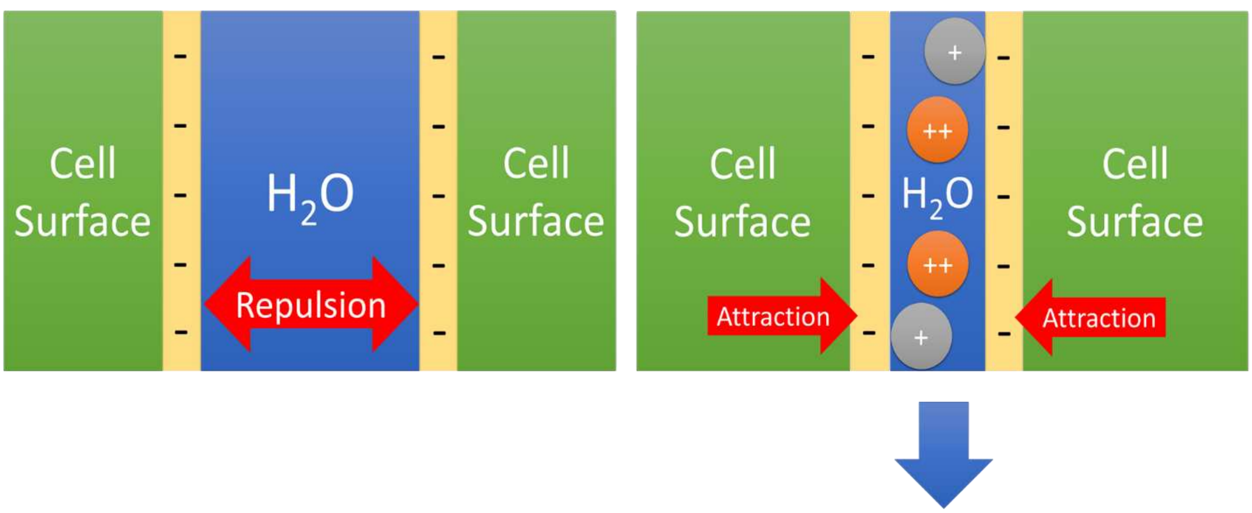

Figure 2.3 Divalent cations (orange circles) create an attraction between cell surfaces freeing otherwise immobilized water.

For anaerobically digested solids, one recent study reported that the addition of monovalent cations (potassium and sodium) to the digester feed led to higher final cake solids (Ngwenya, 2017). The theory proposed was that the addition of monovalent cations frees up water that is bound to the negatively charged sites on EPS, thus increasing the amount of recoverable water. While the exact mechanisms are still unknown, cations have been observed to influence the dewatering characteristics of solids. Research in this area continues to this day.

2.3. Solids Pre-Treatments

Process changes can impact dewatering characteristics of solids. Facilities that use Bio P have reported increased chemical conditioning requirements to maintain target cake solids. Biological phosphorus removal was observed to coincide with a decline in the dewatering characteristics of both secondary and anaerobically digested solids (Cavanaugh et al., 2016; Higgins et al., 2014). Post digestion phosphorus recovery was reported to increase cake solids and reduce polymer consumption compared to a non-recovery option (Wisdom et al., 2017). Solids pre-treatment technologies such as thermal hydrolysis drastically change the nature of the solids, thus affecting both polymer demand and final cake solids (Barber, 2016). For this research, an overarching objective was to estimate the full-scale impact on final dewaterability from solids pre-treatments Secondary Solids Release and Thermal Hydrolysis.

Secondary Solids Release is a biological solids pre-treatment process that leverages the anaerobic PAO metabolism to change the phosphorus and cation characteristics of the solids prior to the anaerobic digestion process. The process takes waste activated sludge (WAS), or a combination of WAS and primary solids, and holds the solids under anaerobic conditions. As discussed in Chapter 2.2.3, PAOs under anaerobic conditions are reported to release intercellular phosphorus and cations from the solids to the liquid stream. The pre-treated sludge solids are thickened prior to sending to anaerobic digestion. The process has been reported to reduce the phosphorus and cation content of the solids feeding anaerobic digestion (Schauer et al., 2009), thus possibly impacting cake solids and polymer demand.

Thermal Hydrolysis Process (THP) is a solids pretreatment process typically located prior to digestion. The process has been used by WRRFs to increase the capacity of existing solids processing infrastructure by changing the nature of the solids feed to digestion. The application of temperatures above 121 ˚C, and rapid pressure drops from ranges of 3 to 6 bar down to ambient pressure, increase the rate of solids hydrolysis prior to anaerobic digestion (Barber, 2016). In addition to changing other characteristics of the solids, THP increases post-digestion cake solids content by approximately 10 percentage points (Barber, 2016; De Clippeleir et al., 2016), and increases the soluble chemical oxygen demand, ammonia, and cation content (Nicholson et al., 2016). It has been reported that the EPS content is reduced during the THP process (Neyens et al., 2004).

2.4. Dewaterability Assessment Methods

Key operating parameters for solids management for WRRFs are polymer demand and cake solids. Polymer demand is a measure of the amount of polymer, a chemical solids conditioner, required to destabilize the solids to form large floc particles, which increases the rate at which water is released from the solids matrix during dewatering. Cake solids are the dewatered solids that are produced during dewatering. Determining an optimal polymer demand at bench-scale can be done using methods based on solids filterability. Filterability and dewaterability of biosolids are often used to describe the same phenomena, but their meanings are different. Filterability of biosolids describes the rate that water releases from the solids matrix. Dewaterability has a broader meaning that encapsulates the kinetics of water released from the biosolids, the dryness of the final dewatered biosolids, and the amount of dewatering conditioner required to achieve a target dry solids content. Commonly used filterability assessment methods for determining an optimal polymer demand are capillary suction time

(CST) and specific resistance to filtration (SRF) (Feng et al., 2008; Fontmorin & Sillanpää, 2015; Murugesan et al., 2014; Yu et al., 2009). The CST method is a quick and easy way to assess the rate at which water can be released from a solids matrix, and is useful as an indicator of the efficacy of conditioning. However, the CST measurement itself is specific to characteristics of the biosolids being tested, and the instrument and chromatographic paper being used, such that CSTs determined in one lab have been reported to be incomparable to CSTs determined in another lab (Vesilind, 1988). The SRF test relies on the theory of filterability as a way to evaluate the kinetics of dewatering (Christensen & Dick, 1985). However, it is very time-consuming to perform, and from the perspective of a municipality, the outputs from the method are not easily relatable to standard operating control measures (Novak & Knocke, 1985). Both the CST and the SRF methods can reveal polymer doses that maximize the rate at which water can be removed from the solids matrix. However, these methods are limited because they are not designed to measure final cake solids content (Ngwenya et al., 2017). For this research, the selected dewaterability assessment methods have been reported to produce cake solids from conditioned solids, and the conditioning was assessed using traditional methods such as CST and other methods described in subsequent sections. The following sections provide background on the different dewaterability assessment methods used for this research.

2.4.1. Drying Rate and Polymer Charge Neutralization

Cake solids can be derived from water evaporation rate curves. This method of assessing final dry solids content is known as the drying rate method. A small pre-centrifuged sample of wet solids is placed in a climate-controlled (constant air flow and temperature) testing chamber, and dried slowly over a 24 – 48 hour time frame while the evaporation rate and water content are recorded (Figure 2.4 - Left graph). The sample is dried slowly at low temperatures, because the theory is that the slope of the evaporation rates correlate with how water is bound to the solids (Kopp & Dichtl, 2001; Vaxelaire & Cezac, 2004). The linear portion of the drying rate versus moisture content graphed on an arithmetic scale is theorized to be associated with the free water content (Figure 2.4 - Right graph), and the maximum cake solids content is determined from the point of inflection (mark A on the graph). Per the method, this point of inflection is referred to as the dried solids at point A (DS(A)), and is theorized to be the maximum mechanically achievable cake solids using current dewatering technology (Kopp & Dichtl, 2001; Tsang & Vesilind, 1990; Vaxelaire & Cezac, 2004).

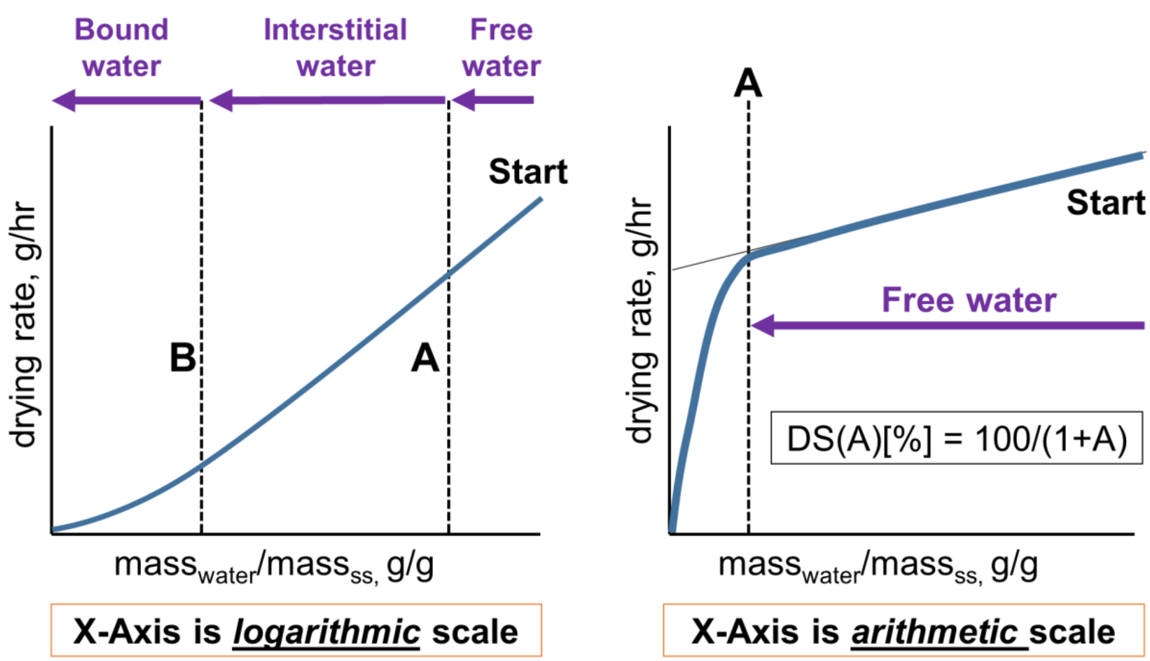

Figure 2.4 Thermogravimetric cake solids analysis. Left graph is drying rate versus moisture content with the X-axis graphed logarithmically. Right graph is the same data with X-axis graphed arithmetically. Figure adapted from Kopp et al. (2016).

A modified version of the drying rate method used in this research was developed by Kopp & Dichtl (2001) and is now performed at Klarenlagen Beratung Kopp (KBKopp) laboratory in Lengede, Germany. Polymer conditioners are not used during the drying rate test because an increase in polymer from 0 to 12 kg/metric ton (tonne) did not significantly change the derived cake solids point for three different solids types (Kopp & Dichtl, 2001). A similar observation was reported by Tsang and Vesilind (1990) when they examined the impact of polymer doses from 0 – 14.3 kg/tonne on water distribution as determined by the drying rate method. The fraction of water that separated during centrifugation and the remaining free water content as determined by the drying rate did change. With increasing polymer dose, the amount of water released during centrifugation increased, and the remaining free water content decreased. However, the total free water content (sum of water separated during centrifugation plus the remaining free water as determined by the drying rate) did not change. They observed no changes to the interstitial, surface, or bound water fractions (Tsang & Vesilind, 1990). In research by Smollen (1990), with increasing polymer dose, a decrease in the immobilized water and an increase in chemically bound water were observed, but no comments about changes in free water were made because all of the free water was assumed to be separated during centrifugation (Smollen, 1990). These results disagree with observations from Tsang & Vesilind (1990) and Kopp & Dichtl (2001). Although the impact of polymer on the moisture content may differ based on drying rate methods tested in these studies, the authors agree that polymer does increase the rate at which water is released from the solids matrix. This could explain why the conditioned solids only require a shorter retention time to achieve target dry solids content in full-scale centrifuges.

When using the drying rate test to determine cake solids content, separate methods are used to determine polymer demand. For this research, the lab that performed the drying rate test used the charge demand method to determine an optimal polymer demand (OPD). Determining polymer demand using charge demand is based on surface chemistry. The theory is that biological surfaces, and byproducts from microbial metabolism such as EPS are negatively charged. A cationic polymer is added to a solids slurry to neutralize the electrostatic forces binding water to the biological products, allowing the water to be released from the solids particles. The total polymer required to neutralize the charge is defined as the OPD (Kopp, Yoshida, & Forstner, 2016).

2.4.2. Modified Centrifuge Technique

The modified centrifuge technique (MCT) combines polymer conditioning with laboratory centrifuge dewatering in modified centrifuge cups to produce cake solids. The method was developed by Dr. Matt Higgins at Bucknell University. For conditioning, mixing intensity is controlled by establishing a calibrated mixing speed and time that minimizes the CST of a baseline solids sample at a fixed polymer dose. The CST is used to determine the conditions that maximize the velocity at which water is removed from the solids. After mixing is calibrated, the polymer dose is varied until a dose is found that minimizes the CST (Ngwenya et al., 2017). The CST works by measuring the rate of filtrate capillary suction from the conditioned slurry. The solids form a layer on the bottom of the CST funnel that basically resists the flow of water. Better conditioned solids have been associated with low CSTs because conditioning is suggested to increase floc size, which frees up water immobilized in the solids matrix, and thus water faces less resistance through the solids (Chen, Lin, & Lee, 1996; Scholz, 2005). The CST is the time it takes for the water to travel the distance in the chromatography paper between electrodes located in the frame plates of the CST apparatus. The polymer dose resulting in a minimum CST is defined as the OPD.

The OPD-conditioned sample is then dewatered in modified centrifuge cups. The conditioned sample is free drained through sieves lined with belt filter press material, and then the solids sample is loaded into modified centrifuge cups. The modified centrifuge cups house an elevated platform lined with belt filter material. The solids sample is dewatered for two, 2-minute intervals at increasing centrifugation, and then centrifuged at high speeds for 10 minutes. The final cake that is produced is measured for TS (Ngwenya et al., 2017). Figure 2.5 shows a schematic of the entire procedure.

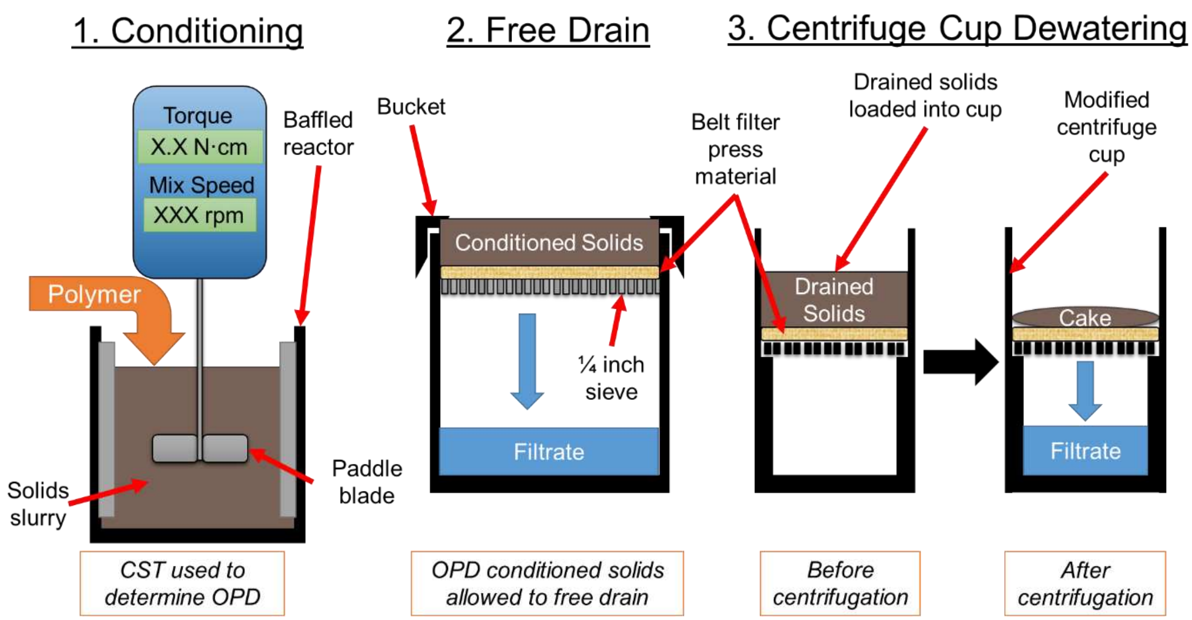

Figure 2.5 Overview of the entire MCT dewaterability assessment method.

Dewatering efficiency, or the cake solids produced, through the MCT was reported to resemble full-scale belt filter press solids (Ngwenya et al., 2017; To et al., 2016). In an optimally conditioned sample, factors that have been reported to affect the final cake solids from the MCT are dry solids loading to the centrifuge cups, centrifugation intensity, and mixing the sample during the high-speed centrifugation period (Ngwenya et al., 2017; To et al., 2016).

2.4.3. Press Method

The Press method produces cake solids from conditioned solids by squeezing the sample through two steel plates at a set pressure of 1.4 bar for 5 minutes. The steel plates are lined with 300-micron filter screen mesh material that allow the filtrate to pass through, leaving behind cake solids (Figure 2.6). Per the Press methodology, polymer doses are varied, and the conditioned sample is dewatered. An optimal polymer dose is determined through qualitative examination of polymer curves (graphs of cake solids vs. polymer).

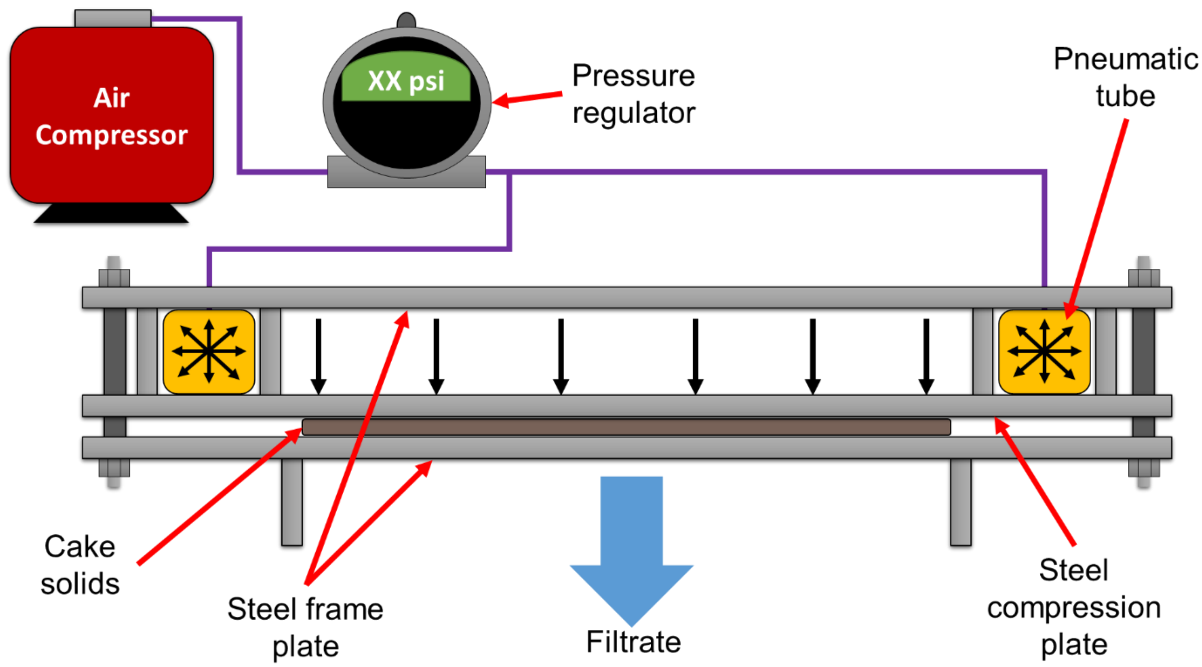

Figure 2.6 Schematic of pneumatic press used to produce dewatered cake - Image adapted from schematic in Bensich and Neethling (2015).

2.4.4. Limit Dryness Test

The Limit Dryness Test (LDT) dewaters conditioned samples in a Fournier (Quebec, Canada) LDT Apparatus (Figure 2.7). The LDT apparatus is pressurized to 6.8 bar and held for 10 minutes. The air pressure applies a force on the surface of a conditioned sample, and the water is forced through belt filter press material that lines the bottom of the apparatus. The mass of water captured is recorded over time. Polymer doses are varied, and water release curves are evaluated to determine the optimal polymer dose. The minimum time required to reach 60% percent water capture is reported to be the optimum polymer dose (green line in Figure 2.8), under-conditioned samples do not reach a water capture of > 60% over a 10-minute period (red line in Figure 2.8), and over-conditioned samples require longer time to reach the 60% water capture threshold (Ginisty et al., 2014; Schermerhorn et al., 2015). After dewatering, the cake solids are collected from the LDT apparatus and analyzed for TS.

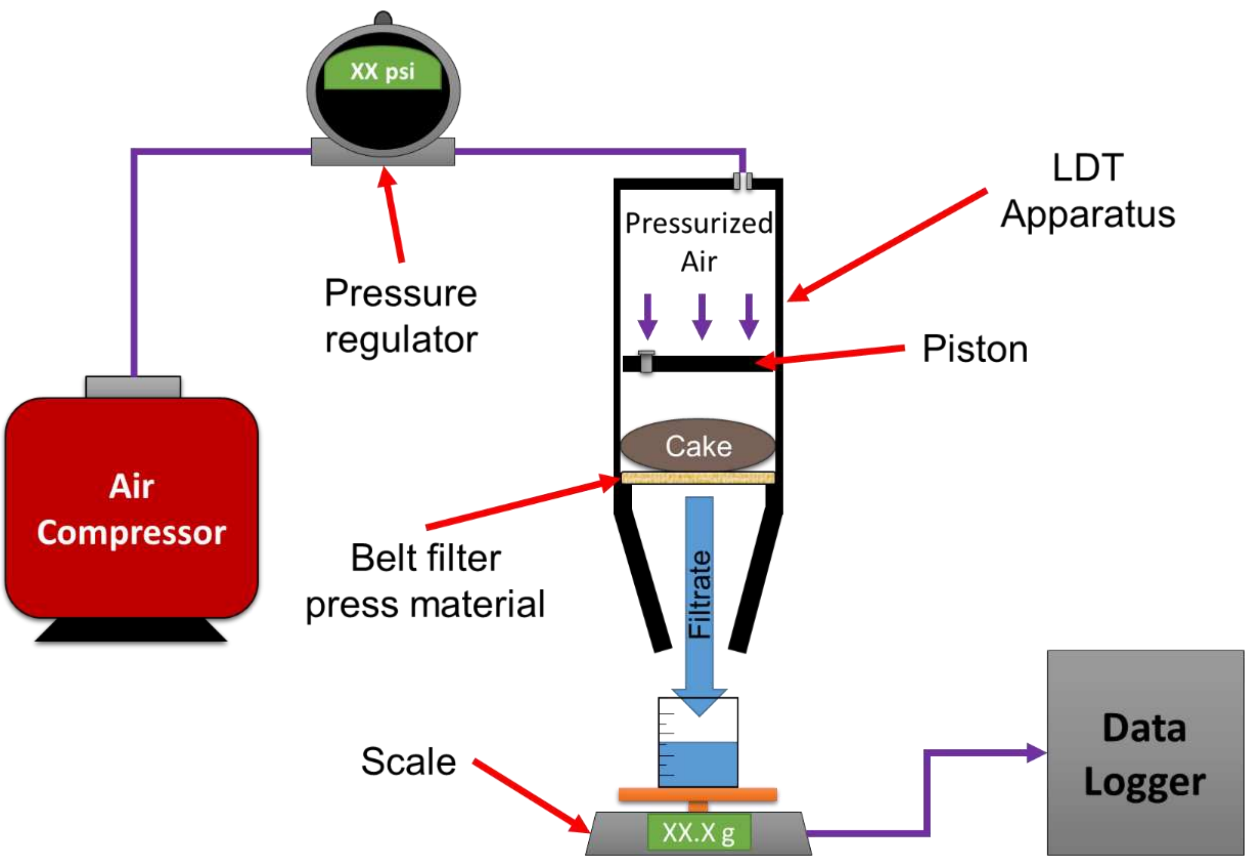

Figure 2.7 Schematic of LDT apparatus.

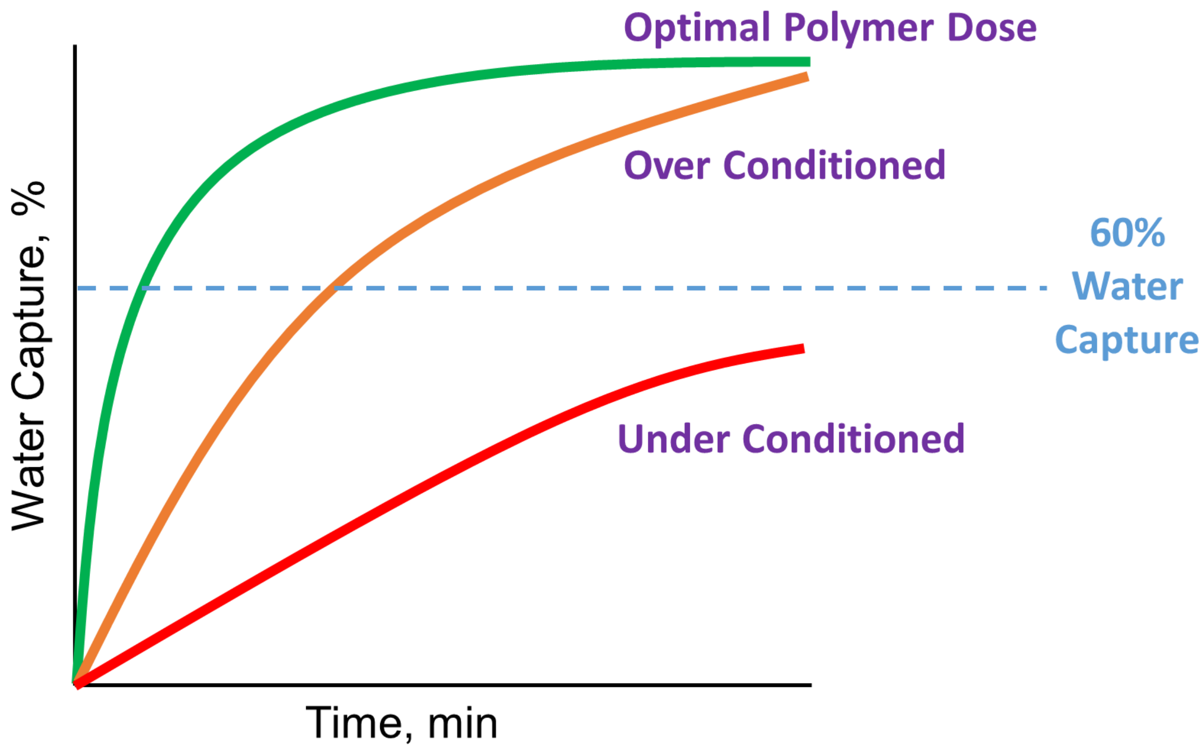

Figure 2.8 Characteristic polymer dose response curves produced via LDT.

CHAPTER 3: METHODS AND MATERIALS

3.1. Secondary Solids Release Anaerobic Digester Pilot

Metro Wastewater Reclamation District (MWRD) operated a pilot anaerobic digester system from January 18, 2017 to June 10, 2017. Two trains, a Secondary Solids Release and Control train, were operated. The pilot anaerobic treatment trains were fed mixtures of thickened primary (PS) and waste activated solids (WAS). For the Control train, the WAS was allowed to settle in the Solids Thicken Reactor for 0.5 to 2 hours, depending on settleability of solids, and the supernatant was wasted. For the Secondary Solids Release train, the WAS was allowed to react under anaerobic conditions for 48 hours to strip phosphorus and cations from the solids, and then the supernatant was wasted to produce stripped WAS (SWAS).

The effluent WAS from the Solids Thicken Reactor on the Control Train had average total solids (TS) = 0.9%, while the SWAS on the Pre-Treatment train had average TS of 1.2%; a complete process flow diagram is provided in Figure 3.1. The WAS and SWAS were each combined with Clarifloc CE-1817 (Polydyne Inc., Georgia, US) polymer at a dose of 14 kg/tonne, and dewatered in a PWTech Volute ES-131 (Rosedale, MD) dewatering screw press. The dewatered solids (cake) produced from the screw press averaged around 11% for the Secondary Solids Release train, and around 10% for the Control Train. The cake was diluted using the filtrate produced from dewatering. After dilution, the thickened SWAS (TSWAS) averaged TS = 4.1% and the thickened WAS (TWAS) TS = 4.0%. For all digester feedings, 1.0 g of solids from both feed streams was loaded into an Ohaus MB35 Moisture Analyzer (Greifensee, Switzerland) to determine Primary to Secondary Mass Loading (Pri:Sec) ratios to each train. The thickened secondary solids were combined with primary solids (average TS = 6%) and fed to homogenizers for mixing and to control feed to each digester. Each digester was continually mixed to ensure homogeneity, and fed 5.7 L six times per day for a total of ~34.2 L/day; the SRT was maintained at 17.8 days via overflow wasting of solids.

A higher fraction of primary solids was fed to the digester during the period of January 18 – May 6, 2017 (Phase 1a). The Pri:Sec ratio was 2.3 g/g for the Secondary Solids Release train and 2.4 g/g for the Control train. A higher fraction of WAS was fed to the digester pilot during the period of May 7 – June 10, 2017 (Phase 1b). The Pri:Sec ratio was 1.1 g/g for both the Secondary Solids Release train and Control train. During Phase 1b, the Control train mimicked full-scale digester operation. The digester effluent (digestate) was sampled and analyzed for solids content, nutrients, and metals on a weekly basis by the MWRD analytical laboratory per standard methods.

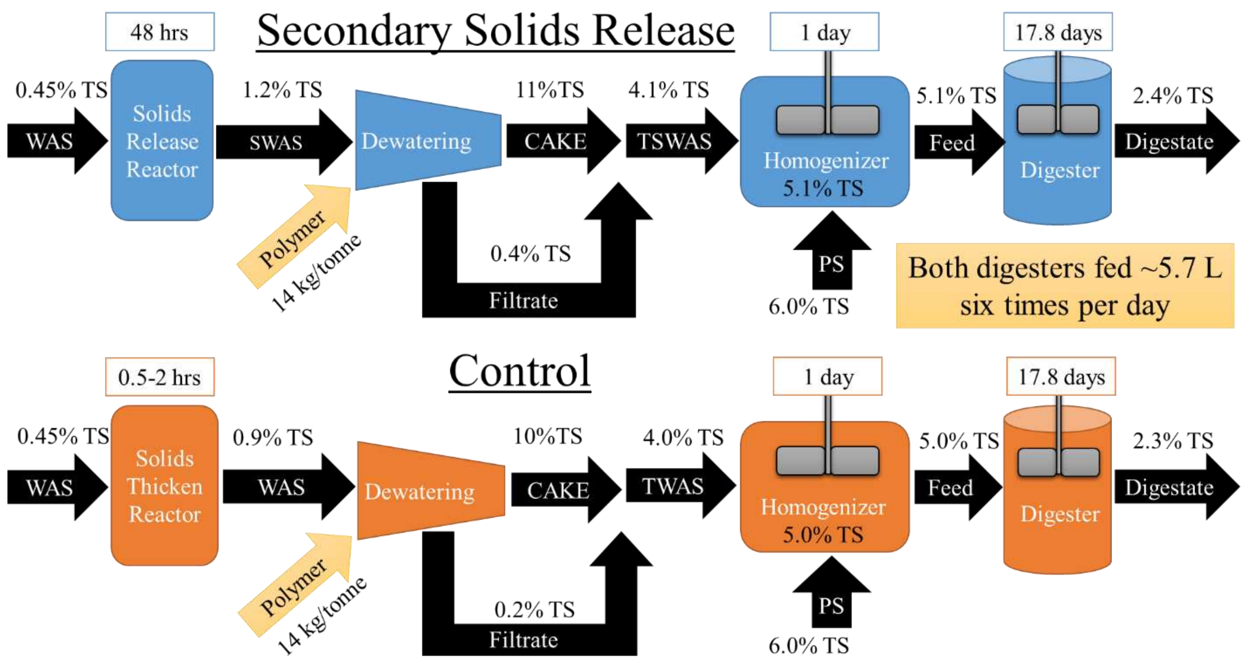

Figure 3.1 Process flow diagram of the digester pilot system operated from January, 2017 to June, 2017. Phase 1a was operated from January 18, 2017 – May 6, 2017. Phase 1b was operated from May 8, 2017 – June 10, 2017.

3.2. Thermal Hydrolysis Anaerobic Digester Pilot

From July 24, 2017 to January 5, 2018, the Secondary Solids Release anaerobic digester pilot was operated in a modified configuration that incorporated a thermal hydrolysis process (THP) pilot system (Biotheylis by Kruger/Veolia, Cary, NC). One of the goals of the THP pilot was to evaluate the impacts of thermally hydrolyzed secondary solids pre-digestion on polymer demand and cake solids post anaerobic digestion. Two different THP pre-treatment schemes followed by anaerobic digestion were evaluated: WAS only Thermal Hydrolysis Process to Anaerobic Digestion (WAS THP-AD in Phase 2a), and WAS and Primary Solids Thermal Hydrolysis Process to Anaerobic Digestion (WAS+Pri THP-AD in Phase 2b).

For the WAS THP-AD pilot (Phase 2a), the WAS (TS = 0.5%) was allowed to settle in the Solids Thicken Reactor for 0.5 to 2 hours, depending on settleability of solids, and the supernatant was wasted; a complete process flow diagram is provided in Figure 3.2. The thickened WAS was held under anaerobic conditions for 48 hours. The thickened SWAS (TSWAS) was combined with Clarifloc CE-1817 (Polydyne Inc., Georgia, US) polymer at a dose of 14 kg/tonne and dewatered using a PWTech (Rosedale, MD) Volute ES-131 dewatering screw press. The dewatered SWAS (DSWAS) TS averaged around 11%. For the WAS THP-AD train, the DSWAS was thermally hydrolyzed in a batch process at 165˚C and 7 bar for 30 minutes. For all digester feedings, 1.0 g of solids from both feed streams were separately loaded into an Ohaus MB35 Moisture Analyzer (Greifensee, Switzerland) to determine Pri:Sec ratios to each train. The thermally hydrolyzed WAS (TS = 6.5%) was combined with PS (TS = 6.3%) at a targeted Pri:Sec ratio of 1.0 g/g. An average Pri:Sec ratio of 1.5 g/g was achieved and added to a homogenizer for mixing and feed control. For the Control train, the dewatered solids from the screw press were diluted to an average TS = 4.2% using the filtrate produced from dewatering.

The thickened secondary solids were combined with PS (average TS = 6.3%) at a targeted Pri:Sec ratio of 1.0 g/g. The average Pri:Sec ratio of 1.1 g/g was added to the Control homogenizer during the pilot. The differences observed in the Pri:Sec ratio from the targeted loading of 1.0 g/g for both trains were likely due to the challenge of loading a representative sample for TS measurement on the moisture balance prior to feeding, and two different operators handling digester feeding may have contributed to differences observed. Each digester was continually mixed to ensure homogeneity and fed 6.3 L six times per day for a total of ~37.9 L/day; the SRT was maintained at 16 days via overflow wasting of solids.

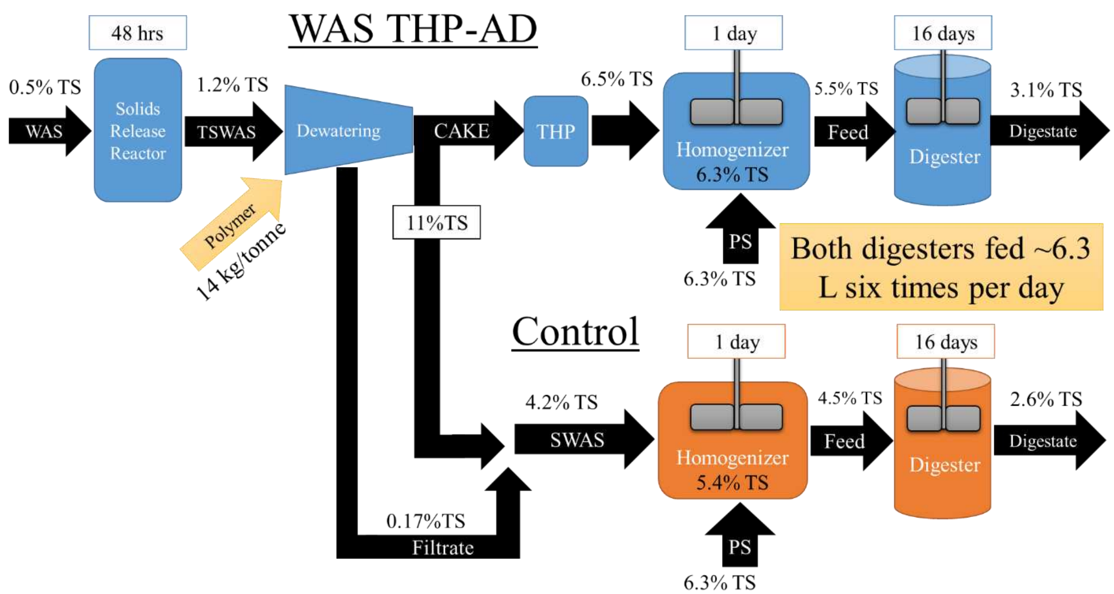

Figure 3.2 Process flow diagram of WAS THP-AD pilot operated from January, 2017 to June, 2017 (Phase 2a).

For the WAS+Pri THP-AD (Phase 2b) train (Test), mixtures of PS (TS = 6.3%) and concentrated WAS (CONWAS) (average TS = 4.5%) were combined in an average Pri:Sec ratio of 1.0 g/g (Target Pri:Sec ratio was 1.0 g/g), and held under anaerobic conditions for 4 hours. A complete process flow diagram for this testing phase is provided in Figure 3.3. The SWAS was combined with Clarifloc CE-1817 (Polydyne Inc., Georgia, US) polymer at a dose of 4 kg/tonne and dewatered using a PWTech (Rosedale, MD) Volute ES-131 dewatering screw press producing a cake with TS = 19%. The undiluted cake was thermally hydrolyzed in a batch process at 165˚C and 7 bar for 30 minutes. The thermally hydrolyzed WAS+Pri mixture (TS = 13.5%) was diluted with tap water (normally used plant water was not available) to an average TS = 8.1% to test higher digester solids loading typically performed with a THP system, before adding to the homogenizer. For the Control train, the goal was to mimic full-scale digester operation. CONWAS (average TS = 4.5%) was combined with PS (average TS = 6.3%) in the Control homogenizer in an average Pri:Sec ratio of 0.9 g/g (target was 1.1 g/g). The differences observed in the Pri:Sec ratio from the targeted loading of 1.0 g/g for both trains was likely due to issues with getting a representative sample for TS measurement on the moisture balance prior to feeding. Each digester was continually mixed to ensure homogeneity and fed 6.3 L six times per day for a total of ~37.9 L/day; the SRT was maintained at 16 days via overflow wasting of solids.

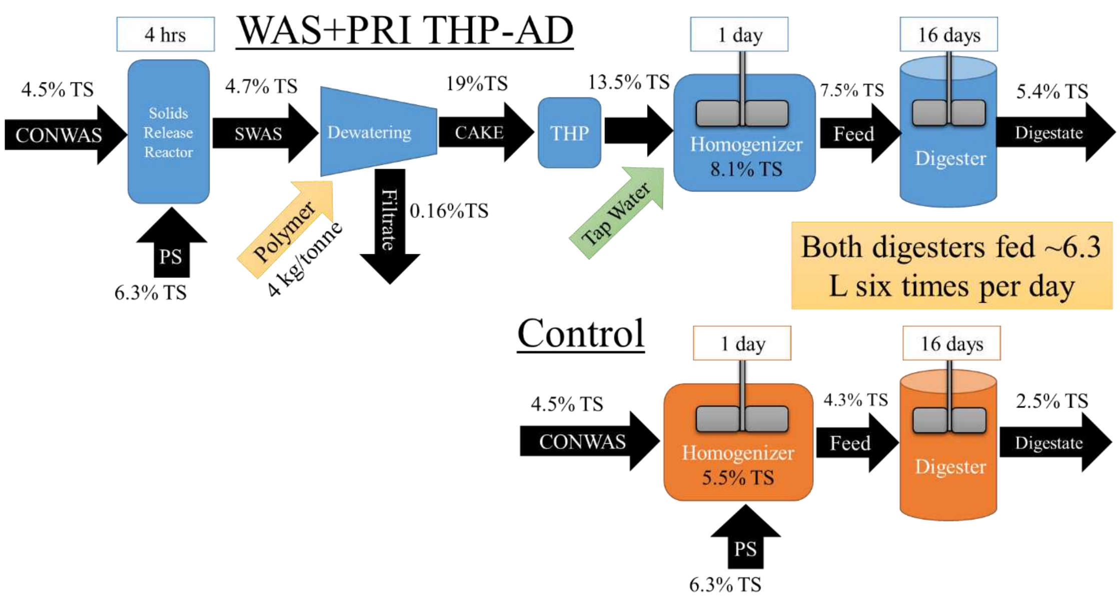

Figure 3.3 Process flow diagram of WAS+PRI THP pilot operated from January, 2017 to June, 2017 (Phase 2b).

3.3. Sample Handling

A total of five sample pairs (Test and Control) of digestate were shipped to KBKopp in Lengede, Germany for analysis by drying rate for cake solids, and charge demand for polymer demand over the entire testing period. On Tuesdays during sampling weeks, 7.5 L of sample were flushed from the sample collection port prior to sampling, and then 10 L of sample were collected and stored overnight at 4˚C with the cap off to allow gas release from the sample. The samples were degassed to prevent them from exploding during shipment. Samples were shipped on ice the following day (Wednesdays) via FedEx and typically arrived within five days. Upon arrival at the KBKopp laboratory, portions of the samples were immediately processed for thermogravimetric analysis. For polymer testing, the remaining sample was stored at 4˚C until polymer testing at which point it was brought back up to room temperature of 20˚C.

A total of eleven sample pairs were shipped to Bucknell University, Pennsylvania for MCT Bucknell analysis over the entire testing period. On Tuesdays during sampling weeks, 7.5 L of sample were flushed from the sample collection port prior to sampling, and then 10 L of sample were collected and stored overnight at 4˚C with the cap off to allow gas release from the sample. The samples were degassed to prevent them from exploding during shipment. Samples were shipped on ice the following day (Wednesdays) via FedEx and usually arrived at the laboratory the following day. Samples were typically processed within a day or two after arrival. Samples were stored in the laboratory at room temperature prior to analysis.

Three methods were performed at the lab at MWRD. For MCT MWRD, a total of fifteen sample pairs were processed. Prior to sampling, 7.5 L of sample were flushed from the sample collection port, and then 8 L of digestate from each anaerobic digester train were collected on Mondays and allowed to cool to room temperature before processing the same day. A total of fourteen samples were processed using the Press method. On Wednesdays, 7.5 L of sample were flushed from the sample collection port prior to sampling, and then 8 L of digestate from both the Test and Control train were collected and allowed to cool to room temperature before analysis the same day. A total of five sample pairs were evaluated with the limit dryness test, which was performed either Thursday or Friday during testing weeks, and only during Phase 1a and Phase 1b. Prior to sampling, 7.5 L of sample were flushed from the sample collection port, and then 8 L of digestate pairs (Test and Control) were collected and allowed to cool to room temperature. Samples were processed the same day. Due to limitations in time and resources during the course of the pilot, unequal sample numbers were processed from each dewaterability method. Sample pair counts and the number of replicates run from each method and pilot are reported in Table 3.1. Figure 3.4 summarizes a typical sample collection week, and indicates when samples were collected and processed from each lab.

Table 3.1 Sample and replicate measurement count for each method for dewatered solids and polymer.

Lab:	**KBKopp**		**Bucknell**		**MWRD**					
Cake Solids/Polymer Method:	Drying/Charge		MCT/CST		MCT/CST		Press/CST		LDT/LDT	
	Sample Pairs	*Reps*	*Sample Pairs*	*Reps*	*Sample Pairs*	*Reps*	*Sample Pairs*	*Reps*	*Sample Pairs*	*Reps*
Phase 1a	2	3*	8	1	5	1	3	1	3	1
Phase 1b	1	3*	1	1	3	1	2	1	2	1
Phase 2a	1	5*	1	4*	4	1	5	1	0	0
Phase 2b	1	5*	1	2	3	1	4	1	0	0
Total in Analysis	**38**		**30**		**30**		**28**		**10**	
** Only single average value reported for polymer demand*										

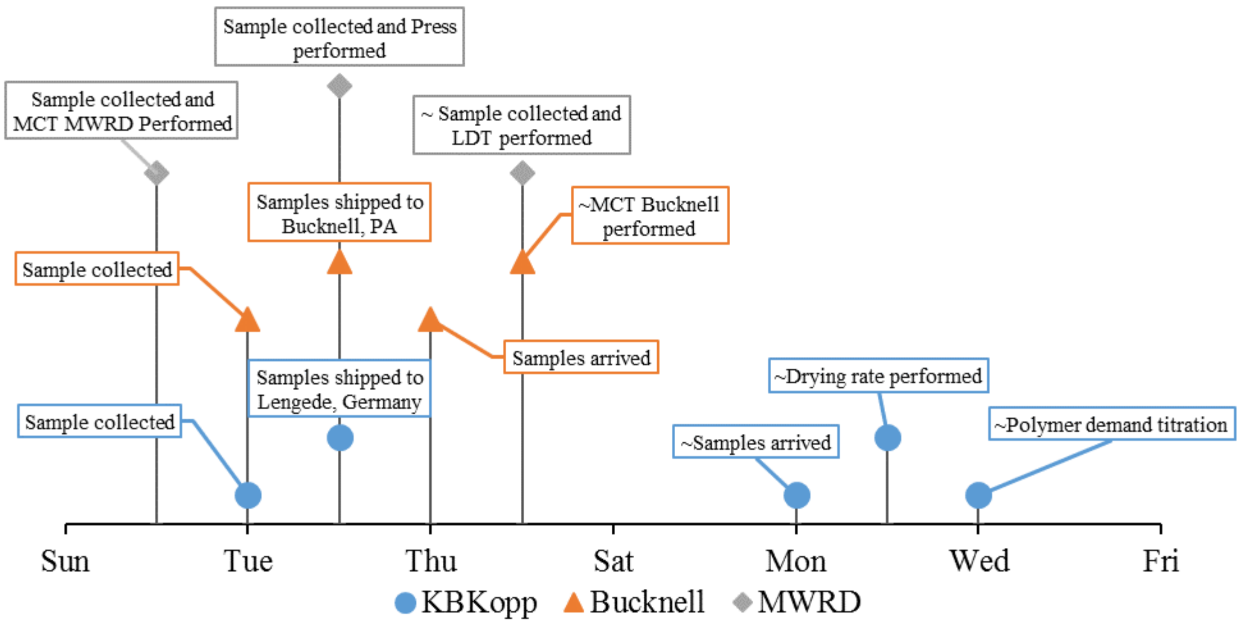

Figure 3.4 Typical timeline for sample handling and processing for each laboratory during sampling events.

3.4. Conditioning and Cake Solids

Cake solids and polymer demand were measured using the methods at the labs listed in Table 3.2. BASF Zetag 8849 FS emulsion polymer is a branched polymer with a high molecular weight, and a very high charge density. The polymer has a specific gravity of 1.03 and an active concentration of 40%. For dewaterability testing at MWRD, a polymer stock solution with an active concentration of 0.36% was made using chlorinated plant water. The chlorinated plant water is secondary effluent that has been dosed with sodium hypochlorite to maintain a residual chlorine concentration of 0.4 mg/L. Chlorinated plant water and polymer was supplied with each set of samples sent to KBKopp and Bucknell University for their respective dewaterability assessments. Methodology used for each method is summarized in Table 3.3.

Table 3.2 Methodology, location, and reference.

Lab	Cake Solids	Polymer Demand	Method Reference
Bucknell University	MCT Bucknell	CST	Ngwenya et al., 2017
KBKopp	Drying rate	Charge demand	Kopp et al., 2016
MWRD	MCT MWRD	CST	Ngwenya et al., 2017
	Pneumatic Press	CST	Benisch and Neethling, 2014
	Limit Dryness Test		Schermerhorn et al., 2015

Table 3.3 Summary of cake solids and polymer demand methods used in this research.

Laboratory	Cake Solids		Polymer Demand		Reference
	Cake Solids Method	*Key Method Details*	*OPD Method*	*Key Method Details*	
Bucknell University	MCT Bucknell	• Centrifuged then mixed at: • 140 x g for 2 min • 350 x g for 2 min • 2000 x g for 10 min • Cake measured via Standard Methods	CST Bucknell	• Polymer dose varied • Flash mix: G ~1000/s for 10 s • Floc mix: G ~100/s for 90 s • Min CST = OPD	Ngwenya et al., 2017
KBKopp	Drying Rate	• Sample dried slowly • Slope of drying rate equated to free water content • Cake solids derived graphically	Charge demand	• Cationic polymer titration • Streaming current detector used to measure charge • Point of zero charge = OPD	Kopp et al., 2016
MWRD	MCT MWRD	• Centrifuged then mixed at: • 200 x g for 2 min • 490 x g for 2 min • 1970 x g for 5 min • 1970 x g for 5 min • Cake measured via Standard Methods	CST MWRD	• Polymer dose varied • Flash mix: G ~1600/s for 10 s • Floc mix: G ~40/s for 200 s • Min CST = OPD	Ngwenya et al., 2017 Avila et al., 2018
	Press	• Sample drained and then pressed at 1.4 bar for 5 minutes. • Cake measured via Standard Methods	CST Press	• Polymer dose varied • Flash mix: G ~1600/s for 10 s • Floc mix: G ~40/s for 200 s • Min CST = OPD	Ngwenya et al., 2017 Avila et al., 2018 Benisch and Neethling, 2014
	LDT	• LDT apparatus pressurized at 6.9 bar for 10 minutes • Cake measured via Standard Methods	LDT	• Ferric constant • Polymer dose varied • Mix 1: G ~400/s • Mix 2: G ~300/s • Dose with min time yielding >60% water capture is OPD	Schermerhorn et al., 2015

3.5. Statistical Analysis

Rstudio Statistical Software (R Foundation for Statistical Computing, Vienna, Austria. URL https://www.R-project.org/) was used to perform Wilcoxon rank sum tests to compare the means in cake solids and polymer from each method between the test digester and the control digester from each phase of the study. Additionally, the means of cake solids and polymer from each method were compared using the same software and method. The ggpubr package (https://CRAN.R-project.org/package=ggpubr) in R was used to analyze the data sets and generate comparison graphs.

3.6. Total Solids Analysis

MWRD hauls their biosolids to be land applied at farms throughout Colorado. Changes in cake solids impact the amount of wet tons hauled, and a difference of 0.5% in cake solids content equates to around 11 – 22 wet tonnes of biosolids (Wisdom et al., 2017). It is important to MWRD to closely estimate impacts of cake solids on hauling, but struvite dehydration during the TS test reduces the mass of TS, thus under-predicting the wet tons hauled. The conditions found in anaerobic digestion are highly suitable for struvite precipitation. At MWRD, struvite has been reported to account for anywhere from 5% to 11% of the mass of the cake solids (Wisdom, et al., 2017). In one study, struvite was reported to lose 30.7% and 45% of its mass when heated at temperatures of 90 ˚C and 100 ˚C, respectively (Kurtulus & Tas, 2011). Another study observed the partial dehydration of struvite ($MgNH_4PO_4{\cdot}6H_2O$) to dittmarite ($MgNH_4PO_4{\cdot}H_2O$) at temperatures of 100 ˚C when applying heat at increasing temperatures over time (Ramlogan & Rouff, 2016). By stoichiometry, the dehydration of struvite to an anhydrous form results in a loss in mass of 44%, which would produce lower than expected TS measurements using the Standard Methods 2540 G procedure (Equation 3.1).

$$MgNH_4PO_4 \cdot 6H_2O = MgNH_4PO_4 + 6H_2O \tag{3.1}$$

The drying rate method used at KBKopp to assess cake solids does not subject the sample to an elevated temperature, thus no adjustments for struvite were applied. However, the standard method for TS analysis was utilized for cake solids measurements from MCT Bucknell, MCT MWRD, Press, and LDT. Cake solids results were adjusted to account for the loss in struvite mass from dehydration that occurs at the TS test standard temperature of 105˚C (Equation 3.2) (Wisdom et al., 2017).

$$TS_{Adjusted}(\%) = TS_{measured}(\%) \times (1 + \mathrm{f}_{Struvite\ dehydration} \times \mathrm{f}_{Struvite\ in\ digestate}) \qquad (3.2)$$

- where TS is the % total solids (adjusted for struvite mass loss or measured),
- $f_{struvite\ dehydration}$ is the mass fraction loss due to struvite dehydration during the TS test (0.44 based on stoichiometry), and
- $f_{struvite\ in\ digestate}$ is the mass fraction of struvite present in anaerobic digester effluent.

Additionally, reported polymer doses were adjusted to account for the adjusted digestate total solids using Equation 3.3.

$$Polymer_{Adjusted}\left(\frac{kg}{tonne}\right) = Polymer_{Measured}\left(\frac{kg}{tonne}\right) \times \frac{TS_{Measured}(\%)}{TS_{Adjusted}(\%)} \qquad (3.3)$$

- where Polymer is the polymer dose (adjusted for struvite loss or measured).

An acidification test used to estimate struvite content was performed monthly. 7.5 L of digester sample were collected and wasted to flush the sample line, and then sample was collected for testing. Initial conditions of the slurry (volume, pH, TS, VS, total magnesium, total phosphorus, ammonia, soluble phosphorus, soluble magnesium) were measured using standard methods. Stock solutions of 10%, 1%, and 0.1% hydrochloric acid were used to titrate the sample to a pH of 4.5, and then the slurry final volume, TS, VS, total magnesium, total phosphorus, ammonia, soluble phosphorus, and soluble magnesium were measured. The differences in soluble magnesium, ammonia, and soluble phosphorus concentrations were assumed to be solely associated with struvite dissolution, and these concentrations were converted to equivalent struvite mass based on the stoichiometry of the struvite precipitation (Equation 3.4). Soluble magnesium was used as the basis for the struvite concentration when ammonia and phosphorus were in stoichiometric excess. The mass of struvite in the sample was converted to a fraction by dividing by the mass of total solids in the sample.

$$MgNH_4PO_4 \cdot 6H_2O \; = \; Mg^{2+} + NH_4^+ + PO_4^{3-} + 6H_2O \qquad (3.4)$$

XRD Testing was performed by an external laboratory (H&M Analytical Services, New Jersey, US) only during Phase 1b and Phase 2a. 7.5 L of digester sample were collected and wasted to flush the sample line, and then sample was collected for testing. 75 mL of sample from the Test and Control digester were dried in an incubator at 25 ˚C for 72 hours. 5 g of dried solids were ground with a mortar and pestle, placed in 15 mL Falcon™ tubes (Fisher Scientific, New Hampshire, US) and shipped to H&M Analytical Services for analysis. Results were provided to MWRD analytical laboratory upon completion of analysis.

Results from estimating struvite from each phase of the pilot are displayed in Table 3.4. H&M Analytical reported that high amorphous content resulted in poor diffraction patterns and reduced confidence in some of the identified crystalline phases. Weekly values from acidification testing were used to adjust the cake solids and polymer values measured during each dewaterability test.

Table 3.4 Results from struvite quantification methods.

		% Mass Struvite			
Digester		**Test**		**Control**	
Pilot	*Treatment*	*Acidification*	*XRD*	*Acidification*	*XRD*
Phase 1a	Secondary Solids Release/High Primary Fraction	5.3%	--	6.8%	--
Phase 1b	Secondary Solids Release/High WAS Fraction	7%	6%	10%	8%
Phase 2a	WAS THP-AD	12%	19%	10%	11%
Phase 2b	Secondary Solids Release/Pri+WAS THP-AD	4%	--	5%	--

CHAPTER 4: RESULTS AND DISCUSSION

4.1. Cake Solids

This section examines and discusses the cake solids results from the dewaterability assessments performed during all study phases. The cake solids produced from each method are compared to evaluate any differences stemming from the methods. Differences in cake solids produced from each laboratory are compared and evaluated. Finally, each method is evaluated for the ability to detect changes in the cake solids from the upstream solids pre-treatments as compared to a control.

4.1.1. Method Comparison

The cake solids produced from each method for both the Test and Control digester were compared to one another as presented in Figure 4.1. The Drying Rate method produced statistically greater cake solids than mechanically produced cake solids from other methods in Phase 1a. During Phase 1b, no statistical differences were identified between the cake solids produced by any of the methods, likely due to low sample count from all methods. Similarly, Wilcoxon method is for comparison of samples not replicates, so similar differences observed for Drying Rate during Phase 2a and Phase 2b are not statistically confirmed. One possible explanation for the differences observed between Drying Rate and the other methods is that the Drying Rate is fundamentally different from all of the other methods tested. The Drying Rate method derives a cake solids result from an evaporation rate curve versus mechanically producing cake solids using Press, MCT Bucknell, MCT MWRD, and LDT methods.

Cake solids results derived from the Drying Rate are postulated to represent the theoretical limit of free water able to be removed (Kopp & Dichtl, 2001; Tsang & Vesilind, 1990). However, the LDT's mechanically produced cake solids and Drying Rate-derived cake solids did not statistically differ. Annual average full-scale centrifuge cake solids of 20.9 ± 1.0% were produced at MWRD (Figure 4.1). Drying rate derived cake solids were more consistently similar to full-scale cake solids (Table 4.1).

Table 4.1 Relative percent difference between method average cake solids content from the Control train and full-scale centrifuge cake solids for comparable pilot phases.

Pilot	**Drying Rate**	**MCT Bucknell**	**MCT MWRD**	**Press**	**LDT**
Phase 1b	-3%	-20%	-3%	-11%	11%
Phase 2b	0%	-39%	-20%	-35%	

The percent standard deviation in the cake solids results between all of the methods was within a range of 10% - 23% for both treatment trains in all phases (Table 4.2). Percent standard deviation from each method was below 20% from all phases (Table 4.3). A key observation is that while the methods were observed to produce statistically different results from one another, the percent standard deviation between all of the methods suggests that the differences are slight.

Table 4.2 Average cake solids results from all methods for test and control trains for each phase.

Location	**Test**			**Control**		
Pilot	*Average*	*Std. Dev.*	*% Std. Dev*	*Average*	*Std. Dev.*	*% Std. Dev*
Phase 1a	20.8	2.9	14%	19.6	3.2	16%
Phase 1b	20.3	2.3	11%	19.8	2.4	12%
Phase 2a	26.7	6.1	23%	16.7	2.7	16%
Phase 2b	37.1	3.8	10%	16.0	3.7	23%

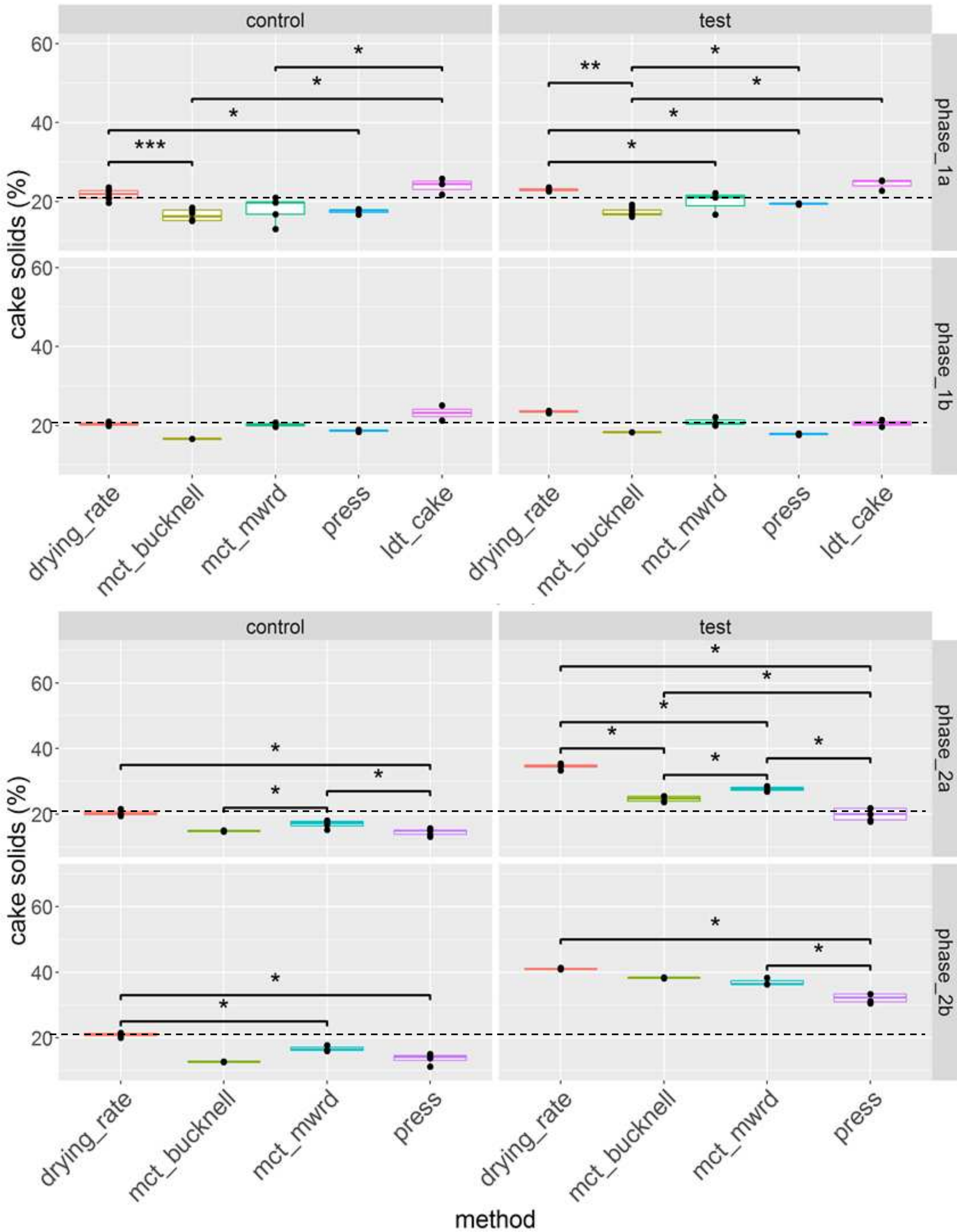

Figure 4.1 Comparison of cake solids between each method from the test and control digesters over all study phases. Only significant statistical differences are identified with asterisks above bracket comparisons. *: p <= 0.05, **: p <= 0.01, ***: p <= 0.001. Black dotted line indicates MWRD annual average full-scale centrifuge cake solids = 20.9 ± 1.0%.

Table 4.3 Percent standard deviation of cake solids from each method within each pilot phase. Average and standard deviation for the Test and Control reported at the bottom of the table.

Pilot	Treatment Train	Drying Rate	MCT Bucknell	MCT MWRD	Press	LDT
Phase 1a	*Test*	1.7%	6.2%	14.3%	0.9%	5.9%
	Control	6.6%	9.1%	17.7%	4.0%	8.6%
Phase 1b	*Test*	1.4%	--	5.3%	1.4%	6.1%
	Control	2.8%	--	3.7%	2.1%	11.6%
Phase 2a	*Test*	2.4%	3.7%	2.6%	9.5%	--
	Control	5.2%	1.1%	7.3%	7.3%	--
Phase 2b	*Test*	0.5%	0.6%	3.1%	4.6%	--
	Control	2.8%	1.3%	5.5%	12.5%	--
Test	*Average*	1.5%	3.5%	6.3%	4.1%	6.0%
	Std. Dev.	0.8%	2.8%	5.4%	4.0%	0.2%
Control	*Average*	4.4%	3.8%	8.6%	6.5%	10.1%
	Std. Dev.	1.9%	4.6%	6.2%	4.6%	2.1%

4.1.2. Laboratory Comparison

Figure 4.2 compares the cake solids results produced from each laboratory. The MWRD laboratory grouping included LDT, MCT MWRD, and Press cake solids during Phase 1a and Phase 1b, and only the MCT MWRD and Press methods during Phase 2a and Phase 2b. In terms of sample handling, all samples processed at MWRD were processed within 2 to 4 hours after sample collection. Samples shipped to KBKopp on ice took approximately five days to arrive, and were stored at 4˚C until processed. Samples shipped to Bucknell University on ice typically arrived in two days, and were stored in the collection vessel under ambient conditions until processed. The shipping time and storage of the sample may have allowed for additional digestion of the solids to occur. Increasing solids retention time within anaerobic digestion was reported to decrease the CST of anaerobically digested solids, which suggests that dewatering characteristics of the solids changed (Verma et. al, 2005). Analyzing the data in Figure 4.2 corroborates the previous result that cake solids produced at KBKopp laboratory (Drying Rate) typically yielded greater cake solids than those produced at MWRD and at Bucknell, and suggests that either differences in sample handling or methodological differences influenced cake solids produced KBKopp.

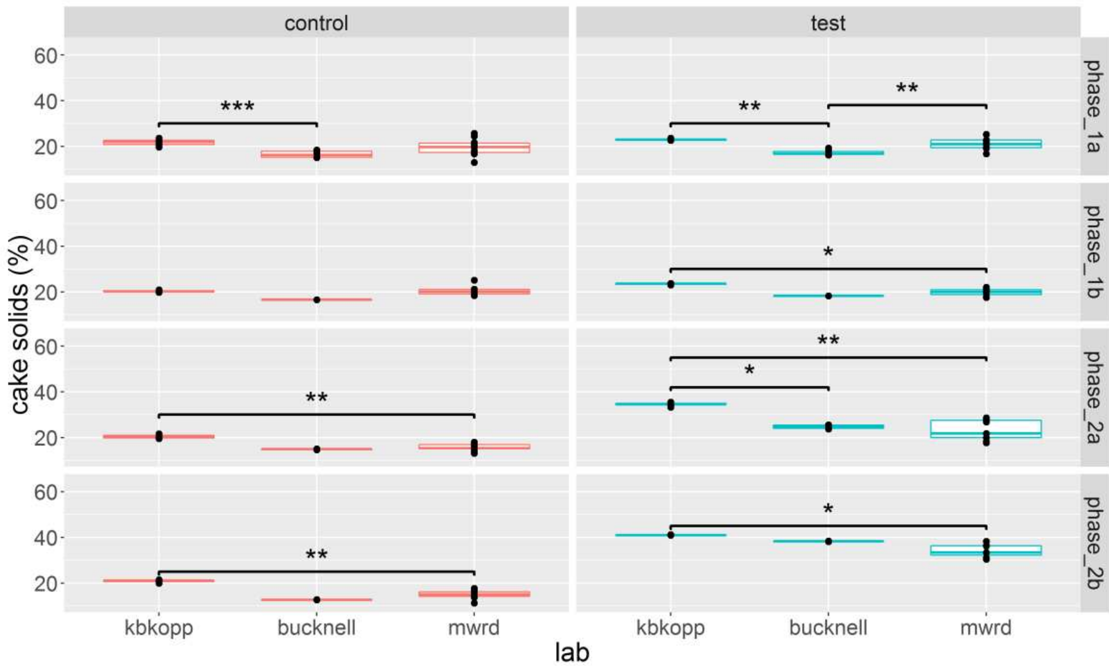

Figure 4.2 Comparison of cake solids produced by each lab over all study phases. Only significant statistical differences are identified with asterisks above bracket comparisons. *: p <= 0.05, **: p <= 0.01, ***: p <= 0.001.

4.1.3. Test vs. Control Comparison

Average cake solids from each pilot phase are presented in Figure 4.3, which compares the cake solids produced from the Test and Control digesters from each phase using each method. During Phase 1a and Phase 1b, the differences in means were not statistically significant. One study using the MCT method to evaluate the impact of Secondary Solids Release on cake solids observed an average cake solids of 13.4 % from the Secondary Solids Release pre-treated stream versus control cake solids of 12.8% (Higgins et al. 2014). Statistical significance of the differences was not discussed and the observed differences were nominal. Results from this study suggest that pre-treatment of Secondary Solids Release tested during

Phase 1a and Phase 1b does not alter the solids characteristics enough that dewaterability improvements were quantifiable given typical variance associated with the methods. Grouping samples collected over periods of time may also have captured process variability over time, thus reducing statistical significance of any differences.

During Phase 2a, Test digester cake solids produced by the Drying Rate, MCT Bucknell, MCT MWRD, and Press methods were statistically greater than the cake solids from the Control digester. During Phase 2b, the differences were only statistically significant for Drying Rate and Press methods. While differences were not significant for MCT Bucknell and MCT MWRD methods, the magnitude of the percent differences observed for MCT Bucknell and MCT MWRD were within the range of, or greater than, the percent differences observed for Drying Rate and Press. Confidence in the observed differences may be low due to fewer than four total samples being used in the comparison. In Phase 2a for all methods, and for Phase 2b for Drying Rate and Press, comparisons that were statistically different used sample numbers greater than four (Table 3.1). The cake solids (32.1% to 41.1%) observed during Phase 2b are greater than those from previous full-scale evaluations where final cake solids of 26-33% have been reported from WAS+Pri THP-AD pretreatment (Barber, 2016; De Clippeleir et al., 2016).

The results presented in this section show that under the conditions tested in this study, the cake solids produced from each method had a percent standard deviation ranging from 10 – 23%, and statistically significant differences were observed. Cake solids derived from the Drying Rate method typically were greater those produced by MCT Bucknell and by two methods (MCT and Press) at MWRD laboratory. The cake solids produced from LDT did not statistically differ from the cake solids derived by Drying Rate. All methods showed a statistically significant increase of 37% - 69% in cake solids in Phase 2a (WAS Only THP-AD pretreatment). Drying

Rate and Press methods captured statistically significant differences during Phase 2b (WAS+Pri THP-AD pretreatment). In Phase 2a, all methods had four or more data points included in the statistical comparison, and the differences were statistically significant. However, in Phase 2b, MCT Bucknell and MCT MWRD only had two and three data points, respectively, which always yields a p-value greater than 0.05 (Wilcoxon et al., 1973). Thus, the limited sample size likely reduced confidence in the observed differences.

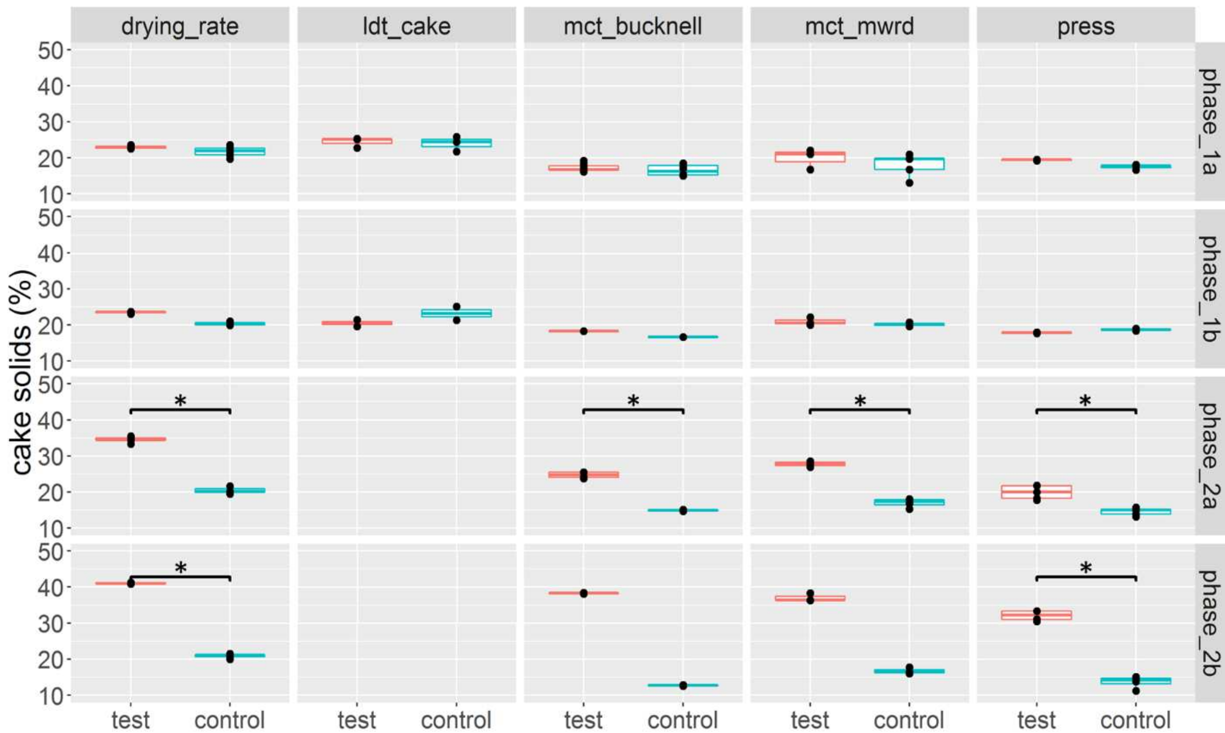

Figure 4.3 Comparison of cake solids between Test and Control from each dewatering method and pilot over all study phases. Statistical significance identified with asterisks above bracket comparisons. *: p <= 0.05.

4.2. Optimal Polymer Demand

Similar to the organization of the previous section, this section evaluates the optimal polymer demand (OPD) results from all methods during all study phases. Polymer demand results are compared to evaluate any differences between the methods. Differences in polymer demand between laboratories are compared and evaluated. Finally, the methods are assessed for the ability to detect changes in polymer demand from the upstream solids pre-treatments as compared to a control.

4.2.1. Method Comparison

For Phase 1a Test and Control digester, CST Bucknell OPD is statistically greater than OPD determined by the other methods (Figure 4.4). The greater OPD as measured by CST Bucknell did not translate to similar statistically significant differences in cake solids during the same phase (Figure 4.1). For example, CST Bucknell OPD was statistically greater than CST MWRD and CST Press, but no statistical differences were observed in cake solids during Phase 1a (Figure 4.1). Conversely, for 52% and 37% less polymer for the Test and Control, the LDT produced 42% and 45% drier cake solids than MCT Bucknell, respectively. No statistically significant differences were identified during Phase 1b. For Phase 2a, only one comparison was statistically significant: for the Control digester, CST Bucknell OPD required 63.5% more polymer than CST MWRD OPD, consistent with polymer results from Phase 1a, and produced a 14.1% wetter cake (Figure 4.1) than MCT MWRD. Differences in OPD from Phase 2b are not statistically significant. Annual average full-scale polymer demand is 21 ± 2.0 kg/tonne (Figure 4.4). During pilot phases where the Control digester was operated similar to full-scale anaerobic digesters (Phase 1b, Phase 2b), CST Bucknell OPD was similar to full-scale polymer demand. However, depending on operational needs, the full-scale plant switches between using emulsion polymer or dry polymer, possibly influencing these comparisons.

Table 4.4 Relative percent difference between method average OPD from the Control train and full-scale polymer demand for comparable pilot phases.

Pilot	Charge Demand	CST Bucknell	CST MWRD	CST Press	LDT
Phase 1b	-1%	7%	-26%	-25%	-31%
Phase 2b	29%	-7%	8%	-15%	

The variability as measured by percent standard deviation in the mean OPD as determined by all methods ranged from 5% - 32% (Table 4.5). Percent standard deviation from each method varied more for polymer than for cake solids in all phases (Table 4.6). Analysis of Figure 4.4, Table 4.5, and Table 4.6 suggests that OPD results can differ between methods, and that the differences in polymer do not necessarily translate to statistical differences in cake solids.

Table 4.5 Average OPD from all methods for test and control trains for each phase.

Location	**Test** OPD (kg/tonne)			**Control** OPD (kg/tonne)		
Pilot	*Average*	*Std. Dev.*	*% Std. Dev.*	*Average*	*Std. Dev.*	*% Std. Dev.*
Phase 1a	19.9	5.0	25%	18.9	6.1	32%
Phase 1b	15.5	2.7	17%	17.7	3.5	20%
Phase 2a	22.5	4.8	21%	15.4	3.5	23%
Phase 2b	22.2	1.1	5%	21.6	4.0	19%

Table 4.6 Percent standard deviation of OPD from each method within each pilot phase. Average and standard deviation for Test and Control reported at the bottom of the table.

Pilot	**Location**	**Charge**	**CST Bucknell**	**CST MWRD**	**CST Press**	**LDT**
Phase 1a	*Test*	1.1%	14.5%	6.7%	10.9%	12.0%
	Control	5.6%	12.9%	3.5%	14.8%	0.4%
Phase 1b	*Test*	--	--	23.6%	4.6%	0.0%
	Control	--	--	20.2%	3.9%	0.0%
Phase 2a	*Test*	--	--	10.2%	13.4%	--
	Control	--	--	0.0%	13.1%	--
Phase 2b	*Test*	--	0.3%	9.4%	10.7%	--
	Control	--	0.0%	11.4%	28.8%	--
Test	*Average*	--	7.4%	12.5%	9.9%	6.0%
	Std. Dev.	--	10.0%	7.6%	3.8%	8.5%
Control	*Average*	--	6.5%	8.8%	15.2%	0.2%
	Std. Dev.	--	9.1%	9.0%	10.3%	0.3%

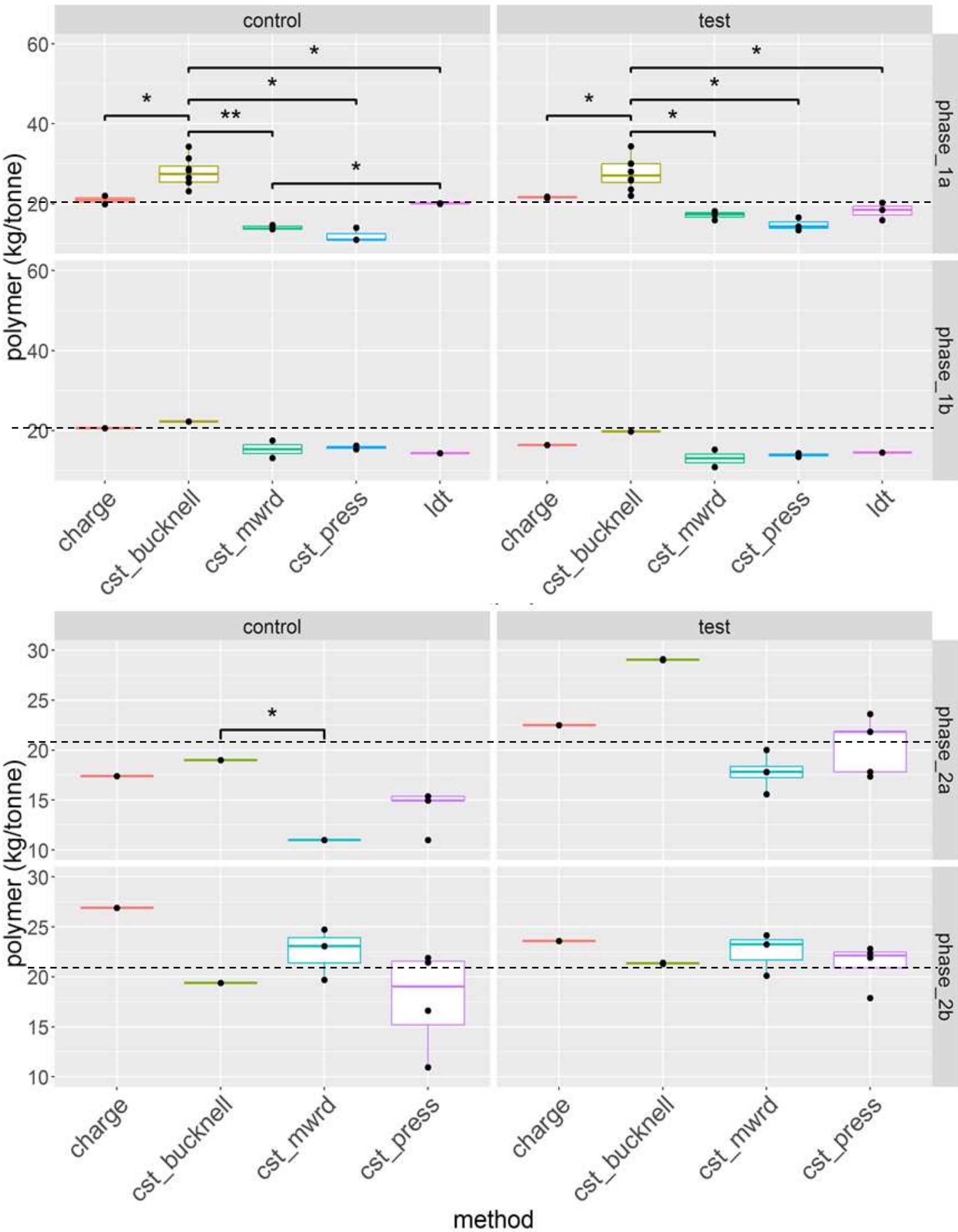

Figure 4.4 Comparison of OPD between each method from the test and control digesters over all study phases. Only significant statistical differences are identified with asterisks above bracket comparisons. *: p <= 0.05, **: p <= 0.01. Black dotted line indicates MWRD annual average full-scale polymer demand = 21 ± 2.0 kg/tonne.

4.2.2. Laboratory Comparison

With the exception of Phase 2b, the observed differences in OPD determined by the three laboratories are generally consistent between all phases (Table 4.7). The OPD determined externally by KBKopp ranged from 8% - 37% different than OPD determined on site at MWRD, and OPD determined at Bucknell ranged from -3% to 78% different than OPD determined onsite at MWRD. However, statistical significances were only observed in the OPD results determined between certain laboratories during Phase 1a and Phase 2a. During Phase 1a, KBKopp reported statistically greater OPD than MWRD for the Test digester, and Bucknell reported statistically greater OPD for both the Test and the Control than both KBKopp and MWRD (Figure 4.5). During Phase 2a, Bucknell OPD was statistically greater than MWRD OPD. These results suggest that something fundamentally different in the methodology caused the mean OPD reported by Bucknell during Phase 1a and during Phase 2a to be greater than the OPD reported by MWRD laboratory, and greater than OPD determined at KBKopp laboratory during Phase 1a.

Table 4.7 Comparison of percent difference in OPD between external labs (KBKopp and Bucknell) and MWRD.

Location	**Test**		**Control**	
Pilot	*% Difference KBKopp and MWRD*	*%Difference Bucknell and MWRD*	*% Diff Difference KBKopp and MWRD*	*%Difference Bucknell and MWRD*
Phase 1a	29%	66%	36%	78%
Phase 1b	19%	43%	36%	47%
Phase 2a	18%	52%	37%	50%
Phase 2b	8%	-2%	34%	-3%

The statistical differences observed between OPD determined at Bucknell and MWRD could be due to methodological differences or differences in sample handling.

Low variance observed in TS and VS analyses from Phase 1a and Phase 2a (Appendix A, Table A.1 and Table A.3) suggest that the statistical differences were not from sample collection. Of the MWRD methods (CST MWRD, CST Press, and LDT), conditioning methodology of CST Press and CST MWRD was similar to that for CST Bucknell (Table 3.3). No statistical differences were observed in OPD between CST MWRD and CST Press during Phase 1a and Phase 2b for both the Test and Control (Figure 4.4), and different analysts performed CST MWRD and CST Press. This suggests that method variability was unlikely to stem from different analysts or the method itself.

Samples were shipped to Bucknell University on ice, and typically arrived in two days. Samples not processed the same day were stored in the collection vessel in ambient conditions in the lab for an additional day or two until processed. Samples shipped to KBKopp on ice took five days to arrive, and were stored at 4˚C until processed. All samples processed at MWRD were processed within two to four hours after sample collection. The additional shipping time and storage of the sample may have allowed for additional digestion of the solids to occur. Increasing polymer demand has been reported to be associated with increasing soluble EPS stemming from increased digestion time (Novak et al., 2003). Additional digestion could explain why polymer demand determined at Bucknell was greater than polymer demand determined with fresh samples at MWRD. KBKopp did not provide replicate OPD results for all polymer samples processed, and only processed a single sample during Phase 1a, Phase 2a, and Phase 2b. Therefore, statistical comparisons of OPD between KBKopp and the other two labs could not be performed.

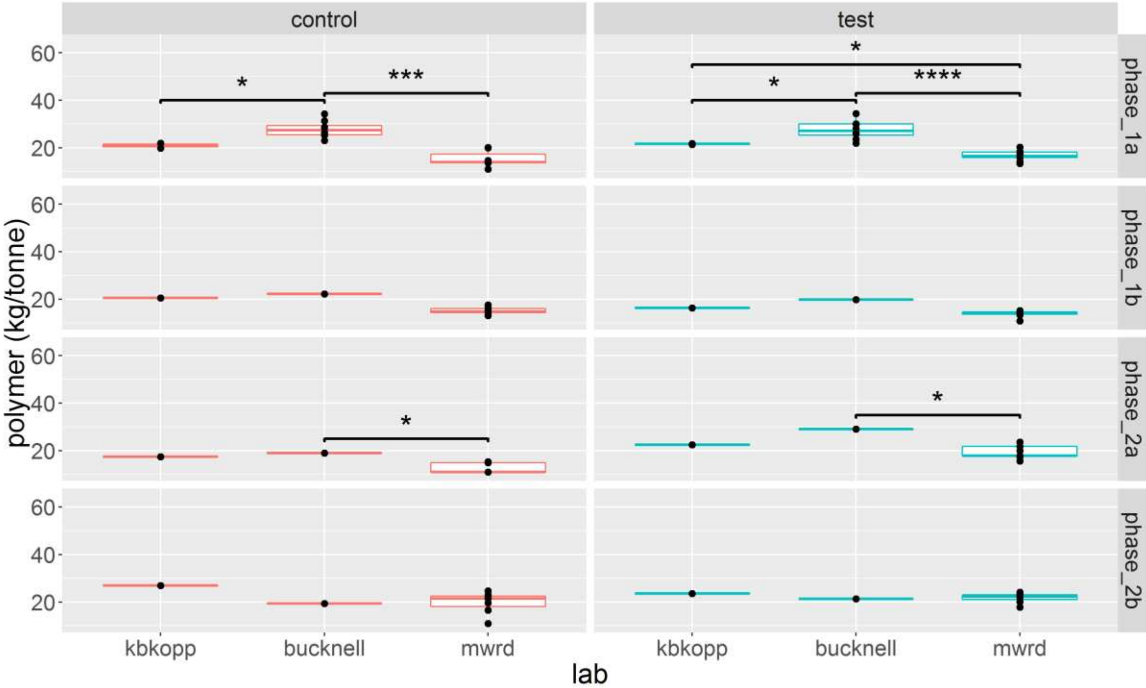

Figure 4.5 Comparison of OPD produced by each lab over all study phases. Only significant statistical differences are identified with asterisks above bracket comparisons. *: p <= 0.05, **: p <= 0.01, ***: p <= 0.001.

4.2.3. Test vs. Control Comparison

Statistical comparisons of the impact of the solids pre-treatments on the OPD are presented in Figure 4.6. Figure 4.6 shows that very few statistically significant differences were observed. During Phase 1a, optimal polymer demand determined via CST MWRD showed that Secondary Solids Release pre-treatment with a high fraction of primary solids required an additional 3.2 kg of polymer / ton to gain 1.8% in cake solids content. Differences from other methods during Phase 1a and all methods during Phase 1b were not significant.

During Phase 2a, the mean OPD determined by the CST MWRD and CST Press for the Test digester was statistically greater than that of the Control digester. The results from these methods suggest that a WAS THP-AD process may increase polymer required to dewater by 20 – 30%. For Phase 2b (Pri+WAS THP AD), no clear impacts of this process on OPD were observed.

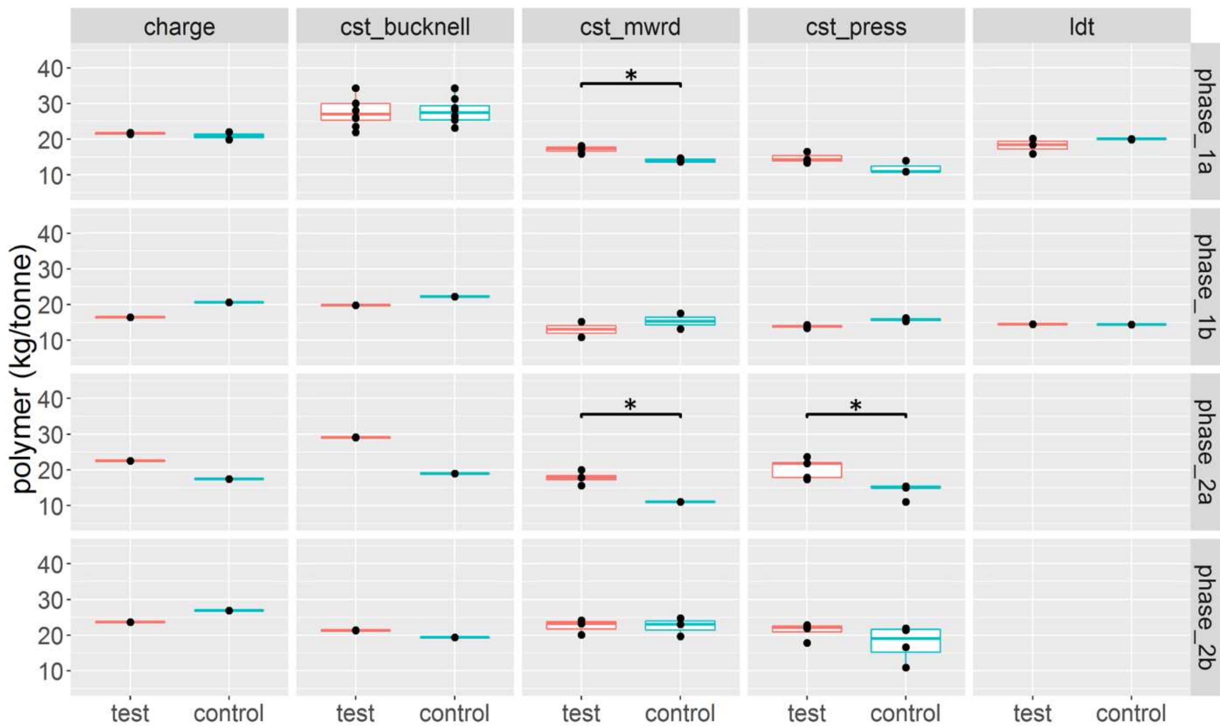

Figure 4.6 Comparison of OPD between the Test and Control digestate measured by each method during each pilot phase. Statistical significance identified with asterisks above bracket comparisons. *: p <= 0.05.

Analysis of the OPD results suggests that less variability was observed in OPD results than in cake solids. Statistical differences in OPD were observed between labs, notably from OPD results from Bucknell during Phase 1a. It is possible that storing the sample under ambient conditions caused additional digestion of the solids samples, leading to increased polymer demand. However, the results in this research cannot confirm this hypothesis. The differences in OPD between MWRD laboratory and the other labs suggest that more control needs to be exerted over sample handling in determining polymer demand. As recommended previously, the influence of sample hold time and temperature on OPD as determined by these methods should be evaluated. This evaluation could reveal to what extent these variables affected OPD, and what steps experimenters should take to address.

4.3. Method Applicability to WRRFs

This section provides a qualitative discussion of the applicability and usefulness of these methods to a utility. Pros and cons of each method are discussed, followed by a discussion of implications that the data generated during this research have on operating costs for a full-scale WAS THP-AD system for MWRD's Robert W. Hite Treatment Facility (RWHTF).

The Drying Rate method was the only non-mechanical method tested. The cake solids value derived is independent of the OPD determined using charge demand. The cake solids result was greater than the cake solids content from other methods (Figure 4.1). An OPD determined via charge demand was provided for each given cake solids derivation from drying rate. However, the decoupling of the OPD from the cake solids derivation makes it difficult to conclude whether the OPD provided would yield the cake solids derived via drying rate on the full-scale. The main limitations in the drying rate method are that its use requires technical knowledge possibly beyond what smaller utilities might have available, fully drying a sample can take up to two days, and the method does not reveal any information on water capture efficiency. However, the data provided did seem to indicate a higher end for cake solids from a given process change, which could be used in conjunction with other methods to determine expected ranges in cake solids content from process changes.

The MCT method to produce cake solids was a relatively easy method to perform. The method produces cake solids, as well as filtrate that can be evaluated for water capture efficiency. The method as written has more variables to control up front than the Press method such as: solids loading to the centrifuge cup, centrifuge speed and time with or without mixing, and number of times to mix during centrifugation. All of these variables have been reported to influence final cake solids produced, and therefore are important to consider prior to utilizing this method (Ngwenya et al., 2017). A centrifuge and specially manufactured centrifuge cups are required to utilize the method. For larger utilities with resources, such as with MWRD, procurement of the supplies needed and method adaptation were not issues. However, this could possibly reduce the viability of this method for other utilities with limited budget and time.

Utilizing a pneumatic press to produce cake solids was the quickest and easiest of the methods tested. However, special manufacturing of stainless steel components was required to build the press (the press used in this study was the original press used in the Benisch and Neethling (2015) study). Increasing applied pressure correlated with an increase in cake solids, and the measured TS content of the outer 3.8 cm of a 15.2 cm cake solids product was 12.5% less than the TS of the center 11.4 cm diameter cake (Benisch & Neethling, 2015). In this study, only a single applied pressure and dewatering time was evaluated, and in some cases produced lower cake solids than some of the other methods (Figure 4.1). Additionally, the entire cake solids sample was collected and evaluated for TS. Filtrate from the sample can be collected and evaluated for water capture efficiency. The biggest limitation to using the press equipment in this study was the weight. The stainless steel plates have a total weight of 45 kg. However, given the simplicity in its ability to produce cake solids from conditioned solids, the press is very attractive to a utility looking for a quick laboratory method to produce cake solids.

The use of the LDT apparatus to determine cake solids and OPD requires the entire sample be dewatered. For this study, only samples meeting the OPD criteria discussed in Chapter 3.4 were evaluated for cake solids, and the analyst who performed the test did not provide the water release curves. However, the LDT both produces a cake and generates polymer dose-response graphs in an all-in-one test. While not performed for this research, the filtrate can be evaluated for water capture efficiency, and the LDT has also been reported to be able to estimate a theoretical limit in dewatering (Ginisty et al., 2014). However, compared to CST and charge demand, the LDT is a time- and labor-intensive test, taking a full six to eight-hour day just to test three different doses of polymer for only two samples. Per the method, if the time to capture 60% of the water was a minimum, this polymer dose was selected as the well-conditioned state. If a greater number of polymer doses were tested, it is possible that a different dose could have been defined as the OPD. Only a partial evaluation of the method was performed for this study, and it was very time-consuming, hence this evaluation stopped after Phase 1b. The time required to perform the LDT and evaluate the data might make the test less attractive to smaller utilities that have fewer resources and less time. However, this was the only method tested that simultaneously quantifies polymer dose and produces cake solids, and could be extended to evaluate dewatering limit of solids, which could be very powerful. Continued evaluation of this test as a laboratory-based method for dewatering characterization is recommended.

CST was used to determine an OPD for CST Bucknell, CST MWRD and CST Press. Using traditional CST apparatus to determine the ideal polymer requirements has limitations. Per the methods used in this research and in other studies (Lynch, 1989; Nicholson et al., 2016; To et al., 2016), a 10 mL pipet with the end cut off of the pipet tip is used to collect the conditioned sample from the baffled reactor and place the sample into the CST funnels. A representative sample of conditioned slurries is difficult to get into the pipette because the dense particles settle immediately. The size of the modified pipette tip opening can potentially restrict larger floc particles from entering the pipette. Also, quickly placing the sample into the CST apparatus is challenging, and spills can influence results because water can seep around the outer diameter of the CST funnel. Fluid flow parallel to the grain of Whatman No. 17 chromatography paper is greater than the fluid flow perpendicular to the grain, leading to inconsistent fluid dynamics

(Herwijn, 1996; Scholz, 2005). Given these challenges, the CST method provides a quick means to estimate the dewatering impact of a polymer dose, making it particularly attractive for utilities looking for quick ways to assess dewaterability.

Utilizing charge demand to determine an OPD has its own limitations in complex solids matrices. Different results for OPD have been observed to be produced by two different analysts performing the same charge demand method, and one of the reported issues was due to differences in the rate at which the titrant (polymer) was added to the solution (Hubbe, 2005). Slower addition of polymer to a charged solution was theorized to allow time for polymer to sorb onto particle surfaces in the presence of an electric field, thus reversing the direction of the charge of the solution. This phenomenon occurs during gel electrophoresis (Viovy, 2000) and could bias lab-measured OPD results higher than required at full-scale. These challenges associated with using charge demand to measure an OPD might make the test inaccessible to smaller utilities that have fewer resources and time to evaluate data.

The cake solids and OPD results evaluated in this research were used as key assumptions in a pre-design effort performed by Stantec Engineering of a THP solids pre-treatment versus digester expansion for MWRD's Robert W. Hite Treatment Facility. The data provided design estimates for required cake and polymer storage, as well as O&M costs for hauling and polymer use. The results showed that for a WAS THP-AD system, daily storage requirements for cake solids would decrease by 175 wet tonnes, while annual polymer required would increase by 205 tonnes. The reduction in cake solids results in 3,700 fewer trucks being hauled annually which equates to a savings of $580,000. However, the increase in polymer would cost $1.02 million (Figure 4.7).

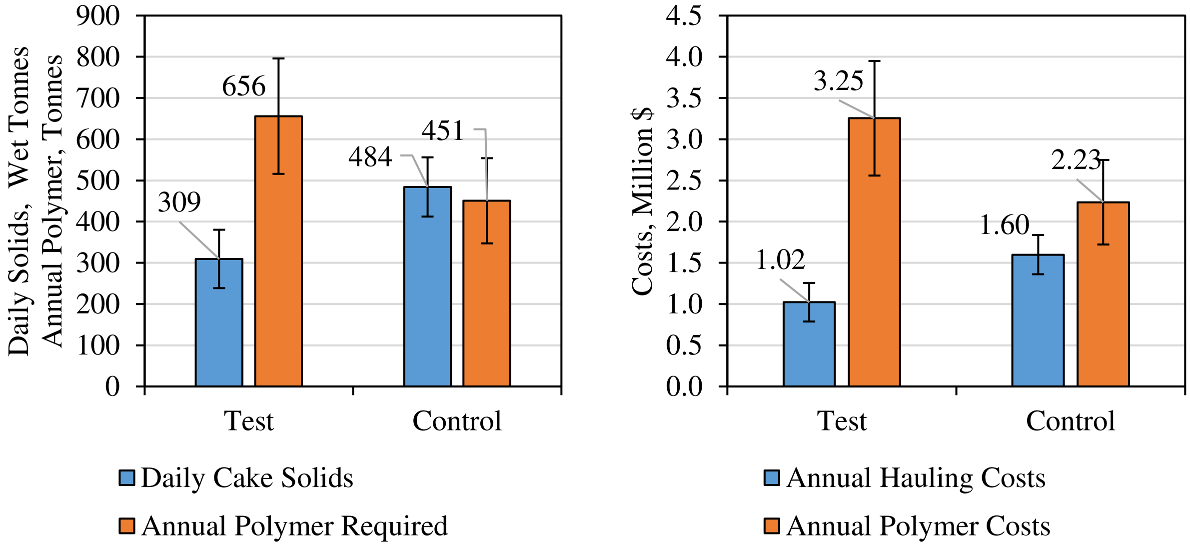

Figure 4.7 Daily solids storage and annual polymer requirements in tonnes (left), and annual hauling and polymer costs in millions of dollars (right).

This information was valuable to the MWRD's planning for the Robert W. Hite Treatment Facility because it yielded return on investment models based on data generated from the facility's own solids, allowing MWRD to make a more informed decision regarding future infrastructure upgrades.

For utilities, research facilities, or academic institutions looking to assess process change impacts on cake solids and polymer, the methods tested were relatively consistent. Selecting a method for use boils down to what research question is being asked, and available labor and costs investments. An understanding of strengths and limitations of each method is important to determine whether the method can address a particular research question. For this thesis, only cake solids and polymer were evaluated, however, only MCT, Press, and LDT provide data on water capture efficiency. While MWRD had the means to test all of the methods, labor constraints restricted fully evaluating some of the methods through all phases. This research demonstrated that quick and easy assessments provided results similar to more involved and controlled tests.

CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS

Dewaterability tests used in this research tested two hypotheses: (1) lab-based methods that provide cake solids and optimal polymer demand (OPD) data can capture differences stemming from process changes, and (2) cake solids and OPD will not differ across methods if the same solids and polymer are used.

This research demonstrated that the methods could capture differences associated with process changes, confirming the first research hypothesis. Drying Rate, MCT Bucknell, MCT MWRD, and the Press method showed an average of 58.7% increase in cake solids from WAS only thermal hydrolysis upstream of anaerobic digestion. For polymer during Phase 2a, CST MWRD and CST Press showed a 52.5% increase in the OPD. Pre-treatment from Secondary Solids Release (Phase 1a and Phase 1b) likely did not change the solids characteristics enough to improve the dewaterability, or the method resolution is not great enough to capture the changes. For polymer during Phase 1a, CST MWRD showed that Secondary Solids Release with high primary fraction would increase OPD by 22.4%. To confirm these results in future studies, it is recommended that a minimum of three replicate samples be collected for analysis.

Methodological or sample handling differences likely resulted in differences across methods, which rejects the second research hypothesis. The Drying Rate method derived a cake solids result that was greater than the MCT MWRD, MCT Bucknell, and Press. During Phase 1a, LDT produced cake solids similar to Drying Rate and greater than some of the other methods. Method differences in Drying Rate and LDT could explain why these methods derived or produced greater cake solids than some of the other methods evaluated. In addition, statistically significant differences in cake solids and OPD were observed between labs. Notably, Test Digester cake solids from KBKopp were observed to be greater than cake solids from MWRD and Bucknell during Phase 1a and Phase 2a. The OPD determined at Bucknell was statistically greater than OPD determined at KBKopp and MWRD during Phase 1a. These observations suggest that differences in sample hold time and temperature could have influenced cake solids and OPD. Shipping and storing samples may have allowed additional digestion of the solids, leading to changes in the dewaterability characteristics. However, the results in this research cannot confirm this hypothesis. To determine whether sample handling influenced cake solids and OPD results, a simple test evaluating the influence of holding time and temperature on cake solids should be performed. This would reveal whether these variables played any role in the final cake solids content and OPD, and would inform whether sample handling practices for these analyses need to be modified.

For interested parties, method selection is dependent on understanding the strengths and limitations of each method, knowing what dewaterability question one is trying to answer, and financial and labor resource availability. This research demonstrated that quick and easy assessments provided cake solids and polymer results similar to more involved and controlled tests. However, for some utilities, maximizing water capture efficiency is a priority because it maintains clean filtrate and minimizes biosolids loss back into the plant. Only MCT, Press, and LDT methods provide data on water capture efficiency. It is recommended that these methods be evaluated further to compare the water capture efficiency across methods, and determine whether these tests can anticipate changes in capture efficiency associated with process changes.

Outputs from these methods were used to provide assumptions for cake solids and polymer for WAS THP-AD pre-design work performed for Metro Wastewater Reclamation District. The cake solids and polymer data informed sizing and provided estimates of biosolids produced daily, polymer requirements, and required mechanical upgrades for cake solids handling equipment. The results showed that a savings in hauling costs of $580,000 could be expected, however, this would come with an expected increase in polymer costs of $1.02 million. The use of actual plant data in return on investment models better informed decision-making regarding future process upgrades and the expected whole plant impacts of a WAS THP-AD process upgrade. However, care must be used in interpreting operating estimates based on results from these methods as they could differ from results actually produced on the full-scale.

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APPENDIX A: SUPPLEMENTAL DATA

This appendix includes supplemental data that were not included in the main body of the thesis but were still pertinent to include. The digester effluent characteristics from all pilot phases are reported in Table A.1 – Table A.4. The values reported are averages over the period that all dewaterability testing was performed. During Phase 1a (high primary solids fraction and Secondary Solids Release), higher alkalinity, greater total solids and ammonia, and lower total and soluble phosphorus were observed in the WAS Pretreatment AD effluent than in the Control AD effluent (Table A.1).

Table A.1 Average digestate characteristics during Phase 1a.

Analyte	Units	Secondary Solids Release AD		Control AD	
		Value	Std Dev	Value	Std Dev
pH	--	7.40	0.04	7.41	0.04
Alkalinity	*mg $CaCO_3$/L*	5,307	273	4,683	644
Total Solids	*%*	2.32	0.07	2.24	0.11
Corrected Total Solids	*%*	2.40	--	2.34	--
Volatile Solids	*%*	1.8	0.06	1.7	0.09
Ammonia	*mg N/L*	1482	155	1374	186
Total COD	*mg/L*	28,563	3,817	27,494	3,517
Total Phosphorus	*mg P/L*	661	60	721	48
Total Calcium	*mg/L*	663	35	635	55
Total Iron (II)	*mg/L*	216	16	207	22
Total Magnesium	*mg/L*	67	4	64	18
Total Potassium	*mg/L*	214	14	234	65
Total Sodium	*mg/L*	125	6	112	31
Soluble Phosphorus	*mg P/L*	150	26	192	23
Soluble Calcium	*mg/L*	40	5	33	3
Soluble Iron (II)	*mg/L*	0.56	0.08	0.54	0.08
Soluble Magnesium	*mg/L*	17	3	16	2
Soluble Potassium	*mg/L*	193	13	233	10
Soluble Sodium	*mg/L*	119	5	118	4

During Phase 1b (high WAS fraction and Secondary Solids Release) greater alkalinity and ammonia, lower total and soluble phosphorus, and lower total and soluble potassium were observed on the Secondary Solids Release AD than in the Control AD (Table A.2). The reduction in total phosphorus and total potassium are indicative of PAO anaerobic activity occurring within the Solids Release Reactor.

Table A.2 Average digestate characteristics during Phase 1b.

Analyte	Units	Secondary Solids Release AD		Control AD	
		Value	Std Dev	Value	Std Dev
pH	--	7.41	0.01	7.38	0.03
Alkalinity	*mg $CaCO_3$/L*	5,237	255	4,567	67
Total Solids	*%*	2.62	0.09	2.59	0.03
Corrected Total Solids	*%*	2.71	--	2.69	--
Volatile Solids	*%*	2.0	0.07	1.9	0.02
Ammonia	*mg N/L*	1453	51	1330	53
Total COD	*mg/L*	31,000	1,015	28,533	1,501
Total Phosphorus	*mg P/L*	718	13	826	21
Total Calcium	*mg/L*	705	66	697	21
Total Iron (II)	*mg/L*	284	15	294	14
Total Magnesium	*mg/L*	72	2	77	5
Total Potassium	*mg/L*	234	7	293	3
Total Sodium	*mg/L*	126	2	125	3
Soluble Phosphorus	*mg P/L*	211	47	269	30
Soluble Calcium	*mg/L*	32	1	26	0
Soluble Iron (II)	*mg/L*	0.76	0.04	0.75	0.07
Soluble Magnesium	*mg/L*	14	1	12	1
Soluble Potassium	*mg/L*	205	11	262	14
Soluble Sodium	*mg/L*	117	7	117	5

During Phase 2a, effluent solids from the WAS THP-AD were greater than Control AD solids. Total and soluble COD was greater in the WAS THP-AD, an expected result from thermal hydrolysis of WAS. Total calcium was present in greater quantities in WAS THP-AD than in the Control AD. Higher concentrations of ammonia were observed in the WAS THP-AD than in the Control AD. No differences were observed in phosphorus and potassium, which is an expected result from the WAS from both trains undergoing anaerobic reaction in the solids release reactor. However, total magnesium and calcium would also be expected to be similar (Table A.3).

Table A.3 Average digestate characteristics during Phase 2a.

Analyte	Units	WAS THP-AD		Control AD	
		Value	*Std. Dev.*	*Value*	*Std. Dev.*
pH	--	7.5	0.10	7.4	0.07
Alkalinity	*mg CaCO3/L*	6,368	393	4,840	706
Total Solids	*%*	3.06	0.09	2.56	0.05
"Corrected" Total Solids	*%*	3.22	0.09	2.67	0.05
Volatile Solids	*%*	2.3	0.09	2.0	0.81
Ammonia	*mg/L*	1,877	187	1,312	83
Total COD	*mg/L*	39,633	2,453	29,980	2,416
Soluble COD	*mg/L*	4,300	444	629	45
Total Phosphorus	*mg/L*	1,008	78	930	32
Total Calcium	*mg/L*	747	23	584	37
Total Iron (II)	*mg/L*	288	50	254	22
Total Magnesium	*mg/L*	105	23	69	10
Total Potassium	*mg/L*	284	60	291	36
Total Sodium	*mg/L*	100	11	110	3.8
Soluble Phosphorus	*mg/L*	346	56	352	42
Soluble Calcium	*mg/L*	33	3.0	27	2.2
Soluble Iron (II)	*mg/L*	3	0.57	1	0.09
Soluble Magnesium	*mg/L*	7	1.1	11	1.2
Soluble Potassium	*mg/L*	315	65	292	42
Soluble Sodium	*mg/L*	110	7.5	113	4.6

During Phase 2b, effluent solids from the Pri+WAS THP-AD were greater than Control AD solids, and the variance was also greater than the previous phases. One explanation for the greater solids content in the effluent from the Pri+WAS THP AD is that there was a loss in volatile solids reduction (VSR) compared to the Control. This loss in VSR was due to digester instability during this phase. Total and soluble COD was greater in the Pri+WAS THP-AD, an expected result from thermal hydrolysis. Other differences included greater concentrations of total and soluble cations present in the Pri+WAS THP AD than the Control AD (Table A.4).

Table A.4 Average digestate characteristics during Phase 2b.

Analyte	**Units**	**Pri+WAS THP-AD**		**Control**	
		Value	*Std. Dev.*	*Value*	*Std. Dev.*
pH	--	7.4	0.10	7.5	0.06
Alkalinity	*mg CaCO3/L*	7878	947	4783	398
Total Solids	*%*	5.46	0.81	2.51	0.06
"Corrected" Total Solids	*%*	5.56	0.81	2.56	0.06
Volatile Solids	*%*	4.4	0.73	1.9	0.04
Ammonia	*mg/L*	2,718	572	1,505	39
Total COD	*mg/L*	100,850	46,603	27,450	6,106
Soluble COD	*mg/L*	19,600	9,840	667	190
Total Phosphorus	*mg/L*	1696	133	1087	57
Total Calcium	*mg/L*	934	88	577	15
Total Iron (II)	*mg/L*	392	57	209	11
Total Magnesium	*mg/L*	274.1	16.0	126.9	11.2
Total Potassium	*mg/L*	440	52	397	18
Total Sodium	*mg/L*	563	453.5	124	8.1
Soluble Phosphorus	*mg/L*	869	143	462	28
Soluble Calcium	*mg/L*	24.3	3.9	24.2	1.7
Soluble Iron (II)	*mg/L*	11.91	2.55	0.58	0.03
Soluble Magnesium	*mg/L*	8	0.9	10	1.8
Soluble Potassium	*mg/L*	441	110	394	16
Soluble Sodium	*mg/L*	525	375.5	116	5.3

Average cake solids and polymer results produced from each method are shown in

Table A.5 and Table A.6. Secondary Solids Release did not increase the cake solids content as drastically as thermal hydrolysis did. Polymer demand as compared to the control increased during Phase 2a and Phase 2b.

Table A.5 Average cake solids results from each method for the Test and Control digester effluent from all study phases.

Location	**Test Digester - Cake Solids (%)**					**Control Digester - Cake Solids (%)**				
Lab	**KBKopp**	**Bucknell**	**MWRD**			**KBKopp**	**Bucknell**	**MWRD**		
Method	*Drying or DS(A)*	*MCT Bucknell*	*MCT MWRD*	*Press*	*LDT*	*Drying or DS(A)*	*MCT Bucknell*	*MCT MWRD*	*Press*	*LDT*
Phase 1a	23.0	17.2	19.9	19.4	24.4	21.8	16.5	18.1	17.5	24.0
Phase 1b	23.5	18.3	21.3	17.8	20.5	20.4	16.6	20.2	18.7	23.2
Phase 2a	34.6	24.7	27.8	19.9	--	20.4	14.9	17.0	14.6	--
Phase 2b	41.1	38.3	37.0	32.1	--	20.9	12.7	16.7	13.7	--

Table A.6 Average optimal polymer demand results from each method for the Test and Control digester effluent from all study phases.

Location	**Test Digester - Polymer (kg/tonne)**					**Control Digester - Cake Solids (kg/tonne)**				
Lab	**KBKopp**	**Bucknell**	**MWRD**			**KBKopp**	**Bucknell**	**MWRD**		
Method	*Charge Demand*	*CST Bucknell*	*CST MWRD*	*CST Press*	*LDT*	*Charge Demand*	*CST Bucknell*	*CST MWRD*	*CST Press*	*LDT*
Phase 1a	21.6	27.7	17.2	14.7	18.2	21.0	27.4	14.0	12.0	20.0
Phase 1b	16.4	19.8	13.0	13.9	14.5	20.6	22.3	15.3	15.8	14.3
Phase 2a	22.5	29.1	17.8	20.5	--	17.4	19.0	11.0	14.3	--
Phase 2b	23.6	21.4	22.5	21.3	--	26.9	19.4	22.5	17.7	--

Figure A.1 – Figure A.4 show cake solids and optimal polymer demand (OPD) from each method over time for all of Phase 1 and Phase 2. Figure A.1 shows cake solids from all methods over time during Phase 1a and Phase 1b.

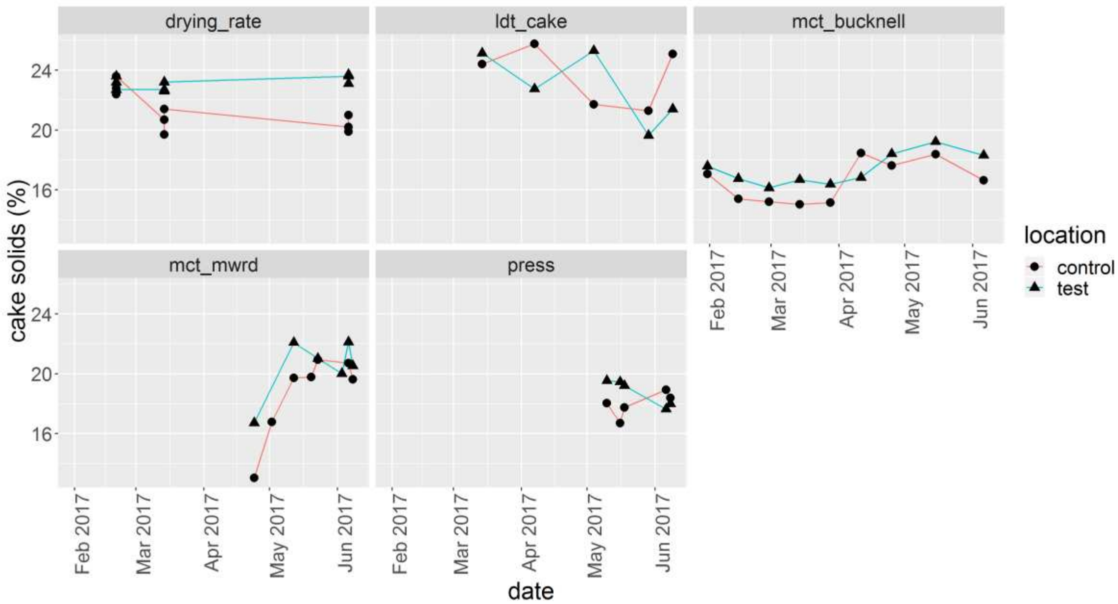

Figure A.1 Cake solids over time from each method during Phase 1a and Phase 1b.

Figure A.2 shows cake solids from all methods over time during Phase 2a and Phase 2b. Note that the LDT method for cake solids was not continued during Phase 2 due to a lack of time and resources.

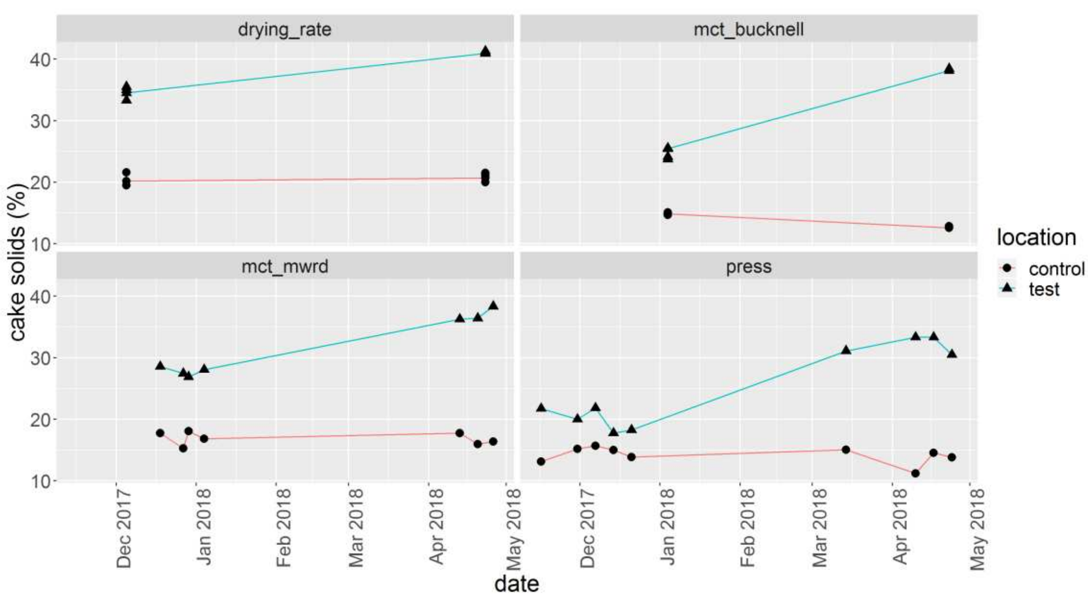

Figure A.2 Cake solids over time from each method during Phase 2a and Phase 2b.

Figure A.3 shows OPD from all methods over time during Phase 2a and Phase 2b.

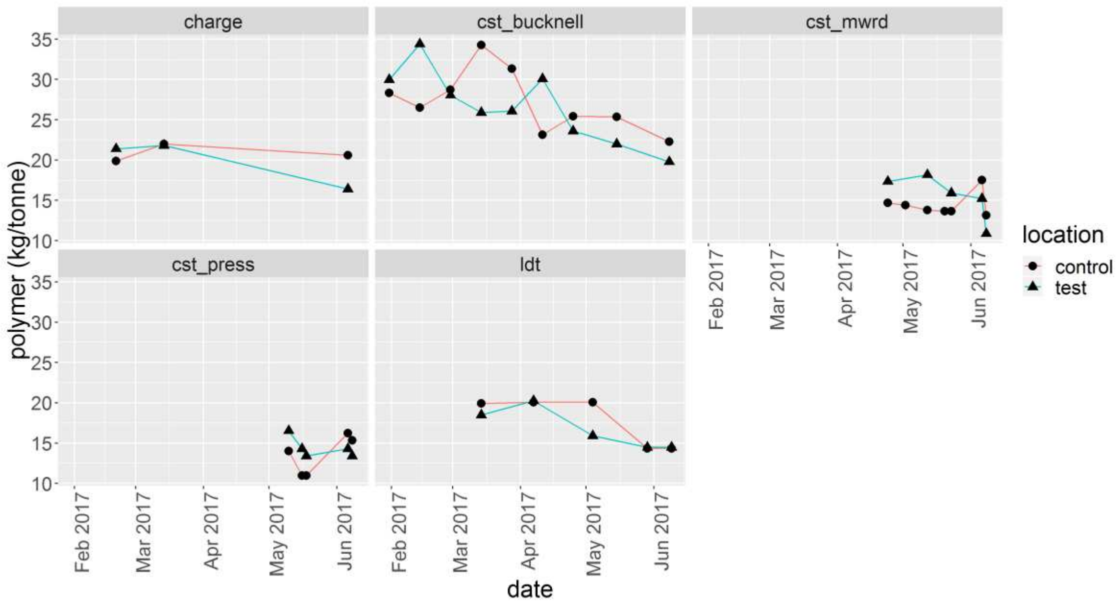

Figure A.3 Polymer demand over time from each method during Phase 1a and Phase 1b.

Figure A.4 shows OPD from all methods over time during Phase 2a and Phase 2b. Note that the LDT method was not performed due to a lack of time and resources.

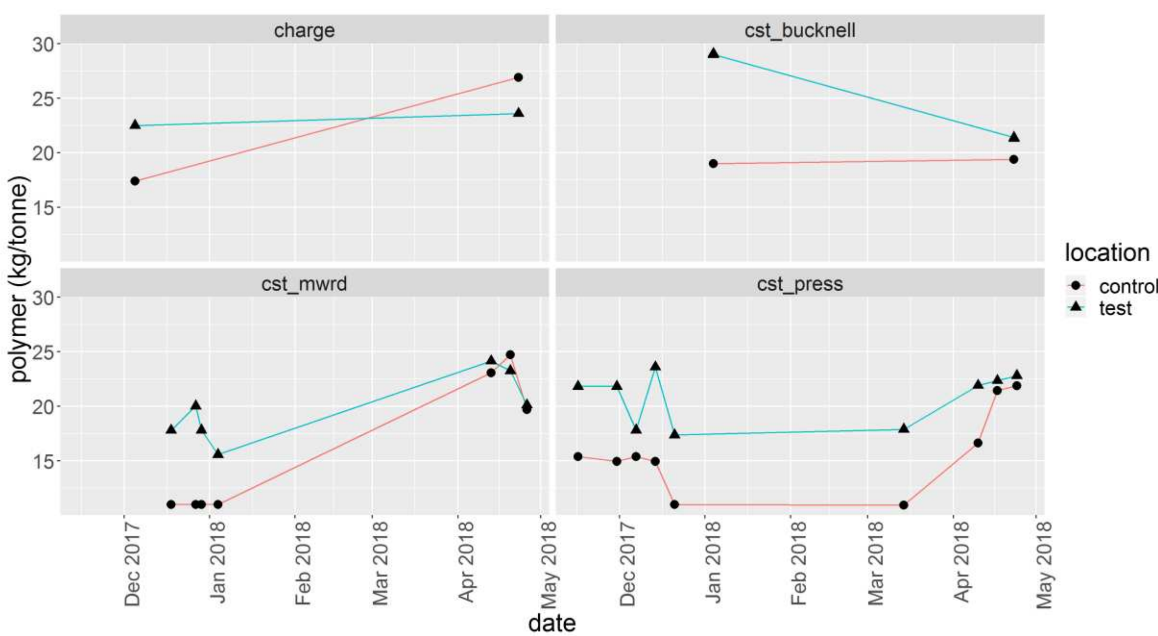

Figure A.4 Polymer demand over time from each method during Phase 2a and Phase 2b.

APPENDIX B: SUPPLEMENTAL ELECTRONIC FILES

This appendix shows the electronic files associated with this thesis. Two files are included. The raw data files included are the raw cake solids and polymer data, and the R code used for the analysis and comparison figures.

Data File Name	**Description**
Cake_Solids_and_Polymer_Raw_Data.csv	CSV file containing raw cake solids and polymer data.
Sp2019_Avila_Thesis_DatAnalysis_NTBK.nb.html	R code used with R studio to perform statistical analyses and generate comparison figures.