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# CHEMICAL PRETREATMENT AND DEWATERABILITY OF ANAEROBICALLY DIGESTED BIO-P BIOSOLIDS

by

Eileen M. Kennedy

A Thesis submitted to the Faculty of the Graduate School,
Marquette University,
in Partial Fulfillment of the Requirements for
the Degree of Master of Science

Milwaukee, Wisconsin

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# ABSTRACT

# CHEMICAL PRETREATMENT AND DEWATERABILITY OF ANAEROBICALLY DIGESTED BIO-P BIOSOLIDS

Eileen M. Kennedy

Marquette University, 2019

Phosphorous (P) effluent regulations from water resource recovery facilities (WRRFs) have become more stringent to reduce the damage of eutrophication caused by excess amounts of P. Enhanced biological phosphorus removal (EBPR) is a popular method to help comply with these regulations combined with other practices such as filtration. However, the sludges from facilities that employ EBPR (bio-P sludges) are difficult to dewater and require more polymer than conventional sludge, thereby increasing the cost of solids handling at these facilities. The monovalent to divalent (M/D) cation ratio, which is important in dewatering, is reportedly altered during the EBPR process. P speciation has also been suggested to affect dewaterability. Dewaterability is defined as the ease of separating water from the solids and was quantified by capillary suction time (CST). Yet, it is not well understood how the M/D cation ratio or P speciation correlate to dewaterability for different types of biosolids. Laboratory experiments were conducted in the Water Quality Center at Marquette University to meet the following objectives: i) determine the effects of the addition of different cations at various doses on dewaterability as CST of non-digested sludges, ii) determine how the M/D cation ratio is correlated to dewaterability as CST for various digested biosolids, and iii) determine if the alteration of P speciation correlates to dewaterability as CST for various biosolids. Batch studies were performed on primary and bio-P sludge to evaluate the effects of the addition of different chemicals on CST and P speciation. Using results from the batch study, a chemical pretreatment was chosen to be implemented in a lab-scale anaerobic digestion study because it improved the dewaterability of the primary and bio-P sludges. The pretreatment chosen was an addition of 200 meq/L calcium hydroxide followed by the addition of hydrochloric acid for pH adjustment. Lab-scale anaerobic digesters were fed with the chemically pretreated sludge to determine how altering the M/D cation ratio and P speciation affected dewaterability as CST. The pretreatment significantly improved the CST of the bio-P anaerobically digested biosolids relative to the control digesters but had little to negative effect on the digested primary biosolids. The chemical pretreatment decreased the M/D cation ratios for both types of biosolids, but the pretreatment had a positive correlation with CST for only the bio-P biosolids. Interestingly, these results reveal that the M/D ratio might not universally affect dewaterability as CST for all sludges and biosolids. P speciation may also play a role in dewaterability as CST. Higher amounts of particulate P improved the CST of the bio-P biosolids, but again, this was opposite for the primary biosolids. The role of specific P species on dewaterability as CST requires further investigation.

# ACKNOWLEDGEMENTS

Eileen M. Kennedy

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# DEDICATION

I would like to dedicate this thesis to my family for their continuous support throughout my life, and to my friends whose encouragement gives me strength every day.

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# 1 INTRODUCTION

## 1.1 Motivation

Regulations on phosphorus (P) effluent concentrations from water resource reclamation facilities (WRRFs) have become more stringent, with some regulations as low as 5-10 μg/L (Mayer, et al., 2016). P is essential to all life but can cause severe aquatic health issues downstream of WRRFs. An excess of P in the presence of nitrogen can cause eutrophication, or algae blooms, and create dead zones that are detrimental to aquatic life (Macintosh, et al., 2018). A popular method for biological wastewater treatment for removal of P is enhanced biological phosphorus removal (EBPR) (Yang, Shi, Ballent, & Mayer, 2017).

Research has shown that EBPR biosolids are more difficult than conventional biosolids to dewater, where biosolids are sludge that have been anaerobically digested and dewatering is the separation of water from solid sludge matter (Vesilind, 1994). Difficulty in dewatering results in higher solids handling costs (Higgins, Bott, Schauer, & Beightol, 2014). The sludge produced from EBPR, i.e. bio-P sludge, releases phosphate during anaerobic digestion, and phosphate readily binds with divalent cations. It has been suggested that a high monovalent to divalent (M/D) cation ratio negatively affects dewaterability, which might explain why EBPR sludge dewaters more poorly than conventional sludge (Higgins & Novak, 1997). Research that investigates the role of the M/D ratio on dewaterability of various types of sludges is sparse. Moreover, recent research has started to consider there are different forms of P in sludge (i.e. reactive, nonreactive, particulate, soluble (Venkiteshwaran, McNamara, & Mayer, 2018)), but the role of P speciation on sludge dewaterability has not been identified.

This thesis research investigated how differences in P speciation and the M/D ratio affected dewaterability. Lab-scale anaerobic digesters were used to generate primary digested biosolids and bio-P digested biosolids to determine if the M/D ratio correlated to dewaterability in the same way across various sludge types.

## 1.2 Objectives

The goal of this research was to understand if and how dewaterability as a function of capillary suction time (CST) is correlated with different characteristics, such as the M/D cation ratio and P speciation, of various types of biosolids produced by a chemical pretreatment for anaerobic digestion. Though, the enhancement of anaerobic digestion by the pretreatment was not evaluated. Various characteristics can affect dewaterability, including sludge type, floc structure, M/D cation ratio, and others. It has also been suggested that P speciation could affect dewaterability (Higgins, et. al., 2014), but this has not been proven, and therefore is a knowledge gap this study aims to fill. t. The three specific objectives of this research were to:

1. Determine the effects of the addition of different cations at various doses on dewaterability as CST of non-digested sludges
2. Determine how the M/D cation ratio is or is not correlated to dewaterability as CST for various digested biosolids
3. Determine if the alteration of P speciation correlates to dewaterability as CST for various biosolids

## 1.3 Approach

The research focused on improving the dewaterability as a function of CST of anaerobically digested biosolids and how the CST correlates to P speciation and M/D cation ratio using a chemical pretreatment. This pretreatment was selected because batch tests indicated it improved the CST of different sludge types (see Section 4.1).

The methods were designed to achieve the stated objectives. For each objective, a hypothesis was formulated (stated in Chapter 2) and then tested. A batch study was designed to test how various chemicals affected the CST and to select a pretreatment to use later in lab-scale studies. Lab-scale digesters were used to examine how the chemical pretreatment impacted the CST of different types of anaerobically digested biosolids. Digesters receiving pre-treated sludge and control digesters receiving sludge that was not pre-treated were operated in parallel. Half of the digesters were fed with primary sludge and the other half were fed with a blend of primary and waste activated sludge (WAS) bio-P sludge. The M/D cation ratio and P speciation were measured in effluent biosolids from all digesters and correlation analysis was conducted with biosolids characteristics and dewaterability as a function of CST. Graphpad Prism (Graphpad Software Inc., CA, USA) and the open statistical program ‘R’ were used to determine correlations (R Core Team, 2018).

## 1.4 Thesis Structure

A literature review describing sludge characteristics and how the characteristics relate to dewaterability is presented in **Chapter 2**. The experimental methods are presented in **Chapter 3** to provide a detailed account of how results were obtained. **Chapter 4** presents the experimental results and discussion. Conclusions drawn from the results and a summary of the research is presented in **Chapter 5**. Appendices are attached with supporting graphics and data.

# 2 LITERATURE REVIEW

## 2.1 General Goals of Biosolids Dewatering

Dewatering of sludge is the separation of water from solid sludge matter (Vesilind, 1994). Dewatering differs from thickening, as it aims to reduce the water adhered to sludge particles, where thickening reduces overall water content (Vesilind, 1994).Dewatering is often completed before the solid sludge reaches its final destination, although it is not required (Metcalf & Eddy, Inc., 2003). Sludge matter must be stabilized before disposal but can be dewater before or after stabilization. The handling of these solids is important for nutrient reuse and solids disposal and is related to the capital and operating costs of water resource recovery facilities (WRRFs). The cost of solids handling may equate to 25% to 50% of the total operations cost of a WRRF (Karr & Keinath, 1978). These high costs are influenced by high polymer costs used to dewater and trucking costs to transport the solids. To decrease the cost of solids handling and disposal, the solid sludge matter is often dewatered (McNamara & Lawler, 2009). The sludge has a high-water content and the water can be released through thickening and dewatering systems such as gravity belt thickeners (before anaerobic digestion) and belt filter presses or centrifuge systems (after anaerobic digestion). These processes can be costly due to the required addition of polymers to aid the dewatering (Christensen, Keiding, Nielsen, & Jørgensen, 2015). The dewaterability of different sludges is influenced by sludge characteristics.

## 2.2 Wastewater Solids Characteristics that Affect Dewaterability

### 2.2.1 Monovalent to Divalent (M/D) Cation Ratio

The monovalent to divalent (M/D) cation ratio is associated with bioflocculation, or the physical arrangement of bacterial flocs, and can also be used to predict dewaterability of activated sludge (Higgins & Novak, 1997). According to the divalent cation bridging theory (DCBT), when phosphate is released from the cell along with magnesium and potassium, the amount of soluble magnesium and calcium decreases because phosphate binds to these divalent cations. The release of potassium will then increase the monovalent cation concentration and deteriorate the floc strength (Higgins & Novak, 1997; Sobeck & Higgins, 2002). The DCBT also suggests that decreasing the M/D cation ratio will improve the dewaterability of activated sludge (Sobeck & Higgins, 2002). Divalent cations, such as calcium and magnesium, were also added to industrial activated sludge to decrease polymer use and improve overall dewaterability (Higgins, Tom, & Sobeck, 2006). These studies focused on dewaterability and soluble M/D ratio of activated sludge. However, additional research is required to determine if this ratio correlates to dewaterability in anaerobically digested biosolids.

### 2.2.2 Floc Structure

Characteristics influencing sludge dewaterability include residual moisture of the cake (bound water) and ease of filtration (Chen, Lin, & Lee, 1996). Specifically, floc structure and the water contained within the floc play a large role in dewatering. Flocs contain three different types of water: interstitial, vicinal, and bulk or free water. The bulk water is the water surrounding the floc. As shown in Figure 2.1, interstitial water is the water trapped in the spaces between particles and organisms (Vesilind, 1994). This water is held in the floc structure or microbe structure and can be released if the structure is destroyed. Vicinal water is the water on the surface of the particle. This layer is held by hydrogen bonds that causes the water to adhere to the particle's surface. The water of hydration is the water chemically bound to the particles and can only be removed by heating the solids (Vesilind, 1994). Vesilind (1994) determined that adding chemicals, such as polymer, will only aid in releasing the interstitial water in the dewatering process.

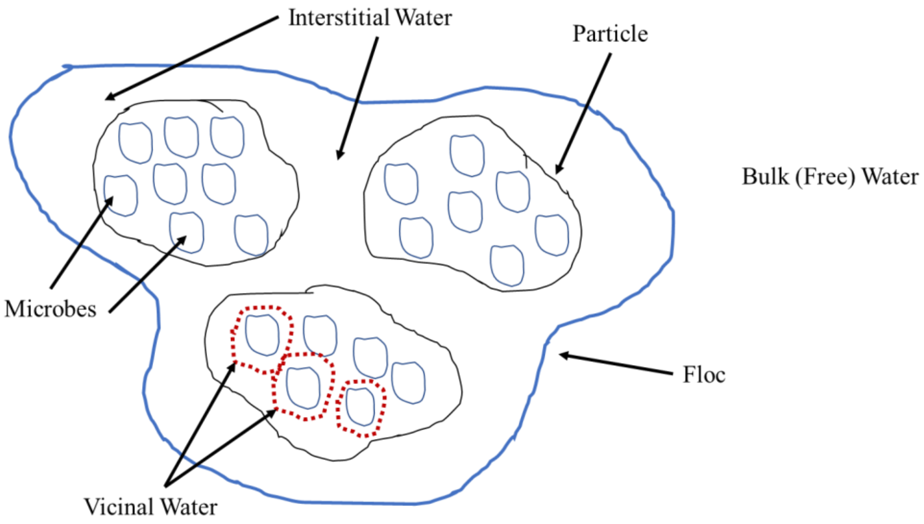

***Figure 2.1: Diagram of a sludge floc including free and bound water.*** *Adapted from Vesilind, 1994.*

Extracellular polymer (ECP) is the organic polymer that allows microorganisms to adhere to the surfaces of particles and other cells and can also affect dewaterability. Increasing the amount of ECP in raw and digested sludges has been found to decrease dewaterability (Houghton, Quarmby, & Stephenson, 2001). ECP is plentiful in activated sludge and is dependent on pH. At neutral pH, the ECP is negatively charged (Christensen, Keiding, Nielsen, & Jørgensen, 2015). This charge affects the structure of the floc and how easily the activated sludge can be dewatered. The pH can also affect particle size. At a high pH, there are more small particles and suspended ECP present that negatively affect dewaterability (Christensen, et. al., 2015). Karr and Keinath (1978) note that in anaerobic digestion specifically, dewaterability is poor at first, but improves once the small particles are destroyed. According to Lawler et al. (1986), the specific surface area directly impacts dewaterability of digested biosolids. If a sludge is digested properly, the small particles are destroyed and allow for less surface area for water to adhere to, therefore improving dewaterability.

### 2.2.3 Phosphorus Speciation

The P found in water can be divided into four fractions: soluble reactive P (sRP), particulate reactive P (pRP), soluble nonreactive P (sNRP), and particulate nonreactive P (pNRP) (Venkiteshwaran, McNamara, & Mayer, 2018). Soluble P is defined as the P that passes through a 0.45 μm filter, whereas particulate P is retained on the filter (APHA, 1998). The reactive P is also known as orthophosphate, or inorganic P, and is readily available to react with cations (Venkiteshwaran, et. al., 2018). From this, it can be inferred that reactive P is not favorable for dewaterability because it readily binds with divalent cations. No studies have been performed that definitively determined how other P fractions affect dewaterability (Anderson, 2018).

## 2.3 Impacts of Enhanced Biological Phosphorus Removal (EBPR) on Dewaterability

Enhanced biological phosphorus removal (EBPR) results in a biological phosphorus (bio-P) sludge that is often more costly to dewater than conventional WAS. According to Higgins (2014), EBPR processes negatively affect dewaterability of sludge after anaerobic digestion. EBPR decreases the monovalent to divalent (M/D) cation ratio of the sludge and weakens floc structure resulting in poor dewaterability. The M/D cation ratio directly relates to the concentration of cake solids produced after dewatering and the amount of P directly relates to the M/D cation ratio (Higgins, et. al., 2014). The presence of soluble P was also detrimental to the dewaterability of the bio-P sludge (Higgins, et. al., 2014).

## 2.4 Alkaline Pre-Treatment to Improve Dewaterability

Many studies have added cations to activated sludge to improve dewaterability, but the cations can also affect the pH of the sludge. High pH decreases dewaterability and the addition of acid does not produce a noticeable effect on dewatering (Christensen, et. al., 2015). When soluble calcium was added to activated sludge, the porosity of the flocs decreased and created a stronger floc structure and dewaterability was improved. Multivalent cations adhered to particle surfaces and reduced the amount of ECP by neutralizing the charge and strengthening the floc (Christensen, et. al., 2015). The addition of calcium and other cations affects the M/D cation ratio. More divalent cations leads to better dewaterability (Higgins, Bott, Schauer, & Beightol, 2014). The soluble calcium concentration was found to correlate with good dewaterability of anaerobically digested WAS (Alm, Sealock, Nollet, & Sprouse, 2016). Low doses of calcium added as a pretreatment also aided in anaerobic digestion of WAS (Ray, Lin, & Rajan, 1990).

## 2.5 Summary of Research Needs

Existing literature suggests that the addition of multivalent cations will improve dewaterability of anaerobically digested biosolids. There are gaps in the current understanding of how P speciation influences the dewatering of different sludges and biosolids. Understanding the roles of the M/D cation ratio and P speciation in dewaterability will allow WRRFs to reduce solids handling costs. In this study, various anaerobically digested biosolids were produced using a chemical pretreatment to assess the effects of the M/D cation ratio and P speciation. Three hypotheses were tested that corresponded to the three specific objectives:

Objective 1: Determine the effects of the addition of different cations at various doses on dewaterability as CST of non-digested sludges

*Hypothesis: Cations with higher valences, and higher doses of these multivalent cations, will improve CST.*

Objective 2: Determine how the monovalent to divalent cation ratio is correlated to dewaterability as CST for various digested biosolids.

*Hypothesis: A lower M/D cation ratio (more divalent cations present) will correlate to better CST.*

Objective 3: Determine if the alteration of P speciation correlates to dewaterability as CST for various biosolids

*Hypothesis: The addition of divalent cations will alter P speciation and breakdown small sludge particles. The divalent cations will bind with the soluble P and cause more particulate P to be present, decreasing the amount of small sludge particles and free P, therefore improving CST.*

# 3 METHODOLOGY

## 3.1 Batch Studies to Assess Impacts of Chemical Pretreatments on Dewaterability as Capillary Suction Time

Batch tests were performed on different sludges to assess the effects of the addition of different cations at various doses on dewaterability as CST (Objective 1). It was proposed that adding cations with higher valences (trivalent > divalent > monovalent) and higher concentrations of the multivalent cations would improve CST. Primary sludge was collected from South Shore Water Reclamation Facility (SS) in Oak Creek, WI and bio-P sludge was collected from Fond du Lac Wastewater Treatment & Resource Recovery Facility (FDL) in Fond du Lac, WI. Hydrochloric acid, sodium hydroxide, calcium hydroxide, sodium chloride, calcium chloride, and ferric chloride were added to 50 mL samples of primary sludge. The chemicals were added at doses of 0.2, 2, and 200 meq/L (Table 3.1). The sludge samples were stirred for approximately 5 minutes on a magnetic stir plate before batch tests were performed. After this time, the pH of the samples was measured and adjusted to the original pH of the sludge using hydrochloric acid or sodium hydroxide. The dewaterability of each sample was measured using capillary suction time, described in section 3.1.1. P speciation tests were performed using the ascorbic acid method (Standard Method 4500-P E.).

***Table 3.1: Doses of chemicals added to different sludge types.*** ** indicates samples where pH was adjusted.*

| Chemical | Dose (meq/L) | pH | Sludge Type |
|---|---|---|---|
| HCl | 200 | 1.29 | Primary |
| | 20 | 5.13 | |
| | 2 | 5.63 | |
| NaOH | 200 | 12.92 | Primary |
| | 20 | 6.93 | |
| | 2 | 5.88 | |
| Ca(OH)2 | 200 | 12.54 | Primary |
| | | 5.17* | |
| | | 5.74* | Bio-P |
| NaCl | 200 | 5.53 | Primary |
| CaCl2 | 200 | 5.41 | Primary |
| FeCl3 | 200 | 2.79 | Primary |
| | | 5.93 | |

### 3.1.1 Capillary Suction Time (CST) for Dewatering

Dewaterability was determined using a multi-purpose CST apparatus (Triton Electronics Limited, Great Dunmow, Essex, England). The CST apparatus measured the time for water to move across filter paper (GE Whatman, Grade 17 7x9cm) from an inner diameter (31.75 mm) near an input well to an outer diameter (45.72 mm) (Figure 3.1). The CST was measured as the time it took for water to travel through the filter from the inner diameter to the outer diameter. The time was measured in seconds, and a high CST value indicates poor dewaterability while a low CST value indicates good dewaterability. Although using a CST device has shortcomings, such as limitations on measuring bond-water content and percent of cake solids, it is the most widely used method to measure dewaterability (Ngwenya, Higgins, Beightol, & Murthy, 2017; Scholz, 2005). CST has been used to measure dewaterability in multiple studies for more than forty years because the test is simple to conduct and it is precise (Vesilind, 1988; Houghton, Quarmby, & Stephenson, 2001; Feng, et al., 2009; Yu, et al., 2009).

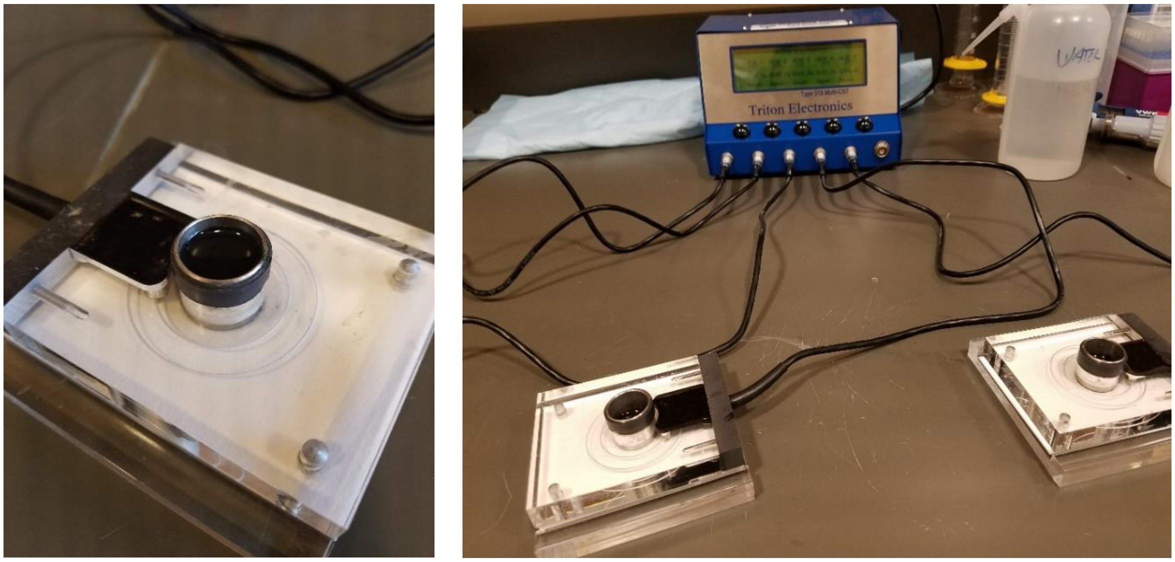

***Figure 3.1 CST set up for dewatering test.*** *The CST measuring device is shown on the right with a close-up view of the input well and inner and outer diameters on the left.*

## 3.2 Anaerobic Digester Lab-Scale Experiments

### 3.2.1 Purpose of Experiments

Lab-scale anaerobic digesters were operated to generate different biosolids types and asses the correlations between M/D cation ratio, P speciation, and CST, as described in the following research objectives to test the following hypotheses:

Objective 2: Determine how the M/D cation ratio is correlated to CST for various digested biosolids.

> *Hypothesis: A lower M/D cation ratio (more divalent cations present) will correlate to better CST.*

Objective 3: Determine how the alteration of P speciation correlates to dewaterability as CST for various biosolids

*Hypothesis: The addition of divalent cations will alter P speciation and breakdown small sludge particles. The divalent cations will bind with the soluble P and cause more particulate P to be present, decreasing the amount of small sludge particles and free P, therefore improving CST.*

### 3.2.2 Influent Sludge Sources

Two types of sludge were used as influent feed for these lab-scale anaerobic digestion experiments. Screened primary sludge was collected from SS. The sludge was collected on site approximately every two weeks and stored at 4 °C until use (at most 1.5 weeks). The bio-P sludge used was a blend of primary and WAS from FDL. The bio-P sludge was shipped weekly on ice to the Water Quality Center at Marquette University in Milwaukee, WI. According to the wastewater superintendent, the activated sludge system at Fond du Lac was run intermittently as bio-P approximately 50-60% of the time. A daily log from FDL indicating the days the activated sludge system was running as bio-P can be found in the Appendix. The bio-P sludge was stored in 4 °C fridge until use (at most 1.5 weeks).

### 3.2.3 Digester Set Up & Operation

Four sets of duplicate digesters were used in this study (Figure 3.2). Two were duplicate control digesters fed with untreated bio-P sludge from FDL. Effluent samples from these two digesters are referred to as BC1 and BC2 because they were bio-P influent sludge operated under control conditions (i.e. no pre-treatment). Two duplicate digesters were fed with chemically treated bio-P sludge. Effluent samples from these two digesters are referred to as BT1 and BT2 because they were bio-P influent sludge operated under treated conditions. Two duplicate control digesters were fed with untreated primary sludge from South Shore. Effluent samples from these two digesters are referred to as PC1 and PC2 because they were primary influent sludge operated under control conditions (i.e. no pre-treatment). The final two duplicate digesters were fed with chemically treated primary sludge. Effluent samples from these two digesters are referred to as PT1 and PT2 because they were primary influent sludge operated under chemically treated conditions. Table 3.2 provides a summary of the four sets of digesters with the corresponding abbreviation, sludge type, origin location, and color used in data presentation.

***Table 3.2 Abbreviations for lab scale digesters**. The colors correspond to colors used in Results figures.*

| Name | Abbreviation | WWRF | Sludge Type | Color |
|---|---|---|---|---|
| Primary Control 1 | PC1 | SS | Primary | 🟦 |
| Primary Control 2 | PC2 | SS | Primary | 🟦 |
| Primary Treated 1 | PT1 | SS | Primary | 🟦 |
| Primary Treated 2 | PT2 | SS | Primary | 🟦 |
| Bio-P Control 1 | BC1 | FDL | Blended | 🟩 |
| Bio-P Control 2 | BC2 | FDL | Blended | 🟩 |
| Bio-P Treated 1 | BT1 | FDL | Blended | 🟩 |
| Bio-P Treated 2 | BT2 | FDL | Blended | 🟩 |

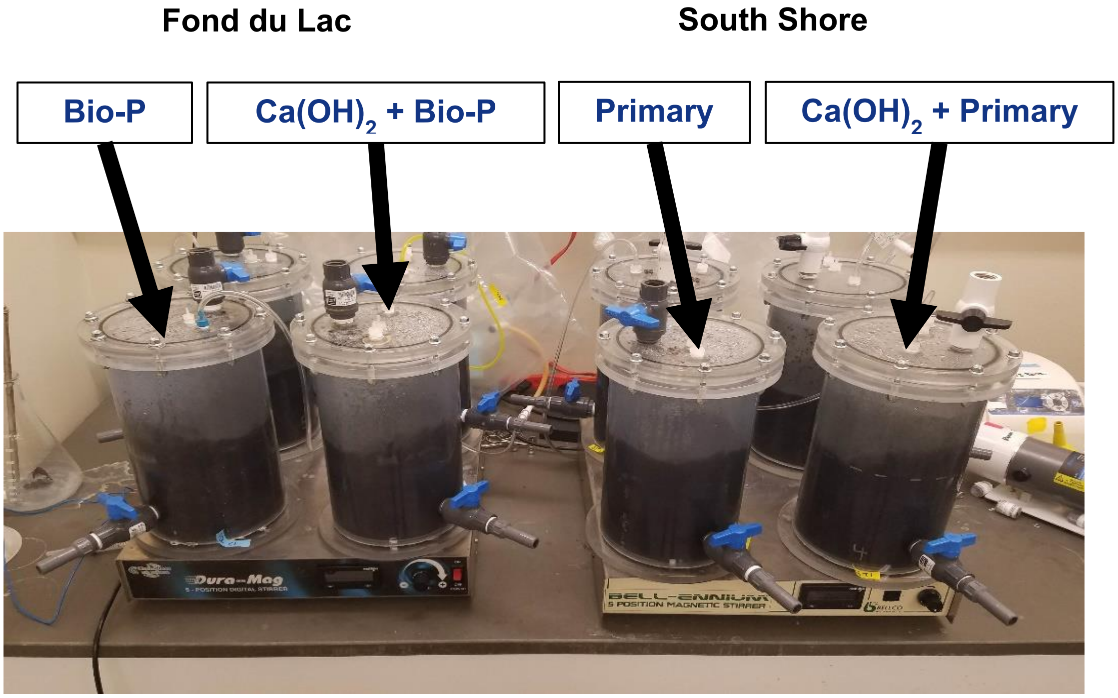

***Figure 3.2 Digester influent type and location.** Each digester had a solids residence time (SRT) of 15 days. 150 mL of effluent was taken out each day and 150 mL of the specific influent was added each day.*

All lab-scale digesters were seeded with digester effluent from South Shore. The operating volume of each digester was 2.25 L. Each digester was sparged with 30% carbon dioxide and 70% nitrogen gas at the start of operation. A small port on the lid of each digester was connected to a Tedlar bag for gas release and collection until gas analysis. The digesters were operated with a 15-day SRT for 78 days total (i.e., more than five SRT values to ensure quasi steady state was reached and to conduct quasi steady state analysis). In accordance with the SRT, 150 mL of biosolids were removed as effluent daily and 150 mL of feed sludge was added to each digester (Figure 3.2). Quasi steady state was considered to be achieved after three SRT values (after 45 days). Digesters were operated at 35 °C in a temperature-controlled room and stirred on magnetic stir plates operated at 140 to 150 rpm for 7 hours a day. The magnetic stir plates operated on a timer and digesters were stirred before, during, and after feeding.

The chemically treated influent was made by amending the control influent sludge with 200 meq/L calcium hydroxide and mixing for two minutes using a stir bar and magnetic stir plate. The sludge pH was then lowered to approximately 8.5 using hydrochloric acid. Initially during Days 1-40, the pH was lowered to a neutral pH (7 to 7.5) but this method was adjusted to keep the effluent pH from dropping and prevent the digesters from souring. A decline in pH was beginning to occur in the chemically treated influent, possibly from acid addition. The switch to an influent pH of 8.5 occurred on Day 41.

Effluent biosolids parameters were measured during operation to monitor digester health including:

- pH (measured daily)

- biogas methane concentration (measured twice per week as previously described by Venkiteshwaran, et al. (2017) using gas chromatography coupled to a thermal conductivity detector (GC-TCD) (GC System 7890A, Agilent Technologies, Irving, TX, USA)
- biogas volume (measured twice per week using a wet test meter (Precision Test Company, San Antonio, Texas, United States))
- total and volatile solids (measured twice per week using Standard Method 2540-G)
- alkalinity (measured twice per week according to Standard Method 2320 B: Alkalinity by Titration)
- volatile fatty acids (VFAs) (measured twice per week via gas chromatography (GC System 7890A, Agilent Technologies, Irving, TX, USA) using a flame ionization detector as described in Venkiteshwaran, Benn, Seyedi, & Zitomer (2019)).

The following effluent biosolids characteristics were measured for dewaterability correlation analysis:

- Cation concentration (total concentration measured once per week using nitric acid digestion and inductively coupled plasma mass spectrometry (ICP-MS) as previously used in Kappell, Harrison, & McNamara (2019)). Monovalent (sodium and potassium) and divalent (calcium and magnesium) cations were quantified and used to determine the monovalent to divalent cation ratio.

- P speciation (measured once per week using the ascorbic acid method (Standard Method 4500-P E.) The ascorbic acid method only measures total P (TP), total soluble P (sTP), reactive P (RP), and soluble reactive P (sRP). The non-reactive P (NRP) fractions and particulate P factions were determined by subtraction with the knowledge that NRP and RP is equal to the TP and that sTP and particulate P (pTP) is equal to TP).
- Dewaterability (measured two to five times per week via CST).

### 3.2.4 Statistical Analyses

Ordinary one-way analysis of variance (ANOVA) multiple comparisons tests were used to compare digester data for each characteristic. Graphpad Prism (Graphpad Software Inc., CA, USA) was used to determine if data was statistically significant (p-value < 0.05).

Statistical analysis for the comparison of multiple characteristics was performed by Dr. Anthony Kappell using the open statistical program ‘R’ (R Core Team, 2018). All corresponding statistical plots were generated from the analysis. Analysis of variance using the ‘anova’ function on ‘lm’ function of linear model was used to determine significant factors contributing to the differences observed in the CST values for the main factors. The main factors examined were measurements taken from the same sample (i.e. CST, pH, M/D cation ratio). Consistent factors were the source of the sludge and if it was treated or untreated. Groups for analysis consisted of P measurements (TP, sRP, pRP, and sNRP), cation measurements (Mg, Ca, Na, K, divalent, monovalent, and M/D cation ratio), the production of biogas and methane, and solids (total solids and volatile solids).

Post hoc multiple pairwise comparisons were conducted using Tukey's honestly significant differences (HSD) by the 'TukeyHSD' function. Pearson correlations for the variables were calculated using the 'cor.test' function on measurements taken from the same sample. Significant correlations were visualized utilizing 'corrplot' function from the 'corrplot' R package (Wei & Simko, 2016). Significant differences were defined as p-value less than 0.05.

# 4 RESULTS & DISCUSSION

## 4.1 Preliminary Batch Capillary Suction Time Results

Different chemicals were added to primary sludge to see how they affected CST. The pH was adjusted with either hydrochloric acid or sodium hydroxide after chemical addition when there was a large change in pH to determine if the CST results were related to the chemical addition or change in pH. The optimal chemical for CST improvement was chosen and then the test was performed on bio-P sludge to determine if the chemical provided similar results.

Cation addition affected CST, and the effect was not just due to changes in pH (Figure 4.1). Experiments with calcium hydroxide with and without pH adjustment both resulted in improved CST (p-values $< 0.0001$, one-way ANOVA) (see 3rd and 4th bars compared to 1st bar in Figure 4.1). The addition of calcium hydroxide improved CST more than the addition of calcium chloride, but the difference was not due to pH. The addition of monovalent cations, via sodium hydroxide addition, had a negative impact on CST. The addition of ferric chloride also improved CST, but not to the extent that the addition of calcium hydroxide improved CST.

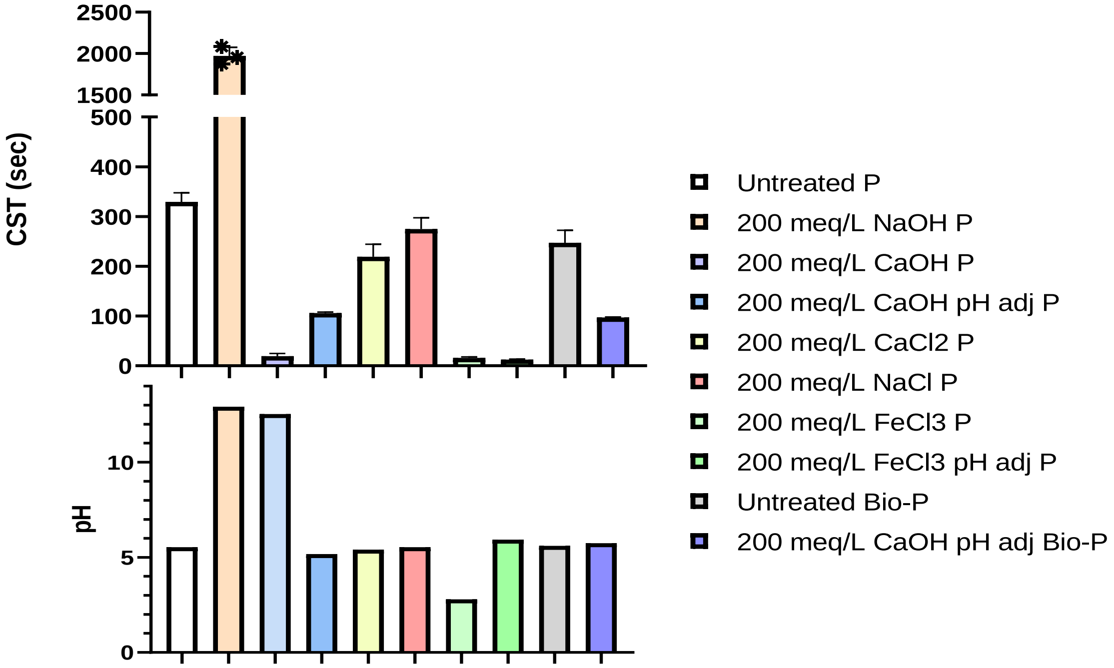

***Figure 4.1: Preliminary average CST results (top) and corresponding pH of each sample (bottom).*** ** Indicates data point off the plot. 200 meq/L NaOH resulted in a CST of 1970 seconds. Error bars represent one standard deviation of three replicate measurements. Some error bars are small and not visible.*

Improved CST was not dependent on the pH of the sludge but was dependent on the cations added. Calcium hydroxide raised the pH of the sludge and ferric chloride decreased the pH, but both chemicals improved the CST of primary sludge significantly (p-values <0.0001). Since ferric chloride is a common metal used for the removal of phosphorus and residual ferric chloride does not already improve dewaterability, calcium hydroxide with hydrochloric acid was chosen for the pre-treatment base addition. Calcium hydroxide with hydrochloric acid was chosen over calcium hydroxide alone because the CST was still improved when the pH was changed. The combination of calcium hydroxide and hydrochloric acid was also used so that a near-neutral pH could be fed to digesters to prevent the digesters from becoming extremely basic. This combination was then added to bio-P sludge and the treatment provided similar improved CST as it did for the primary sludge (see and compare last two columns on righthand side in Figure 4.1). There was significant difference between the untreated and the chemically treated bio-P samples (p-value = 0.0019, one-way ANOVA).

### 4.2 Digester Health & Performance

Digester health and performance were monitored by measuring methane production, pH, alkalinity, cumulative biogas production, and VFAs. Overall, digester health and performance were similar between the four digester conditions, though PC2 had some outlier performance data.

On average, all digesters produced biogas with a methane concentration of over 60% during quasi steady state (Figure 4.2). One-way ANOVA comparison tests showed no significant difference among the biogas methane concentrations from all digesters (p-values > 0.7).

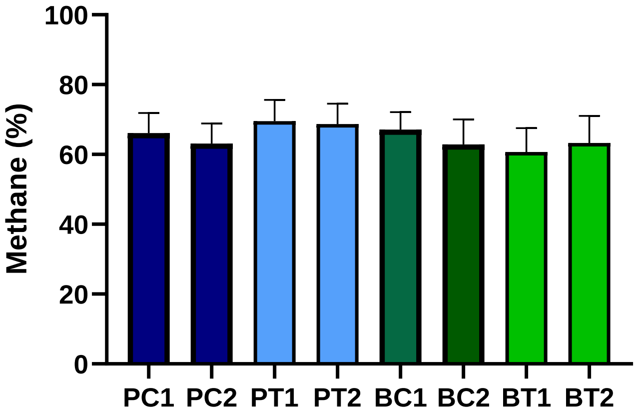

***Figure 4.2 Average methane production in percentage of biogas from all digesters during quasi steady state.*** *Error bars represent one standard deviation of all measurements taken during quasi steady state.*

The average effluent pH during quasi steady state for each digester was near neutral and above 6.8, implying that methanogenic activity was not inhibited by low or high pH (Metcalf & Eddy, Inc., 2003, p. 635) (Figure 4.3). One-way ANOVA comparison tests showed significant differences between the treated and the control effluent pH for each sludge type (p-values <0.0001).

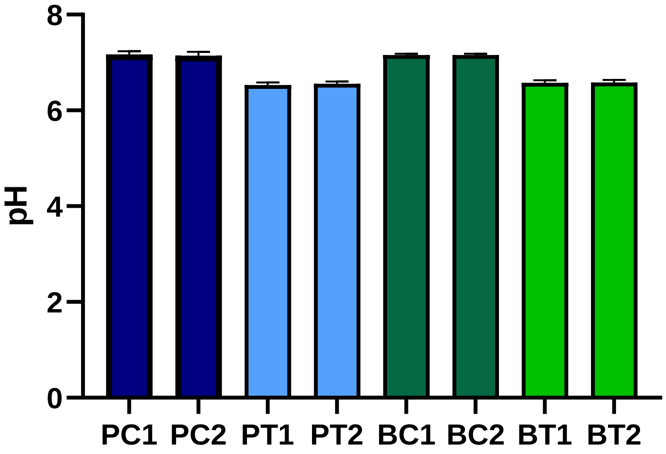

***Figure 4.3 Average effluent pH of all digesters during quasi steady state****. Some error bars are small and not visible.*

For the four digester conditions, the average alkalinity during steady state was between 5500 and 6500 mg/L as $CaCO_3$ (Figure 4.4). This range is higher than the range of 3000 to 5000 mg/L for typical anaerobic digestion (Metcalf & Eddy, Inc., 2003, p.

635). A one-way ANOVA comparison test showed no significant difference between the control and treated effluent alkalinity for each sludge type (p-values > 0.05).

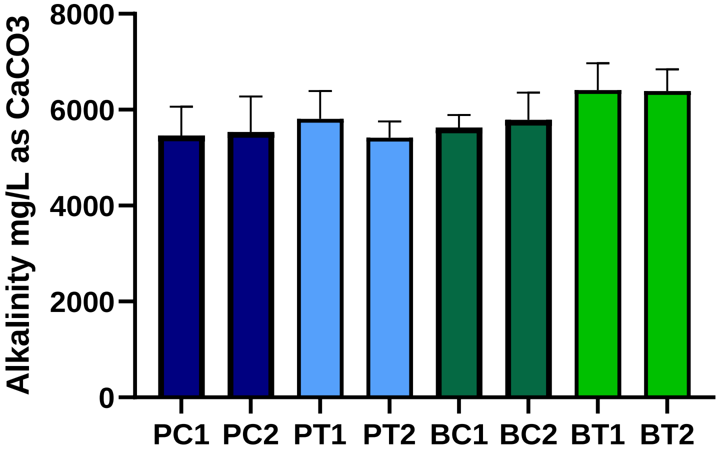

***Figure 4.4 Average alkalinity in mg/L as CaCO$_3$ of all digesters during quasi steady state.*** *The error bars correspond to one standard deviation of all measurements taken during quasi steady state.*

The cumulative biogas production for the bio-P digesters was less than that of the primary digesters during quasi steady state (Figure 4.5). There was not much variation in the cumulative gas production of the bio-P digesters (approximately 1.9 and 1.5 L biogas/ day for controls and treated, respectively). The treated primary digesters produced more biogas than the primary control digesters (2.3 L biogas/ day). The second control for the primary digesters (PC2) produced less biogas than its duplicate for a period during steady state, but simple linear regression analysis determined that biogas production resumed. The biogas production rates for both primary controls were almost the same (1.7 and 1.8

L biogas/day for PC1 and PC2, respectively). PC2 for the conventional control digesters was determined to not be a true duplicate based on more analysis.

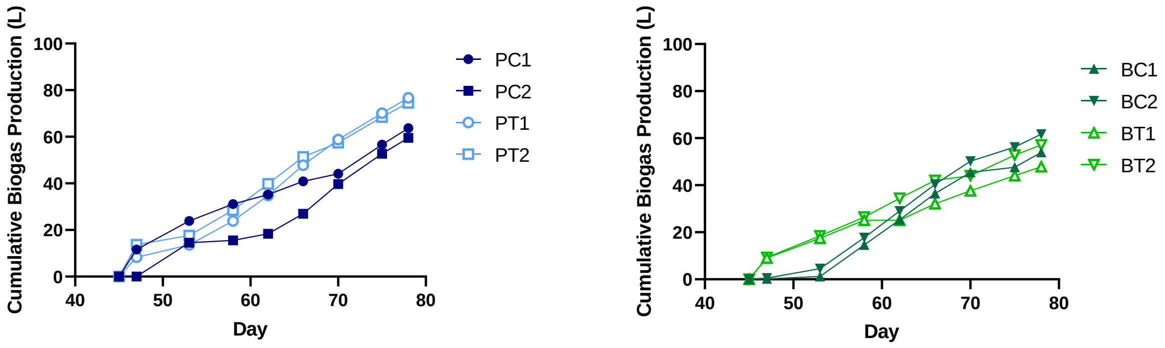

***Figure 4.5: Conventional (left) and bio-P (right) cumulative biogas production during quasi steady state.***

PC2 had significantly higher total VFAs than the other digesters (Figure 4.6). The effluent biosolids from PC2 became less viscous than its duplicate at the start of the quasi steady state. The digester was fed with 50 mL of effluent from PC1 to reduce digestate VFA concentration for one week after Day 46. It was determined later, at the end of this study, that the stir bar got caught in solids built up and was not stirring properly. Although the primary control duplicate malfunctioned, the digester still produced effluent sludge that was used for analysis. The objective of this study was not to test the impact of pre-treatment on anaerobic digestion, but to produce sludge of variable characteristics for correlation analysis of CST and biosolids characteristics.

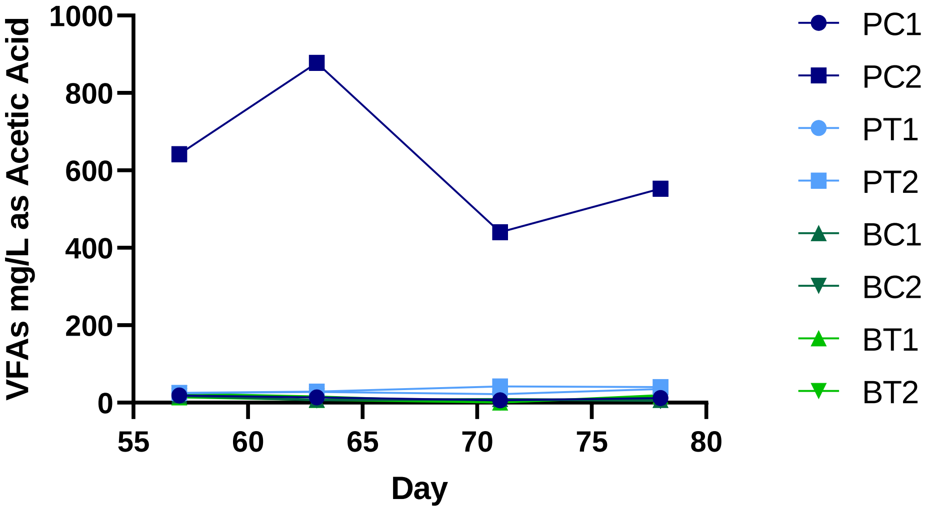

***Figure 4.6 Volatile fatty acids measured during quasi steady state.*** *Single measurements taken at a time.*

### 4.3 Capillary Suction Time of Effluent Biosolids from Bench Scale Digesters

The time series of CST data for the bio-P digester biosolids provides evidence that the pretreatment improved dewaterability after anaerobic digestion; the digesters fed pre-treated solids yielded biosolids with much lower CST values than the control digesters (Figure 4.7). Since EBPR changes the M/D cation ratio, the pretreatment likely shifted the ratio so that CST was improved (more divalent cations present), as suggested by Sobeck and Higgins (2002). The data provide evidence that the divalent cation bridging theory (DCBT) can also be applied to bio-P biosolids and not only activated sludge.

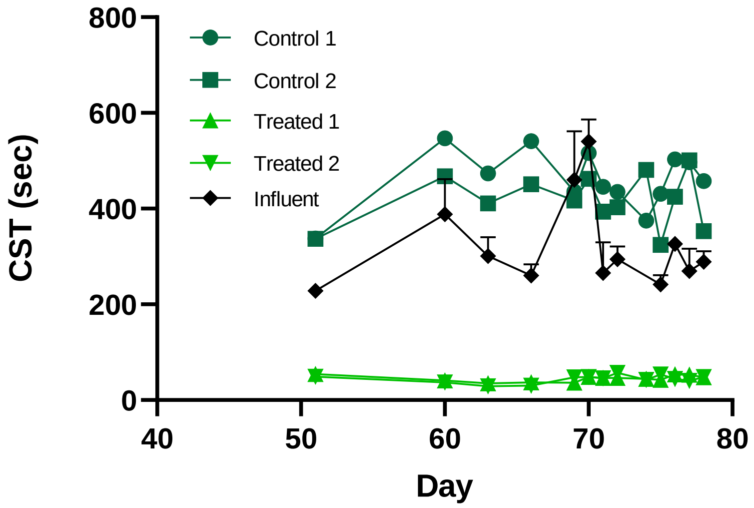

***Figure 4.7: Capillary suction time (CST) of digesters over quasi steady state for digesters fed with bio-P sludge.*** *Each measurement was performed in triplicate.*

Interestingly, the pretreatment did not improve the CST of the primary biosolids (Figure 4.8). This provides evidence that the DCBT, as suggested by Sobeck and Higgins (2002), cannot be applied to all sludge types. The M/D cation ratio may be a better indicator of CST for some biosolids over others. The M/D cation ratio may not be an indicator of improved CST for primary biosolids and may be only applicable to biosolids similar to bio-P biosolids.

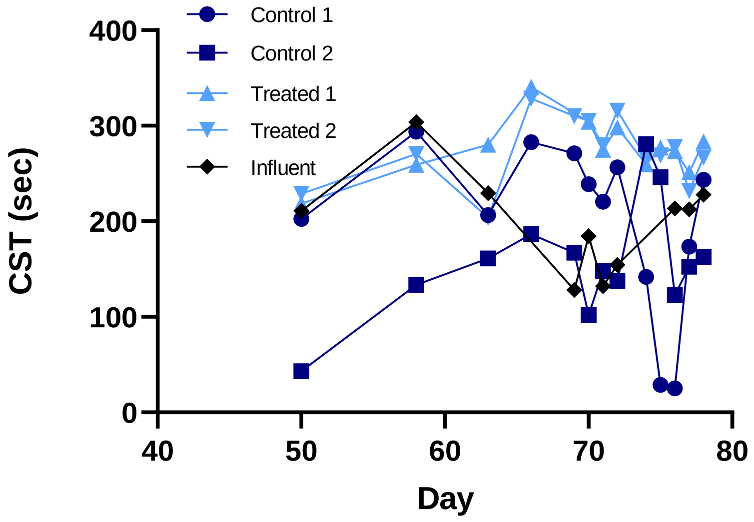

***Figure 4.8: Capillary suction time of digesters over quasi steady state for digesters fed with primary non-bio-P sludge.***

There is a statistical difference between the average CST of the control and treated digesters for the bio-P biosolids (Figure 4.9, right). A multiple comparison test revealed a statistically significant difference between the bio-P control and treated effluents and influent (p-values < 0.0001, one-way ANOVA). The average CST during quasi steady state for the control and treated primary digester effluent were not significantly different (Figure 4.9, left). A one-way multiple ANOVA comparisons test revealed no statistical difference between the primary control and treated effluents and influent (p-values > 0.05). However, this was not true for the second primary control duplicate (p-values < 0.1, one-way ANOVA). The chemical treatment significantly improved the CST of the bio-P digester effluent, but not the primary effluent.

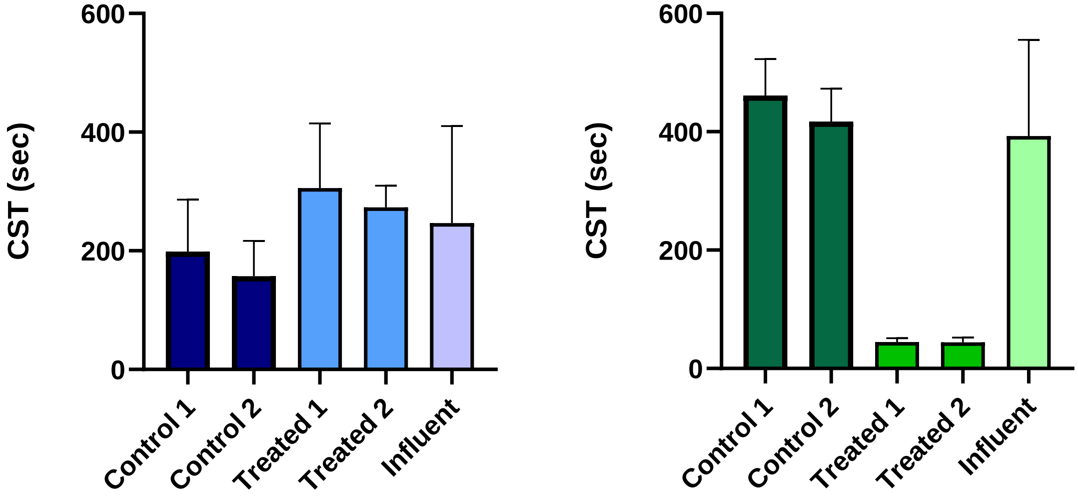

***Figure 4.9 Average capillary suction time (CST) during quasi steady state of digesters fed with primary sludge (left) and bio-P sludge (right).*** *The influent is untreated conventional and untreated bio-P sludge. The error bars correspond to one standard deviation.*

## 4.4 Bench Scale Digesters: Sludge Characteristics

The chemical pretreatment significantly altered the M/D cation ratio of the treated effluent biosolids (Figure 4.10). A one-way ANOVA multiple comparisons test revealed statistical difference between the primary control and treated effluents (p values < 0.001). However, there was a difference between the primary control duplicates. The ANOVA multiple comparisons test also revealed statistically significant differences between the bio-P control and treated effluents (p value < 0.0001). The increase in divalent cations significantly lowered the M/D ratio for both sludge types.

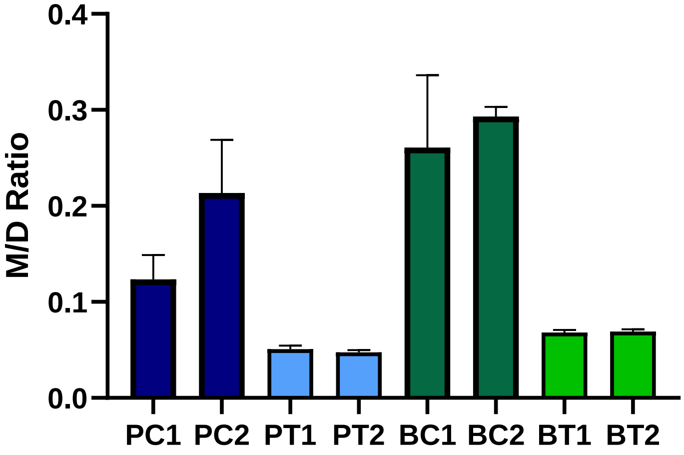

***Figure 4.10 Average effluent total monovalent to divalent cation ratio (M/D ratio) of all digesters during quasi steady state.*** *The error bars represent one standard deviation of all measurements taken during quasi steady state.*

Using R software, the linear correlation between dewaterability as CST and the M/D cation ration was graphed (Figure 4.11). The red line corresponds to the bio-P effluents. The data yielded a positive correlation between the M/D ratio and CST for bio-P digesters. When the M/D ratio increased, the CST also increased, indicating poor dewaterability. The blue line corresponds to the primary effluents. In this case, there was a negative correlation between M/D ratio and CST. The linear correlation plots provide evidence that a low M/D cation ratio may only be an indicator of improved CST for bio-P biosolids but poor CST for primary biosolids. It should be noted that the M/D cation ratio measured was the total ratio which includes free and complex metals. Further research is required to fully understand the correlation of the M/D cation ratio and CST across biosolids types from different WRRFs, specifically how free metals can improve CST.

Supplemental linear correlation plots for various sludge characteristics are presented in the Appendix.

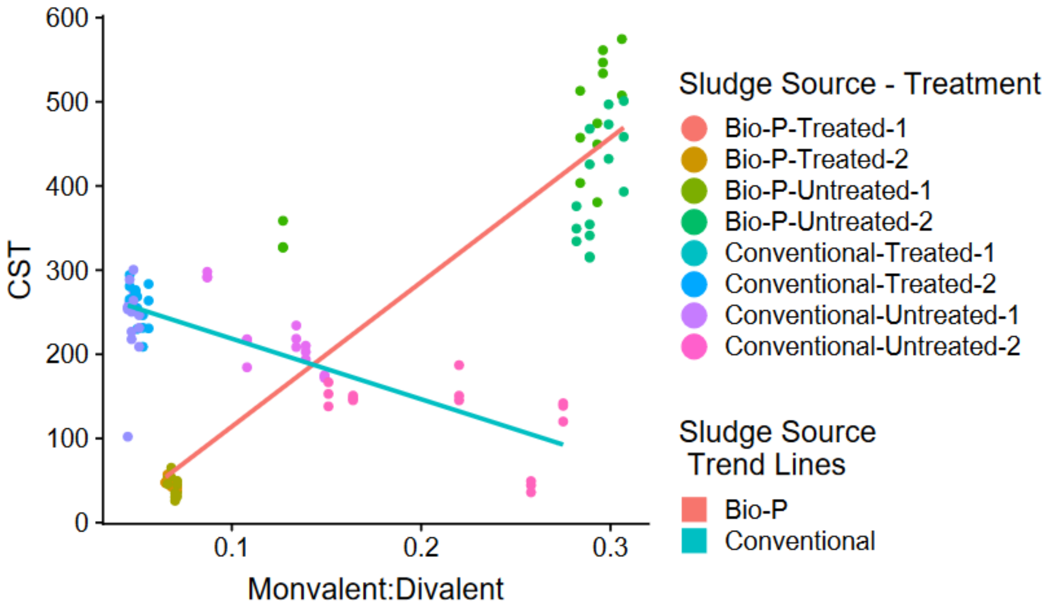

***Figure 4.11 Correlation plot comparing CST and M/D cation ratio.*** *Note that the total M/C cation ratio was measured and compared to CST values.*

The chemical pretreatment also altered the phosphorus speciation by shifting soluble P to particulate P (Figures 4.12 and 4.13). The amount of total P measured in this study should be the same for influent and effluent samples, but that is not the case. The total P values for bio-P biosolids are similar, but they are greater than the influent. The total P values of primary-digested samples are more variable. While P speciation should change during digestion, total P should not change. The differences in the amount of total P could be due to limitations in the methods or due to variable nature of influent sludge over time and, therefore, P speciation cannot be used as a clear indicator of CST in this study, but the information gathered was still used in correlation analysis to gain insight into how P speciation could be correlated to CST. More research must be performed to fully understand the role of P speciation in reference to CST.

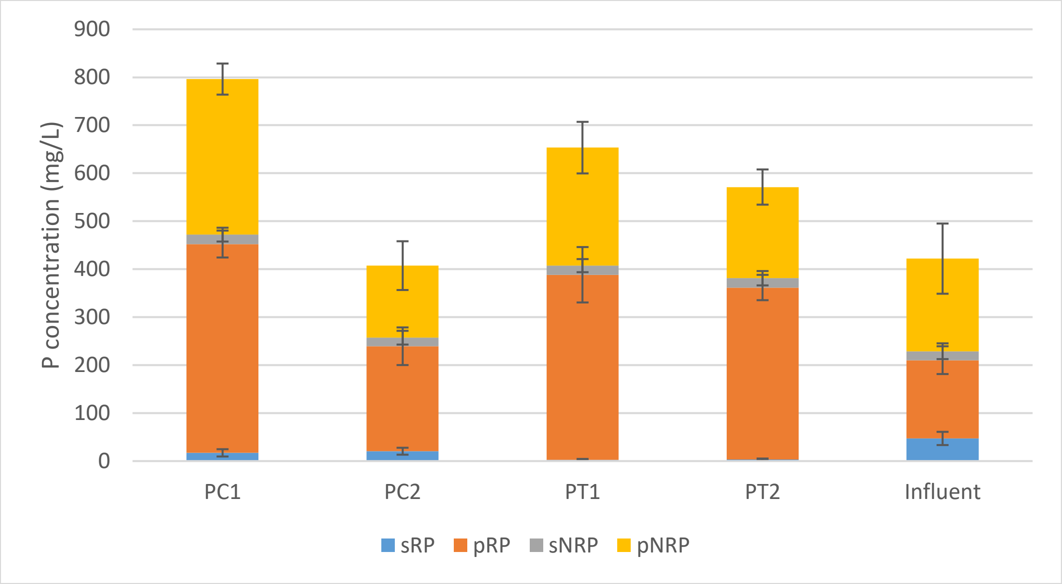

***Figure 4.12 Average effluent phosphorus concentrations of conventional sludge-fed digesters and untreated influent.*** *The different P species are identified by different colors. The species are stacked to represent the total P concentration.*

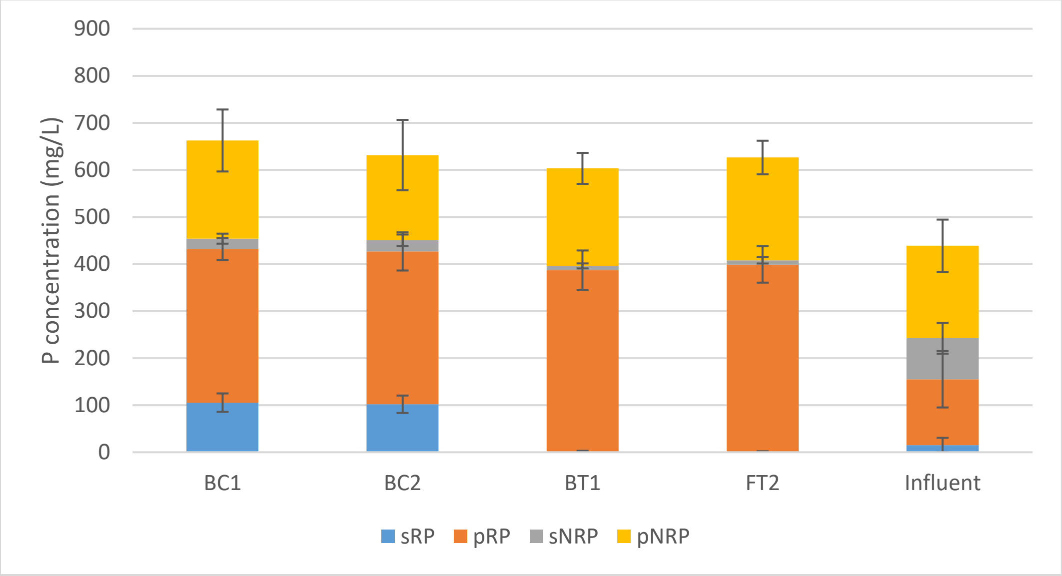

***Figure 4.13 Average effluent phosphorus concentrations of bio-P sludge-fed digesters and untreated influent.*** *The different P species are identified by different colors. The species are stacked to represent the total P concentration.*

The primary biosolids, except for PC2, contained more total phosphorus than the bio-P biosolids. The bio-P and primary biosolids had similar average amounts of soluble reactive and nonreactive P. The available soluble reactive P possibly bonded with the added Ca to form particulate P. The goal of the pretreatment was to precipitate out the reactive P by binding it with the added calcium ions. Improved CST was still achieved for the bio-P biosolids because the free calcium added as a pre-treatment lowered the M/D cation ratio.

Using R software, CST was compared to P speciation, solids content, and cation quantities. Though total P values did not match between influent and effluent, P speciation of effluent samples were included in analysis of effluent samples only to glean if P speciation might have an impact on CST. P speciation values were entered in as fractions of total P for analysis. The analysis provides evidence that lowering the M/D ratio could improve CST of sludge, but it is dependent on the sludge type. The CST of bio-P biosolids improved when the amount of divalent cations increased, but the opposite was observed for the primary sludge. The amount of particulate P could also impact CST because the increase of particulate P positively impacted the CST of the bio-P biosolids but negatively impacted the CST of the primary biosolids. This indicates that biosolids type matters when understanding the role of individual characteristics on CST. More research is required to understand why trends are opposite between these two types of biosolids. A plot generated from this analysis can be found in the Appendix.

It is unclear why the increase in particulate P improved the CST of the bio-P biosolids but not the primary biosolids. It was expected that an increase in particulate P would decrease CST for the primary biosolids because more particulate P is associated with more organic matter. The organic matter creates higher amounts of ECP which corresponds to poor dewaterability (Christensen, et. al., 2015). It was unexpected that the opposite was true for the bio-P biosolids. More research should be conducted to better understand if the improved CST could be explained chemically or biologically.

Anaerobic digestion and EBPR weaken floc structure, allowing for more particles and surface area for water to adhere (Higgins, et.al., 2014). More small particles decreases dewaterability. The chemical pretreatment increased the amount of divalent cations and these cations, instead of vicinal water, possibly adhered to particle surfaces and strengthened the structure of the bio-P flocs. The primary influent sludge may not have had as many free particles for the calcium to adhere to as the bio-P influent sludge. The lack of available particles in the primary sludge possibly did not allow the calcium to adhere to its surfaces and the vicinal water could not be released, causing poor dewaterability as CST.

# 5 CONCLUSIONS

The goal of this research was to determine the effect of changing the M/D cation ratio, where the total cations were measured, and P speciation using a chemical pretreatment on various anaerobically digested biosolids and how these characteristics correlate to dewaterability as CST. A pretreatment with 200 meq/L calcium hydroxide with the addition of hydrochloric acid for pH adjustment was selected because it improved the CST of the primary and bio-P sludge in the batch study. A lab-scale study was performed to test the impact the proposed pretreatment would have on CST after anaerobic digestion, M/D cation ratio, and P speciation. The following conclusions are based on the lab-scale anaerobic digestion study performed at the Marquette University Water Quality Center:

1. The chemical pretreatment, calcium hydroxide-hydrochloric acid, was selected from the initial batch study. The batch study revealed that the improved CST was not dependent on pH, but due to the cations added. The batch study also revealed that the improved CST could be attributed to the cation addition because improved CST was seen before and after acid (anion) addition. This study also revealed that ferric chloride can also improve CST. Further work should be done to determine the effect of multivalent cations with the same anion on other sludge and biosolids types and further provide evidence that improved CST is dominated by cation addition.

2. The calcium hydroxide-hydrochloric acid pretreatment significantly improved the dewaterability as CST of the bio-P biosolids (p-value s<0.0001, one-way ANOVA). Contrarily, the pretreatment had little to negative effect on the primary biosolids. Future research should be conducted to determine the optimal dose of calcium hydroxide for improvement of dewaterability as CST.
3. The chemical pretreatment decreased the M/D cation ratios for both types of biosolids. Based upon linear correlation analysis, the M/D cation ratio and CST have a positive correlation for only the bio-P biosolids. Further work should be performed to determine why the CST was not improved when the M/D cation ratio was decreased in the primary digested biosolids. Future work should also be performed on multiple biosolids of the same type from different WRRFs. These results indicate that the M/D cation ratio is not universally correlated to CST and the type of biosolids affects the correlation.
4. The chemical pretreatment altered the P speciation in favor of the particulate P form. The amount of particulate P impacted CST for both sludge types. More particulate P in bio-P biosolids may have improved CST, but the opposite occurred for the primary biosolids. Future work could focus on the addition of other cations to sludge and biosolids and determining how they might affect P speciation. Calcium phosphate species should also be investigated to determine if and how P binds to calcium.

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# APPENDIX

***Table A.1: Chemical usage log****: record of ferric addition. Days where no ferric was added were considered to produce bio-P sludge, although some residual ferric may have still been in the system. * represents days when sludge was pulled from the line and shipped for use in this study. This was done on a weekly basis, usually every Monday.*

| Date | Ferric addition by operators | |
|---|---|---|
| 9/20/2018 | 0 | * |
| 9/21/2018 | 991.1 | |
| 9/22/2018 | 0 | |
| 9/23/2018 | 0 | |
| 9/24/2018 | 0 | * |
| 9/25/2018 | 0 | |
| 9/26/2018 | 0 | |
| 9/27/2018 | 0 | |
| 9/28/2018 | 474.3 | |
| 9/29/2018 | 0 | |
| 9/30/2018 | 174.4 | |
| 10/1/2018 | 87.5 | * |
| 10/2/2018 | 0 | |
| 10/3/2018 | 0 | |
| 10/4/2018 | 686.1 | |
| 10/5/2018 | 171.2 | |
| 10/6/2018 | 0 | |
| 10/7/2018 | 0 | |
| 10/8/2018 | 230.6 | * |
| 10/9/2018 | 473.2 | |
| 10/10/2018 | 0 | |
| 10/11/2018 | 658.1 | |
| 10/12/2018 | 0 | |
| 10/13/2018 | 0 | |
| 10/14/2018 | 0 | |
| 10/15/2018 | 0 | * |
| 10/16/2018 | 182.9 | |
| 10/17/2018 | 0 | |
| 10/18/2018 | 0 | |
| 10/19/2018 | 0 | |
| 10/20/2018 | 0 | |
| 10/21/2018 | 0 | |
| 10/22/2018 | 0 | * |
| 10/23/2018 | 0 | |
| 10/24/2018 | 0 | |

| | | |
|---|---|---|
| 10/25/2018 | 0 | |
| 10/26/2018 | 226 | |
| 10/27/2018 | 0 | |
| 10/28/2018 | 0 | |
| 10/29/2018 | 0 | * |
| 10/30/2018 | 0 | |
| 10/31/2018 | 0 | |
| 11/1/2018 | 0 | |
| 11/2/2018 | 0 | |
| 11/3/2018 | 0 | |
| 11/4/2018 | 0 | |
| 11/5/2018 | 247.5 | * |
| 11/6/2018 | 146.3 | |
| 11/7/2018 | 123.8 | |
| 11/8/2018 | 228.3 | |
| 11/9/2018 | 419.7 | |
| 11/10/2018 | 298.1 | |
| 11/11/2018 | 0 | |
| 11/12/2018 | 437 | * |
| 11/13/2018 | 53.2 | |
| 11/14/2018 | 195.6 | |
| 11/15/2018 | 107.8 | |
| 11/16/2018 | 916.5 | |
| 11/17/2018 | 0 | |
| 11/18/2018 | 0 | |
| 11/19/2018 | 1,028.40 | * |
| 11/20/2018 | 956.7 | |
| 11/21/2018 | 0 | |
| 11/22/2018 | 0 | |
| 11/23/2018 | 118 | |
| 11/24/2018 | 641.3 | |
| 11/25/2018 | 337.5 | |
| 11/26/2018 | 0 | * |
| 11/27/2018 | 47.7 | |
| 11/28/2018 | 0 | |
| 11/29/2018 | 0 | |
| 11/30/2018 | 641.3 | |
| 12/1/2018 | 0 | |
| 12/2/2018 | 281.3 | |
| 12/3/2018 | 570.4 | |
| 12/4/2018 | 899.9 | |
| 12/5/2018 | 180 | |
| 12/6/2018 | 0 | |
| 12/7/2018 | 0 | |

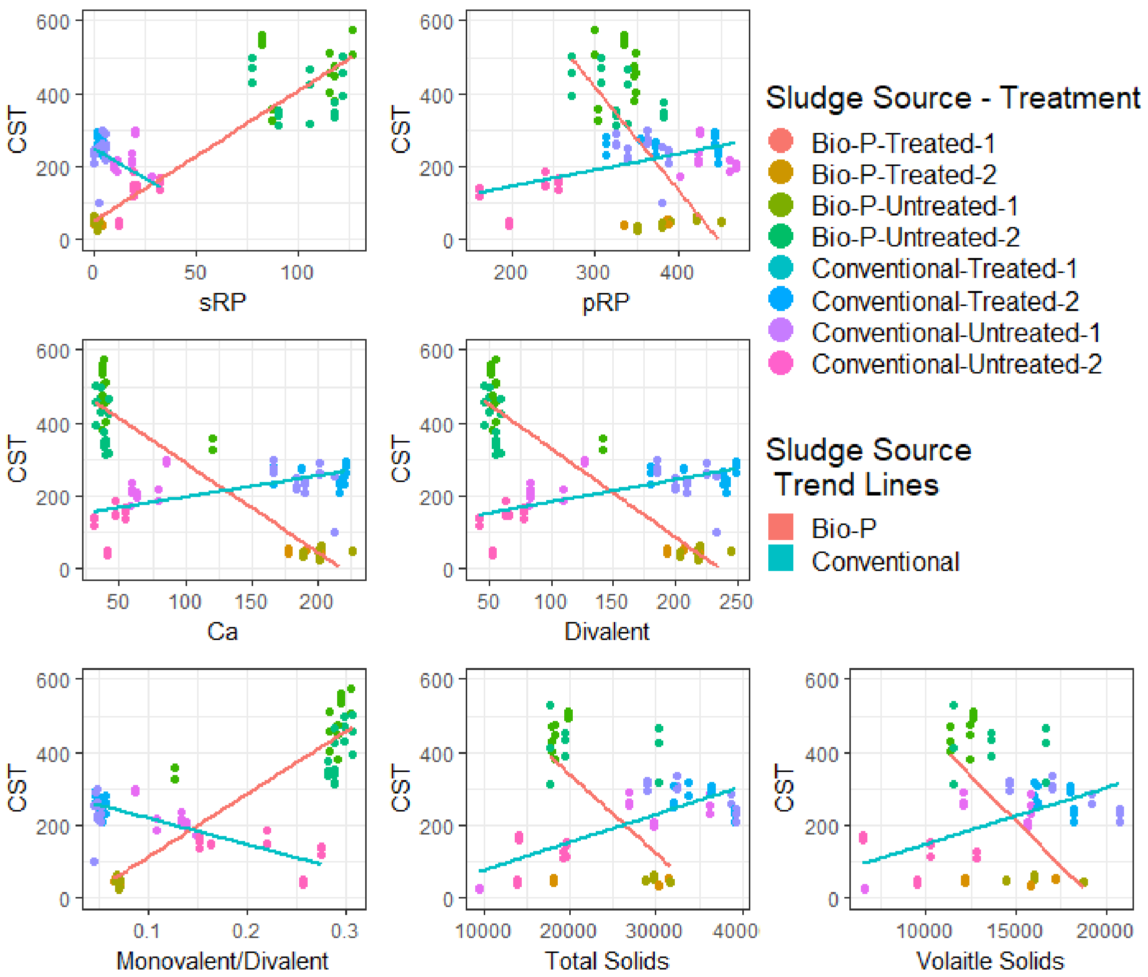

***Figure A.1: Additional correlation plots comparing CST to various biosolids characteristics.***

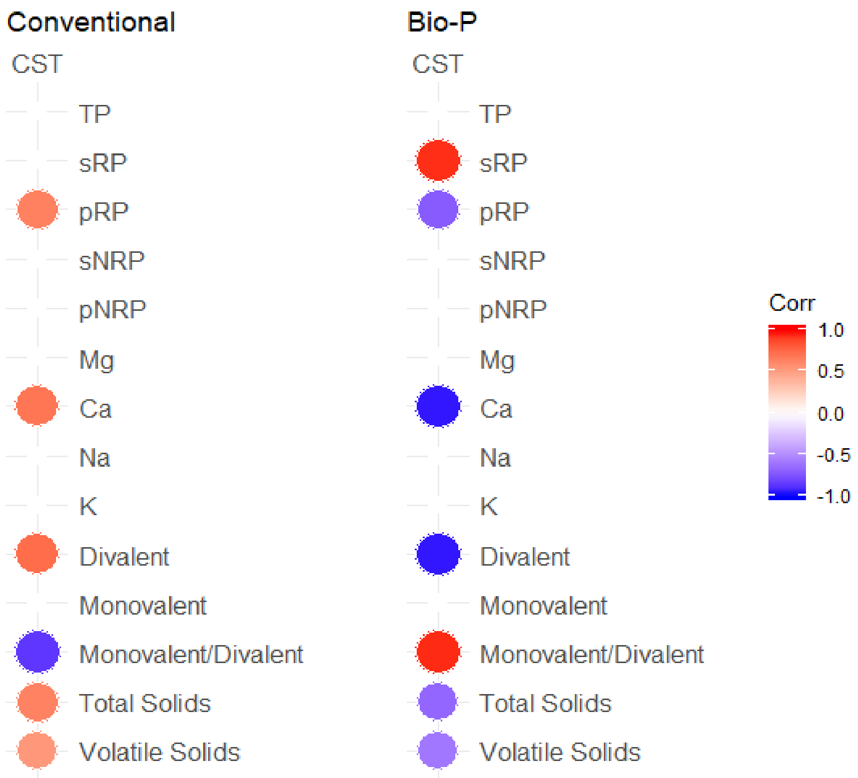

***Figure A.2: Correlation plot comparing the M/D ratio, cations, and monovalent and divalent sums to CST.*** *The red indicates that as the parameter increases, the CST increases, indicating poor dewaterability. The blue indicates that as the parameter increases, the CST decreases, indicating improved dewaterability.*