

ABSTRACT

Title of Thesis: MANAGING NUTRIENT LEACHING
 BEHAVIOR IN DC WATER'S CAMBI
 MATERIAL

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 Environmental Engineering

DC Water's Cambi biosolids material (CM) is formed from a new thermal hydrolysis procedure. CM nutrient leaching characteristics was examined employing 20 column studies and various leaching experiments. The materials were found to leach high concentrations of nitrogen, phosphorus, and potassium when tested with 10% and 15% CM (dry mass). The material was found to have a long-lasting leaching ability and to act differently when dry compared to wet. The dominant forms of nitrogen leaching were ammonium and organic N; the forms of phosphorus changed over time. Initially, organic P and particulate P leached and over time ortho-phosphate leached. Soil amendments for phosphorus and nitrogen mitigation were tested. Aluminum-based water treatment residuals (WTR) were found to be effective at decreasing phosphorus concentrations in the material leachate at ratios of 35%, 50%, and 75% WTR:CM. Results suggest this material to have long-term slow release benefits as a land application and organic fertilizer.

MANAGING NUTRIENT LEACHING BEHAVIOR IN DC WATER'S CAMBI
MATERIAL

by

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Chapter 1: Introduction

1.1 Background

1.1.1 Material Description

In the fall of 2014, DC Water's Blue Plains Advanced Wastewater Treatment Plant implemented a thermal hydrolysis process to advance their biosolids, or resulting product from the wastewater treatment process, from Class B to Class A quality. Their new product, the Cambi material (CM) does not contain a detectable level of pathogens. The non-detectable level of pathogens makes the material safer and usable on a broader scale. The previous biosolids treatment process used by DC Water was liming, which is a common procedure that raises the pH level to reduce odors and kill most pathogens, but does not get the material to EPA's "exceptional quality".

The "exceptional quality" title means the material can be used on public land and is safer for the general public to use as a fertilizer for their lawns and gardens. This could be very beneficial for DC Water economically because it could reduce the amount of biosolids being shipped away for disposal on agricultural land. In addition, the CM could be manufactured and sold for a profit as a Class A organic fertilizer and soil amendment similar to other Class A biosolids fertilizers such as Top Cut®

In other studies, the Cambi thermal hydrolysis process (THP) pretreatment procedure has been shown to increase biogas production, while reducing both organic matter by approximately 60%, and overall sludge volume to a total solids of about 30% (Carrère et al. 2010). The higher production of biogas can be used as a source of electricity.

At DC Water the THP is followed by anaerobic digestion. Other wastewater biosolids digestions/treatments include composting, aerobic digestion, and drying. The methods are conducted differently, and they result in different products. Typically biosolids do not have organic matter content higher than 50% (Lu et al. 2012). Composted biosolids differ from digested biosolids in that they have reduced organic matter through decomposition. In addition, composted biosolids have a decrease in $\text{NH}_3\text{-N}$ by volatilization, a decrease in potassium (K) through leaching, but an increase in concentrations of phosphorus (P) and trace metals (Lu et al. 2012). The CM is predicted to have higher levels of iron (Fe) which may make it more similar to a compost in that it has the potential to provide plant available Fe (Lu et al. 2012).

DC Water produces more than 1200 wet tons of CM a day (approximately 65 truck loads full) (DC Water 2016) and is interested in finding different uses for the material. The CM contains a high amount of nutrients and organic matter. It is known that biosolids treated with lime contain lower amounts of nitrogen (N), phosphorus (P) and potassium (K), and higher levels of calcium (Ca) than conventionally digested biosolids (Lu et al. 2012). When the change in biosolids processing goes from liming to thermal hydrolysis, the Ca levels will be much lower, and it is predicted that both the iron (Fe), and aluminum (Al) levels will be much higher. The variation of nutrient levels will change the way the biosolids act and make the CM an interesting material to study from a nutrient leaching perspective because it may have different leaching properties than conventionally digested biosolids.

1.1.2 Land Applications of Biosolids

Many different forms of biosolids have been used as organic fertilizers in agricultural fields and tested throughout the world because of their high nutrient value, organic content, large production amount, and possible benefits. Many studies have been done that compare different types of biosolids and composts for land applications. The nutrients that biosolids contain are generally in organic form and have been shown to have slow nutrient release when used in field applications (Lu et al. 2012). This slow release can be very beneficial for plants and in long-term agriculture.

It is also seen, however, that when biosolids are used for general land applications, they are usually applied at a rate based on the needed nitrogen content, or, plant available nitrogen (PAN). PAN takes into account the concentrations of ammonium, nitrate and organic N in municipal biosolids (Gilmour and Skinner 1999). The high levels of applied nitrogen provided by land applied biosolids are beneficial for plant growth; however, when biosolids are used in land applications these PAN levels often exceed the phosphorus levels needed for plant growth (Penn and Sims 2002). Typically, PAN accounts for 10-30% of the total nitrogen content in biosolids (Gale et al. 2006).

1.1.3 Phosphorus from Biosolids Applications

The over-application of phosphorus from biosolids has been addressed in some past studies. According to Maguire et al. (2001), a typical biosolids land application adds approximately 135 kg P/ha, which is a considerable amount compared to the 15 to 40 kg P/ha that is used by the crops. The over application of P leads to excess P, leaching, and

runoff of P that can reach surface water and further lead to environmental issues such as eutrophication (Maguire and Sims 2002). Some field application studies have proposed applying biosolids at P based rates, however these applications prove to be at too low of rates for plant nitrogen requirements, making them both uneconomical and impractical (Agyin-Birikorang et al. 2008).

An alternative to applying an overall smaller application of biosolids is to apply them in a mixture with soil amendments. Many methods of minimizing or eliminating P leaching with different soil amendments have been studied. Alum, water treatment residuals (WTR), and other materials have been used in studies in an effort to reduce the nutrient leaching.

Aluminum-based WTR starts as alum, and becomes primarily $\text{Al}(\text{OH})_3$. It is formed during the water purification process at drinking water treatment plants through flocculate colloid creation (Ippolito et al. 2009). The high aluminum content in the WTR has been shown to be effective at reducing phosphorus leaching through adsorption mechanisms (Elliott et al. 2002). Elliott et al. (2001) has shown in field studies that when the Al- WTR is mixed with biosolids in a land application, the WTR is effective at immobilizing the P from leaching to surface waters.

In addition to land applications, WTR has been studied as a P adsorbent in bioretention and compost mixtures in bioretention. Bioretention studies have shown that when Al-WTR is mixed with bioretention soil media (BSM) alone (no biosolids incorporated), it has been effective at sorbing P from stormwater influent at low P concentrations (O'Neill and Davis 2012). When WTR is added to soil or biosolids it decreases the phosphorus saturation index (PSI), that is the molar ratio of phosphorus to

aluminum and iron. The PSI is a calculation employed to predict the potential for P leaching from soils (Agyin-Birikorang and O'Connor 2007). The lower the PSI, the less likely the mixture will leach P. A study was completed on stormwater bioretention with added mixtures of biosolids compost and WTR to obtain three different PSI values, 1.0, 0.5 and 0.1 (Brown et al. 2015). Brown et al. found that when they incorporated the mixture at PSI values of 0.5 and 1.0, there was P leaching; however at 0.1, there was P removal. The study implies that little to no leaching will occur as the PSI value drops below 0.5.

1.1.4 Nitrogen from Biosolids Applications

Although most of the literature on biosolids land applications focuses on the P leaching due to the high amount of PAN needed for plant uptake, N leaching is also a risk in land applications and other uses for the material. Biosolids contain a high amount of nitrogen, however not all of the nitrogen is PAN. The organic forms of N in biosolids are converted to ammonium by mineralization (He et al. 2003). If the land application does not involve immediate tillage incorporation, ammonium in the biosolids will volatilize as ammonia gas (Gale et al. 2006). According to Haralambous et al. (1992), ammonium is the most important nitrogen species for eutrophication formation, and therefore the leaching of it can be very dangerous. In addition, some of the ammonium can also be oxidized to nitrate, which is a highly soluble form of nitrogen, making it more likely to leach and runoff (Gilmour and Skinner 1999). Due to the loss of some of the nitrogen to different N storage pools and leaching, biosolids are applied at a higher amount often to

make up for the nitrogen losses. The N leaching possibility needs to be examined with the CM because N leaching can reach and contaminate surface water sources.

Some soil amendments have been studied for their ability to adsorb and remove nitrogen. Biochar and zeolite are two materials that have been studied with nitrogen uptake in general, however the literature does not contain studies on them mixed with biosolids specifically. Biochar, a carbon-rich product created from thermal decomposition of organic material under low oxygen conditions (Lehmann et al. 2011) has been studied and shown in some instances to adsorb ammonium (Clough and Condron 2010). Studies on biochar differ in their conclusions because the biochars produced in the studies are often completed at different temperatures and produced from different organic sources.

Many studies have examined different biochars at different temperatures and produced from different materials (Cui et al. 2016; Ding et al. 2010; Takaya et al. 2016; Yao et al. 2012; Zheng et al. 2013). Many different conclusions have been developed from these studies that produce varying results. For example, Cui et al. (2016) reported efficient NH_4^+ removal from adsorption kinetic studies with six different wetland plants (*C. indica*, *P. purpureum* Schum, *T. dealbata*, *Z. caduciflora*, *P. australis* and *V. zizanioides*) when they were created at 500 °C. Ding et al. (2010) and Yao et al. (2012) both found NH_4^+ removal with bamboo-derived biochar (temperature not stated by Ding et al. (2010) and 600 °C for Yao et al. (2012)). In addition, Zheng et al. (2013) reported successful NH_4^+ removal with a giant reed grass derived biochar (temperature not given). Takaya et al. (2016) on the other hand did not find that the biochar production temperature had a significant relationship to the NH_4^+ removal.

Despite the literature showing results from very different types of biochars, the general conclusion to the studies are that biochars typically have high success rates when being tested for ammonium adsorption.

In addition to biochar, clinoptilolite zeolite has been studied for its ability to adsorb ammonium due to its high cation exchange capacity (CEC). Zeolite has been studied in adsorption isotherm kinetics studies and in studies involving ammonium adsorption from wastewater and constructed wetlands. Clinoptilolite is a form of zeolite often suggested for wastewater treatment applications due to its high CEC, and affinity for ammonium (Hedström 2001). In lab studies completed with zeolite adsorption isotherms, the results showed that ammonium was the preferred ion for zeolite and the adsorption was a very quick process (Haralambous et al. 1992). Often times, zeolite is recommended for wastewater treatment for ammonium removal in tertiary treatment (Hedström 2001). Zeolite is frequently used in ion exchange because compared to other ion exchange materials, it is much more affordable (Haralambous et al. 1992). Based on the literature, mixing various ratios of clinoptilolite zeolite with the CM may help with possible ammonium and total nitrogen leaching by adsorbing the ammonium and immobilizing it from further leaching.

1.1.5 Biosolids Nutrient Leaching and Uses

The scope of the project aims to determine the initial nitrogen and phosphorus leaching activity from the CM. With background and base knowledge on the leachate produced, the project will incorporate various amendments in an effort to decrease/manage nutrient leaching. It is important to understand the leaching of the

material to better predict how it may act in the environment, and what amendments should be added, and at what ratios to obtain a desired nutrient content and leaching capacity.

A better understanding of the nutrient characterization and leaching of the CM is desirable because it will aid in recommending different beneficial uses for the material. Most biosolids reuse technologies include land application. Some consider land application to be the best biosolids technology available because of the natural high nutrient value, organic content, and the complete reusability of the material (Wang et al. 2008). However, there may be more uses for such a high nutrient class A material.

Some studies have stated that biosolids can be used in mixtures with carbon sources as topsoils for land reclamation in degraded land in places such as mining fields and landfill sites (Brown et al. 2003). Often the application of biosolids to these degraded lands is questioned because of the possibility of nitrate leaching; this is why when biosolids are used for these applications they are mixed with carbon sources such as wood ash and other vegetation materials (Brown et al. 2003). Wang et al. (2008) states that one of the main issues with biosolids reuse is public acceptance. Public education is important. People should be aware that the CM is class A and safe to apply to land and for human contact.

With a material that is safer to use by the public, more material reuse options may become available. Possible uses such as usage in stormwater management technologies and as fertilizer materials will be taken into consideration. The leaching properties of the CM will be better understood through various lab procedures and experiments, and can

then be compared to typical material leaching properties used in stormwater management technologies.

In addition, another topic that needs to be addressed is the storage effect on the CM nutrient leaching and how the material could change/ behave after it air-dries while in storage. Letting the material sit and possibly become wet and re-dried could have an effect on the material characterization and lead to a material different than the fresh material right off the belt press. Wu and Ma (2001) completed a study on biosolids compost stability as a function of storage methods. The study shows that when biosolids compost is stored in a freezer versus air –drying, the water-soluble organic carbon composition of the material differed (Wu and Ma 2001). Although the CM is not compost, differing storage methods could pose different changes to the CM during storage and need to be looked at in this study.

1.2 Research Goals and Objectives

The overall goal of this project is to characterize the CM nutrient leaching behavior and develop CM amendments, processes and recommendations that will allow it to be used beneficially throughout the (sub) urban landscape. Some constraints that affect this project include time and location. Urban areas, such as Washington, DC do not have a lot of space to work with. With such a high nutrient containing material, it may be difficult to use a significant amount of CM compared to the vast amount produced daily by DC Water. In addition, the project duration of one year may pose a time constraint and be a determining factor for experiments based on their needed time frames.

The objectives of this study are specifically related to the uses of the CM produced, specifically (i) what the physical properties and nutrient makeup are and how they can be useful in an urban area such as Washington DC and other types of land areas, (ii) what is the extent it can be used beneficially, i.e., land applications, stormwater control measures, and (iii) if substances such as WTR, zeolite and/or biochar can and should be used to help manage and control nutrient leaching. These objectives will be met through batch and column lab experiments and analyses that involve studying the CM from a physical and nutrient leaching perspective. The results of the experiments will be compared to other studies with materials used for various land purposes and conclusions and recommendations for the CM will be made.

Chapter 2: Methodology

Numerous experiments, extractions, lab tests and analyses were completed to better understand the nutrient characterization of the CM, and to develop predictions for how it will act in the environment in different situations. The timeline of the project was developed in a manner that started with nutrient extractions on the material itself followed by various types of column study experiments, and aging experiments.

2.1 Material Collection

The samples of CM were taken from DC Water approximately once every two weeks. The CM was sampled from the belt filter press at DC Water, placed in Ziplock bags and taken immediately to the environmental engineering lab at the University of Maryland and stored in a refrigerator (3.5° C).

Other materials used throughout the project include soils, water treatment residuals, biochar, and zeolite. Table 2-1 lists the sources of these materials, storage, and when they were sampled.

Material	Sample Manufacturer	Manufacturer Location	Storage Location	Date of Sample
CM	DC Water	Washington, DC	Refrigerator-ENCE Lab*	bi-monthly
Soils, Sandy loam and loamy sand	Creative Landscapes by Gregory	Frederick, MD	UMD Greenhouse	Sept-15
Al-WTR	Dalecarlia Water Treatment Plant	Washington, DC	ENCE Lab *	Jul-15
Zeolite, Clinoptolite	Bear River Zeolite Co. Inc.	Preston, ID	ENCE Lab *	Mar-16
Biochar,	Soil Reef Biochar	Berwyn, PA	ENCE Lab *	Jun-16

Table 2- 1. The material types, source locations, sample dates, and storage locations for the materials used in the project.

*ENCE Lab is the environmental engineering laboratory at the University of Maryland.

2.2 Lab Tests and Procedures

All lab samples/extracts were kept in a refrigerator or freezer, depending on the specific procedure protocol.

2.2.1 Moisture Content and LOI

Moisture content and loss on ignition (LOI) tests were completed on the CM, WTR and soils. The moisture content of a material was tested to find the amount of liquid present in the material and the LOI was tested as an estimate of the organic content in the material being tested. LOI is the most commonly used method for measuring the organic matter in soils (Frogbrook and Oliver 2001). Although some additional weight loss occurs during the ignition, LOI is a very good representation of the organic matter material. The procedures for moisture content and LOI testing for the CM and soils were done according to EPA Standard Method 1684. The mass of material used for the testing was approximately equal each time the test was run in order to minimize replicate error (Heiri et al. 2001).

The moisture content on the WTR was done in accordance with EPA standard methods; however, the LOI testing was done differently. The LOI was done at three temperatures- 180°C, 350°C, and 550°C. The reason for the three temperatures is due to trapped water that exists in the WTR (Elliott et al. 2002). In addition, the material is not

fully understood, and is therefore heated at various temperatures to approximate the LOI value.

The moisture content and LOI tests were completed in ceramic crucibles that had been acid washed and pre-heated at 102°C to eliminate any moisture that may have been in the bowl prior to testing. When the samples were taken out of the ovens they were stored in desiccators to eliminate moisture absorption and to cool to room temperature.

2.2.2 Nutrient Extractions

2.2.2.1 Mehlich III Extraction

Mehlich III extractable P/K was developed to find plant available, or the phosphorus and potassium fertility level in soils and other media (Maguire and Sims 2002). The procedure was completed at a 1:10 ratio (w:v) for the CM, soils, and CM mixtures with soil amendments (Kozar et al. 1996). The procedure was done with wet samples, sieved to 2 mm, and completed using Fisher Brand 50 mL centrifuge tubes. The samples were mixed at approximately 90 rpm for ten minutes, centrifuged at 4000 rpm for 10 minutes and filtered with 0.22 µm EMD Millipore MF-Millipore Mixed Cellulose Ester Membranes filter paper into clean 50 mL Fisher centrifuge tubes. Once the samples were filtered and clear of particulate matter, they were frozen for later analysis.

2.2.2.2 Ammonium Oxalate Extraction

Oxalate extractions were used to extract phosphorus (P), iron (Fe), and aluminum (Al). The oxalate extraction is known for its ability to predict and set environmental limits for phosphorus in soils and other media with its calculated phosphorus saturation index (PSI_{ox}) (Maguire and Sims 2002). The oxalate extraction procedure was completed

with an oxalate acidic solution at pH 3. The procedure was completed for the CM, WTR, soils, and mixtures of the CM and WTR.

Wet CM was sieved to 2-mm, and samples were tested at a 1:50 ratio (w:v) (Wolf and Baker 1990). The samples were shaken in the dark for two hours at approximately 90 rpm in Fisher 50 mL centrifuge tubes, centrifuged at 4000 rpm for 10 minutes, and filtered with 0.22 μ m EMD Millipore MF-Millipore Mixed Cellulose Ester Membrane filters. The samples were then frozen for later analysis.

The extractions on the WTR were completed on wet material at two ratios. The WTR was sieved to 2-mm and then extracted at both 1:50 and 1:100 ratios (w:v) with procedures in accordance with Wolf and Baker (1990). The purpose of the smaller ratio was based on the high-predicted aluminum content in the WTR.

Mixtures containing various ratios of WTR to CM were chosen and analyzed for oxalate P, Al, and Fe as well at 1:50 ratios (w:v). The soils were also sieved to 2-mm and tested for oxalate extractable P, Al, and Fe at 1:50 ratios (w:v).

2.2.2.3 0.01 M CaCl_2 Extraction

Water extractable phosphorus (WEP) is the amount of phosphorus predicted to leach from a material into the environment by measuring easily desorbable P in soils and other materials (Maguire and Sims 2002). The CaCl_2 extraction method is used to estimate the WEP. The WEP is often comparable to the dissolved runoff P concentrations in land applied organic materials (Elliott et al. 2005). The CM and soils were tested with a 0.01 M CaCl_2 extraction at a 1:10 (w:v) ratio (Kozar et al. 1996). The samples were shaken at approximately 90 rpm for two hours in Fisher 50 mL centrifuge tubes,

centrifuged at 4000 rpm for 10 minutes and gravity filtered with Whatman No. 42 filter paper. The samples were frozen for later analysis.

In addition to testing for WEP, the 0.01 M CaCl_2 extraction was used on the CM, and mixtures containing various ratios of zeolite: CM for water nitrogen speciation. The procedure was used to analyze total soluble nitrogen, nitrate, ammonium, nitrite, and then organic N is calculated (Houba et al. 2000).

2.2.2.4 2 M KCl Extraction

Potassium chloride (KCl) extractions are used to find levels of extractable inorganic nitrogen in soils and sediments. The 2 M KCl extraction was chosen for its ability to predict the inorganic nitrogen content in soils and other media (Goloran et al. 2013). The method has shown to have very high correlation with quality control standards in studies analyzing the method (Jones and Willett 2006).

The procedure was completed at a ratio of 5:20 (w:v). The extractions were done according to University of Colorado Aridlands Ecology Lab Protocol (Castle 2009). There were minor differences made to the procedure- the ratio of 5:20 was used instead of 10:40 and the extracts were shaken for two hours at approximately 90 rpm instead of for 2 hours at 200 rpm. The samples were shaken in 50 mL Fisher centrifuge tubes. After the samples were gravity filtered through Whatman No. 1 filter paper, they were frozen to be analyzed later.

2.2.2.5 pH

The pH was measured with an Orion pH Meter Model 520A at both a 1:2 and a 1:3 (w:v) ratio of wet CM to deionized water that were mixed in a glass beaker.

2.3 Column Studies

In order to observe how the CM may act in the environment, column studies were employed. The columns used in the study were 200 mm in height and 25 mm in diameter and are clear glass. Two different types of column study experiments were completed during this study, continuous flow columns, and non-continuous flow columns. For both column studies the columns were prepared the same way. Fisher Scientific brand glass wool was placed at both the bottom and top of the column. The material in the column was tightly packed and all had been previously sieved to 2-mm.

2.3.1 Continuous Flow Column Studies

For the continuous column studies, each column was continuously run for 21 days (~120 m water applied) with a constant applied velocity of 30 cm/hr and an influent of 0.01 M NaCl. It was predicted that the nutrient leaching would decrease after approximately 100 years worth of water was applied to it. Although the continuous flow is not a realistic view of land application, the experiments were expected to show the longer term leaching ability of the material. Column effluent samples were taken every hour for 8 hours on each initial column day. Sample times were recorded to get an approximate instantaneous time to report in depth, or volume of water applied. Effluent samples were taken in 125 mL plastic bottles approximately every other day for the duration of the experiment following the initial day of sampling. After taking a sample, all of the samples were frozen for future analysis.

2.3.2 Non-Continuous Flow Column Studies

The non-continuous flow column studies were identical to the continuous flow column studies in flow rate applied (30 cm/hr), column size, and influent type (0.01 M NaCl). The non-continuous flow columns however, only flowed for 24-hour increments and had at least 48- no-flow periods in between them. The columns were run for the same total amount of water applied. This turned out to be seventeen 24-hour increment storms (~120 m water applied), or approximately 100 years worth of Maryland rainfall. The sampling for the non-continuous flow columns consisted of two samples on the initial day of the 24-hour increment storm (hours 1 and 2), and then one sample on the second day in the last hour of the 24-hour storm. As with the continuous columns, the samples from the non-continuous columns were frozen for future analysis.

2.3.3 Column Study Sample Analysis

The samples from both the continuous and non-continuous columns were measured for various nutrients to develop a better understanding of the nutrient leaching in the materials.

2.3.3.1 Total Phosphorus and P Speciation

All of the samples in the column studies were analyzed for total phosphorus (TP). Three to four samples throughout the column study experiments were selected for total phosphorus speciation (TP, Dissolved Phosphorus (DP), and Soluble Reactive Phosphorus (SRP)). TP, DP, and SRP were analyzed following APHA Standard Methods (Eaton et al. 2005). TP was analyzed with the potassium persulfate digestion (4500-B.5). DP and SRP samples were filtered through 0.22 μm EMD Millipore MF-Millipore Mixed

Cellulose Ester Membranes filter paper, then analyzed with methods identical to TP analysis; however, SRP did not undergo the digestion procedure.

Particulate phosphorus (PP) and dissolved organic phosphorus (DOP) concentrations were obtained from P speciation measurements using the following calculations.

$$PP = TP - DP \quad (\text{Eqn 2-1})$$

$$DOP = DP - SRP \quad (\text{Eqn 2-2})$$

2.3.3.2 Total Nitrogen and N Speciation

Every sample from the column studies was analyzed for total nitrogen (TN). The TN was measured with a Shimadzu TOC-TN-L. As with phosphorus speciation, three to four samples from each column study were also chosen for nitrogen speciation. Total nitrogen speciation involves measuring for total nitrogen, nitrate (NO_3^- -N), nitrite (NO_2^- -N) and ammonium (NH_4^+ -N). Nitrite and ammonium were analyzed with standard methods 4500- NO_2^- B, and 4500- NH_3 F, respectively (Eaton et al. 2005). Nitrate was measured via ion chromatography IonPac AS22 column (Dionex ICS-1100) with an anion eluent solution (4.5 mM Na_2CO_3 , 1.4 mM NaHCO_3) and a flow rate of 1.2 mL/min. With these three species of nitrogen, and TN, organic nitrogen (ON) can be calculated with the following equation.

$$ON = TN - (\text{NO}_3^- - N) - (\text{NO}_2^- - N) - (\text{NH}_4^+ - N) \quad (\text{Eqn 2-3})$$

2.3.3.3 Total Potassium

To get a better understanding of the leaching from a fertilizer perspective, potassium analysis was done on the column study samples. Although potassium leaching is not considered to be as high of a concern to nitrogen and phosphorus leaching, it is still important to have an understanding of the K leaching from both a fertilizer and agronomic perspective (Kayser and Isselstein 2005).

The column effluent samples were measured for TK concentration with inductively coupled plasma atomic emission spectroscopy on radial settings (Shimadzu ICPE-9000).

2.3.3 Column Study Descriptions

All of the columns, whether they were for continuous, or non-continuous flow were prepared in the same manner. If CM was used in the column being tested, it was from a sample of CM taken during the same week the columns were prepared/started. The column studies contained columns filled with different mixtures of materials at different ratios and percentages. Table 2-2 shows an overview of the column studies completed and the mixtures they contained. Despite the following figures not showing this, all of the columns were covered with aluminum foil upon starting to eliminate sunlight and produce a more realistic soil layer setting.

Table 2- 2. Summary Table of the various types of columns, and the dates they were run.

Column #	Column Type	Date of study	Material Mixture (% on dry mass)
1	Continuous	7/23/15-8/13/15	10% CM, 90% Sand
2	Continuous	7/23/15-8/13/15	15% CM, 85% Sand
3	Continuous	9/9/15-9/30/15	100% Sandy Loam
4	Continuous	9/9/15-9/30/15	100% Loamy Sand
5	Continuous	9/30/15-10/23/15	15% CM, 85% Sandy Loam

6	Continuous	9/30/15-10/23/15	15% CM, 85% Loamy Sand
7	Continuous	10/28/15-11/18/15	10% CM, 90% Sandy Loam
8	Continuous	10/28/15-11/18/15	10% CM, 90% Loamy Sand
9	Continuous	1/6/16-1/27/16	15% CM, 35% WTR:CM, Loamy Sand
10	Continuous	1/6/16-1/27/16	15% CM, 50% WTR:CM, Loamy Sand
11	Continuous	1/6/16-1/27/16	15% CM, 75% WTR:CM, Loamy Sand
12	Continuous	3/2/16-3/23/16	15% CM, 85% Loamy Sand
13	Continuous	3/2/16-3/23/16	15% CM, 85% Sand
14	Non-Continuous	4/7/16-6/17/16	100% Sandy Loam
15	Non-Continuous	4/7/16-6/17/16	15% CM, 85% Sandy Loam
16	Non-Continuous	4/7/16-6/17/16	15% CM, 85% Sand
17	Continuous	6/29/16-7/20/16	15% CM, 30% Biochar:CM, Soil
18	Continuous	6/29/16-7/20/16	15% CM, 10% Biochar:CM, Soil
19	Continuous	6/29/16-7/20/16	15% CM, 30% Zeolite:CM, Soil
20	Continuous	6/29/16-7/20/16	15% CM, 10% Zeolite:CM, Soil

For the first columns containing CM and sand, the initial formation of the columns was to be in layers, as shown below in the outer columns in Figure 1. However, the layer of CM was too dense for the influent to travel through, so a complete mixed layer was used instead. Figure 2-1 demonstrates the difference in layered columns vis-à-vis mixed columns. The complete mix column formation was used for the rest of the columns in this project as well.

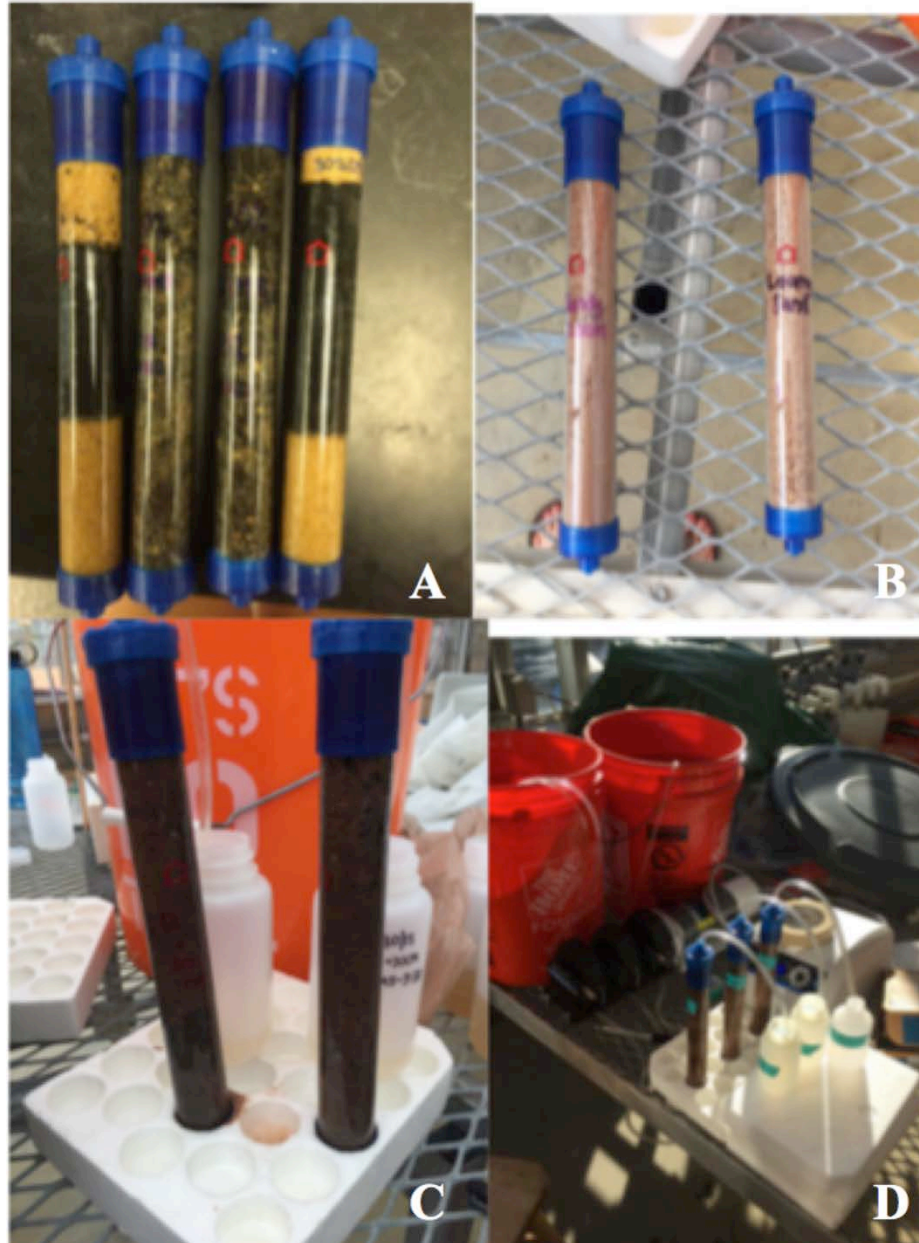


Figure 2- 1. A-The two columns on the outside are the layered CM and sand whereas the two in the middle are the mixed. The middle two columns were used for column study 1 due to the influent not being able to be fed through the dense layer of CM. B- two columns filled with soils; on the left is the sandy loam and on the right is loamy sand. C- the loamy sand and sandy loam columns are filled with 15% CM and 85% soils. D- the loamy sand with 15% CM, and various ratios of WTR: CM columns.

To gain an understanding of both the CM and soils prior to testing them together, a column experiment was completed with two soils alone, loamy sand, and sandy loam

(Figure 2-1B). The soil columns were used to give baseline, or control data for an environment without any soil amendments added.

To gain an understanding of how the CM would leach if it were put in a natural soil environment, the columns with CM at different percentages with the two types of soils were created. Figure 2-1C shows the columns filled with mixed soil and 15% (dry mass) CM.

Phosphorus mitigation was a large consideration to the project. To account for this, WTR was implemented into the column studies at various ratios of WTR: CM. Figure 2-1D shows the setup of the three columns with WTR, CM and loamy sand.

2.4 Cambi and Soil Amendment Ratios

2.4.1 Cambi and WTR Ratios

Various ratios of WTR to CM (dry mass basis) were chosen and tested for extractable nutrients (Mehlich III P, Mehlich III K, CaCl_2 P, KCl N, and oxalate extractable P, Al, and Fe). The different ratios were measured out for dry mass ratio amounts, mixed in ceramic bowls and placed in plastic sampling bottles to sit for at least 24 hours prior to lab work. The WTR is aluminum-based and predicted to have a high sorbing capacity for P losses in soils and other media (Bai et al. 2014). The purpose of completing multiple nutrient extractions was to predict and possibly correlate any changes that may occur in the leaching capacity when WTR is mixed with the CM.

2.4.2 Cambi and Zeolite Ratios

Similar to the CM and WTR ratio study, various ratios of zeolite to CM (dry mass basis) were chosen. The ratios were based on the manufacturers provided CEC value of the zeolite in comparison to the average ammonium concentration in the effluent from previous continuous column studies. The various ratios of zeolite to CM were tested for nitrogen speciation from a CaCl_2 extraction to predict the nitrogen leaching capacity when zeolite was used.

2.4.3 Cambi and Biochar Ratios

Biochar was mixed with the CM at the same ratios as the zeolite (10, 20, and 30% dry mass), and used for CaCl_2 extractions and tested for total nitrogen speciation. The biochar used in this project is from Southern Yellow Pine from a pyrolysis heating system at 550°C .

2.5 Cambi Aging Study

A very large amount of CM is produced at DC Water every day. The material is also not immediately used; there is a possibility that it is stored for days and may vary in moisture levels. To study how aging and varying levels of moisture affect the CM, an aging experiment was done. The experiment involved a total of six buckets filled with the CM. Each bucket had two holes drilled in the bottom to allow for water drainage. The CM was left in the greenhouse to mimic storage conditions. The material was wetted every Monday until the CM appeared saturated, but not flooded (with tap water and a plastic watering can) and sampled prior to Monday wettings. Figure 2-2 shows the bucket experiment setup in the research greenhouse.



Figure 2- 2. The six-bucket setup for the CM Aging Experiment.

The samples were tested for various nutrient extractions (CaCl_2 P, CaCl_2 N speciation, KCl N, and Mehlich III P/K). The experiment was predicted to show how the CM nutrient leaching characteristics change over time and with varying levels of moisture.

2.6 Dry Cambi Leach Test

The CM dries out when it sits outside or is not refrigerated. The material becomes cracked and breaks up into smaller pieces than the cake that initially comes out on the conveyer belts at DC Water. The dry material that forms is very hard and difficult to break apart. It is predicted that the material may get like this if it is stored before used. It is possible that the dry material has different leaching properties than the wet material. To test the difference in leaching, a dry CM leach test was employed in the study.

The dry CM leach test consisted of 4 different sizes of dry CM piece groupings. Figure 2-3 below shows the four different sizes chosen.

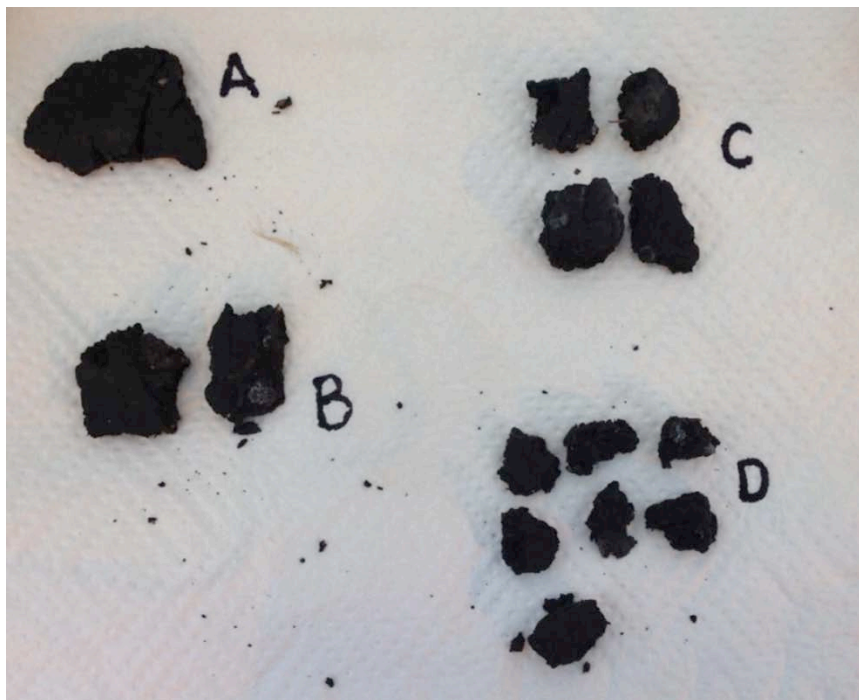


Figure 2- 3. The four different group sizes for the dry leach study. There were three sets of each of these groups to have data in triplicate.

Despite the different sizes, each grouping had approximately the same mass. The mass of each sized group was measured prior to the leaching experiment and at the end of the experiment. The sizes of the CM pieces were assumed to be rectangular prisms; the dimensions of the pieces were estimated with a ruler. The pieces were placed in 250 mL Nalgene sample bottles that had 4 holes drilled in the bottom (Figure 2-4).



Figure 2- 4. The 250 mL Nalgene bottles with holes drilled in the bottom for the dry leach study.

The CM each had 0.01 M NaCl applied at a flow rate of 10-mL/min, which is approximately 4.1 cm/min, for 30 minutes. This value was chosen to simulate a more realistic rainfall in Maryland. The effluent was captured in an additional 250 mL Nalgene sample bottle for further analysis. The samples were stored in the freezer.

All of the effluent samples were tested for total phosphorus speciation, total nitrogen speciation, and total potassium. The simulated “storms” were run once a week for six weeks.

2.7 Analytical Procedures

A summary of the analytical methods used for each nutrient extraction and nutrient concentration measurement are shown in Table 2-3.

Table 2- 3. The various nutrient speciation and concentration measurements and their method and detection limits.

Phosphorus Species	Analytical Methods	Detection Limit	Standards Made from	Standards Range
total phosphorus (TP)*	4500- P B.5, 4500-P E	0.05 mg/L P	RICCA Company PO4-P	0.0-2.5
particulate phosphorus (PP)	PP= TP-DP	XX	PP= TP-DP	N/A
dissolved phosphorous (DP)*	4500-P E	0.05 mg/L P	RICCA Company PO4-P	0.0-2.5
soluble reactive phosphorus (SRP)*	4500-P E	0.05 mg/L P	RICCA Company PO4-P	0.0-2.0
dissolved organic phosphorus (DOP)	DOP=DP-SRP	XX	N/A	N/A
Mehlich III extractable P*	Kozar et al. 1996	0.05 mg/L P	RICCA Company PO4-P	0.0-2.0
CaCl ₂ extractable P*	Kozar et al. 1996	0.05 mg/L P	RICCA Company PO4-P	0.0-1.0
oxalate extractable P**	Wolf and Baker 1990		High-Purity Standards	0.0-50.0
Nitrogen Species				
total nitrogen (TN)*	TOC-TN	0.01 mg/L N	RICCA Company NO ₃ -N	0.0-10.0
ammonium (NH ₄ ⁺)*	4500-NH ₃ F	0.05 mg/L N	Fisher Brand NH ₄ Cl	0.0-1.5
nitrate nitrogen (NO ₃ ⁻ -N)***	IC-Dionex	0.05 mg/L N	RICCA Company NO ₃ -N	0.0-5.0
nitrite nitrogen (NO ₂ ⁻ -N)*	4500-NO ₂ - B	0.01 mg/L N	Fisher Brand NaNO ₂	0.0-1.0
organic nitrogen	ON=TN-NO ₃ --NO ₂ --NH ₃	XX	N/A	N/A
KCl extractable N*	Castle 2009	0.01 mg/L N	RICCA Company NO ₃ -N	0.0-10.0
Potassium Species				
Mehlich III extractable K**	Kozar et al. 1996	0.01 mg/L K	High-Purity Standards	0.0-50.0
total potassium (TK)**	ICP-AES	0.01 mg/L K	High-Purity Standards	0.0-25.0
Aluminum and Iron Species				
oxalate extractable Al**	Wolf and Baker 1990	0.01 mg/L Al	High-Purity Standards	0.0-100.0
oxalate extractable Fe**	Wolf and Baker 1990	0.01 mg/L Fe	High-Purity Standards	0.0-100.0

* Determined with colorimetric procedures and the Shimadzu UV-160U

**Determined with the Shimadzu ICPE-9000

*** Determined with Dionex ICS-1100

XX Determined by equations 2-1,2-2, and 2-3

Table 2-3 shows the different phosphorus methods for different speciations and extractions. The measurements that were determined colorimetrically used the ascorbic acid molybdenum blue method (4500- B.5). The reagent was prepared according to the method written by Sims (Kozar et al. 1996). Mehlich III P samples were diluted. Sample concentrations were found by comparing them to the standards (0.00 - 2.00 mg/L) made from RICCA Company Phosphorus Standards, 1000 ppm and colorimetrically read with the UV-Vis (Shimadzu UV-160U) at a wavelength of 880 nm.

The oxalate extractable P measurements were analyzed with inductively coupled plasma atomic emission spectroscopy (ICPE-9000-AES) and standards were made from High-Purity Standards made according to EPA Method 200.7.

Table 2-3 gives the methodology for various nitrogen species and extraction concentrations and measurements. The measurements completed colorimetrically were determined at different wavelengths. Ammonium is determined at 640 nm (0.0 mg/L-1.0 mg/L), and nitrite is determined at 543 nm (0.0 mg/L – 1 mg/L). Ammonium and nitrite standards were derived from a Fisher Brand ammonium chloride and sodium nitrite powder, respectively.

The total nitrogen is measured, along with the KCl extractable N using the TOC-TN-L (0.0 - 10 mg/L). Nitrate is analyzed with the Dionex-ICS-1100 (0.0 -5 mg/L). When TN, KCl N and nitrate are analyzed, standards checks are made every 8-10 samples to make sure the instrument readings are accurate and reliable. The standards for both nitrate and TN are derived from RICCA Company Nitrate Nitrogen Standard, 1000 ppm.

Table 2-3 shows the two different potassium measurements that are completed on the CM and column study effluent. Both concentrations are determined with the ICPE-9000-AES. Standards, 0 (blank)- 50 mg/L K, were made from High-Purity Standards according to EPA Method 200.7.

Table 2-3 shows both aluminum and iron oxalate extraction measurement procedures. Both metals are analyzed with the ICPE-9000-AES at concentrations of 0.0 - 100 mg/L (Al, Fe) made from High-Purity Standards (Charleston, SC, 29418) according to EPA Method 200.7.

2.8 Quality Assurance/Quality Control (QA/QC)

To ensure high quality and justifiable data, numerous precautions and QA/QC steps were taken in the lab and other experimental areas.

2.8.1 Glassware Cleaning/ Lab wear

When lab work was being completed, the person(s) completing the lab procedures were always wearing lab gloves for their own protection and for lab safety protocol.

Glassware and other lab equipment used was washed with Dionex soap, DI rinsed, completely submerged in 0.5 N HCl acid for 24 hours, and then DI rinsed three times before used for lab procedures. The glassware is not used until it has air dried fully.

2.8.2 Duplicates/Triplicates

All extractions completed on all of the materials used in the project were completed in triplicate to minimize error and develop a sense of precision and accuracy. As with the extractions, every time the analysis of moisture content and LOI were completed, they were also done in triplicate.

Two different continuous columns were duplicated months apart during the study to develop an idea of the reproducibility of the study and the CM.

2.8.3 Standard Checks and Standard Curves

Depending on the procedure being completed, standard curves are created from either vendor purchased stock solution standards, or, by powder chemicals. Table 2-3 shows the various tests completed in the lab, the method of creating the standards for the test, and the range of standards created.

The standards were made every time the tests were completed and were used if they had an R^2 value of 0.999. To further ensure the quality of the data, standard checks were run manually for some tests, and the slopes of the standard curves were compared to one another each time the same test was run again. When using the ICPE-9000-AES, Dionex ICS-1100, and TOC-TN-L, standard checks were placed in the sample tables to be analyzed as normal samples to test the accuracy of the standard curve and to ensure the samples were being read correctly throughout the test runs.

2.9 Statistical Analysis

Statistical analysis was performed on selected nutrient extraction data to compare the results of two data sets. Where applicable, single tailed t-tests were performed using a 5.0% level of significance for extractions with the various soil amendments.

Chapter 3: Cambi Material Results

3.1 Cambi Material Characterization Results

To gain a broad understanding of the background characterization of the CM from both a nutrient and leaching perspective, lab experiments and procedures were completed on the material from June 2015 to September 2016.

3.1.1 Solids, LOI Results

Table 3-1 shows the moisture content and LOI results for the CM.

Table 3- 1. The moisture content and loss on ignition measurements found for the CM.

Cambi Material (CM)	Level	Analysis Date
Moisture Content (% wet)	67.9 ± 0.3	6/10/15
LOI- Organic Content (%)	56 ± 0.06	6/29/15

The moisture content of the CM is about 68%, corresponding to approximately 32% solids. Brandt et al. (2004) observed 41 different types of biosolids, which had a median level of solids at 21% (a moisture content of 79%). Of the biosolids observed, the anaerobically digested biosolids cake (various processing used) ranged from 74.3 to 86.7% moisture content. These values of moisture content were higher than that of the CM. These results indicate the CM may have higher total solids, and less moisture than other biosolids due to the processing it undergoes such as dewatering and gravity thickening.

The LOI testing shows the CM is approximately 56% organic matter. A higher level of organic matter is to be expected with digested biosolids materials than inorganic fertilizers and sewage sludge (Singh and Agrawal 2008). The LOI value of the CM falls

within the range found by O'Connor et al. (2004) of 54.2-78.1. The value of LOI for the CM is on the lower end of the range, which is reasonable since it has been reported that the thermal hydrolysis process has been shown to reduce the organic matter in biosolids by approximately 60% (Carrère et al. 2010).

3.1.2 pH Results

The pH of the CM is 7.99 ± 0.02 (1:2 ratio) and 8.04 ± 0.01 (1:3 ratio). Biosolids do not seem to vary far from neutral; typical levels are fairly neutral (Schroder et al. 2008). Brandt et al. (2004) observed 41 different types of biosolids that had pH values ranging from 5.9-10. The anaerobically digested cake biosolids ranged from 7.13 to 8.51; the CM falls within this range. The pH in biosolids does not seem to significantly affect the pH of soils long term when used as a land application; according to Schroder et al. (2008), the levels fluctuated, but only slightly and therefore it was not an aspect of concern. The CM is not limed so it is not surprising that the pH is not on the higher end of the levels.

3.2 Cambi Material Nutrient Extraction Results

3.2.1 Phosphorus Extraction Results

3.2.1.1 Mehlich III P

Table 3-2 below shows the CM Mehlich III phosphorus levels.

Table 3- 2. The Mehlich III P values for the CM over the course of the project.

Cambi Material (CM)	Average Levels	Analysis Date
Mehlich III P (mg/dry kg)	1330 ± 146	7/20/15
Mehlich III P (mg/dry kg)	1090 ± 10.5	12/15/15
Mehlich III P (mg/dry kg)	1330 ± 20.2	2/24/16
Mehlich III P (mg/dry kg)	894 ± 26.1	4/14/16

Mehlich III P (mg/dry kg)	1020 ± 56.2	6/17/16
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The results of the five Mehlich III P (M3P) extractions ranged from 894 ± 26.1 to 1330 ± 146 mg/kg dry CM. M3P is a predicted value of the plant-available P in soils and other materials. The M3P test predicts the losses of P to runoff and surface waters because it measures the soluble and easily desorbable P (Maguire and Sims 2002). The average overall CM M3P value is 1130 ± 192 mg P/dry kg. To give some comparison to soil M3P values, Hooda et al. (2000) reports soil M3P values ranging from 10 to 567 mg P/dry kg. Tian et al. (2016), Maguire et al. (2001) and Dayton and Basta (2005) found values for soils within this range as well at 26 mg P/dry kg, 49 mg P/dry kg and 315 mg P/dry kg, respectively.

Few studies provide the M3P concentrations for biosolids. Many studies report the values in terms of how much M3P they are applying, but not how much is contained in the biosolids themselves. It is evident that the M3P in the CM is much higher than in soils. Tian et al (2016) completed a study on biosolids as a land application and found them to have a M3P concentration of 21.3 g P/dry kg. This is higher than the CM value by an order of magnitude. When compared to a range of total phosphorus in a group of different types of biosolids given by O'Connor et al. (2004) of 20,000-40,000 mg TP/ kg dry biosolids, the CM M3P values are low.

The CM appears to have lower M3P levels than other types of biosolids, however other nutrients and characterizations such as aluminum and iron need to be taken into consideration when evaluating leachable P concentrations in materials.

3.2.1.2 CaCl₂ P

0.01 M Calcium chloride extractions are often related to water -soluble extractions, or what is likely to leach from soils and other materials by measuring easily desorbable P (Maguire and Sims 2002). Table 3-2 shows the CaCl₂ P values found for the CM.

Table 3- 3. The 0.01 M CaCl₂ extractable P values for the CM.

Cambi Material (CM)	Average Levels	Sample Date
CaCl ₂ P (mg/dry kg)	8.11 ± 1.85	6/24/15
CaCl ₂ P (mg/dry kg)	3.35 ± 0.32	6/30/15
CaCl ₂ P (mg/dry kg)	3.57 ± 0.25	7/20/15
CaCl ₂ P (mg/dry kg)	7.39 ± 0.14	12/15/15
CaCl ₂ P (mg/dry kg)	4.42 ± 0.23	4/14/16
CaCl ₂ P (mg/dry kg)	4.35 ± 0.23	6/17/16

The CM ranges in CaCl₂ P values of 3.35 ± 0.32 to 8.11 ± 1.85 mg P/kg dry CM. These extractable P values are much lower than the values measured from the Mehlich III extraction. This is expected since M3P uses an acidic extracting solution to measure the P. Compared to other studies, the CM appears to have a low water extractable P value. For example, Dayton and Basta (2005) measured biosolids to have 62.5 mg P/ dry kg, which falls in the range of 10 to 8,860 mg P/ dry kg found by Brandt et al. (2004). Although the CM average value of 5.20 ± 2.03 mg P/ dry kg is lower than this range, it is important to note that the biosolids in these studies do not undergo the same treatment of processes that the CM does at DC Water. Many of the biosolids used in these studies use techniques such as liming, alkaline stabilization, aerobic and anaerobic digestion, and, the majority are not “Class A” materials (Brandt et al. 2004).

3.2.1.3 Ammonium Oxalate Extractable P

Ammonium oxalate extractions are used for their ability to predict and set environmental limits for phosphorus in soils (Maguire and Sims 2002). The ammonium

oxalate extraction is used to measure P_{ox} , Al_{ox} and Fe_{ox} . It has been reported that oxalate extractable P is highly correlated to total phosphorus concentrations (Maguire et al. 2001). Table 3-4 shows the values of P_{ox} for the CM.

Table 3- 4. The ammonium oxalate phosphorus extraction values for the CM.

Cambi Material (CM)	Average Levels	Sample Date
Oxalate P (mg/dry kg)	23100 \pm 23.1	7/10/15
Oxalate P (mg/dry kg)	33100 \pm 1210	7/16/15
Oxalate P (mg/dry kg)	36300 \pm 1010	7/29/15
Oxalate P (mg/dry kg)	44500 \pm 1020	12/15/15
Oxalate P (mg/dry kg)	39800 \pm 592	2/25/16

The P_{ox} values for the CM range from 23,100 \pm 23.1 mg P/dry kg to 44500 \pm 1020 mg P/dry kg with an average value of 35400 \pm 8050 mg P/dry kg. The majority of these values fall within the high ranges of P_{ox} that have been reported in other studies that examine various types of biosolids. For example, Maguire et al. (2001) studied eight different types of biosolids in addition to poultry litter and found their range in P_{ox} to be 4900 to 32,900 mg/dry kg. Agyin-Birikorang et al. (2008) reported a range of 11,000-to 22,000-mg/ dry kg, and Dayton and Basta (2005) found a level of 15,300-mg/ dry kg. Most of the values of P_{ox} measured for the CM fall within the range given by Maguire et al. (2001), however they exceed both the range given by Agyin- Birkiorang et al. (2008) and value given by Dayton and Basta (2005).

3.2.2 Nitrogen Extraction Results

3.2.2.1 2.0 M KCl N

Potassium chloride extractions are used for soils and other materials as an estimate of the inorganic nitrogen content (Goloran et al. 2013). The extraction is not a

measurement of the total nitrogen in the CM, however the total extractable KCl nitrogen was found. Table 3-5 shows the KCl extractable N values found in the CM.

Table 3- 5. The 2 M KCl extractable N values for the CM.

Cambi Material (CM)	Average Levels	Sample Date
KCl N (mg/dry kg)	2130 \pm 93.4	7/1/15
KCl N (mg/dry kg)	6920 \pm 144.6	7/20/15
KCl N (mg/dry kg)	7160 \pm 46.6	8/4/15
KCl N (mg/dry kg)	7680 \pm 344	12/15/15
KCl N (mg/dry kg)	7250 \pm 55.1	4/25/16
KCl N (mg/dry kg)	7360 \pm 344	6/13/16

The KCl N measurements ranged from 2130 \pm 93.4 mg N/kg dry CM to 7680 \pm 344 mg N/dry kg CM. The average value is 6420 \pm 2120 mg N/kg dry CM. KCl N concentrations were not found in the literature on biosolids, however, Goloran et al. (2013) reported that the KCl extractable nitrogen is a superior indicator for plant N uptake in soils.

Although KCl extractions are often used as an estimate of the inorganic N content, it is important to take into consideration the total nitrogen values for biosolids to compare the ranges. Chinault and O'Connor (2008) reported TN values of 50,000 to 70,000 mg TN/dry kg and O' Connor et al. (2004) reported TN values of 21,100 to 68,800 mg TN/dry kg. These values are much higher than the inorganic KCl extraction values for the CM. However, if the KCl extraction is mainly the inorganic nitrogen, it is not extracting the organic nitrogen components in the CM which are predicted to be high in biosolids due to their high organic content.

3.2.2.2 0.01 CaCl₂ N

The CaCl₂ extraction procedure has been used to determine the bioavailable nutrients in soils and other material samples (Houba et al. 2000). The extraction is

measured for CaCl₂ TN, and nitrogen speciation (NH₄-N, NO₂-N, NO₃-N). The results of the extraction total nitrogen measurements are given in Table 3-6. The total speciation data is found in the Aging Study results in Chapter 6.

Table 3- 6. The CaCl₂ extractable TN for the CM.

Cambi Material (CM)	Average Levels	Sample Date
CaCl ₂ N (mg/dry kg)	2750 ± 105	6/13/16

The N values for the CaCl₂ extraction are lower than that of the KCl extraction. This is to be expected and has been seen in other studies. The KCl extraction is a stronger extracting reagent than the CaCl₂ reagent. The CaCl₂ extraction has been proposed to mimic the ionic strength in soil solutions and furthermore extract most available N (Goloran et al. 2013). Both Inselsbacher (2014) and Zhou et al. (2012) observed a higher amount of nitrogen in a 2M KCl extraction than in a simple water extraction (comparable to a 0.01 M CaCl₂ extraction).

3.2.3 Potassium Extraction Results

3.2.3.1 Mehlich III K

Potassium is not often discussed in studies about biosolids. Potassium leaching is not well known or understood, however it is important to measure and analyze potassium from a fertilizer perspective. The Mehlich III extraction is completed to find the plant available potassium (van Raij 1998). The values for the CM were measured and are shown in Table 3-7.

Table 3- 7. The Mehlich III K values for the CM.

Cambi Material (CM)	Average Levels	Sample Date
Mehlich III K (mg/dry kg)	1620 \pm 66.8	12/15/15
Mehlich III K (mg/dry kg)	298 \pm 25.0	6/13/16

Potassium content is discussed more in plant sciences literature. According to Kayser and Isselstein (2005), potassium is not of serious environmental concern, however it is important from an agronomic perspective. Plants uptake potassium second to nitrogen, and therefore the levels of K should be most similar to N (Kayser and Isselstein 2005). When the M3K values for the CM are compared to the KCl N values, they are lower, however they are on the same order of magnitude. These are different extractions so they are not fully relatable but they do give an idea of the nutrient content in the material.

3.2.4 Ammonium Oxalate Extraction Results

3.2.4.1 Ammonium Oxalate Extractable Aluminum

Ammonium oxalate extractable aluminum (Al) is often measured because when found with oxalate P and Fe, the concentrations can be used to find the phosphorus saturation index, a widely known and used tool to characterize the potential for phosphorus leaching (Dayton and Basta 2005). The Al_{ox} concentrations for the CM were found and are listed in Table 3-8.

Table 3- 8. The ammonium oxalate extractable Al for the CM throughout the project.

Cambi Material (CM)	Average Levels	Sample Date
Oxalate Al (mg/dry kg)	2890 \pm 2.89	7/10/15
Oxalate Al (mg/dry kg)	3920 \pm 286	7/16/15
Oxalate Al (mg/dry kg)	3950 \pm 191	7/29/15
Oxalate Al (mg/dry kg)	2910 \pm 47.8	12/15/15
Oxalate Al (mg/dry kg)	2730 \pm 31.1	2/25/16

The CM Al_{ox} average concentration was 3280 ± 600 mg / dry kg CM. This value compares with other studies completed on biosolids however it is on the lower side. Agyin- Birikorang et al. (2008) reported a range of Al_{ox} in biosolids of 3100 ± 500 to 25,000 ± 3500 mg/kg dry. O'Connor et al. (2004) reported a range of 4200 to 30,700 mg/dry kg. Some of the Al_{ox} values for the CM fall in the range for the Agyin- Birkorang et al. (2008) study, however they are on the low side of the range. This could be due to different treatments to the biosolids in the wastewater treatment plants. Often times biosolids are treated with amendments such as aluminum sulfate, aluminum based WTR, and other materials that can increase the aluminum levels in the biosolids themselves (Donner et al. 2013).

3.2.4.2 Ammonium Oxalate Extractable Iron

Ammonium oxalate extractable iron levels for the CM were found and are shown in table 3-9 below.

Table 3- 9. The ammonium oxalate extractable Fe values for the CM.

Cambi Material (CM)	Average Levels	Sample Date
Oxalate Fe (mg/dry kg)	87600 ± 87.6	7/10/15
Oxalate Fe (mg/dry kg)	88800 ± 3970	7/16/15
Oxalate Fe (mg/dry kg)	92400 ± 3490	7/29/15
Oxalate Fe (mg/dry kg)	80700 ± 2490	12/15/15
Oxalate Fe (mg/dry kg)	86100 ± 1470	2/25/16

The average Fe_{ox} value for the CM is 87,100 ± 4290 mg Fe/kg dry CM. It is expected that some biosolids have higher levels of iron because it is added during tertiary wastewater treatment to help precipitate phosphorus. These precipitates end up in the solids, or biosolids formations. Compared to other biosolids in studies, the Fe_{ox} values for

the CM are high. Agyin-Birikorang et al. (2008) reported a range in Fe_{ox} of 1200 ± 300 to 71000 ± 800 mg Fe/ kg dry, and O'Connor et al. (2004) reported a range of 900 to 38,900 mg Fe/dry kg. The CM Fe_{ox} levels are on the same order of magnitude as the higher range found by O'Connor et al. (2004), however they are still more than twice the amount. It is predicted that the iron pretreatments that are added to enhance phosphorus precipitation result in a higher overall iron content in the CM biosolids.

3.2.4.3 Ammonium Oxalate PSI

The oxalate phosphorus saturation index (PSI_{ox}), or molar ratio between forms of P to Al + Fe, is an indication of the bonding between P, Al and Fe. Equation 3-1 shows the calculation for determining the PSI_{ox}.

$$PSI_{ox} = \frac{P_{ox}/Moles\ P}{Al_{ox}/Moles\ Al + Fe_{ox}/Moles\ Fe} \quad (Eqn\ 3-1)$$

where P_{ox} , Al_o, and Fe_{ox}, are the phosphorus, aluminum, and iron concentrations, all as moles/kg.

Table 3-10 shows the values of PSI_{ox} for the CM.

Table 3- 10. The ammonium oxalate PSI values for the CM.

Cambi Material (CM)	Average Levels	Sample Date
PSI	0.616 ± 0.015	7/16/15
PSI	0.651 ± 0.010	7/29/15
PSI	0.926 ± 0.005	12/15/15
PSI	0.781 ± 0.018	2/25/16

The average PSI_{ox} for the CM was 0.744 ± 0.141. This value falls within the PSI ranges reported in other studies on biosolids. For example, Agyin-Birikorang et al. (2008) reported a range in PSI_{ox} values of 0.43-2.1, and O'Connor et al. (2004) found a range of 0.5-1.4. Studies completed on compost mixtures prepared to certain PSI_{ox} values have

shown that a PSI_{ox} above 0.5 will result in nutrient P leaching and below 0.1 will result in almost no P leaching and possible nutrient uptake (Brown et al. 2015). With these findings, it was predicted that the CM would have P leaching since most of the PSI_{ox} measurements were above 0.5.

3.2.5 Nutrient Characteristics Discussion

The CM produced nutrient extraction levels both within and outside of the ranges reported in other biosolids studies. The CM itself is not processed the same way as all of the other biosolids it is being compared to. Due to the difference in material processing it is not surprising that the ranges in nutrient extraction data are different.

The extractable nitrogen levels for the CM were not compared to other biosolids nitrogen levels; however when tested for nitrogen speciation, ammonium was the dominant form in both extractions. With these data, it is predicted that most of the nitrogen that will leach from the CM will be in the form of NH_4^+-N and it will be much higher than the EPA recommended level of nitrogen to enter surface waters.

The CM was within range for oxalate extractable P, however lower than reported ranges for both extractable M3P and $CaCl_2$ P. The P extractions can be related to the PSI_{ox} and the predictability of P leaching from the CM. The levels of $CaCl_2$, or water extractable (likely to leach P) are low which means it is predicted P leaching from the CM will be on the lower end of biosolids, however the PSI_{ox} values average 0.744 ± 0.141 . Since this level is above 0.5, it is predicted that the material is likely to exhibit P leaching despite its high levels of Al and Fe.

Chapter 4: Soil and Amendment Material Characterization

4.1 Soils Material Characterization Results

Two soil types; a sandy loam and loamy sand were used throughout this project as experimental controls and in mixes with different materials. A similar physical and nutrient characterization was completed on the soils as was done on the CM.

4.1.1 Solids, LOI Results

The soils were both tested for moisture content and LOI. Table 4-1 shows the values for moisture content and LOI.

Table 4- 1. The moisture content and loss on ignition values for the two types of soils.

Soils	Average Levels	Sample Date
<i>Loamy Sand</i>		
Moisture Content (%)	1.13 ± 0.214	9/10/15
LOI- Organic Content (%)	1.38 ± 0.076	9/10/15
<i>Sandy Loam</i>		
Moisture Content (%)	2.32 ± 0.326	9/10/15
LOI- Organic Content (%)	1.68 ± 0.015	9/10/15

The soils were dry when they were collected, and it had not rained in a couple of weeks so it was not surprising that they had low moisture contents. The sandy loam (SL) had slightly more moisture than the loamy sand (LS), however they both were mostly dry. The two soils used for this project contained a very small amount of organic matter, LS contained approximately 1.38% and SL contained approximately 1.68%. These low levels of organic matter are not surprising; sandy soils used in other studies have been shown to have low levels below 1% (Elliott et al. 2002).

4.1.2 Phosphorus Extraction Results

The soils were tested for CaCl_2 , Mehlich III, and ammonium oxalate extractable phosphorus. The results of the phosphorus extractions are given in Table 4-2.

Table 4- 2. The phosphorus extraction values for the two soil types.

Soils	Average Levels	Sample Date
<i>Loamy Sand</i>		
CaCl_2 P (WEP) (mg/dry kg)	0.461 ± 0.277	9/3/15
Mehlich III P (mg/dry kg)	26.5 ± 1.99	9/3/15
Oxalate P (mg/dry kg)	54.3 ± 25	2/26/16
<i>Sandy Loam</i>		
CaCl_2 P (WEP) (mg/dry kg)	0.364 ± 0.093	9/3/15
Mehlich III P (mg/dry kg)	29.5 ± 1.85	9/3/15
Oxalate P (mg/dry kg)	44.3 ± 2.15	2/26/16

The phosphorus extraction values in the two soils were comparable to other studies with similar sandy and loamy soils. Maguire et al. (2001) found a sandy loam to have a water extractable concentration of 0.3-mg/dry kg, a M3P value of 19-mg/dry kg and an oxalate P value of 55-mg/dry kg. These values are comparable to the values found for the two soils used in this project.

4.1.3 Ammonium Oxalate Extraction Results

The oxalate extractable Al and Fe were also found for the two soils, and with them the PSI_{ox} values for the soils were calculated. The Al_{ox} , Fe_{ox} and PSI_{ox} values for both soils are given in Table 4-3.

Table 4- 3. The ammonium oxalate extractable Fe and Al values, along with the calculated PSI's for both soils.

Soils	Average Levels	Sample Date
<i>Loamy Sand</i>		
Oxalate Al (mg/dry kg)	168 ± 8.12	2/26/16
Oxalate Fe (mg/dry kg)	558 ± 18.3	2/26/16

PSI	0.108	2/26/16
<i>Sandy Loam</i>		
Oxalate Al (mg/dry kg)	194 ± 6.42	2/26/16
Oxalate Fe (mg/dry kg)	601 ± 39.4	2/26/16
PSI	0.080	2/26/16

The soils Al_{ox} values of 168 and 194 mg/dry kg compare well to ranges found in other studies for different types of loamy and sandy soils. Elliott et al. (2002) found Al_{ox} values of 40.1 and 12.5 mg/dry kg, however Maguire et al. (2001) found the Al_{ox} to be 439 mg/dry kg. The soils in this study are within the range of these studies for oxalate extractable aluminum values. The Fe_{ox} values of 558 and 601 measured are much higher than the values found by Elliot et al. (2002) of 10.1 and 85.6 mg/dry kg, and between the values of 335 and 1991 mg/dry kg found by Maguire et al. (2001).

The PSI_{ox} values for the soils were 0.108 and 0.080. These values are comparable to the PSI values found by Elliot et al. (2002) of 0.14 and 0.19. The low values for soils are to be expected since they have very low levels of native phosphorus and are meant to be control materials.

4.2 WTR Material Characterization Results

The WTR used in this project was aluminum-based and tested for various physical and nutrient characterizations.

4.2.1 Solids, LOI Results

The WTR was tested for both moisture content and LOI. The LOI test was completed differently for the WTR than for the CM and soils. Elliott et al. (2002) completed a study on WTR and found that the LOI for both Al and Fe based WTR were

too high to comfortably predict organic matter. Elliott et al. (2002) found that when total organic carbon (TOC) and LOI values for various WTRs were completed, it was found that the LOI values were an overestimation. For this project, the LOI process was completed at three different temperatures. The results for both the LOI and moisture content of the WTR are given in Table 4-4.

Table 4- 4. The moisture content and LOI values for the WTR.

Water Treatment Residual (WTR)	Average Levels	Sample Date
<i>Moisture Content (%)</i>	73.04 ± 0.10	8/18/15
LOI (%) @ 180°C	7.04 ± 0.07	8/18/15
LOI (%) @ 350°C	23.98 ± 0.32	8/18/15
LOI (%) @ 550°C	30.27 ± 0.50	8/18/15

The values of LOI found at the temperatures of 350°C and 550°C of 23.98 ± 0.32 and 30.27 ± 0.50 are comparable to those found by Elliott et al. (2002) of 24%. These values are only an approximation of the organic content in the WTR.

4.2.2 Ammonium Oxalate Extraction Results

Oxalate extractions were completed on the WTR (Table 4-5).

Table 4- 5. The ammonium oxalate extraction values and PSI value found for the WTR.

Water Treatment Residual (WTR)	Average Levels	Sample Date
Oxalate P 1:50 (mg/ dry kg)	2200 ± 14	8/18/15
Oxalate Fe 1:50 (mg/ dry kg)	4600 ± 70	8/18/15
Oxalate Al 1:50 (mg/ dry kg)	796000 ± 127000	8/18/15
PSI _{ox} Molar Ratio	0.0024 ± 0.0004	8/18/15

The WTR contains a very large amount of aluminum, 796 g/kg dry CM. This high level of Al makes the PSI_{ox} of the WTR very low, at a level of 0.0024 ± 0.0004 . Due to the high P adsorption capacity in the aluminum, it is predicted that the addition of WTR to the CM will decrease the phosphorus leaching (Alleoni et al. 2014). When the WTR is

mixed with the CM, it is predicted that the WTR amendments will decrease the overall PSI_{ox} levels of the mixture and therefore decrease some of the P that could leach without the WTR amendment.

4.2.3 WTR and CM Mixture Results

Multiple ratios of WTR: CM (dry mass basis) were chosen and analyzed for various nutrient extractions to develop predictions on the effectiveness of WTR incorporation with the CM for P mitigation. The ratios of WTR: CM that were chosen were 7.5, 15, 25, 35, 50, 60, and 75%. The results of the M3P, M3K, $CaCl_2$ P, and KCl N extractions are shown in Figure 4-1.

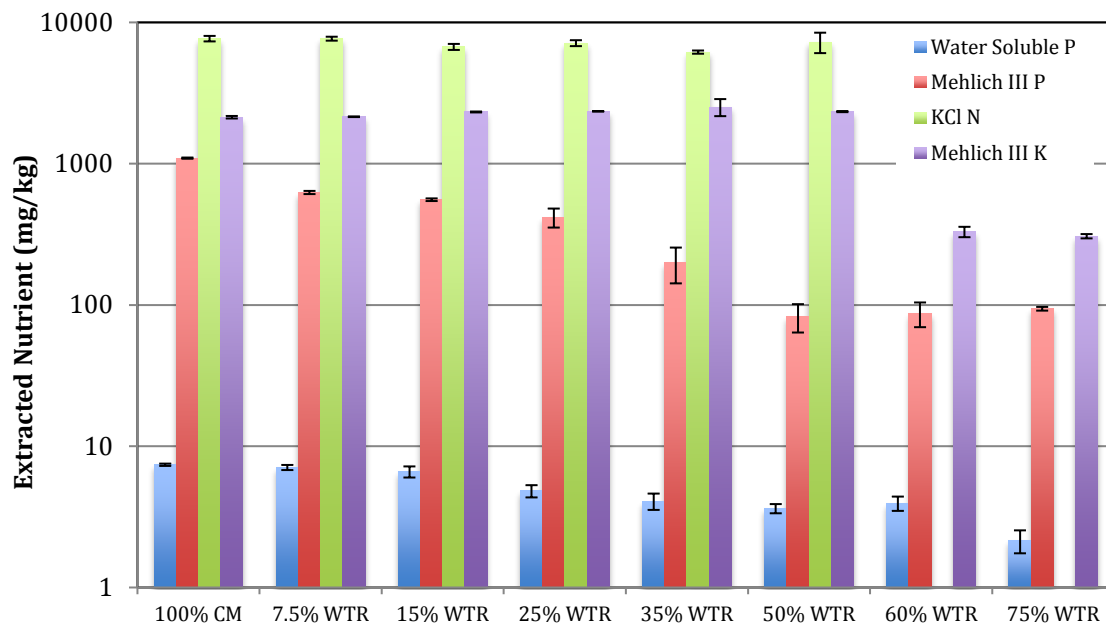


Figure 4- 1. The results of $CaCl_2$ extractable P, M3P, KCl N, and M3K for CM, and CM with various ratios of WTR incorporated. The error bars represent one standard deviation within triplicate samples.

In Figure 4-1, 100% CM (no added WTR) nutrient extraction data are reported on the left side of the figure. As the figure goes from left to right the ratio of WTR: CM

increases. As the WTR amount increases, the phosphorus levels (both M3P and CaCl_2) decrease. The decrease in extractable P levels was predicted with higher amounts of WTR amendments due to the predicted increase in extractable Al_{ox} levels and adsorption capacity.

T-tests were completed on the nutrient extractions for both M3P and CaCl_2 P for the various individual mixtures of WTR and CM and 100% CM. The results showed that at a $\alpha=0.05$ level, all of the mixtures were significantly different than the 100% CM for both M3P and CaCl_2 P. For the M3P the 50% and 60% mixtures were not statistically different from one another, however all of the other mixtures were from each. The CaCl_2 had some mixtures that were not statistically different from one another; for example the 35% WTR: CM mixture was not statistically different from the 50% or 60% mixture, and the 50% was not statistically different from the 60% mixture. The overall findings based on these statistics however were that the WTR was having a significant effect on the P leaching/extractions from the materials. The levels of M3K and KCl extractable N were not significantly changed from the added amounts of WTR.

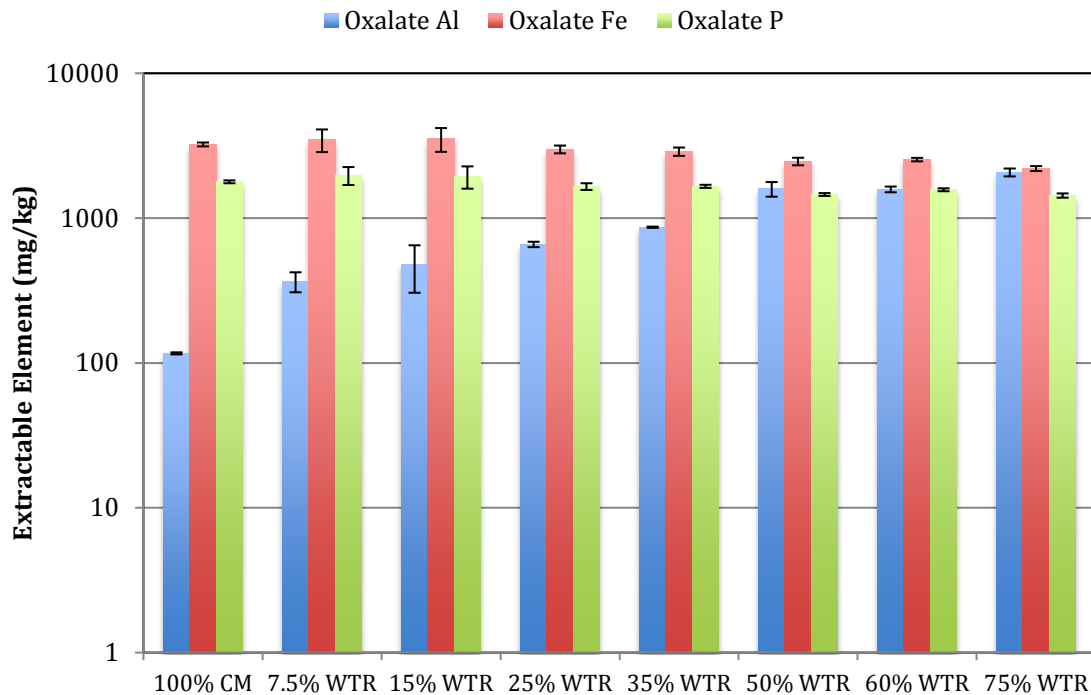


Figure 4- 2. The ammonium oxalate extractable Al, Fe, and P for the CM and CM with various ratios of WTR mixtures. The error bars represent one standard deviation within triplicate samples.

Figure 4-2 shows the levels of oxalate extractable Al, Fe, and P. As predicted, the P_{ox} levels decrease as the WTR added increases. Also as predicted, the Al_{ox} levels increase as WTR added increases. Figure 4-3 shows the PSI_{ox} values calculated for the CM and mixtures of increasing WTR: CM ratios (dry mass basis).

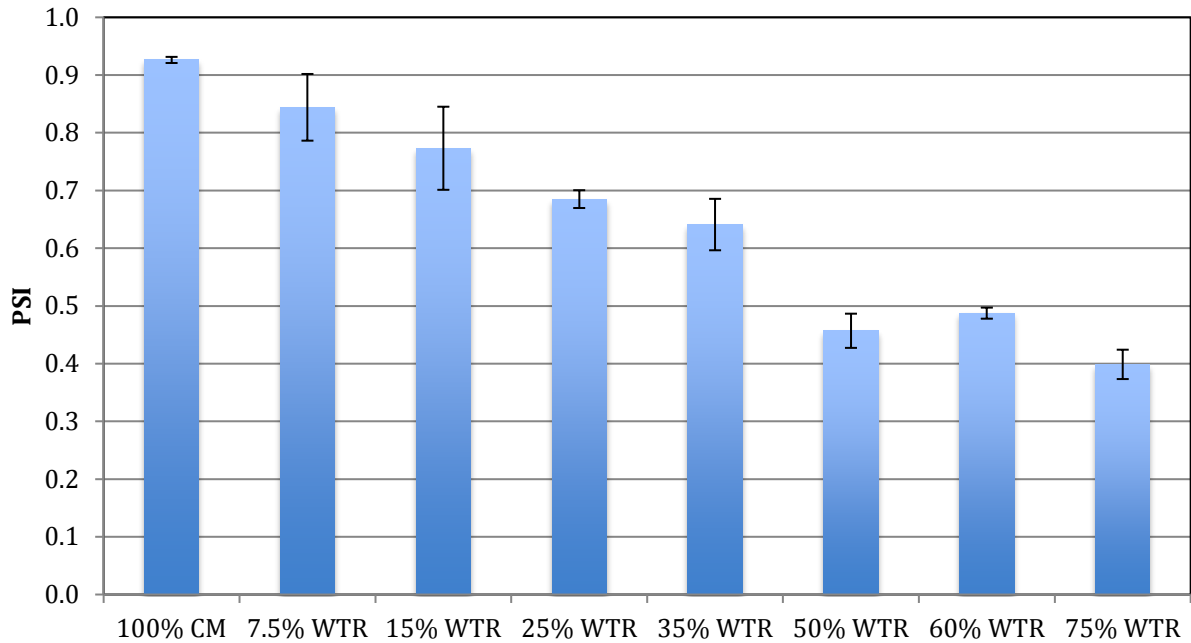


Figure 4- 3. The PSI_{ox} values for the CM and mixtures of CM and increasing WTR amendments. The error bars represent one standard deviation within triplicate samples.

As predicted, with increasing Al_{ox} and decreasing P_{ox}, the PSI_{ox} decreased with increasing WTR amendment additions. The 75% WTR amendment reached a PSI_{ox} value of 0.399 ± 0.025 ; this is more than half of the PSI_{ox} of 0.926 ± 0.005 found for the CM alone. From these PSI_{ox} values further hypotheses were drawn on WTR amendments and their impact on P mitigation. The lower the P_{ox} to Al_{ox} + Fe_{ox} ratios are, or, the higher the Al_{ox} and Fe_{ox} contents are in the biosolids, the less likely the P is to leach. Maguire et al. (2001) concluded that the PSI_{ox} ratio is one of the best indicators and superior to other methods often used in studies on biosolids land applications for determining the likelihood of P leachability. Brown et al. (2015) completed a study on PSI_{ox} in mixtures of compost and WTR and tested three PSI_{ox} values: 0.1, 0.5, and 1.0. Leachate from the mixtures was observed over time to examine possible equilibrium and patterns. Overall, it was concluded that the PSI_{ox} was a good determining factor for P leaching; the 0.1 PSI_{ox}

mixtures of compost and WTR leached minimal P, and in fact removed some of the P from the influent synthetic stormwater (Brown et al. 2015). In addition, Elliott et al. (2002) completed a study on PSI_{ox} ratios and found that when biosolids PSI_{ox} values were above 1.1, they had excess P leaching, whereas at values below 1.0, P adsorption to the Al and Fe was more effective.

Relating these conclusions back to the CM and WTR mixtures, the lowest PSI_{ox} value reached was with the 75% WTR: CM (0.399 ± 0.025). This value falls between the 0.1 and 0.5 in the study by Brown et al (2015) and also below the 1.0 value reported by Elliott et al. (2002). It is predicted that the 75% WTR: CM mixture will result in the lowest amount of P leaching.

4.3 Zeolite Material Characterization Results

Zeolite has a high CEC and is known for its high cationic adsorption capacity. Studies with synthetic wastewater and high ammonium- containing influents have shown zeolite to adsorb and remove a high level of ammonium (Wang and Peng 2010). Table 4-6 shows the physical characteristics for the zeolite used in this project.

Table 4- 6. Physical characteristics for the zeolite. Bear River Zeolites provided the values.

Physical Properties	Values
Cation Exchange Capacity (CEC):	1.4 - 1.65 meq/g as $NH_4^{+}-N$
Bulk Density	55-60 lb/ ft ³
Overall Surface Area	24.9 m ² /g

4.3.1 Zeolite and CM Mixture Results

With the provided physical properties, in particular, the CEC, ratios of zeolite were proposed to be effective at ammonium adsorption. The following calculations were

completed based on estimated concentrations of NH_4^+ leaching which were an average calculated from column study effluents:

$$\text{total avg N accumulation} \cong 150 - 180 \text{ mg/L (120 m water applied, 20 g CM)}$$

$$\text{avg } \text{NH}_4^+ \cong 65 - 70\% \text{ TN}$$

$$\text{NH}_4^+ - \text{N} \cong 108 \text{ mg } \text{NH}_4^+ - \text{N}$$

$$\frac{108 \text{ mg } \text{NH}_4^+ - \text{N}}{20 \text{ g dry CM}} * \frac{m_{eq}}{14 \text{ mg } \text{NH}_4^+ - \text{N}} * \frac{1 \text{ g zeolite}}{1.4 m_{eq}} = 0.28 \frac{\text{g zeolite}}{\text{g CM}} \text{ (Eqn 4-1)}$$

From the calculations in equation 1 it was predicted that a percentage between 25-30% zeolite: CM (dry mass basis) would be effective at ammonium adsorption. With this prediction, three ratios were chosen: 10%, 20%, and 30% zeolite: CM (dry mass basis). The hypothesis was that the 30% would have the biggest impact, but each ratio would be somewhat effective at ammonium adsorption. Figure 4 has the results of the nutrient extractions for each ratio.

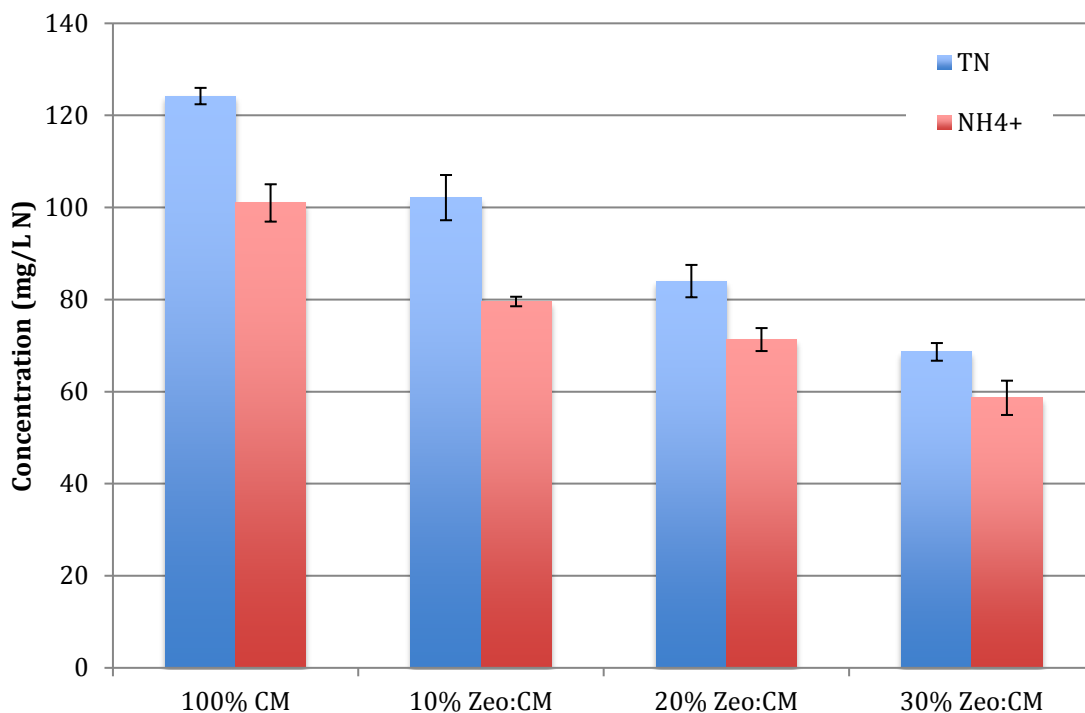


Figure 4- 4. The TN and NH₄⁺ concentrations from CaCl₂ extractions on 100% CM and mixtures of Zeolite: CM (dry mass basis).

Figure 4-4 shows the evident decrease in both TN and NH₄⁺ that result from increasing additions of zeolite. As predicted, the 30% zeolite: CM mixture was most successful at decreasing the both TN and NH₄⁺ concentrations. Zeolite provides more surface area and therefore an increase in CEC and ability of the zeolite to capture the NH₄⁺ from the leachate.

In addition to the evident decreases in TN and NH₄⁺ shown in Figure 4, t-tests completed on the three ratios of Zeolite: CM and the 100% CM mixture show the concentrations of NH₄⁺ to be significantly different at a significance level of 5%. This confirms that the increasing amounts of zeolite are making a significant difference in the ammonium concentrations from a batch test perspective. From this it was hypothesized that the zeolite would be an effective amendment in column studies at mitigating some of the ammonium and TN leached from the CM. Nitrate and nitrite were not measured with

these extractions, however it is predicted that organic N would still leach from the column studies with zeolite amendments.

4.4 Biochar Material Characterization Results

Biochars can be prepared at different temperatures and from different materials. Past studies show that the effectiveness of biochars with ammonium, phosphate and nitrate adsorption differs with the type of biochar and the temperature at which it is produced (Yao et al. 2012). Past studies did not provide any information on biochar produced from Southern Yellow Pine; however most plant derived biochars resulted in successful NH_4^+ adsorption.

Table 4-7 provides the physical properties of the biochar used for this project.

Table 4- 7. Physical properties given for the biochar.

Properties	Values
Moisture Content	25%
pH	9-9.5
Temperature Prep'd:	550° C

Compared to the CM, the biochar has lower moisture content and a higher pH. The pH of the biochar is on the higher end of the range 5.2-9.1 found for other biochars found by Yao et al. (2012), and within the range 9.15-10.11 found by (Cui et al. (2016).

4.4.1 Biochar and CM Mixture Results

Biochar was added to the CM in mixtures based on dry weight and analyzed for TN and NH_4^+ from a CaCl_2 extraction. Three ratios of biochar: CM (dry mass basis) were chosen: 10%, 20%, and 30%. These ratios were kept on the lower end to match with the zeolite percentages chosen, which were based on the CEC of the zeolite. It was predicted

that with more biochar added, there would be an overall decrease in TN and NH_4^+ . Figure 5 shows the changes in TN and NH_4^+ values found for the mixtures.

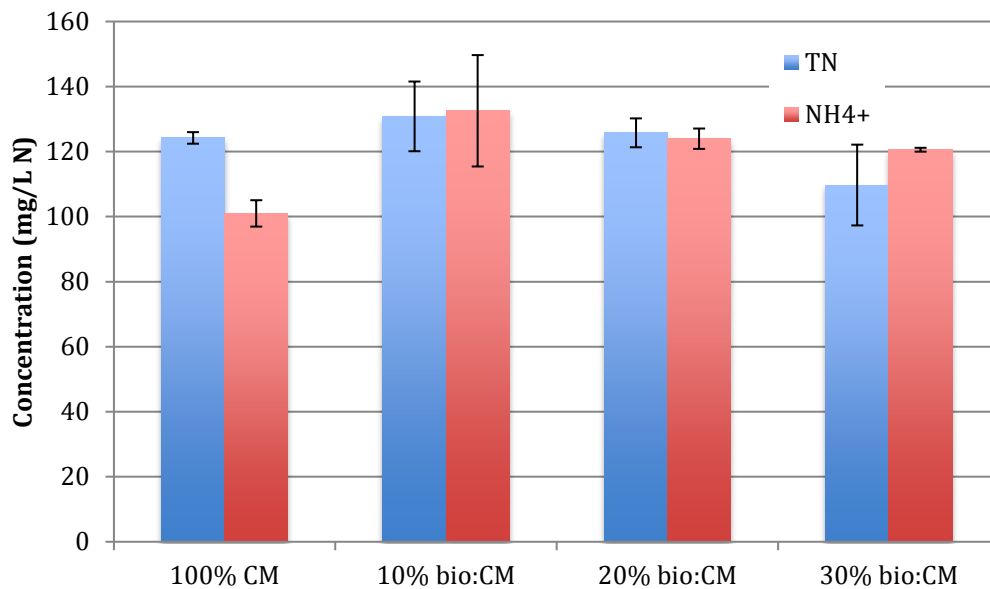


Figure 4- 5. The CaCl_2 extractable TN and NH_4^+ values for CM and mixtures with added biochar.

The biochar mixture results for CaCl_2 mixtures did not produce as effective decreases as the zeolite mixtures. The 10% biochar: CM mixture was higher for TN and NH_4^+ than the CM alone. There was a decrease in TN as the biochar percentage increased; however it was slight. In addition, the NH_4^+ concentrations for each mixture were very similar to the TN values. From these nutrient extractions, it was predicted that biochar had some effect on ammonium adsorption, however it was not going to be as effective as zeolite. T-tests ($\alpha=0.05$) also showed the different biochar amendments were not statistically different from each other, however they were all statistically different from the 100% CM. From the values produced, it appeared that the biochar increased the NH_4^+ amount in the mixtures.

Chapter 5: Column Studies

5.1 Column Study Descriptions

5.1.1 Continuous Column Studies

Seventeen columns were continuously run for three weeks (approximately 120 m water applied) with different amendments and percentages of CM. Table 2-2 in Chapter 2 lists the column experiments that were completed. The purpose of the continuous columns was to develop a better understanding of the nutrient leaching properties of the CM under continuous (fully saturated) conditions. It was predicted that studying the continuous flow effluent nutrient characteristics would result in hypotheses for how the CM would leach if it were placed in the environment. Continuous flow was employed to study the timespan of material leaching and the long term leaching possibilities.

The majority of the continuous columns were completed with 15% CM (dry mass basis). The reported nutrient leaching data in chapter 5 will be from the 15% CM columns, however 10% (dry mass basis) columns were completed and nutrient data is reported in Appendix C.

5.1.2 Non-Continuous Column Studies

The four (three fully completed) non-continuous column studies were significant to the project because they gave a more realistic idea of how the CM could act in the environment. The non-continuous columns allowed the media in the columns to become unsaturated during the draining periods as opposed to constant full saturation in the wet columns. Past studies on soils in natural environments and other environments such as

wetlands have seen increases in N, and P leaching upon rewetting after having a draining period in the soil (Baldwin and Mitchell 2000). The nutrient effluent concentration increase can be attributed to nitrification occurring, or the mineralization of nutrients, which can then be flushed out with the first water application in the next wet period (Venterink et al. 2002). Whether it is either mechanism, it is a common occurrence and was predicted to occur with the CM in the columns.

5.1.2.1 15% Dry CM

The non-continuous columns were completed identically to the continuous columns apart from being stopped for at least 24 hours in between the 24-hour wet periods. Before the non-continuous columns were completed it was predicted they would have an initial washout and intermediate increases, but an overall decrease overtime in nutrient effluent concentrations.

Four non-continuous columns were first prepared. The four columns were: a 100% sandy loam soil column, a 15% CM (dry mass basis) and sandy loam column, a 15% CM (dry mass basis) and sand column, and a 15% CM with 75% WTR: CM (dry mass basis) and loamy sand column. Unfortunately the column with the WTR amendment did not last through the duration of the wet-dry period. The cap of the column completely came off under pressure. Figure 5-1 shows the cap burst that occurred with the WTR column.



Figure 5- 1. The broken column and cap from the column made with the WTR/CM mix.

The CM seems to have clay-like characteristics in it that it holds moisture very well and acts almost like “playdough” in physical texture. Other studies have seen that biosolids slow down water infiltration rates and increase the bulk density of the soil mixture when they are used as soil amendments (McIvor et al. 2012). This decrease in water infiltration may have influenced the column studies and not allowed the water to flow through the column in a timely manner, causing the caps or tubing to burst off.

The cap malfunction was not surprising since the material “playdough” like property was seen in the initial columns in the first column study with the 15% CM and sand. When those columns were initiated it became apparent that the water would not flow through the CM when it was a layer on its own. This is an indication that the material may have an extremely low infiltration capacity. Despite the column break, some of the data with the WTR column are still usable and will be reported.

5.2 Phosphorus

5.2.1 Continuous Column Phosphorus Leaching

All of the continuous columns completed were analyzed for the total phosphorus (TP) concentration in each effluent sample taken. Figures 5-2 and 5-3 show the phosphorus leaching concentrations for three different continuous flow columns.

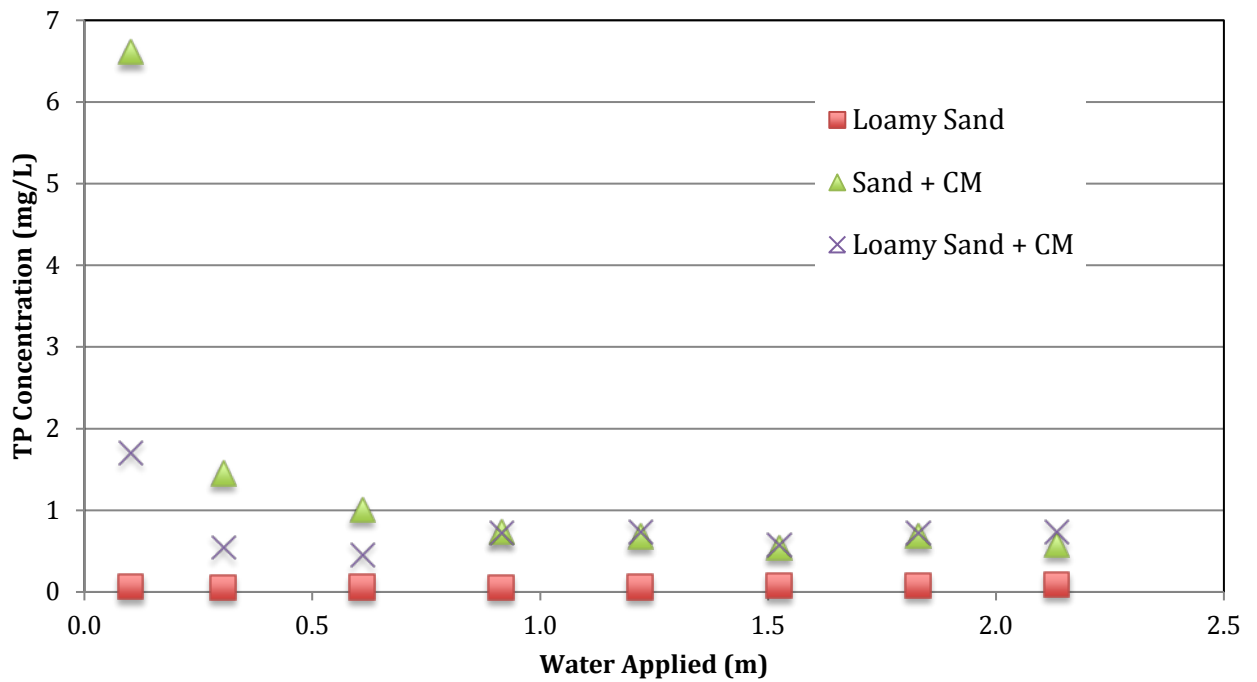


Figure 5- 2. The initial day of TP effluent concentrations in three columns. 100% Loamy Sand soil, 15% CM (dry mass) with 85% Sand, and 15% CM (dry mass) with 85% Loamy Sand.

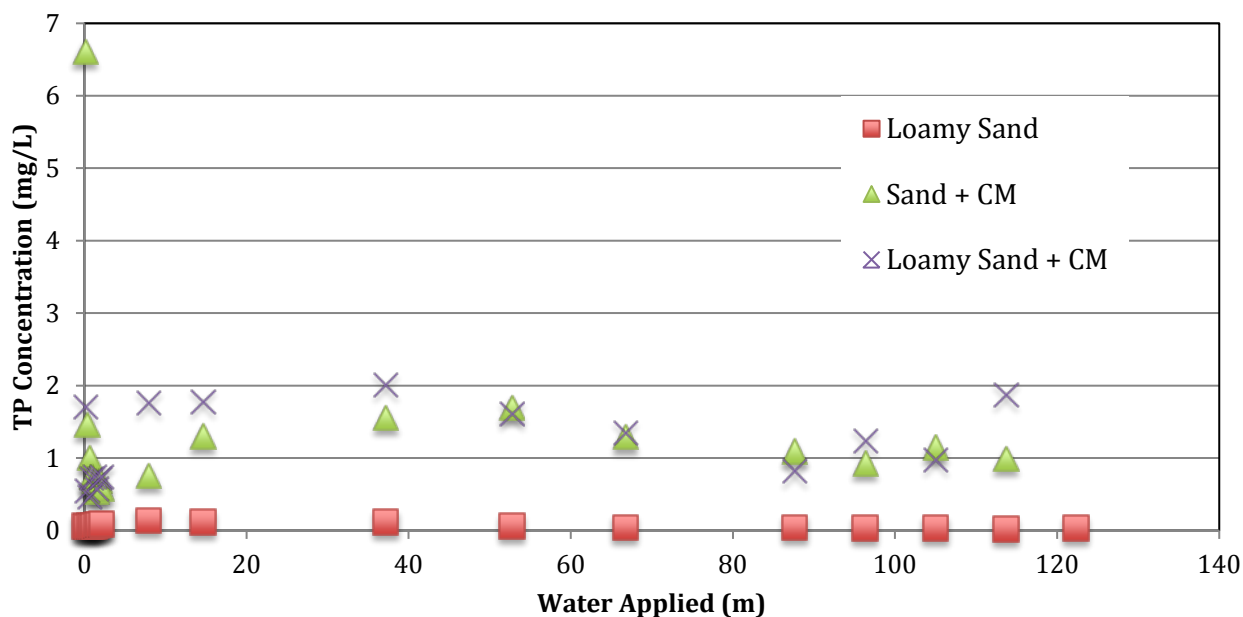


Figure 5- 3. The entire duration of TP effluent concentrations. 100% Loamy Sand soil (red), 15% CM (dry mass) with 85% Sand (green), and 15% CM (dry mass) with 85% Loamy Sand (purple).

The TP concentrations in the columns with 100% loamy sand were completed as an experimental control column and provided an estimate of how the CM would act alone without soil or amendments. The column with the CM and the loamy sand was used to predict the nutrient leaching that could occur if the CM were placed in the environment with natural soils.

Figures 5-2 and 5-3 show that the CM leaches P. Initially, the column with CM and sand leached the highest amount of P, at a concentration of approximately 6.6 mg/L. The effluent concentrations immediately decreased within the first meter of water applied. Despite this initial washout, the P effluent concentrations increased again at around 20 m of water applied in columns containing CM. The increase was not expected since the material was fully saturated and under continuous flow. Initially, it appeared that the column containing soil in addition to CM, rather than with sand had an impact on

the overall P leached from the column; however when the total P leached from the columns was calculated, the two columns (sand with CM and soil with CM) contained comparable levels of P leached. Figure 5-4 shows the P leached for the three columns shown in Figures 5-2 and 5-3.

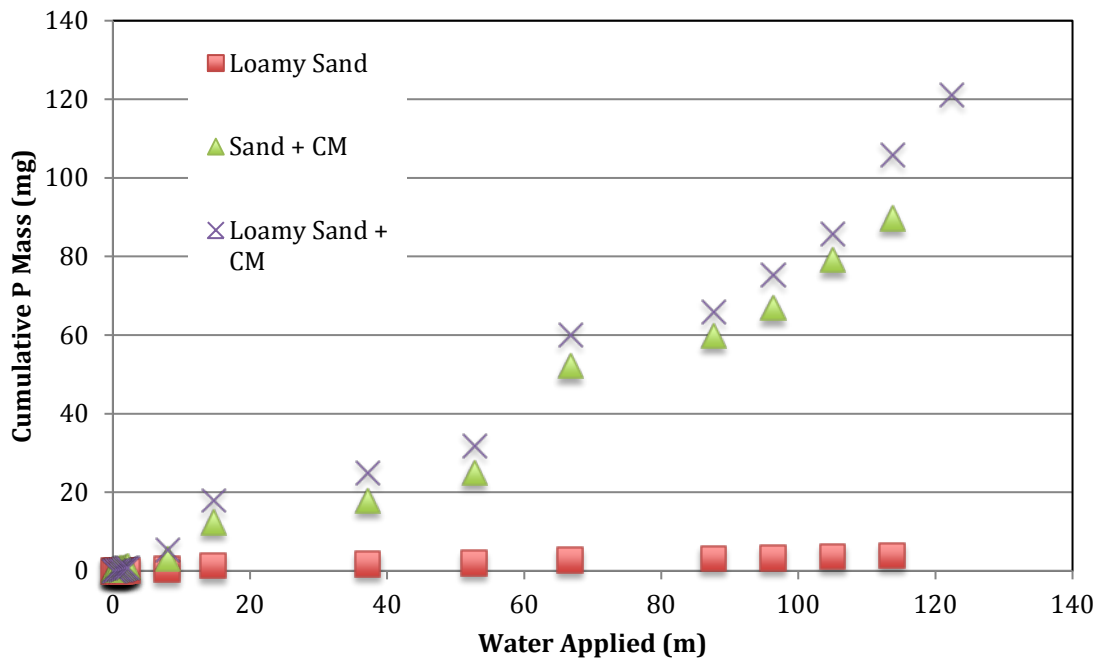


Figure 5- 4.Total P mass leached for three columns: 100% loamy sand, 15% CM and 85% sand, and 15% CM and 85% loamy sand.

The column with 100% loamy sand showed that without CM additions, P was leached at low levels. At around 115 m applied water, approximately 3.8 mg of P had leached from the column. The two columns containing CM had much higher leached P masses than the column with 100% soil. The column with CM and sand had around 90 mg and the column with soil had approximately 106 mg. It was surprising that the column with loamy sand and CM leached comparable (and even higher) levels of leached P to the column with sand. It was predicted that the soil would have some adsorption capacity, however it did not appear to be effective at mitigating P from the CM. Sandy

loam was also used in the columns with CM at the same percentages and resulted in comparable results to that of loamy sand. The data for the sandy loam and CM columns and 100% sandy loam column are in Appendix C.

5.2.2 Continuous Column Phosphorus Mitigation

After the column experiments with the soils and sands did not result in effective P leaching mitigation, WTR was incorporated into the project. From the ratios tested with nutrient extraction analysis, three ratios were chosen for continuous column study testing: 35%, 50%, and 75% WTR: CM. The 35% was predicted to be on the lower side of WTR amendment additions, 50% was thought to be more practical on a mass production basis, and 75% was predicted to be too high, but the most effective. Figures 5-5 and 5-6 show the effluent P concentrations from the continuous columns with and without the WTR amendments.

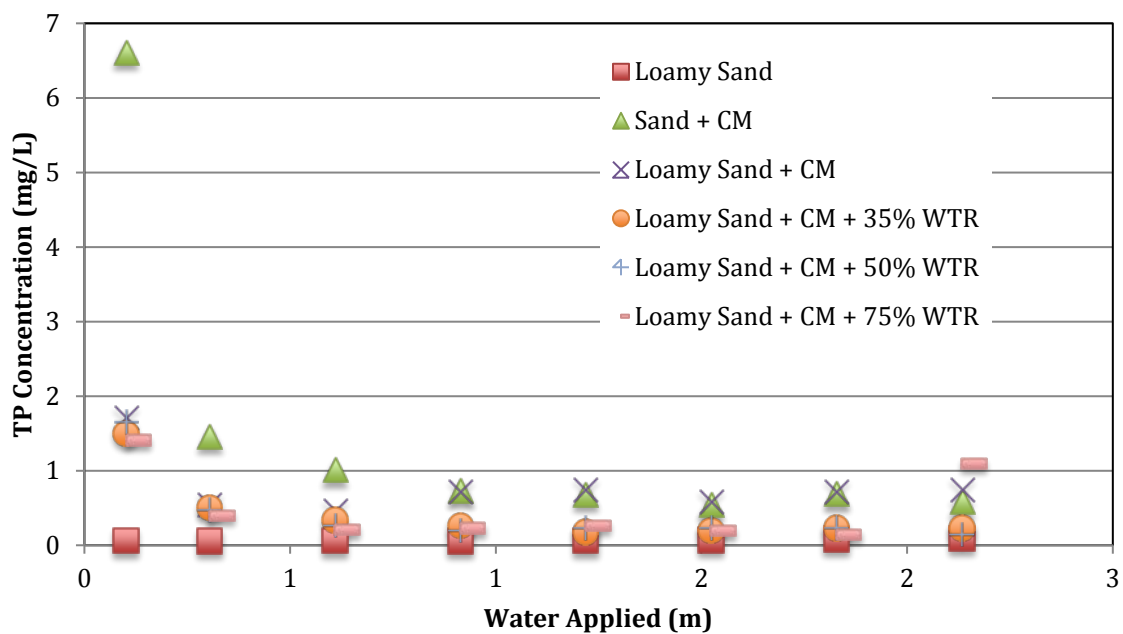


Figure 5- 5. The initial day of P effluent concentrations for columns containing 100% loamy sand, 15% CM with sand, loamy sand, and loamy sand with different ratios of WTR: CM (dry mass basis).

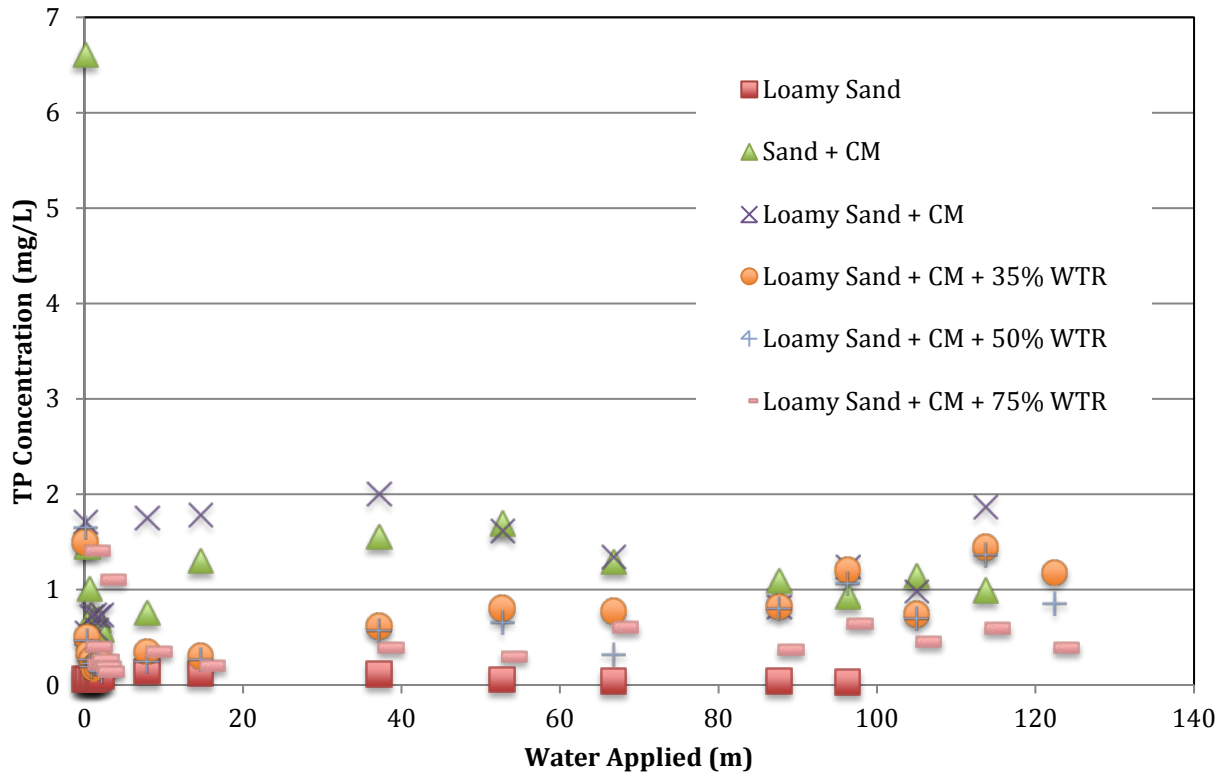


Figure 5- 6. The entire column duration of P effluent concentrations for columns containing 100% loamy sand, 15% CM with sand, loamy sand, and loamy sand with different ratios of WTR: CM (dry mass basis).

Figure 5-5 shows that the initial effluent from the columns containing the three WTR ratios, 35%, 50%, and 75% all were lower than both columns with CM (sand and loamy sand), however they were still higher than the column containing 100% loamy sand. Lower effluent concentrations than CM and soil/sand, but higher effluent than 100% loamy sand was to be expected since the PSI_{ox} values for each of these ratio mixtures was higher or around 0.5 (Figure 4-3).

Unlike the columns without WTR (with CM), P concentrations in the columns containing WTR did not increase after an initial decrease around 20 m of water applied. The WTR columns did however seem to gradually show an increase in P effluent concentrations again at approximately 35 m water applied. This was surprising since

studies with co-applications of CM and WTR have shown to have long term P mitigation (Ippolito et al. 2009). A prediction for this gradual increase is a possible decrease in WTR adsorption capacity. The CM itself may also have a holding capacity for P and release increasing amounts.

Columns with CM and WTR appeared to be lower in column TP effluent concentrations than the columns without WTR. Leached P (Figure 5-7) showed that the WTR amendments decreased the P effluent mass as more water was applied. For example, at approximately 67 meters of water applied the columns without WTR had leached around 50-60 mg of P, respectively, whereas the columns with WTR leached less than half of that at masses between 14-25 mg of P. At about 120 m of water applied the trend continued; columns without WTR had leached around 90-100 mg when the columns with WTR only leached 41-76 mg. There was a decrease in WTR efficiency overtime, however there is an evident decrease in P leached when WTR is incorporated.

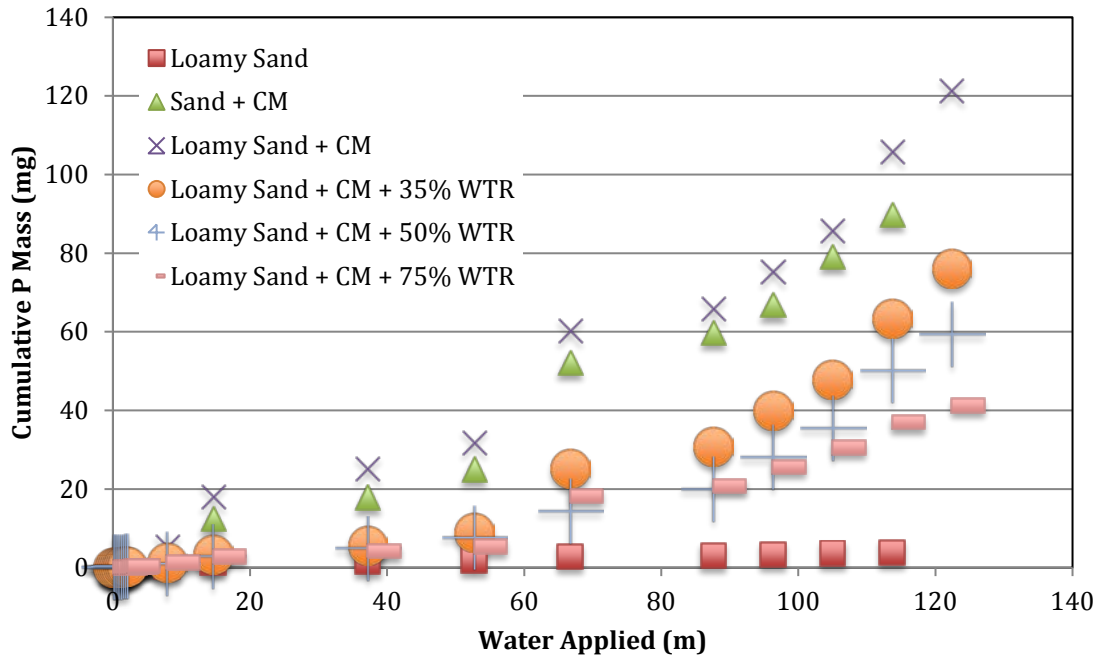


Figure 5- 7. Total column P leached for columns containing 100% loamy sand, 15% CM with sand, loamy sand, or loamy sand and different ratios of WTR: CM (dry mass basis).

Figure 5-7 shows the P leached (mg) over approximately 120 m of water applied.

All of the CM columns containing WTR leached a smaller mass of P than columns without WTR (not considering 100% soil columns). The columns containing 35% and 50% WTR resulted in some P mitigation, with 50% having more success than 35%. The column with 75% WTR was most effective at P mitigation.

The resulting level of effectiveness of the chosen WTR ratios agrees with the PSI_{ox} data. For example, the 35%, 50% and 75% WTR:CM ratios have PSI_{ox} values of 0.641 ± 0.045 , 0.457 ± 0.030 , and 0.399 ± 0.025 , respectively. A PSI_{ox} value below 0.5 has been concluded to decrease P leaching in comparison to PSI_{ox} values above 0.5 (Brown et al. 2015). The columns did however leach more P than was overall expected. The 75% WTR: CM ratio was expected to have longer term effects based on the PSI_{ox} , however it is not surprising that the effectiveness does not reflect extremely well to the

batch study. Batch and column studies have been shown to have varying levels of effectiveness with respect to nutrient adsorption. In a study with WTR, the batch studies had higher levels of adsorption capacity in comparison to the column studies (O'Neill and Davis 2012).

The leached P in the various columns was compared to the nutrient extractions that were completed on the CM and mixtures of CM and WTR. The compared data showed correlating trends that as the WTR increases, the P leached, and the P nutrient extractions decrease. Figure 5-8 shows a graph of the leached P at various levels of applied water against the nutrient extraction for the corresponding mixture type.

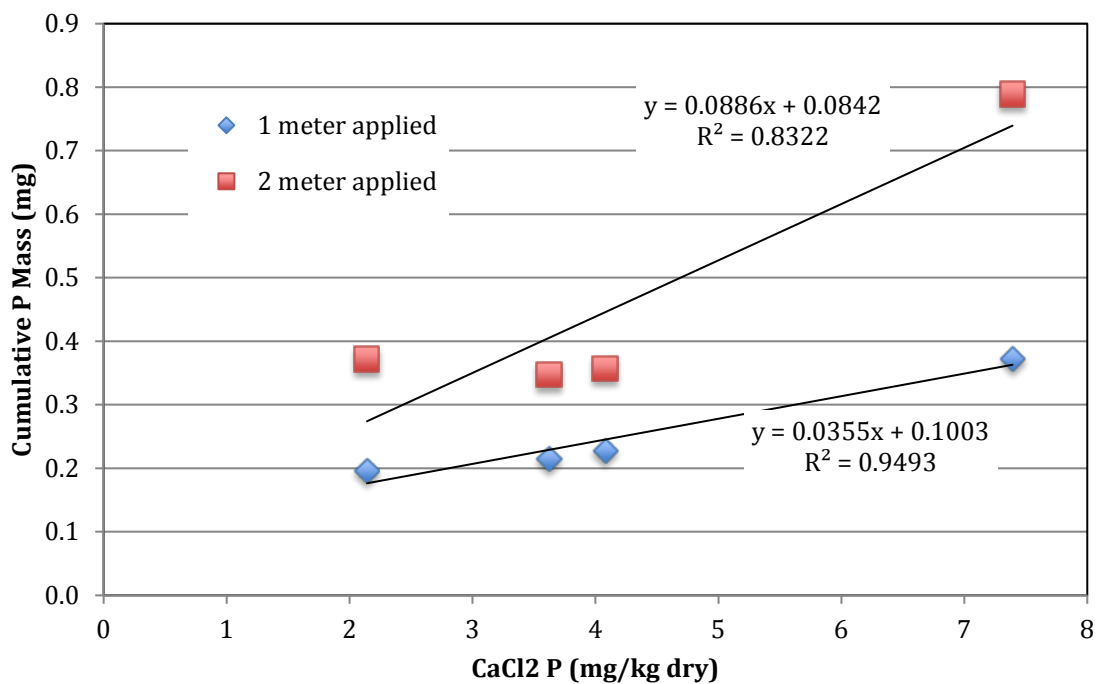


Figure 5- 8. The relationship between the CaCl₂ nutrient extractions for the CM and mixtures of CM and WTR. From left to right are 75% WTR:CM, 50% WTR:CM, 35% WTR:CM, and 100% CM.

As Figure 5-8 shows, the leached P from the columns increases with increasing CaCl₂ P nutrient extraction concentrations. This is not surprising since the CaCl₂ is used

as an estimate of the water extractable P in biosolids. When the nutrient extractions were completed prior to the column studies it was predicted that since the mixtures with the WTR had lower CaCl_2 levels that they would leach less P in column studies and therefore have a smaller amount of leached P overtime. Figure 8 shows this prediction to be true. As the water applied to the column increased over 2 meters, P leaching from CM without WTR increased much higher than the columns with the WTR, making the trend less linear.

Column studies with biochar and zeolite were completed with the intent of N, mainly ammonium mitigation. However they were analyzed for TP concentrations as well to see if the soil amendments had any effects on the P leaching. Figure 5-9 shows the leached P mass that was found from the continuous columns with the soil amendments.

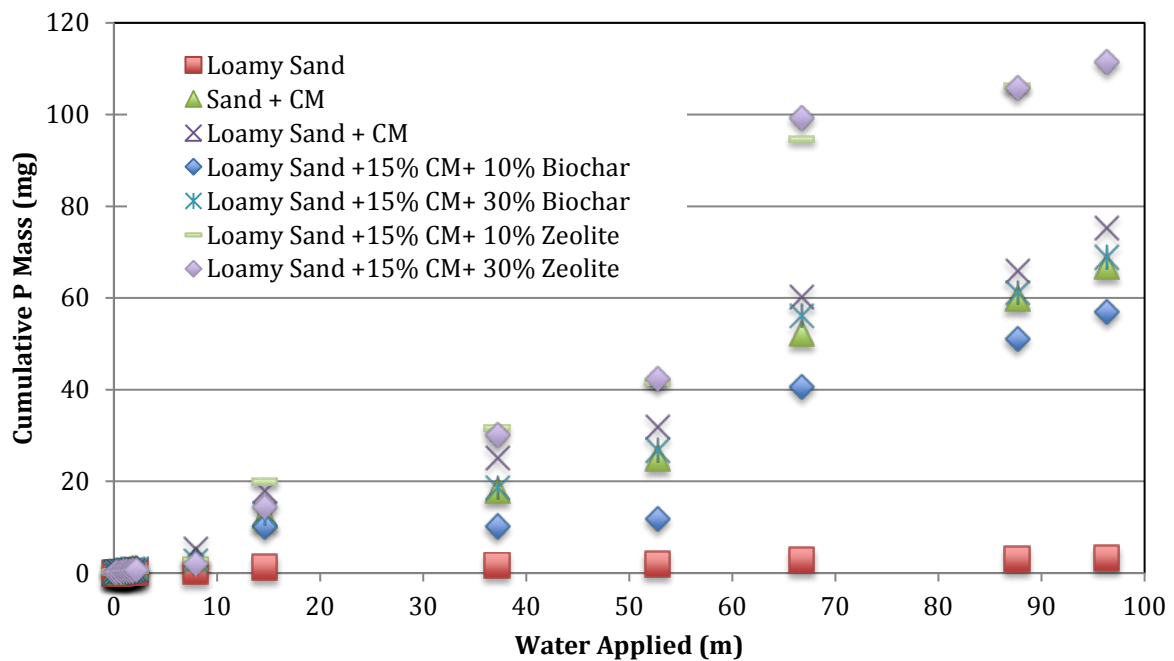


Figure 5- 9. The leached TP mass from the columns containing 15% CM and sand, or loamy sand soil with various ratios of zeolite and biochar.

Figure 5-9 demonstrates that the two soil amendments- zeolite and biochar – did not have noteworthy mitigation effects of TP on the CM leaching. If anything, the zeolite may have had an increasing TP effect on the amount leached. At approximately 67 meters of water applied the zeolite mixture columns had leached about 100 mg of P, whereas the columns without the zeolite (biochar included) leached approximately between 40-60 mg of P.

5.2.3 Continuous Column Phosphorus Duplications

Duplicated continuous studies were completed on the 15% CM with both sand and loamy sand soil. The TP results for the duplicated columns were very similar for both columns, however, the loamy sand duplicated column had a drastic increase in TP overtime compared to the initial column. Figures 5-10 and 5-11 show the duplicated studies TP data.

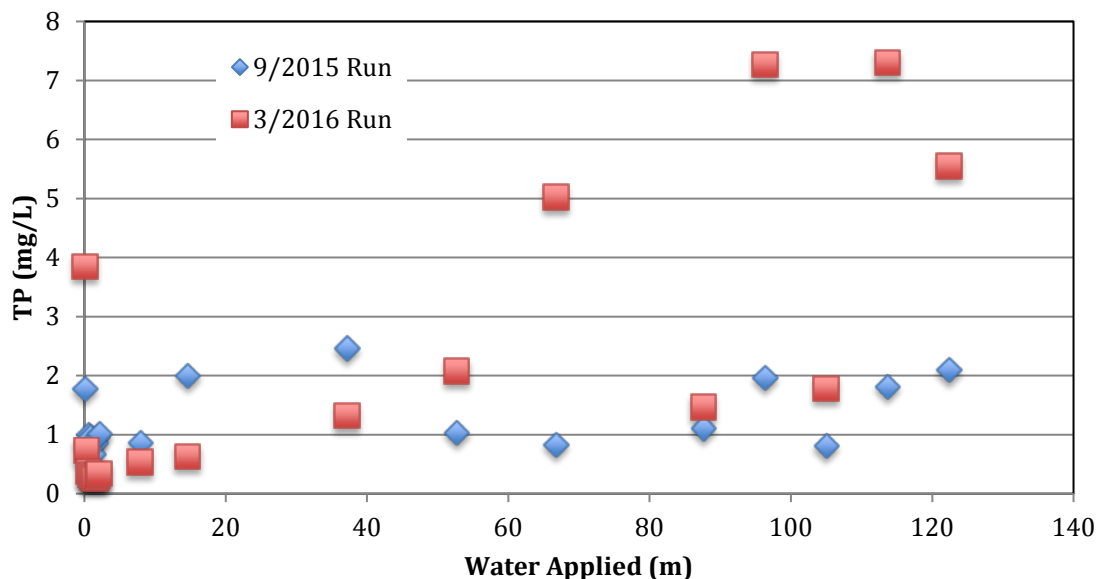


Figure 5- 10. The TP data for the duplicated continuous columns containing 15% CM with loamy sand soil.

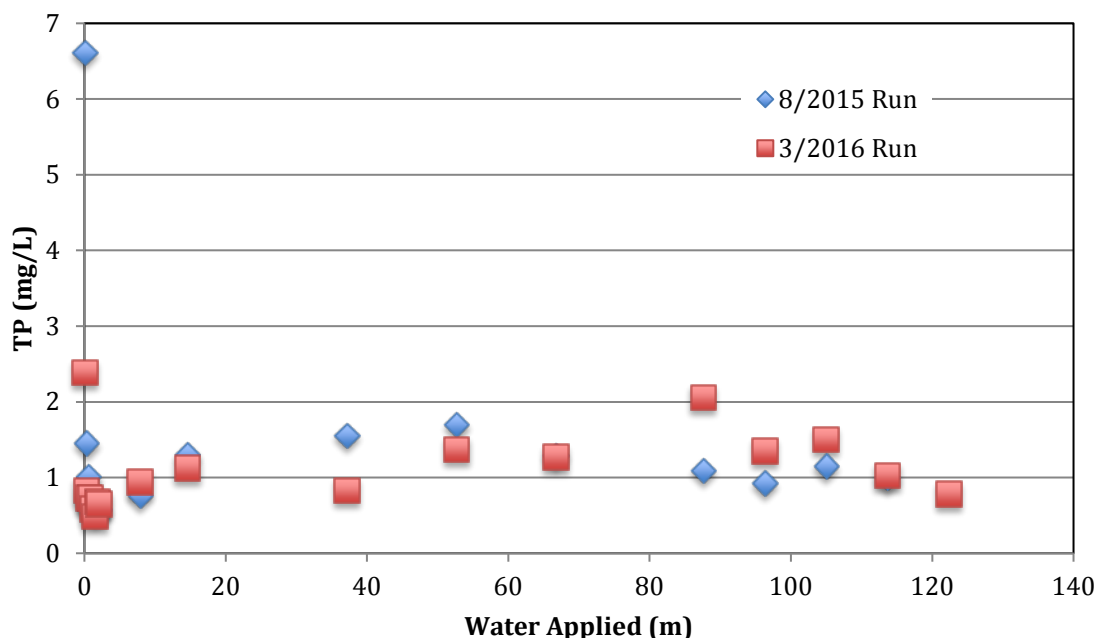


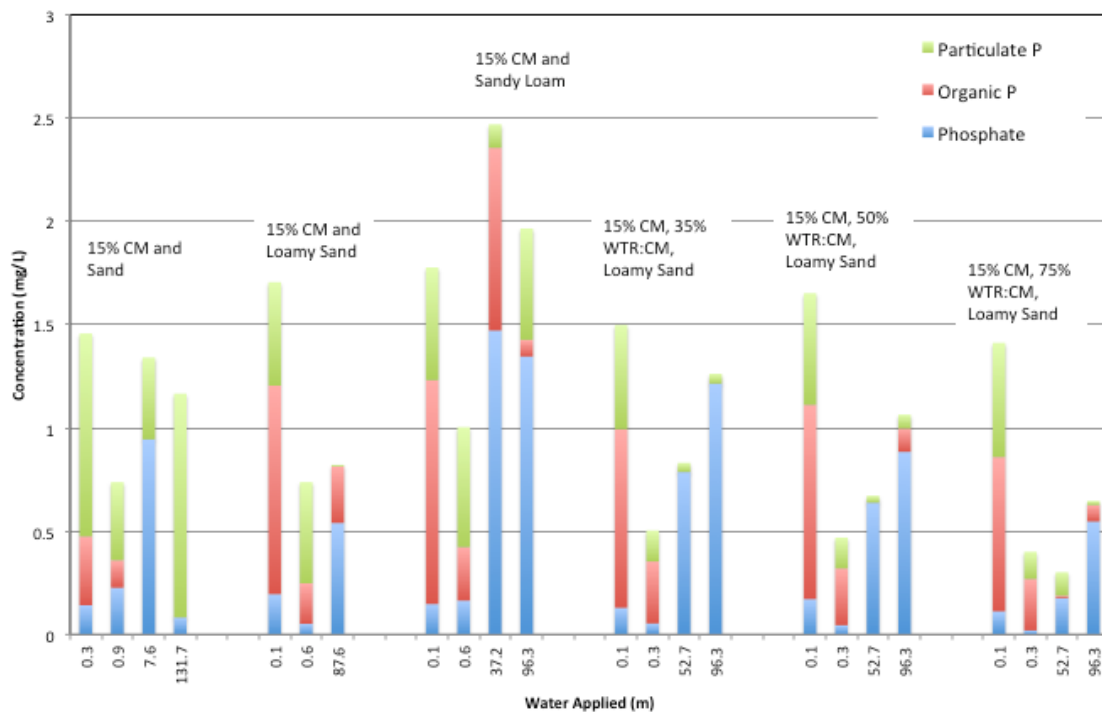
Figure 5- 11. The TP effluent concentration data for the duplicated continuous columns containing 15% CM and sand.

Figure 5-10 shows the evident P increase overtime in the later 15% CM and loamy sand soil column that the initial column did not exhibit. The initial effluent sample for the March 2016 column was also higher in TP than the September 2015 column. Predictions for why the TP increase occurred are variability in the CM. As seen in Chapter 3, the CM nutrient extraction levels vary throughout sampling dates. Based on the variability of the nutrient extraction data, it is not surprising that the March and September data are not identical. The general trends in each column are similar throughout the duplicate studies, however the duplicate columns with loamy sand and 15% CM have different cumulative P mass amounts. For example, at around 50 m water applied both the loamy sand/CM columns leached approximately 20-30 mg of P. The March 2016 duplicate column increased however at around 60 m water applied and by 120 m water applied the column had leached approximately 347 mg, whereas the August 2015 column had leached only 121 mg. The full cumulative mass data is shown in

Appendix C. The duplicate CM/sand columns both leached similar amounts of P- at 114 m water applied, approximately 90-96 mg P.

5.2.4 Continuous Column Phosphorus Speciation

The phosphorus speciation in the sample effluent was analyzed to better understand the forms of P in the effluent. Two samples from the first day of sampling were analyzed for complete P speciation in each column, along with one or two other effluent samples throughout the study. The general trend in P speciation was leaching of organic P in the first meter followed by mostly phosphate and particulate P. Figure 5-12 shows the P speciation breakdown for the continuous column studies.



From Figure 5-12 it becomes apparent that although the media mixes do not have the same speciation, there is a general trend. Initially, the first meter of the column effluent contains mostly organic P (in red), and particulate P (in green). The initial leaching of organic P has been observed in other studies with P applications. Idowu et al. (2008) stated that within the first 2-4 pore volumes of water applied to an agricultural land with biosolids and other organic amendments, organic P was the initial dominant form of P in the leachate. The dominant form of P leachate throughout the entire 20 pore volumes however was inorganic P.

Similar to the study with land-applied biosolids, the columns did not typically leach organic P over the detection limit (0.01 mg/L) after the first meter, but they did leach inorganic P. The overall trend of TP in the effluent is also shown in Figure 5-12 it is clear that there is an initial decrease in TP but gradual increase in TP in the effluent overtime.

It is important to consider the column environmental characteristics as well when analyzing the P speciation. The columns are completely saturated throughout the duration of the study, apart from the initial wetting period. The P speciations of the initial samples are more comparable to land applied biosolids runoff P speciation results since they are newly saturated. Elliott et al. (2005) completed a study on the P speciation from land applied biosolids runoff and found that the total dissolved P (TDP) was the highest percentage of P in the runoff. The TDP is phosphate and organic P together. These results are comparable to what was found in the first day of samples (first meter applied) for this project for the columns containing both soil and CM. The samples taken further in the

studies are not as comparable and should not be compared since they have been fully saturated for longer periods of time.

5.2.5 Non-Continuous Column Phosphorus Leaching

The phosphorus concentrations in the leachate from the non-continuous columns are shown in Figure 5-13. Three samples were taken for each wet period, two within the first two hours, and one in the last hour.

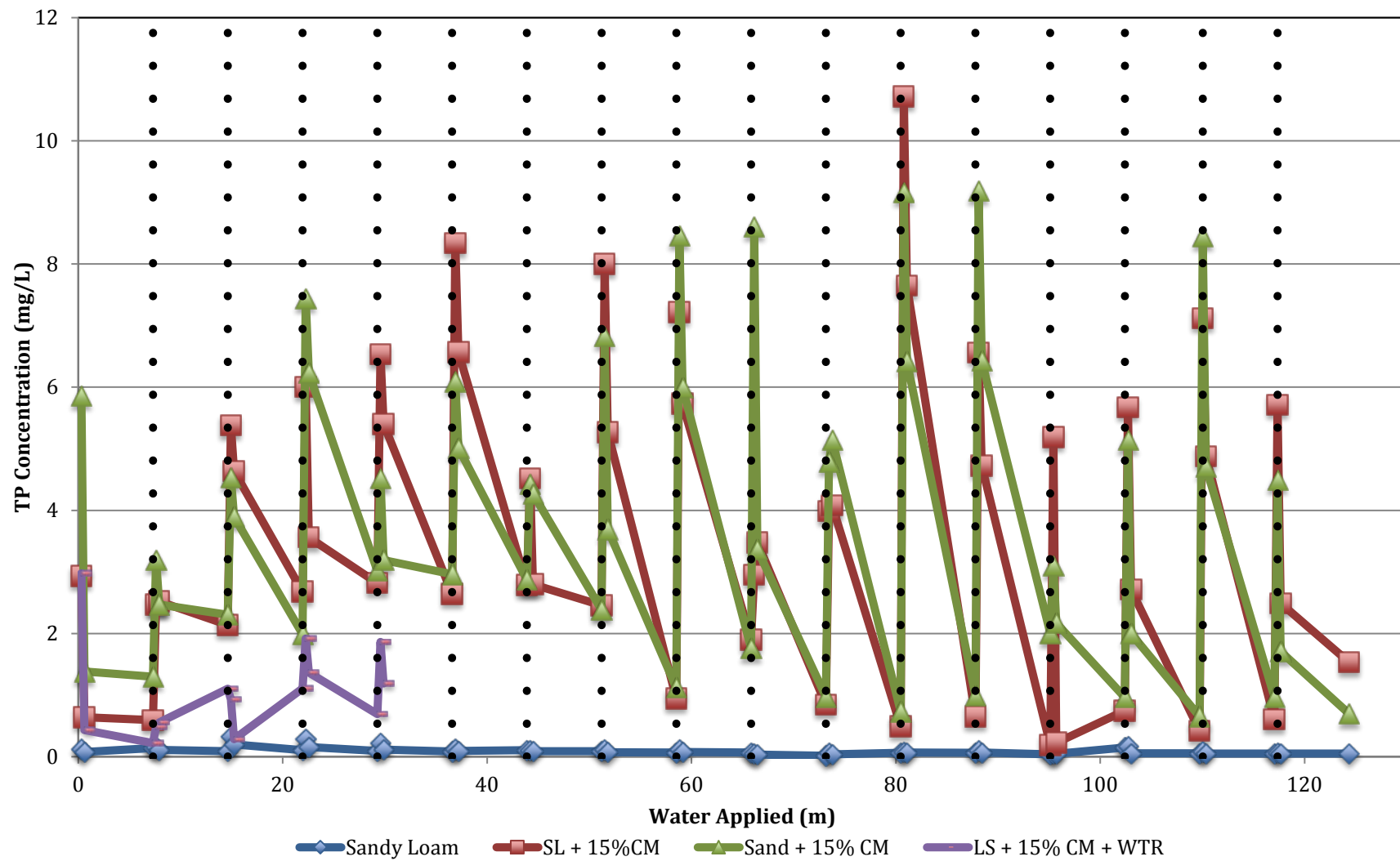


Figure 5- 13. The non-continuous column effluent TP concentrations. Each of the black dotted lines separates the wet periods.

The results of the TP concentrations in the column effluents overtime were different than predicted. The first two initial high TP concentrations followed by an initial drop within the first wet period was expected, as they should match previous continuous columns. However, as the wet periods progressed, the initial TP in the effluent samples began to rise. Many of the samples for the columns with 15% CM and sand or soil reached TP concentrations over 8 mg/L, which had not been seen in previous columns. This is a high value for phosphorus and was not expected since the initial effluent TP values were below 6 mg/L.

The overall trends of the columns within wet periods were expected to be higher then decrease with increasing flow within the wet period. Although increases in effluent TP concentration were expected and have been seen in other studies Venterink et al. (2002), a general decrease over time was expected but did not occur over the 120 meters of water applied column period.

In addition, although the WTR column did not successfully run throughout the entire non-continuous column study, the column TP effluent concentrations did appear to be much lower than the two other columns containing CM. It is predicted that if it had run longer it would have had continued success and effectively mitigated TP. The accumulated P data for the columns in the first 30 meters of water applied are shown in Figure 5-14. The data support the hypothesis that the WTR would have had a longer-term effect on the P mitigation if the column had been successful.

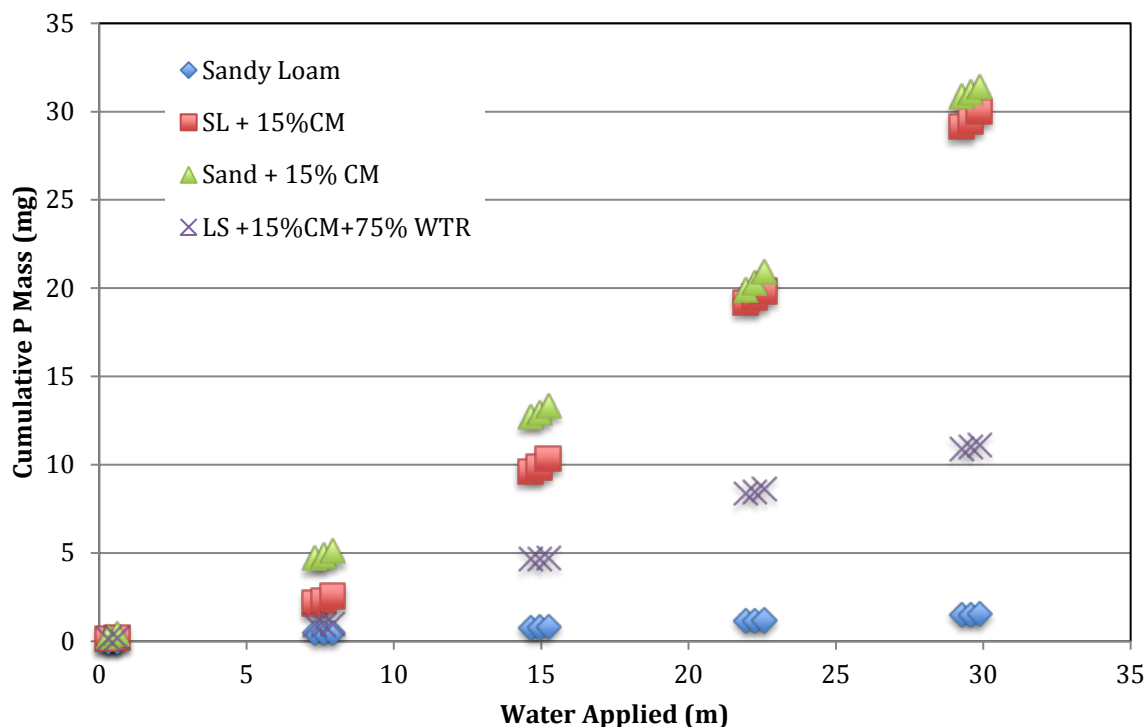


Figure 5- 14. The leached P data for the non-continuous column. The data only goes to 30 m of water applied since the WTR column cap broke after this point.

As shown in Figure 5-14, the column containing the WTR is lower than the columns with CM (without WTR) at 30 m of water applied. The columns without the WTR leached approximately 30 mg of P at 30 m applied water, whereas with the WTR the leached P was approximately 11 mg. A linear fit was calculated for the column with the WTR to predict how the total column cumulative P mass would look in relation to the other three columns throughout the column study. The real data for the three successful columns is shown in Figure 5-15 in addition to the data of the “predicted WTR column”.

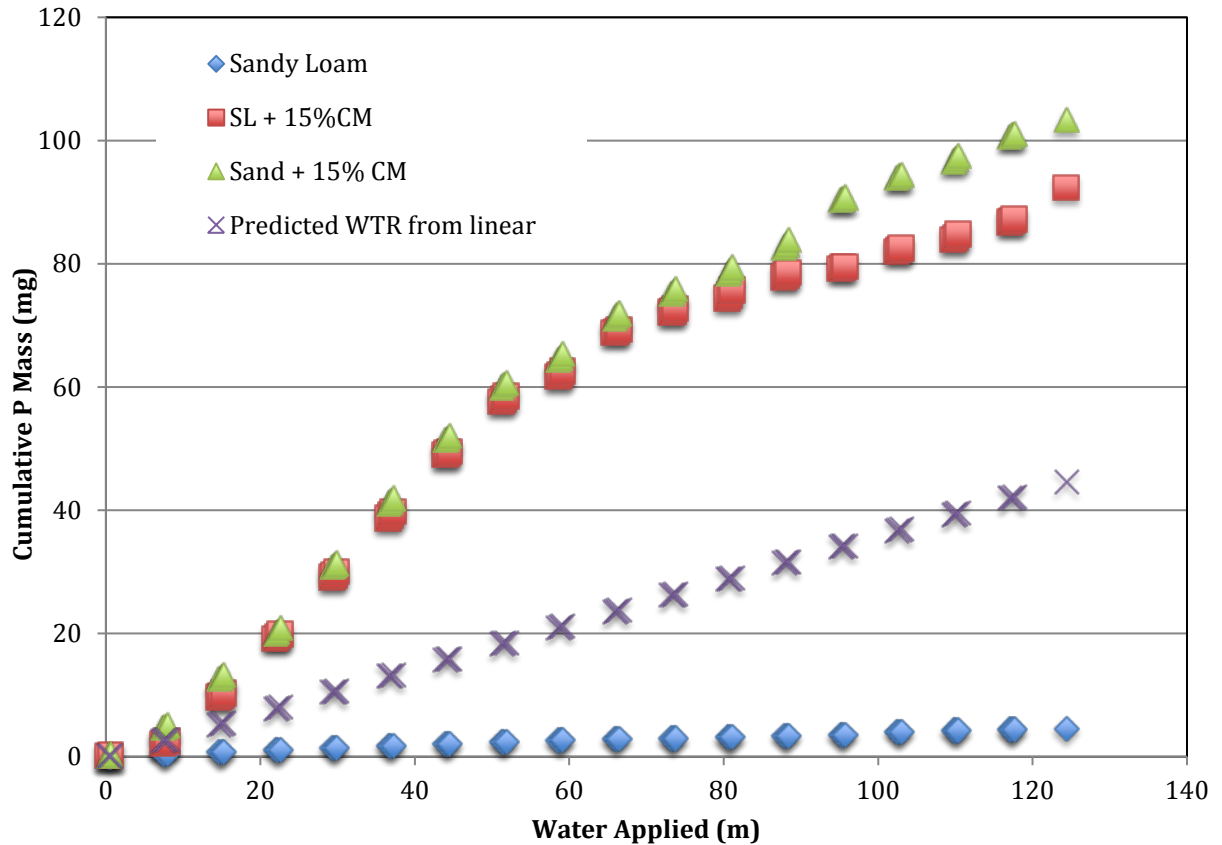


Figure 5- 15. The column P leached for the columns with 100% sandy loam, 15% CM and sandy loam, 15% CM and sand, and the predicted accumulation for the 15% CM, 75% WTR with loamy sand soil.

The cumulative P leached from columns without the WTR (apart from the sandy loam column) appeared to increase much faster than the column with the WTR. Although the column containing WTR is only the proposed P mass, the predicted P leached mass was still about half of what it is with the other columns containing the CM. It is also noteworthy that the non-continuous columns reached a comparable level of leached P mass as the continuous columns. It is likely that when the columns were stopped the phosphorus that would have leached out with the continuous columns built up in the column. When the columns were turned back on, the accumulated phosphorus leached

out at higher initial concentrations, making the continuous and non-continuous columns similar in cumulative P leached.

5.2.6 Non-Continuous Column Phosphorus Speciation

The phosphorus speciation was completed on four samples throughout the non-continuous column study. The non-continuous speciation trends differed from the continuous column study. The non-continuous speciation trends differed from the continuous column speciation trends. The non-continuous speciation is shown in Figure 5-16.

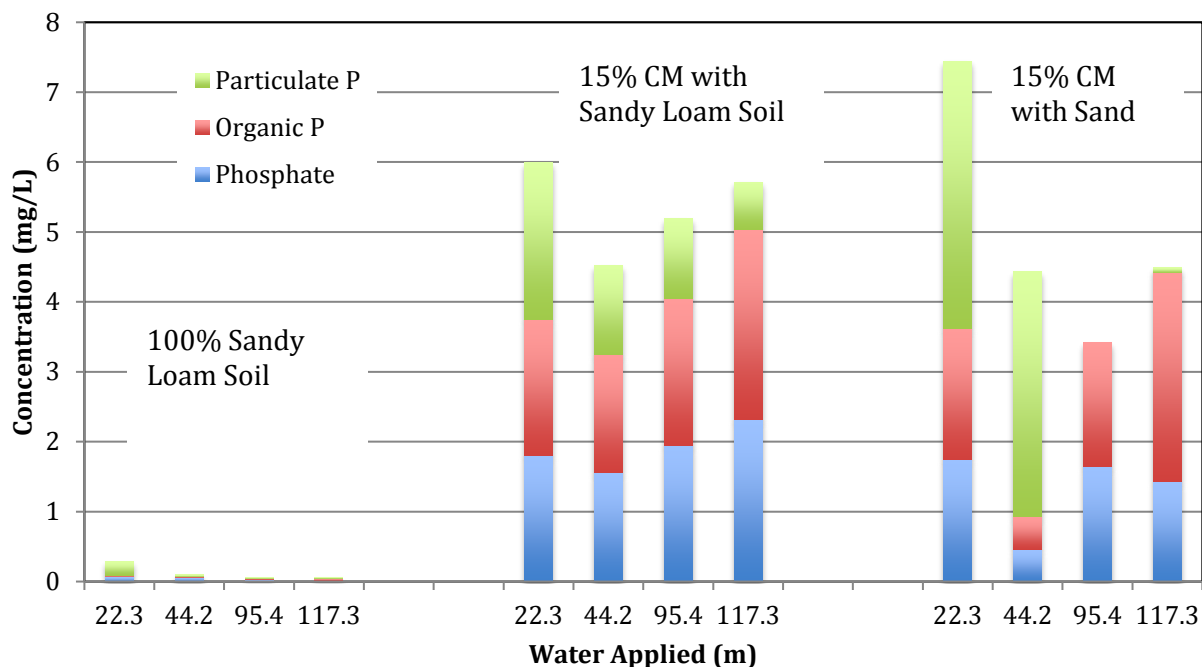


Figure 5- 16. The P speciation for the non-continuous columns. The data is for the soil column and 15% CM with sandy loam and sand columns.

The non-continuous P speciation consisted of organic P leaching throughout the entire study. This P leaching differs from the continuous study because organic P is leached from the columns consistently overtime. In the continuous column study there was a washout of organic P earlier in the study followed by inorganic P leaching. There

are P speciation differences between the two types of columns which can be compared to the study on runoff by Elliott et al. (2005). For example, the non-continuous column with CM and soil produces the highest amount of TDP (phosphate and organic P) throughout the samples taken. These samples were the initial effluent after a draining period. These P species are relatable to land applied runoff more so than the continuous column effluents since they are not continuously fully saturated. This may be a reason for the differences in P speciation between the two types of columns.

The leaching of organic P with the non-continuous columns occurred when the columns were re-wetted. Soils and wetlands are known to have an increase in leachable P in general when they are re-wetted (Venterink et al. 2002). The CM had what was believed to be an initial washout of organic P with the continuous columns, however organic P leached throughout the study. The non-continuous columns show the increase in P leaching upon re-wetting, however they also show that despite the observed decrease in organic P with continuous flow, the organic P may still continuously leach overtime from the CM and be a major species of the total P leachate.

5.3 Nitrogen

5.3.1 Continuous Column Nitrogen Leaching

The nitrogen concentrations in the leachate from the continuous columns were expected to be high due to the high amount of CaCl_2 and KCl extractable TN in the CM. Figures 5-17 and 5-18 show the TN concentrations in three of the continuous column effluents.

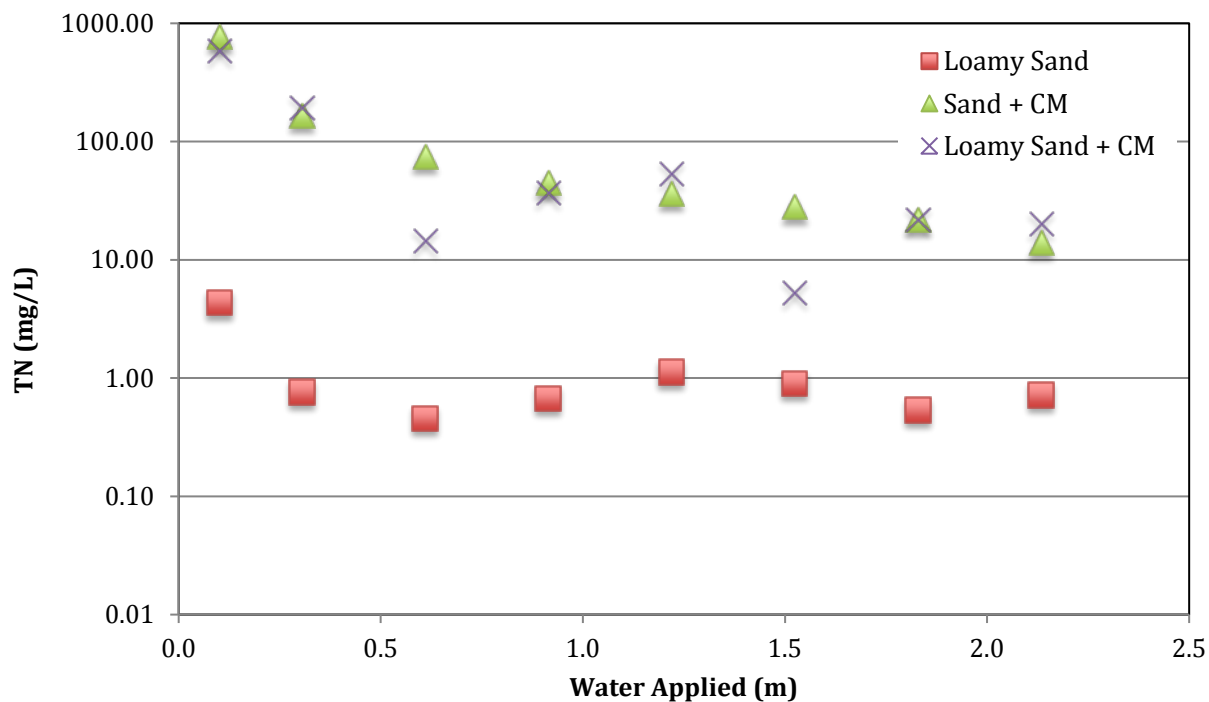


Figure 5- 17. The initial day of column effluent TN concentrations. The y-axis shows the TN concentrations in log scale for a better view of each column effluent concentration.

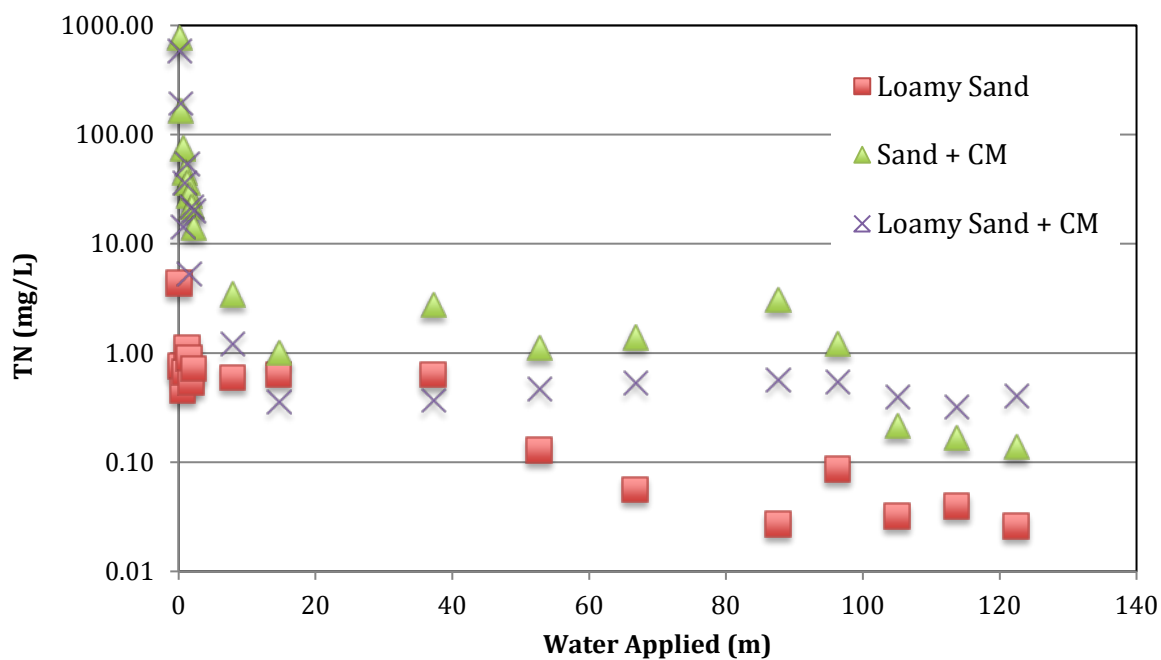


Figure 5- 18. The entire column duration of TN values. The y-axis is in log scale to better view the distribution of TN concentrations.

The TN effluent concentrations from the 15% CM (dry mass) with sand, 15% CM with loamy sand soil and 100% loamy sand columns (Figures 5-17 and 5-18) show the high concentrations in the column effluent and quick decrease of leached TN. It is evident that the columns with CM have a much higher TN in the effluent than the control soil column. For example, the initial effluent TN concentration for the soil control column was approximately 5 mg/L whereas the column with CM and sand initially had approximately 772 mg/L TN leach from the column. When the CM was mixed with soil it leached about 588 mg/L TN. The concentrations in the effluents from columns containing CM were both high, however the soil seemed to have more of a mitigating capacity for the TN leaching than it did for the TP leaching. The soil may have a natural higher adsorption capacity for some of the N leaching rather than P leaching.

5.3.2 Continuous Column Nitrogen Mitigation

Despite the small mitigation effect that the soil seemed to have on the initial TN effluent levels, further amendments were investigated for TN mitigation. Two different percentages (10% and 30%) of both zeolite and biochar were used in continuous column studies to analyze the possible ammonium adsorption and further TN mitigation. These percentages were based on the nutrient data found from the extraction ratios. Figure 5-19 shows the TN from the columns containing these amendments.

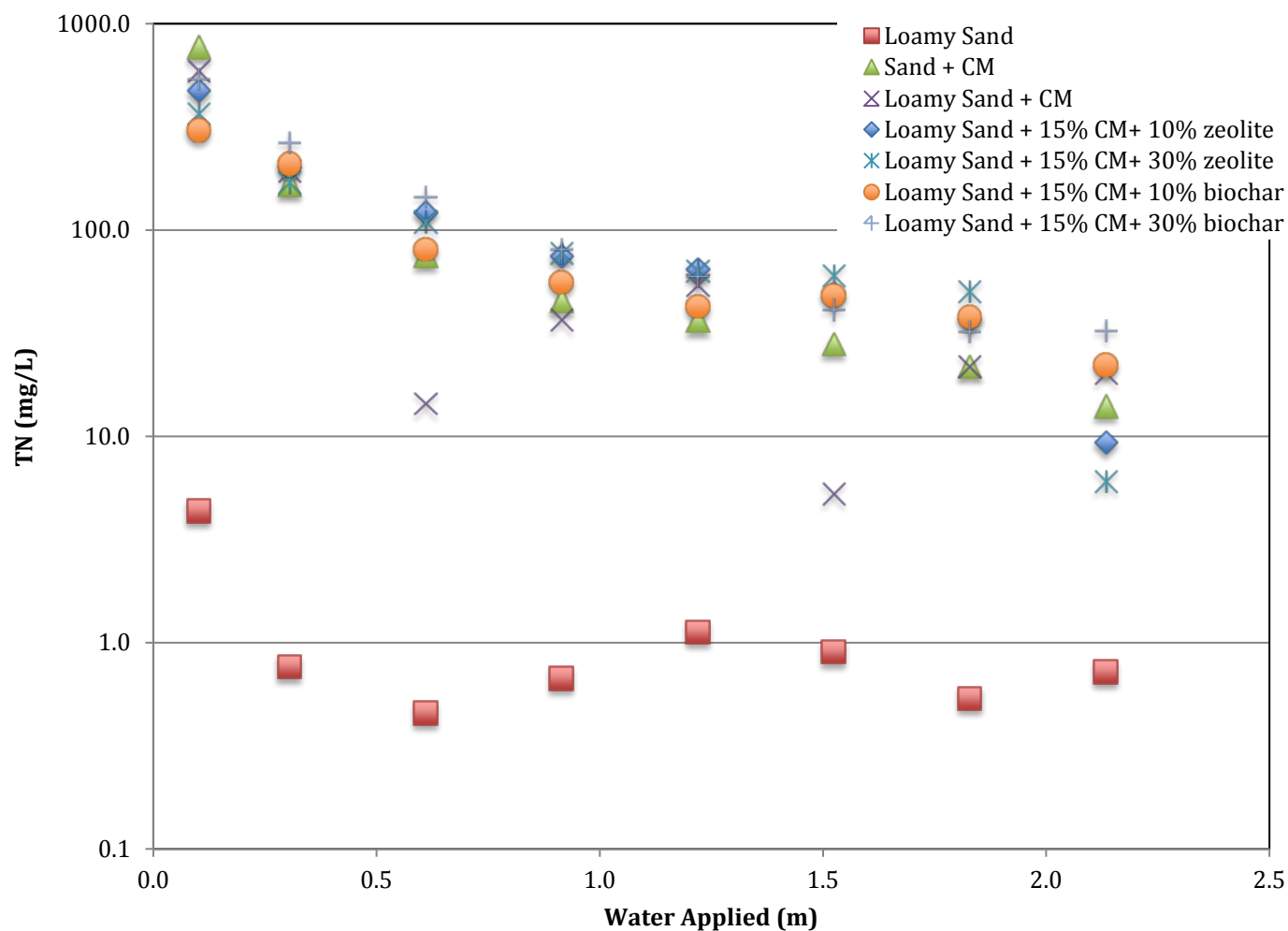


Figure 5- 19. The initial day of column effluent TN concentrations for multiple columns with 15% CM and various soil/amendment mixtures. The y-axis shows the TN concentrations in log scale for a better view of each column effluent concentration.

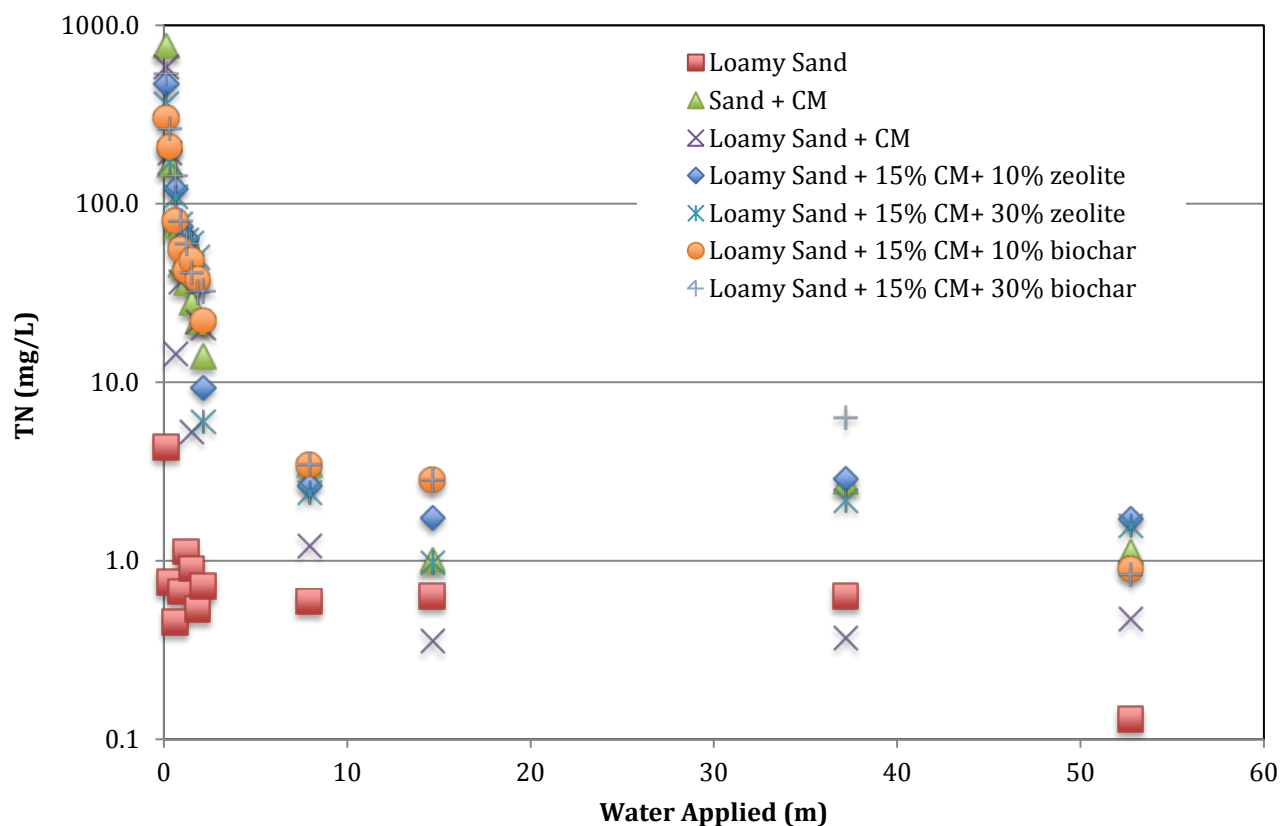


Figure 5- 20. The duration of TN effluent concentrations from columns with 15% and different soil/ amendment mixtures. The columns with zeolite did not run for the entire three weeks due to columns breaking, so the column duration for all of the comparison columns for TN mitigation only go to approximately 52 m applied water.

Figures 5-19 and 5-20 show the decrease in TN effluent concentrations from each of the columns throughout the duration of the continuous flow. The columns with biochar and zeolite both begin with lower concentrations of TN in their initial effluent samples than columns with CM/ sand or CM/ loamy soil (without soil amendments). For example, the initial TN leached concentrations for the zeolite mixtures (10% and 30%) were 470 and 365 mg/L, respectively and the biochar mixtures (10% and 30%) were 300 and 540 mg/L, respectively. All of the columns follow the same trend of immediate decrease followed by a gradual decrease below 1 mg/L TN after the first 2 meters of water applied.

To gage the effectiveness of the soil amendments at TN mitigation, the leached TN was calculated as well. Figures 5-21 and 5-22 show the leached TN from each of the columns.

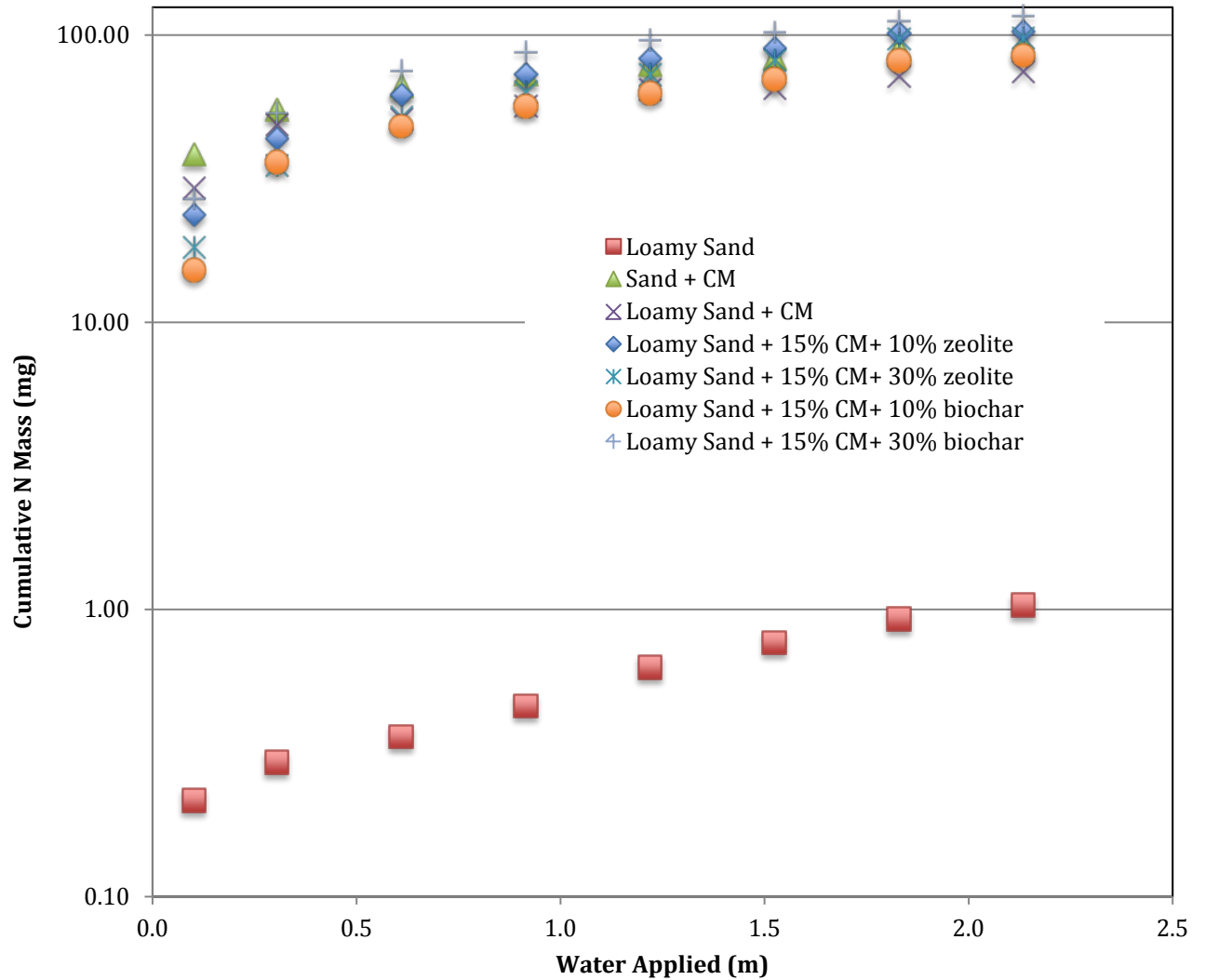


Figure 5- 21. The leached TN (mg) from the columns with 15% CM and soils and soil/amendment mixtures. The 100% loamy sand column is also plotted as a control. The first 2.5 meters of water applied are plotted in this figure to show the initial TN leached.

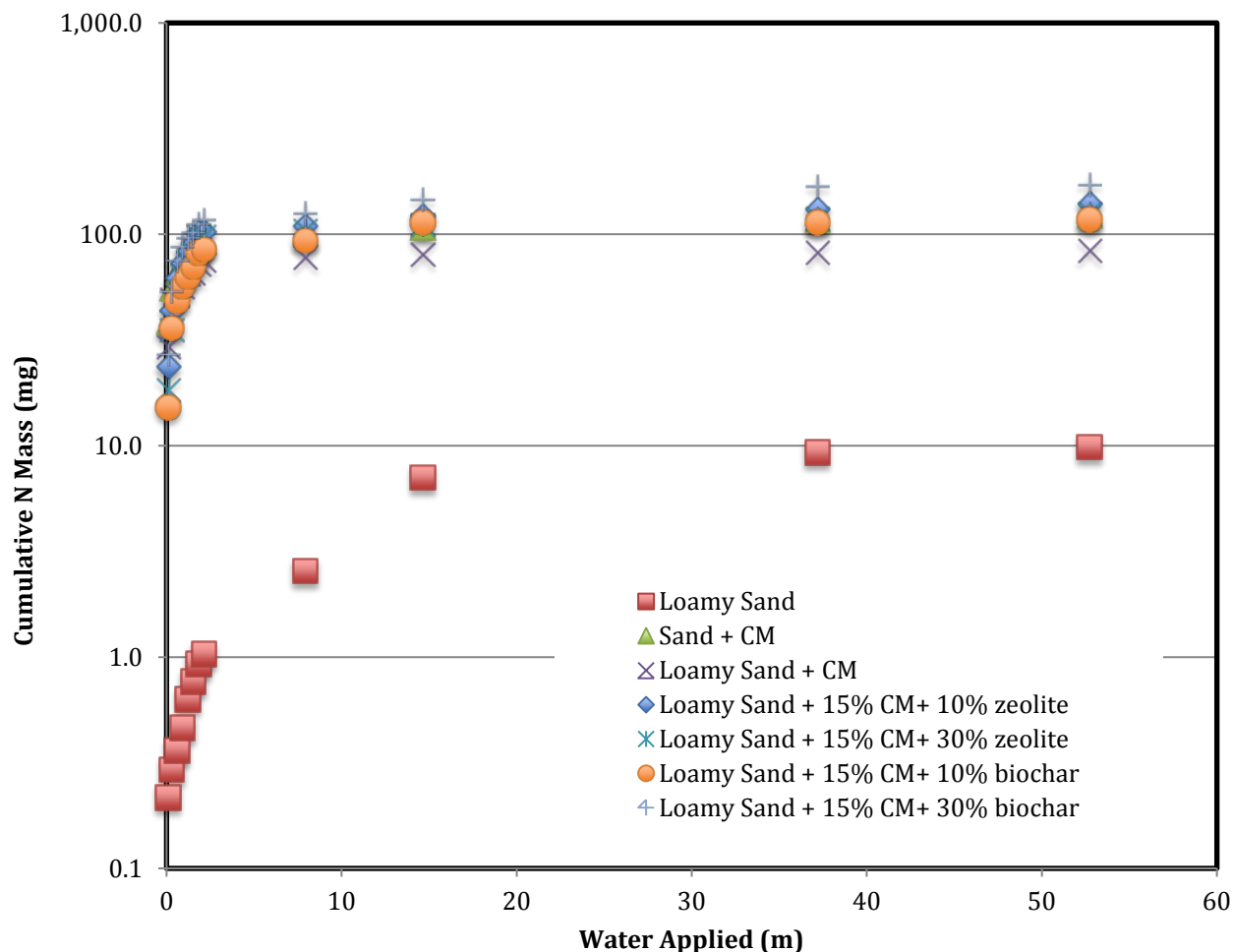


Figure 5- 22. The total cumulative N mass from the columns at approximately 53 meters of water applied.

Despite the initial high TN concentration in the first effluent sample from the columns containing the soil amendments being lower than those without the soil amendments, the leached TN plot trends shows the amendments to be ineffective at decreasing the TN in the leachate from the columns. Figure 5-21 shows the leached TN amounts to be lower for columns containing the soil amendments; however Figure 5-22 shows the leached TN masses with as more water is applied. It is possible that the soil amendments can only mitigate so much N in the initial washout stages of the column study and then leach more N as more water is applied. Knowles et al. (2011) completed a

study on mixes of biosolids and biochar and found them to be successful at nitrate sequestration in the initial 20 L of cumulative drainage. It could be possible that longer term N mitigation is harder to obtain with the ratios chosen for this study.

Although WTR was used for P mitigation, it was also tested for nitrogen leaching concentrations and cumulative leached N. Figure 5-23 shows the cumulative nitrogen leached from the continuous columns with and without WTR mixes.

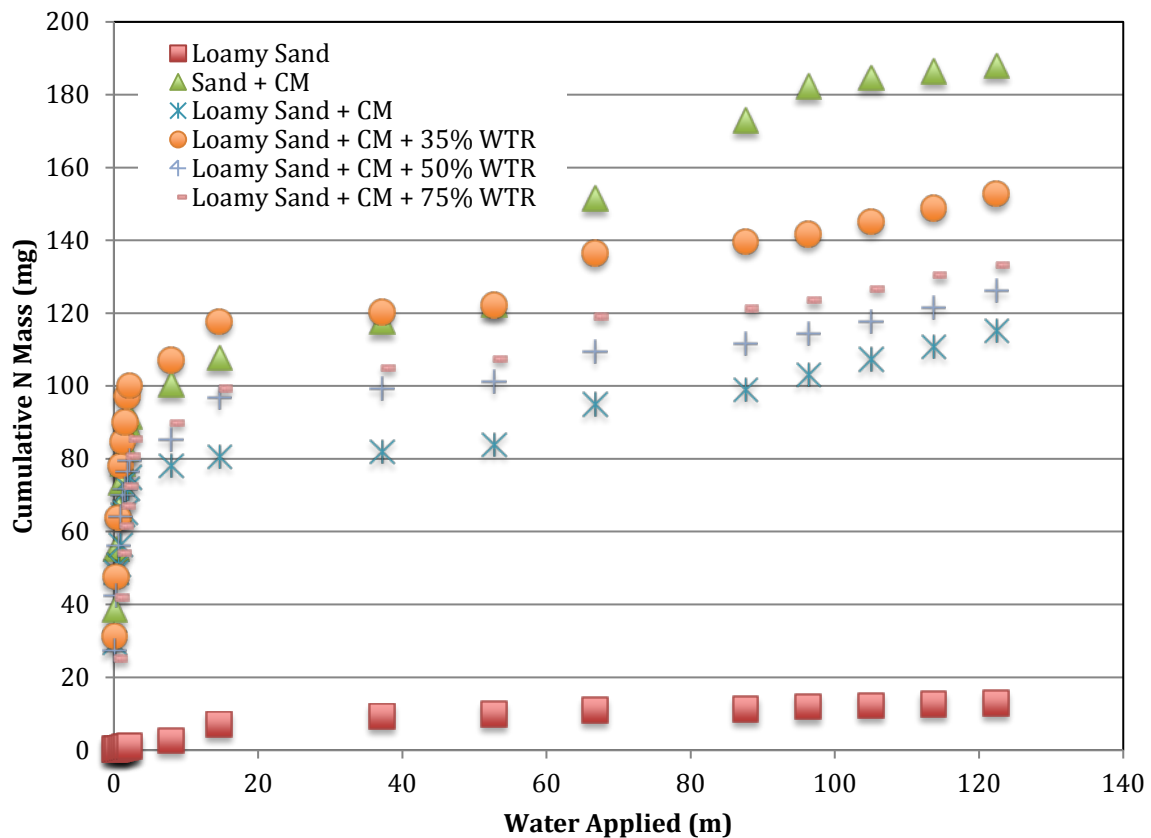


Figure 5- 23. The cumulative N mass from the continuous columns both with and without WTR.

The leached N data for the three columns with WTR- 35%, 50%, and 75% WTR:CM all lie within the data for the columns with CM/sand and CM/soil. From the leached N data, it does not appear that WTR had any significant effect on the nitrogen

leaching. This was not surprising after the KCl extractions with the various ratios of WTR:CM did not differ from the CM extraction alone.

5.3.3 Continuous Column Nitrogen Duplications

The duplicated continuous studies completed on the 15% CM with both sand and loamy sand soil were also compared for TN data. Figures 5-24 and 5-25 show the TN data for the duplicated columns.

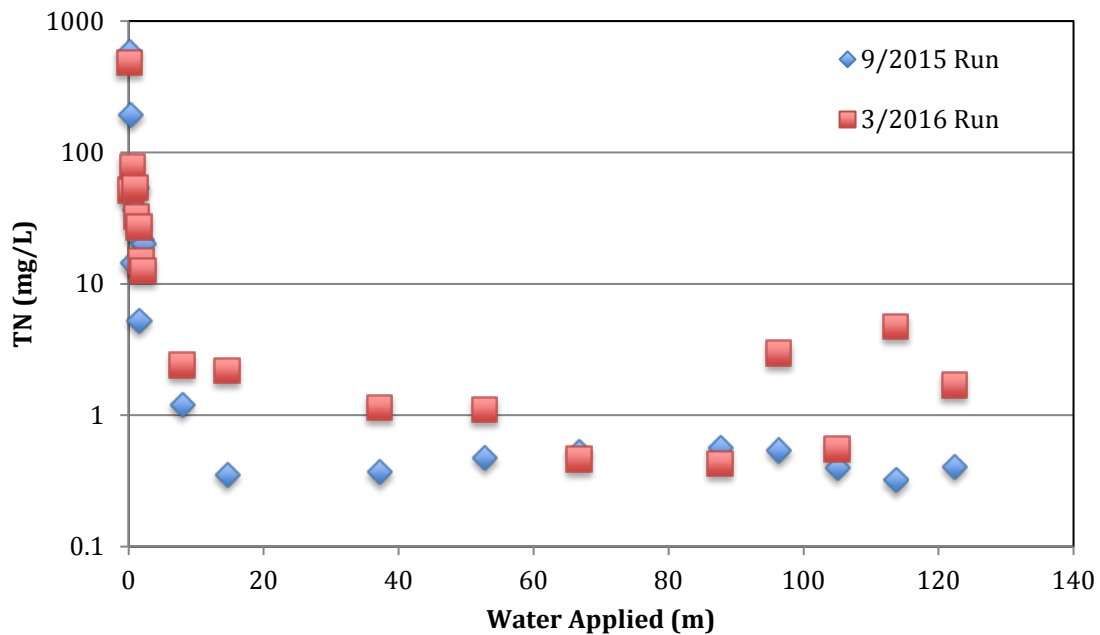


Figure 5- 24. The TN data for the duplicated 15% CM and loamy sand soil columns.

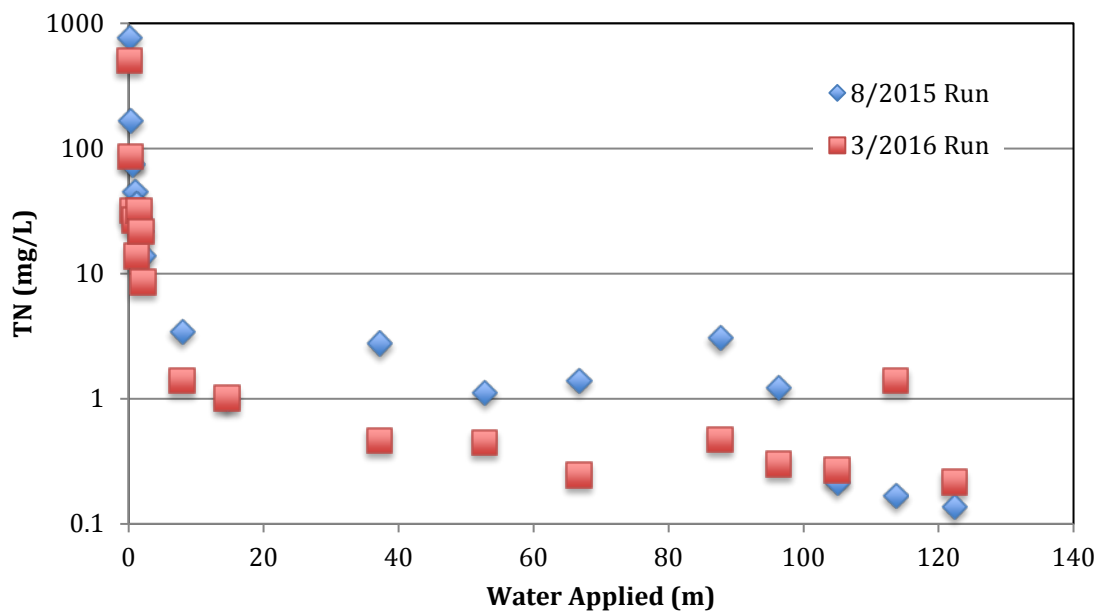


Figure 5- 25. The TN data for the duplicated 15% CM and sand columns.

The TN concentrations had the same trends for the columns completed in both September and March. They both had initial high effluent concentrations above 500 mg/L, respectively followed by the washout and gradual decrease throughout the column study.

5.3.4 Continuous Column Nitrogen Speciation

Total nitrogen speciation was analyzed for 3-4 of the samples from each of the columns tested in the study. Figure 5-26 shows the speciation breakdown for seven continuous columns completed with 15% CM (dry mass basis) and different mixtures.

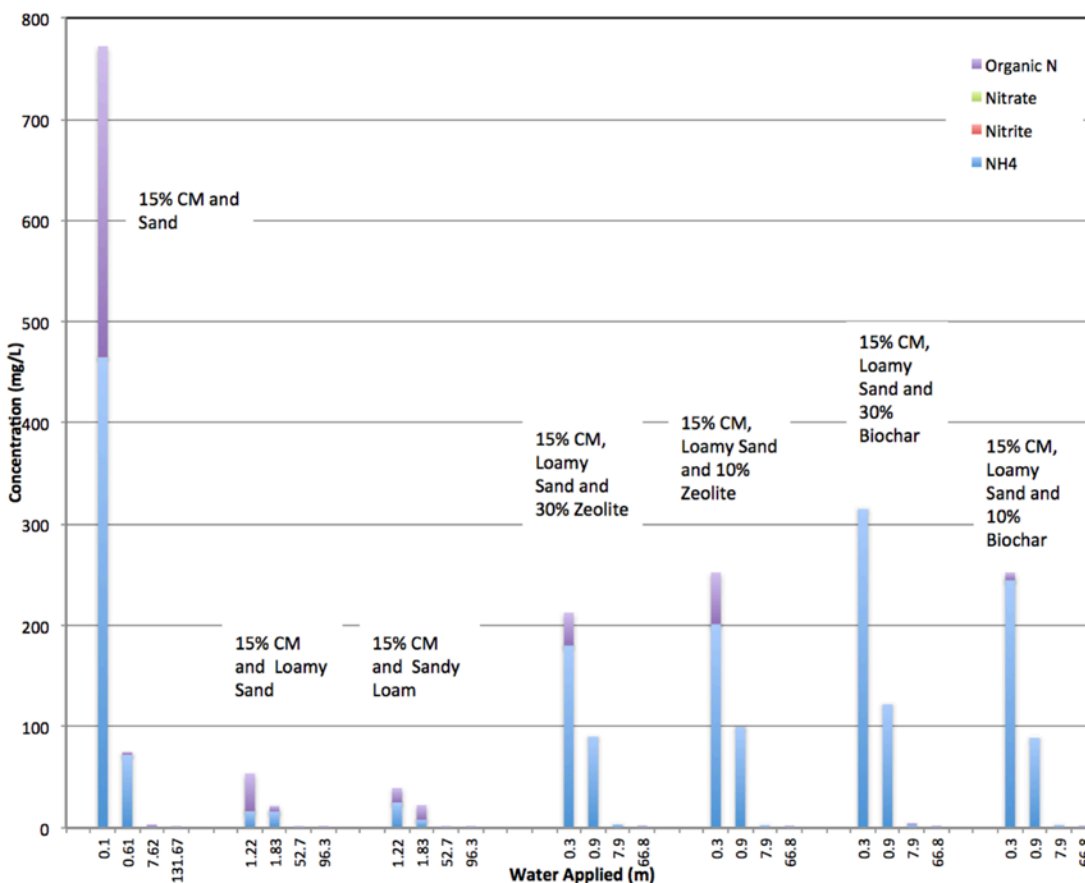


Figure 5- 26. The nitrogen speciation for 7 of the columns containing 15% CM (dry mass basis). The y-axis is in log scale.

From Figure 5-26 it is apparent that ammonium and organic N are the dominant forms of N in the column effluents. Most of the samples from the initial day contain the highest amount of N as ammonium, followed by later samples containing organic N. Nitrate and nitrite were below detection limit (0.05 and 0.01 mg/L) for all samples analyzed. The high amount of ammonium was to be expected with biosolids and has been seen in other studies as well. Peckenham et al. (2008) reported that biosolids leachate often contains up to 90% nitrogen as ammonium and has negligible nitrate and nitrite. The levels of ammonium in the columns with the zeolite and biochar ratios were surprising. It was predicted that both biochar and zeolite would both be effective at

mitigating ammonium and overall TN concentrations; however when comparing the values to the columns without the amendments they do not appear to be effective.

5.3.5 Non-Continuous Column Nitrogen Leaching

The nitrogen leachate from the CM in non-continuous columns is shown in Figure 5-27. It was predicted that the columns would have a large initial leachate concentration within the first wet period followed by an overall decrease in effluent TN concentrations.

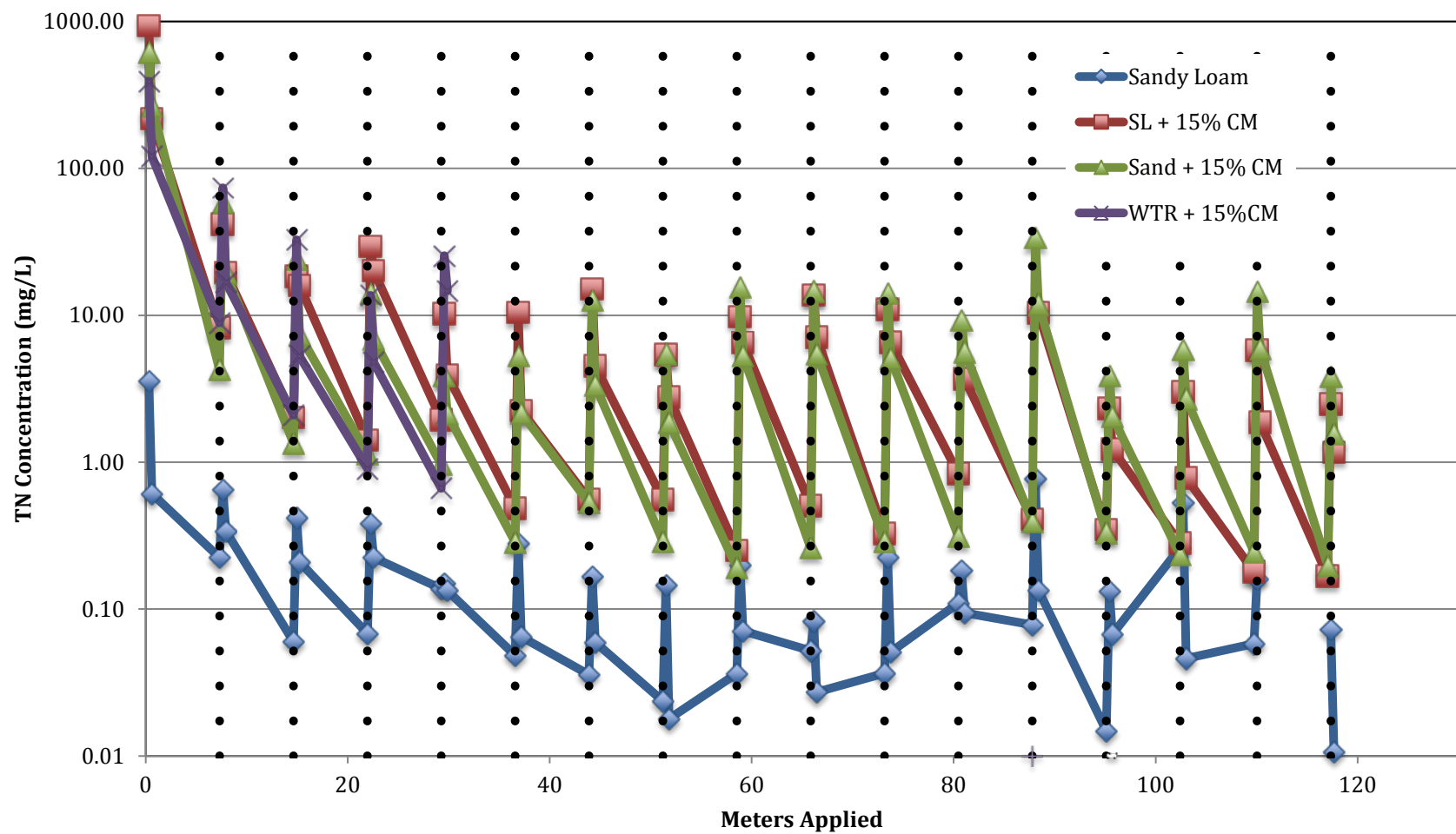


Figure 5- 27. The total nitrogen effluent concentrations from the non-continuous columns.

The TN leaching was similar to the TP leaching in that it increased with every re-wetting period. However, unlike the TP leaching and as predicted, the TN leaching decreased overtime. The initial washout of TN in the effluents for columns containing CM decreases by the second wet period by over an order of magnitude. It is evident that the CM has a high initial washout of TN; this appears to be different than the P leaching since it increased after the first “washout”.

The majority of the TN leaching concentrations remained below 10 mg/L throughout the study after the first 2-3 wet periods. This is still a high TN concentration to be leaching from the material. It is important to consider the long-term nutrient leaching that the CM appears to have. The CM appears to have long-term impacts due to the continuous TN leaching from the material after it is allowed to drain and re-saturate. The long-term impacts also become apparent when the cumulative leached TN mass is observed. Figure 5-28 shows the total TN leached from the non-continuous column study.

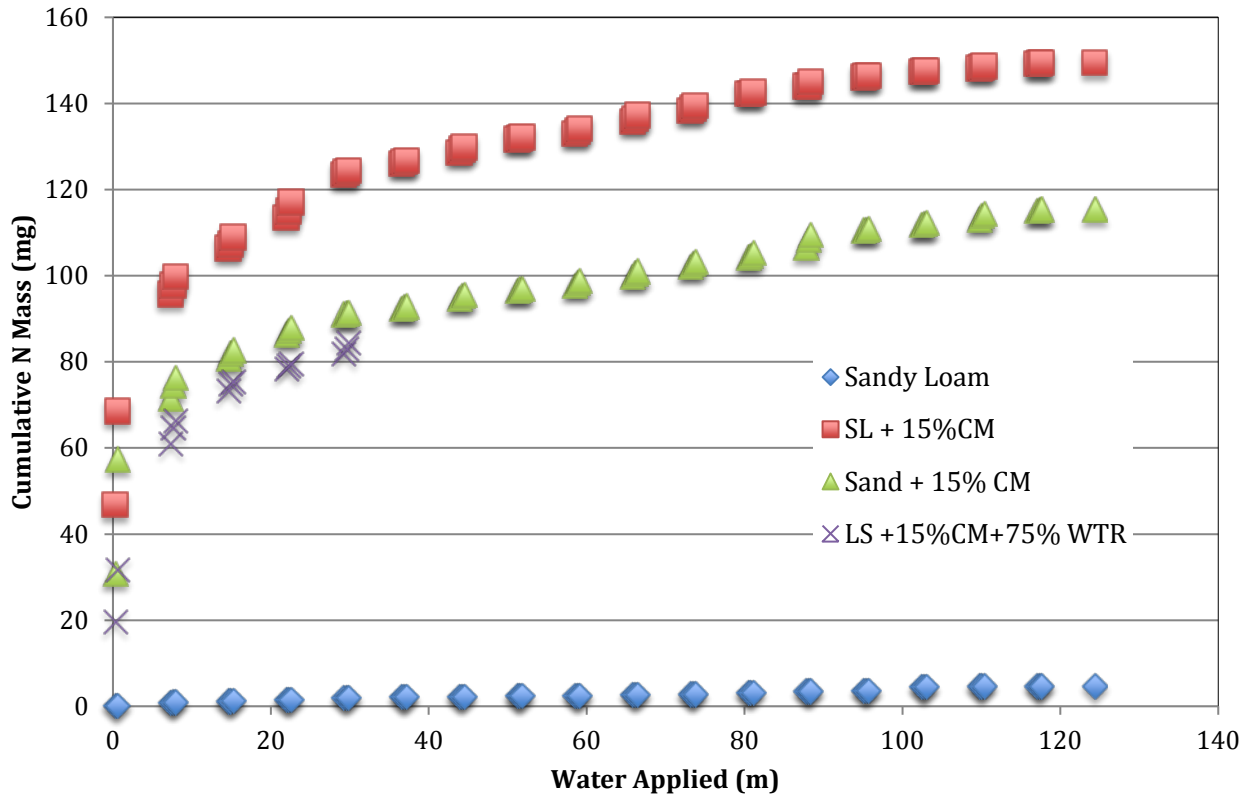


Figure 5- 28. The total cumulative N mass for the columns in the non-continuous column study.

The TN leached clearly shows that the column containing both CM and soil have higher amounts of leached TN than the column of CM and sand. This was the opposite in the continuous columns. Nitrogen leaching from the soil may be a reason why this is occurring. The CM may act differently when it is mixed with the soil instead of sand. Different biological processes may take place and produce different nitrogen outputs.

Similar to the cumulative phosphorus leached, the nitrogen leached similar cumulative masses for both the continuous and non-continuous columns. It is likely that between flow periods the nitrogen built up and was discharged in higher initial concentrations when the flow was resumed.

5.3.6 Non-Continuous Column Nitrogen Speciation

The total nitrogen speciation was analyzed for four samples from the three successfully run non-continuous columns. It was predicted that the column effluent would have similar speciation to the continuous columns- mostly ammonium followed by organic N. The total speciation is shown in Figure 5-29.

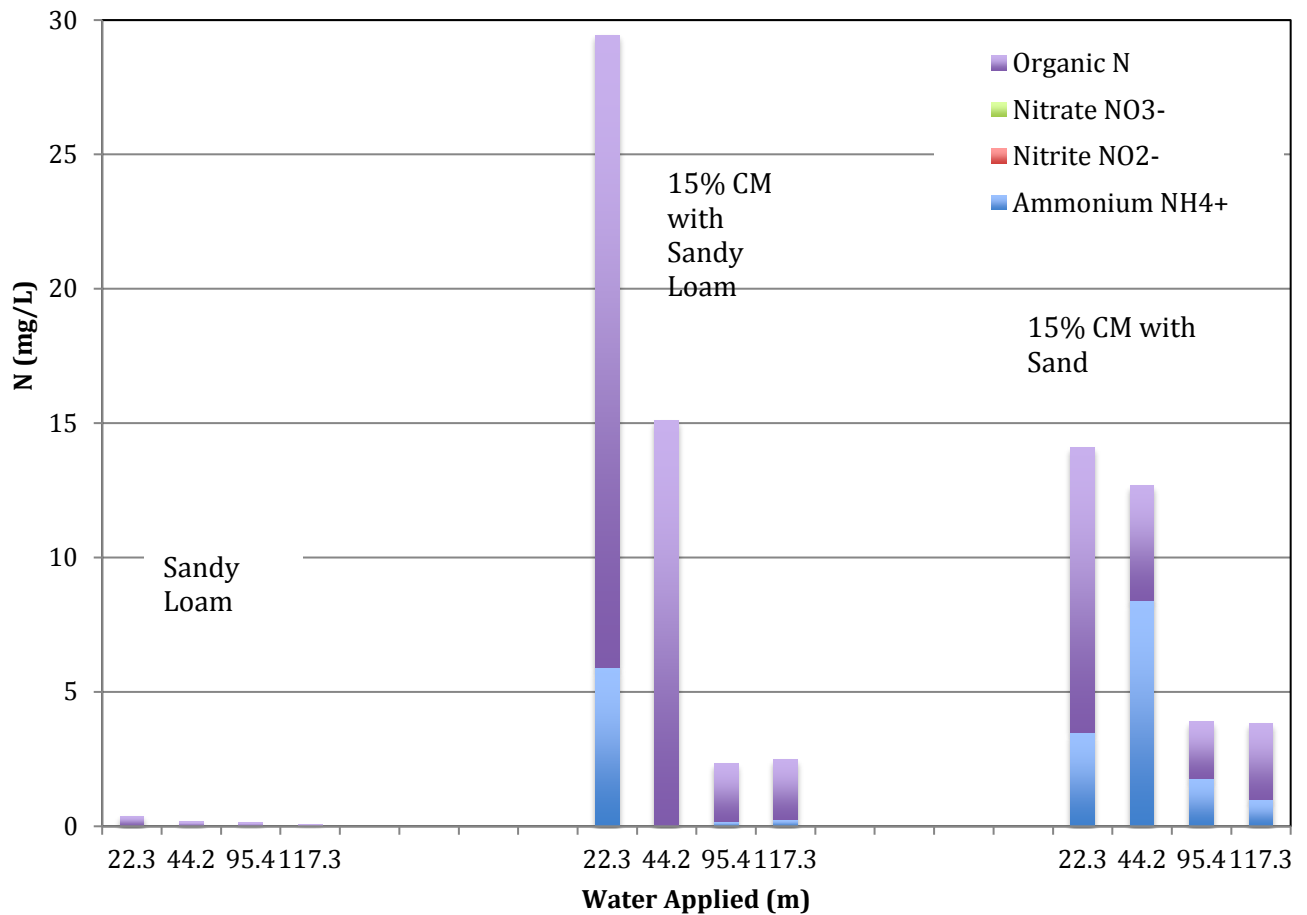


Figure 5- 29. The total nitrogen speciation for the non-continuous columns; the 100% sandy loam, the 15% CM with sandy loam and 15% CM with sand.

The results of the nitrogen speciation were not surprising. The samples contained mostly ammonium and organic N. Most of the effluents contained higher amounts of organic N than ammonium. In the continuous columns this was seen at times, mostly in

the samples towards the later end of the study. This could be an indication that initially a lot of the ammonium washes out of the material and is therefore decreased towards the end of the studies, or as the CM has more water-applied overtime. It was surprising that the nitrate levels were not detectable in the samples. Nitrate is known to build up through nitrification and aerobic environments. Therefore the draining periods of the non-continuous studies were expected to have an increase in nitrate, followed by a leaching of the nitrate in the effluent, however this was not observed in the CM effluent (Singh et al. 2012).

5.4 Potassium

5.4.1 Continuous Column Potassium Leaching

The potassium concentrations were analyzed on most of the columns to gain a better understanding of the fertilizer effects the CM may have and provide. Some of the columns were not tested for potassium earlier in the project. Some of the continuous column total potassium (TK) data are shown in Figure 5-30. For simplicity, only five of the data sets were plotted.

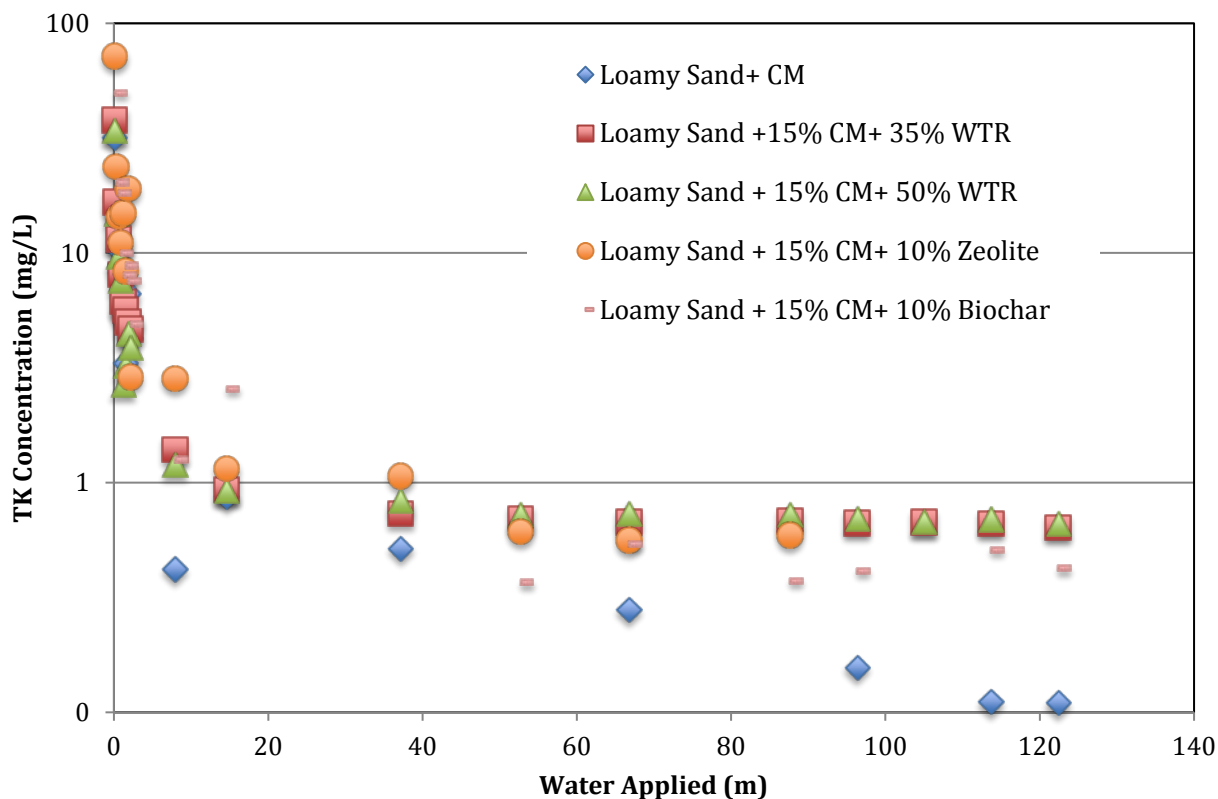


Figure 5- 30. The TK concentration data from the continuous column effluent. The columns shown are select 15% CM (dry mass basis) with mixtures of loamy sand and various soil amendments.

From Figure 5-30 it appears that total potassium (TK) follows a similar trend to TN, where it starts out high, typically around 20-40 mg/L, has an immediate wash out and continues to decrease and stay below 1 mg/L throughout the column study duration. Literature on TK is not as available as it is on nitrogen and phosphorus. This gave little predictions into how the potassium would leach and what trends it would follow. The columns with the soil amendments, in particular the zeolite and biochar had higher initial effluent concentrations of TK compared to the column with just the soil and the columns with the WTR.

5.4.2 Non-Continuous Column Potassium Leaching

The non-continuous columns were expected to have similar K leaching to the continuous columns- an initial higher washout, but overall decrease with some re-wetting increasing peaks. Figure 5-31 shows the K leachate concentrations for the four columns that were completed for the non-continuous column study.

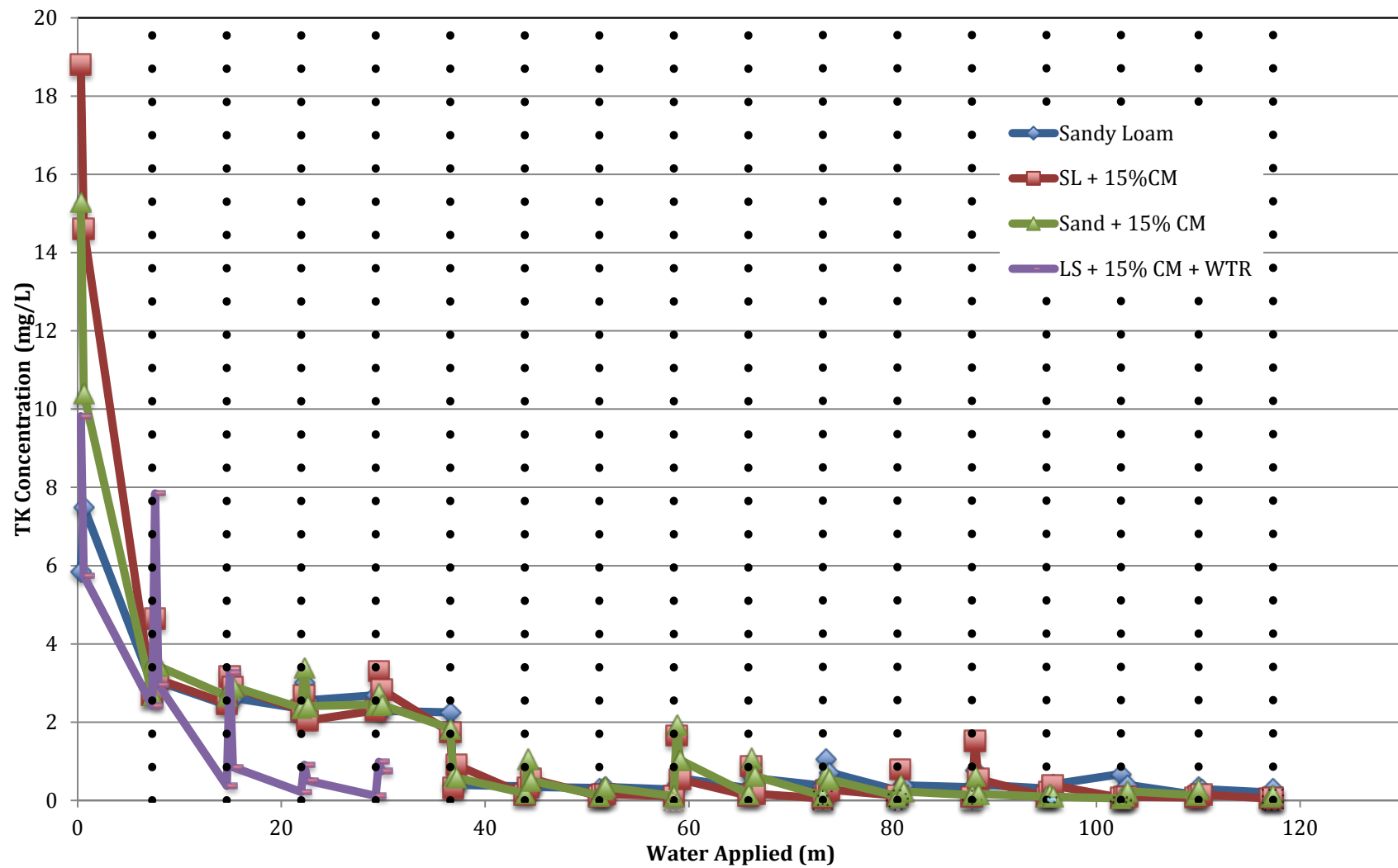


Figure 5- 31. The non-continuous columns potassium leaching data. The black vertical dotted lines mark off each wet period.

The non-continuous column K leaching predictions were reasonably accurate. As shown in Figure 5-31, the levels did have higher initial washout concentrations of potassium with an overall decrease in levels over time. The K leaching is more like the nitrogen than the phosphorus in that it continues to decrease over time and reach a stable level below 2 mg/L. There are some small increasing peaks when the columns are re-wetted, however the spikes that were seen with the phosphorus leaching are not seen with the potassium. The total leached TK is shown in Figure 5-32.

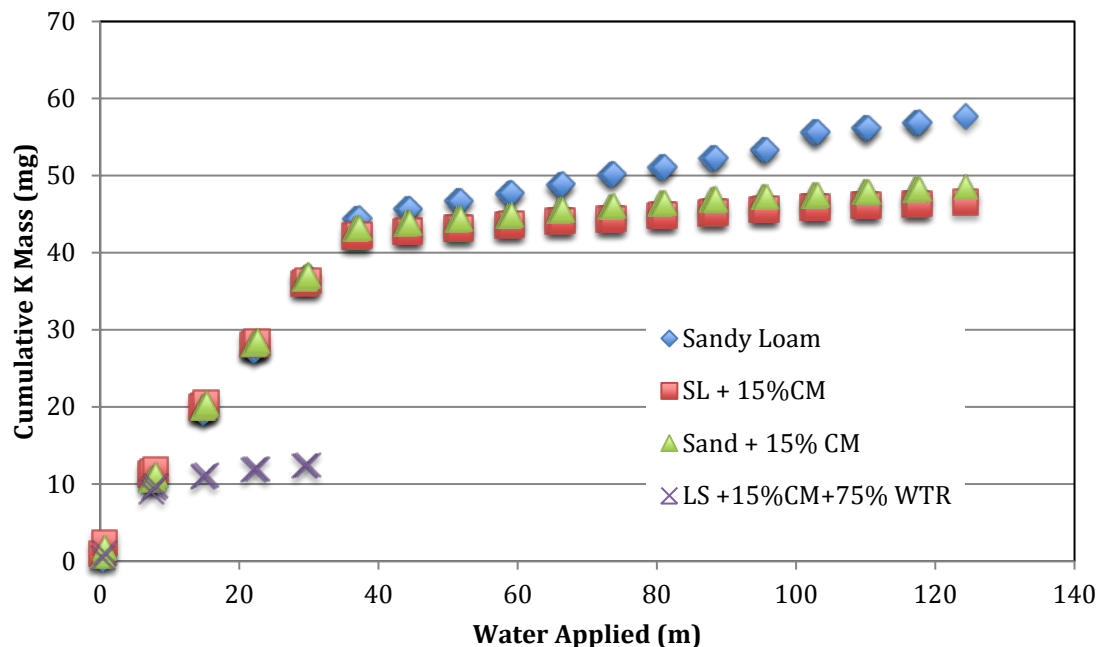


Figure 5- 32. The total leached K for the duration of the non-continuous column study.

There were not too many differences in leached TK among the media mixes; however there seems to be a plateau of leached TK in the WTR column much earlier than the other three columns. This was surprising since the continuous columns with WTR did not seem to have any effect on the TK concentrations. The other three columns plateaued at around 40 meters of water applied. They do not seem to increase much after

this, which is not surprising since the effluent concentrations of TK in the columns all were below 2 mg/L for most of the study.

Chapter 6: Aging Study

From the non-continuous column studies it was observed that the CM exhibited different characteristics over time as it was allowed to dry. Due to these different characteristics, an aging study was completed to study the effects of letting the CM drain and re-wet over a longer period of 12 weeks. The CM was predicted to decrease in nutrient levels over time from drying and being re-wetted. It was expected that some particulate matter would wash off and that ammonification and further volatilization would occur, lowering the extractable ammonium concentrations over time (Singh et al. 2012). It has been shown in studies over time that dried biosolids, whether they are air-dried or heat-dried lose some of their plant available nitrogen (PAN) content (Cogger et al. 2004).

The buckets containing the biosolids used for the aging study are shown in Figure 6-1 after 1 week of the study. The biosolids broke off into smaller pieces as shown in Figure 6-1 and accumulated a white film over them.



Figure 6- 1. The biosolids that had been wetted and re-dried after one week of the aging study.

6.1 Phosphorus Extraction Results

Both the Mehlich III and CaCl_2 extractable P were measured weekly for the first month and then bi-weekly for the second and third month of sampling. It was predicted that the material would decrease in P leachate concentrations over time since some of the P would runoff the material after wetted. The results from the two P extractions are shown in Figures 6-2 and 6-3.

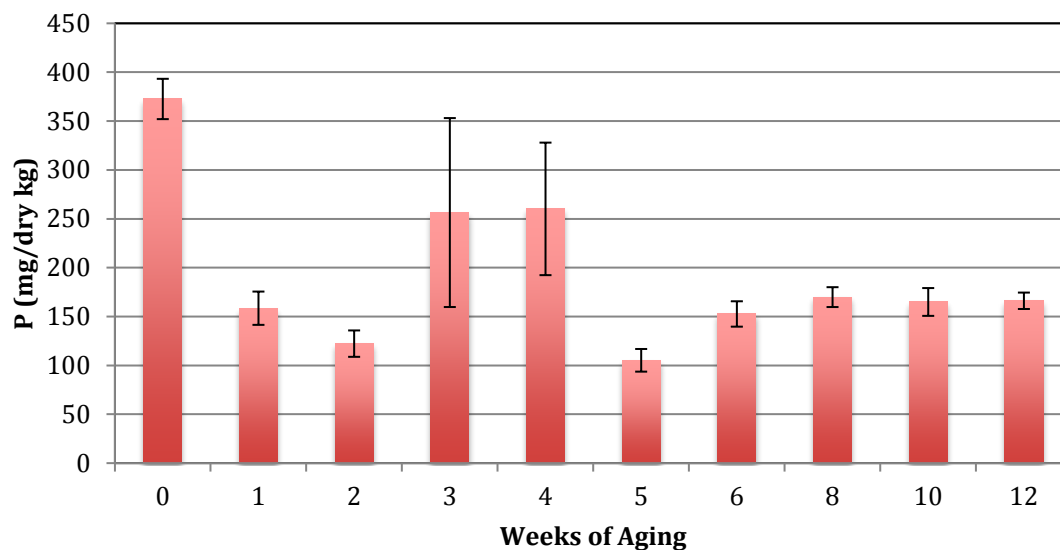


Figure 6- 2. The Mehlich III P extraction data for the CM as it aged over 12 weeks. The concentrations are in mg/ dry kg of CM. The error bars represent one standard deviation within the triplicate samples.

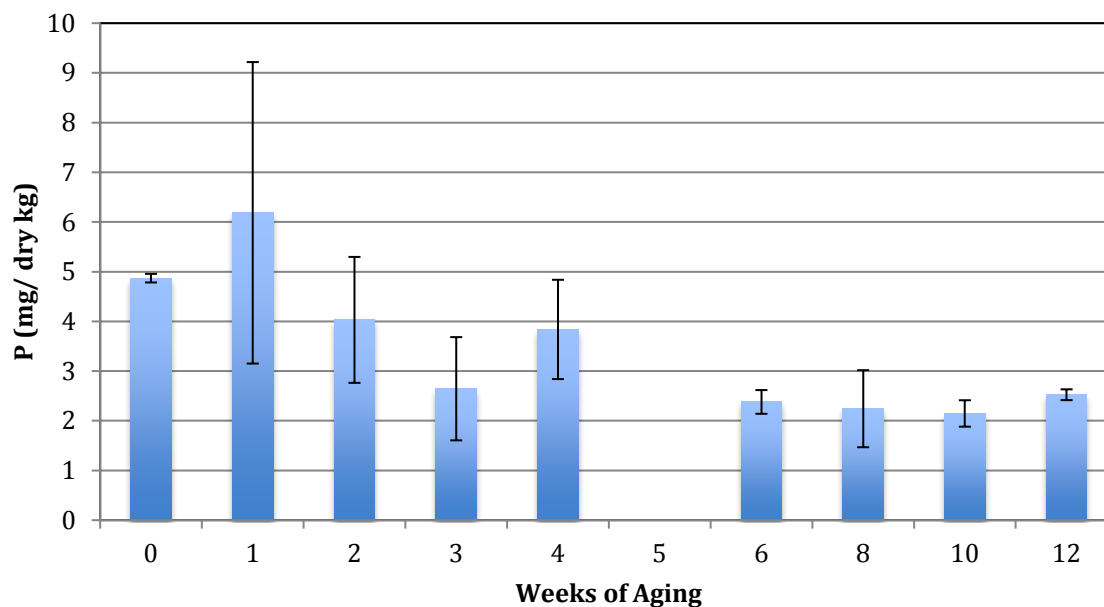


Figure 6- 3. The CaCl_2 P extraction data for the CM as it aged over 12 weeks. The concentrations are in mg/dry kg of CM. Week 5 was not tested for CaCl_2 P. The error bars represent one standard deviation within the triplicate samples.

Figure 6-2 shows the Mehlich III P extraction data throughout the aging study. The initial M3P concentration was much higher than the subsequent concentrations. The overall decrease in concentration was expected as some of the P was thought to wash off of the CM as it was re-wetted each week. Apart from weeks 3 and 4, the data is consistent at approximately 150 mg P/dry kg CM. This is lower than the wet CM that was tested throughout the project at around 1000 mg P/dry kg CM as discussed in Chapter 3.

The CaCl_2 P data was similar to the M3P data in that the initial concentration was higher than the majority of the following samples, however not all of them. The ranges of differences in the P levels for the M3P extractions were greater than the range for the CaCl_2 P extractions. This is not surprising given the much higher magnitude of the M3P extractions.

Although both nutrient extraction concentrations decreased over time, they still leached after 12 weeks of aging and re-wetting/drying. Past studies on dried biosolids have predicted them to have P that is less likely to leach and runoff since the P was extracted only with stronger extractants compared to other organic sources (Ajiboye et al. 2004). Based on the study by Ajiboye et al. (2004) and what has been found with the CM in this study, it is likely that the P in the CM does not leach out quickly. The material has a slow leach process that appears to last over time.

6.2 Nitrogen Extraction Results

The nitrogen extraction data for the CM throughout the aging study is shown in Figures 6-4 and 6-5. The nitrogen extraction concentration results were predicted to

decrease over time, especially the ammonium concentrations via ammonification and volatilization.

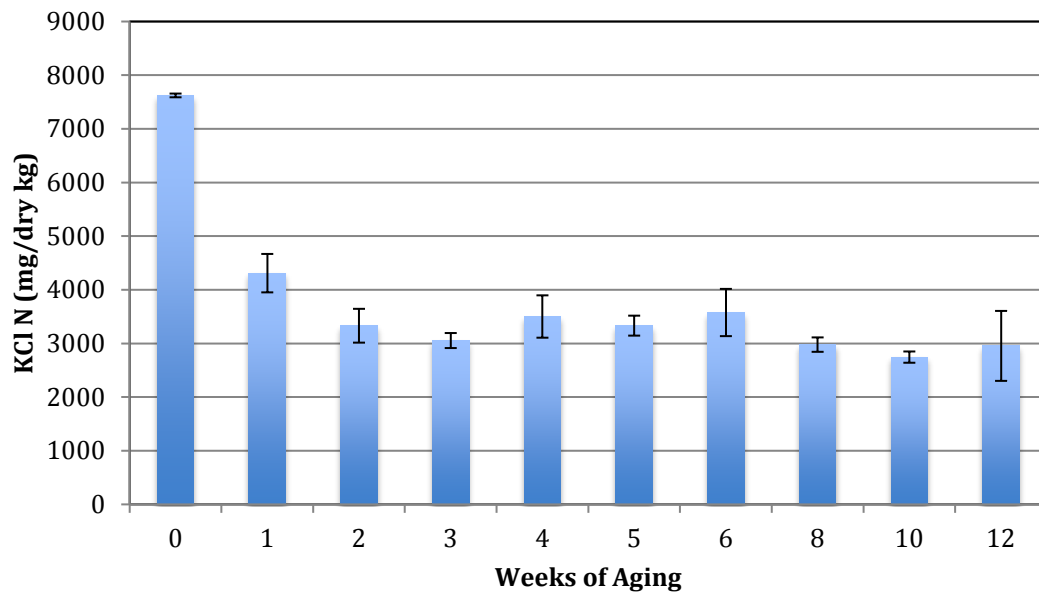


Figure 6- 4. The KCl TN concentration results for the CM throughout the aging study. The data is shown in mg N/dry kg CM. The error bars represent one standard deviation within the triplicate samples.

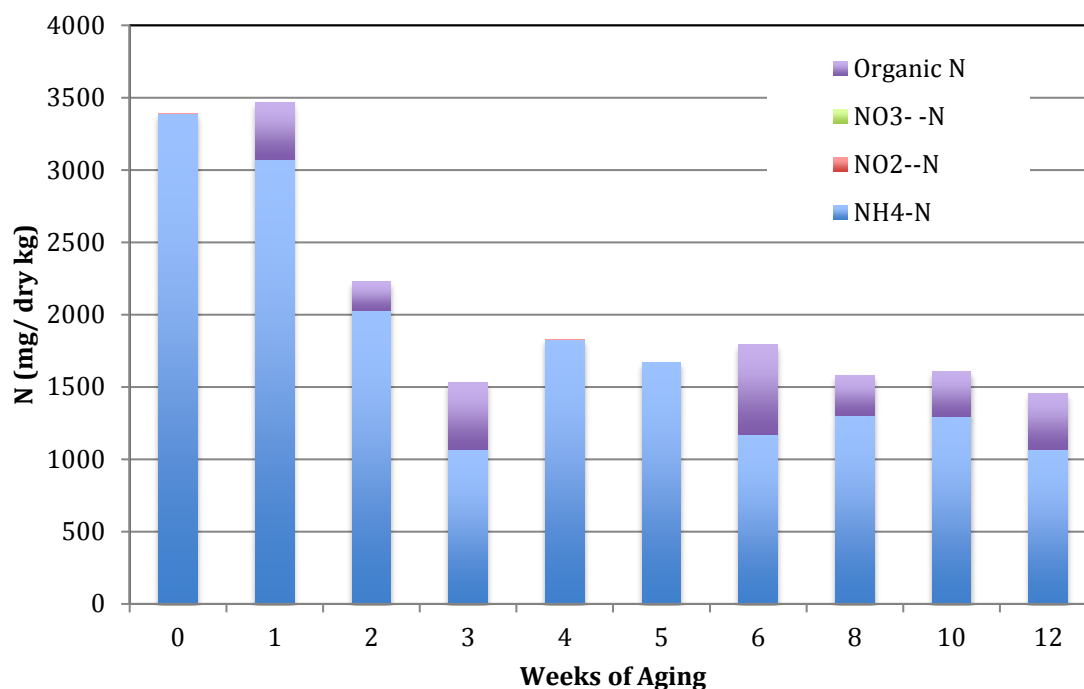


Figure 6- 5. The total nitrogen speciation from the CaCl_2 extraction for the CM throughout the duration of the aging study. The data are shown in mg N/dry kg CM.

The nitrogen extraction data show an evident decrease in concentration from the initial sample of CM. Figure 6-4 shows the initial CaCl_2 TN values to be almost 3500 mg N/dry kg CM whereas after 2 weeks they were at or below 2000 mg N/dry kg CM. This decrease seemed to occur slowly throughout the study. Initially, the extraction contained approximately all ammonium and trace amounts of nitrate, nitrite and organic N. It was not surprising that ammonium was the dominant form of N in the nutrient extractions each sample, as this was seen throughout the entirety of the project in multiple studies. Over time the organic N started leaching out as well. This data correspond to the predictions that over time the ammonium leaching would decrease.

Figure 6-5 shows a more drastic decrease in nitrogen extraction concentration data than Figure 6-4. The KCl N extractions for the initial samples of CM were roughly

7620 mg N/dry kg CM. After drying, the levels dropped to 4000 mg N/dry kg CM or below throughout the remainder of the study. The decrease from the initial sample concentration was almost halved, and the levels continued to decrease very slowly to approximately around 3000 mg N/dry kg CM. As with the study by Cogger et al. (2004), it is predicted that most of the N is leached out after being wetted and the overall PAN decreases in the CM.

6.3 Potassium Extraction Results

The plant available potassium was analyzed for each sample of CM for the duration of the study with a Mehlich III extractant. The results of the potassium extraction are shown in Figure 6-6.

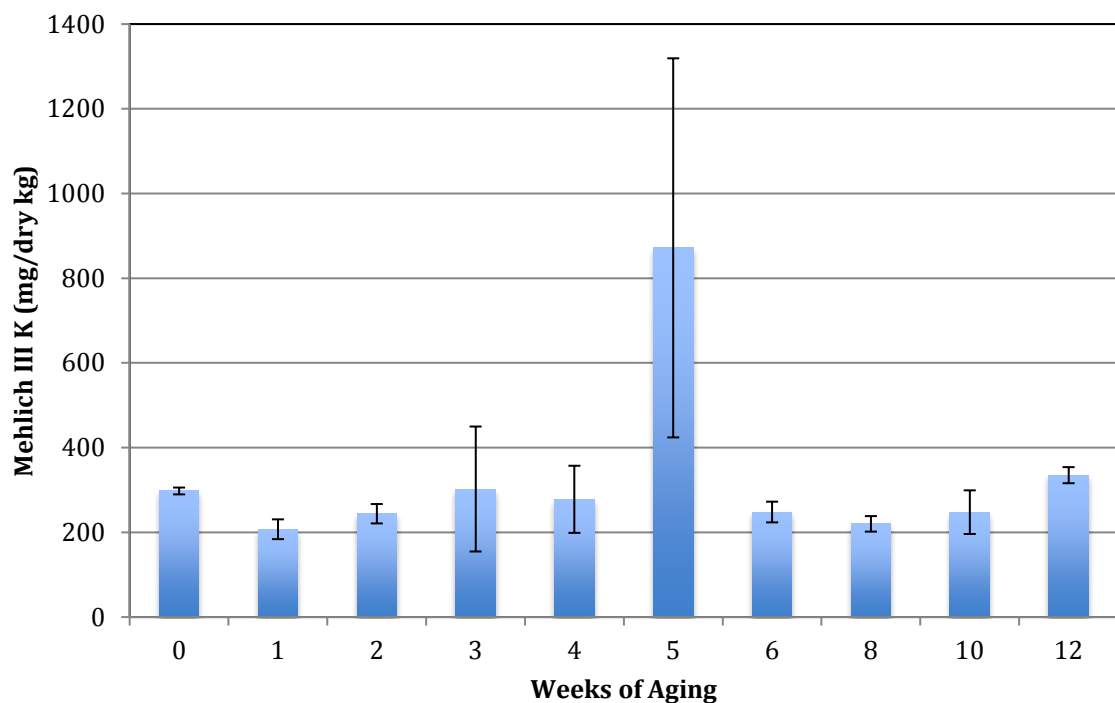


Figure 6- 6. The concentration results for the Mehlich III K. The concentrations are in mg K/dry kg CM. The error bars represent one standard deviation within the triplicate samples that were completed each week (1 per each replicate bucket).

The M3K results did not turn out to be as expected. Apart from the sample M3K concentration at week 5 of aging, the samples did not differ significantly in M3K content. This was surprising since in the column studies where the CM had been wetted, K seemed to washout quicker than both N and P. This could be an indication that the material has longer lasting plant available K.

Chapter 7: Dry CM Leach Study

The non-continuous and continuous column studies gave insight into the nutrient content in the CM, with the non-continuous study providing more realistic results. After completing both types of column studies there became an interest in the actual runoff that would leach from the material if it were dried and land applied. The dry leach study was completed for six weeks to test the runoff that would come off various sizes of CM materials to see how the dried material would leach depending on the size, mass and surface area of the CM. Table 7-1 shows the sizes, masses, and surface areas of the CM used for the dry leach study.

Table 7- 1. The CM volumes, surface areas, and masses for each amount of dried CM.

# CM Pieces	Bottle	Total Volume (cm ³)	Total Surface Area (cm ²)	Mass (g)
1	1A	10.5	34	10.9
1	2A	12.5	40	10.8
1	3A	13.5	42	10.4
2	1B	17.5	59	11
2	2B	12	44	10.8
2	3B	12	44	10.7
4	1C	15.5	63	11
4	2C	12.8	54.5	10.6
4	3C	15	61	10.5
7	1D	11.3	63	10.6
7	2D	16.5	80.5	10.7
7	3D	18.3	84.5	10.7

Each group has approximately the same mass, however as the pieces in the groups increase, the total surface area increases. It was predicted that with a greater surface area, more nutrient leaching would occur. Runoff water from each group of CM was tested weekly for TP speciation, TN speciation, and TK. Similar to the aging study, it was

expected that the nutrient concentrations of the leachate from the material would decrease over time. Also, particulate matter was predicted to leach off initially.

7.1 Phosphorus Results

Based on past column studies and the initial data from the aging study, the phosphorus leaching from the dry leach study was expected to be below the initial column P leaching concentrations; below 1 mg/L, and decrease over time. Figure 7-1 shows the total phosphorus data, and Figure 7-2 shows the total phosphorus speciation for the various sizes of CM over the six weeks.

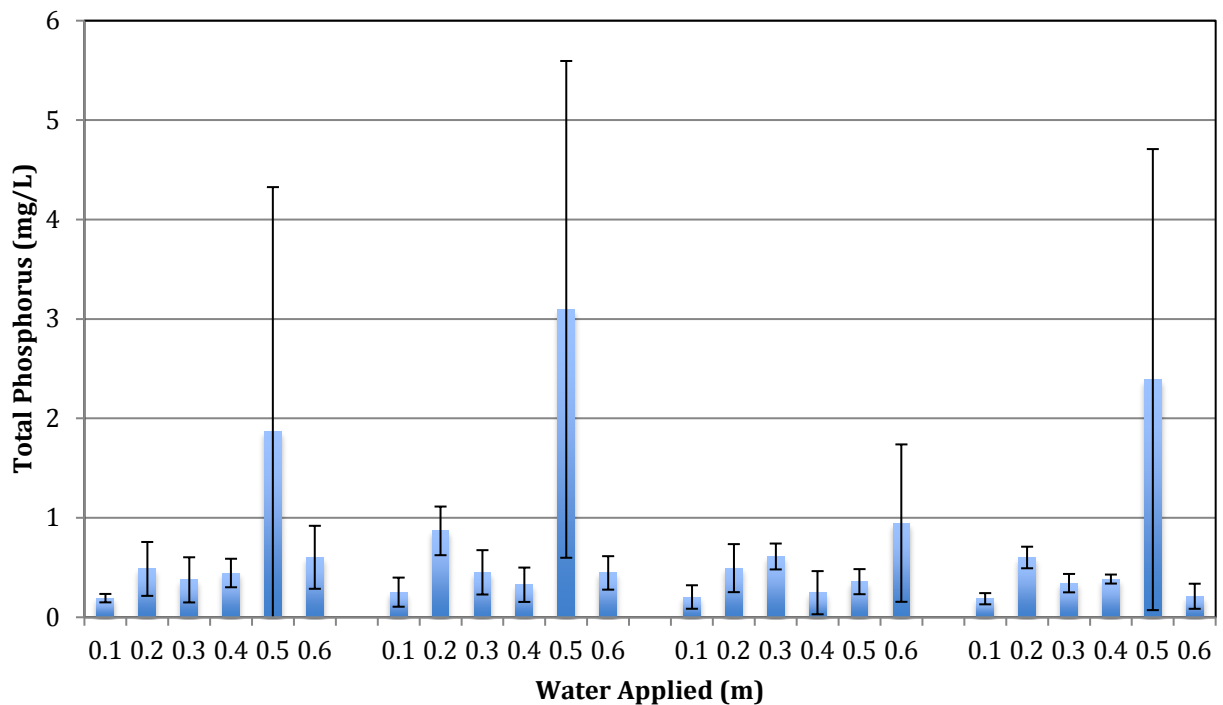


Figure 7- 1. The total phosphorus concentration data for the various sized CM pieces throughout the 6-week dry leach study. The error bars show one standard deviation within the triplicate samples for each size.

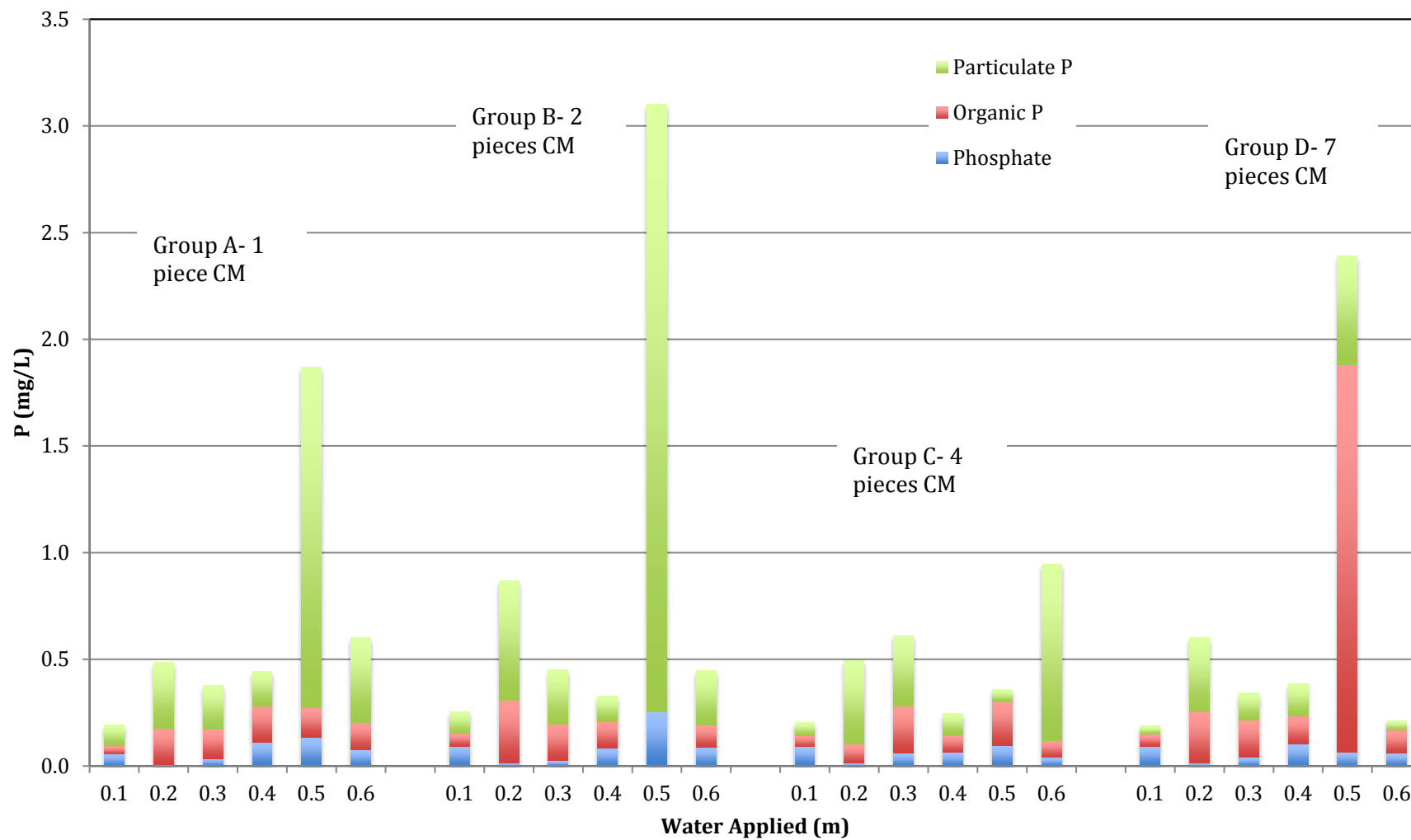


Figure 7- 2. The total phosphorus speciation data for each group of CM for the various amounts of water applied.

Figure 7-1 shows the average total P concentrations from the effluent from the dry leach study for each CM size. The majority of the TP effluent concentrations remained below 1 mg/L, apart from the samples at 0.5 m water applied.

The majority of the leached P for the various groups was particulate P, apart from 3 samples with organic P as the dominant P species. The TP data is not significantly different for any of the groups from each other based on the entirety of the TP data. However there are some significant differences between TP concentrations within weeks between group sizes. Table 7-2 shows the average TP concentrations and significant differences between groups, if any ($\alpha=0.05$). Table 7-3 shows the significant differences within a group as more water is applied. For most of the groups the samples at 0.5 m water applied were significantly different from many other sampling days.

Table 7- 2. The average total phosphorus concentrations for each week and group size. Student t-tests were completed between columns for each week's information.

Week and TP Concentration (mg/L)						
Group	1	2	3	4	5	6
A	0.19	0.49	0.38ab	0.45	1.87ab	0.60
B	0.25	0.69	0.45ab	0.33	4.59a	0.45
C	0.20	0.49	0.61b	0.25	0.36b	0.95
D	0.19	0.60	0.34a	0.38	2.39ab	0.21

* Same letters within columns signify values that are not significantly different ($\alpha=0.05$).

Table 7- 3. The average total phosphorus concentrations for each week and group size. Student t-tests were completed between rows for individual groups TP concentration data.

Week and TP Concentration (mg/L)						
Group	1	2	3	4	5	6
A	0.19a	0.49ab	0.38ab	0.45b	1.87ab	0.60ab
B	0.25a	0.69be	0.45ae	0.33d	4.59c	0.45a
C	0.20a	0.49ac	0.61c	0.25b	0.36b	0.95ac
D	0.19ac	0.60d	0.34b	0.38b	2.39bcd	0.21bcd

* Same letters within rows signify values that are not significantly different ($\alpha=0.05$).

It was predicted that the groups with more surface area (group D has the highest decreasing to A) would have more P leaching. Statistically speaking, the TP leached from the groups with an overall greater surface area was not significantly more than groups with less surface area. It is possible that similar masses of the CM produce similar leaching since the different groups produce similar TP leaching and have similar masses.

Since the dominant species is mostly particulate phosphorus, these data can be compared to the beginning of the continuous columns and the non-continuous column study TP speciations. For the continuous column study, the initial output of P was mainly in the form of particulate P, and for the non-continuous columns the particulate P was mostly dominant as well. When the CM is dried and then re-wetted, the material may leach more particulate P than other forms of P.

The overall trend of TP does not change significantly throughout the study. Apart from the samples for groups A, B, and D at 0.5 m of water applied, the TP stays fairly consistent at around or below 0.5 mg/L P. This leached concentration is lower than what was seen throughout most of the column studies. However, unlike the columns, there is not any evident decrease in the TP in the leachate from the dried material. This could be an indication that the material has a long lasting slow release ability.

7.2 Nitrogen Results

In the past studies done with this project, most of the nitrogen leached from the CM is in the form of ammonium and organic N. From this, it was predicted that most of the dry material leachate would have ammonium as the dominant form of nitrogen. It was also predicted that with a higher surface area, there would be greater amounts of N in the leachate. Figure 7-3 shows the average effluent TN concentrations for each CM group

size and Figure 7-4 shows the TN speciation for each of the sizes throughout the dry leach study.

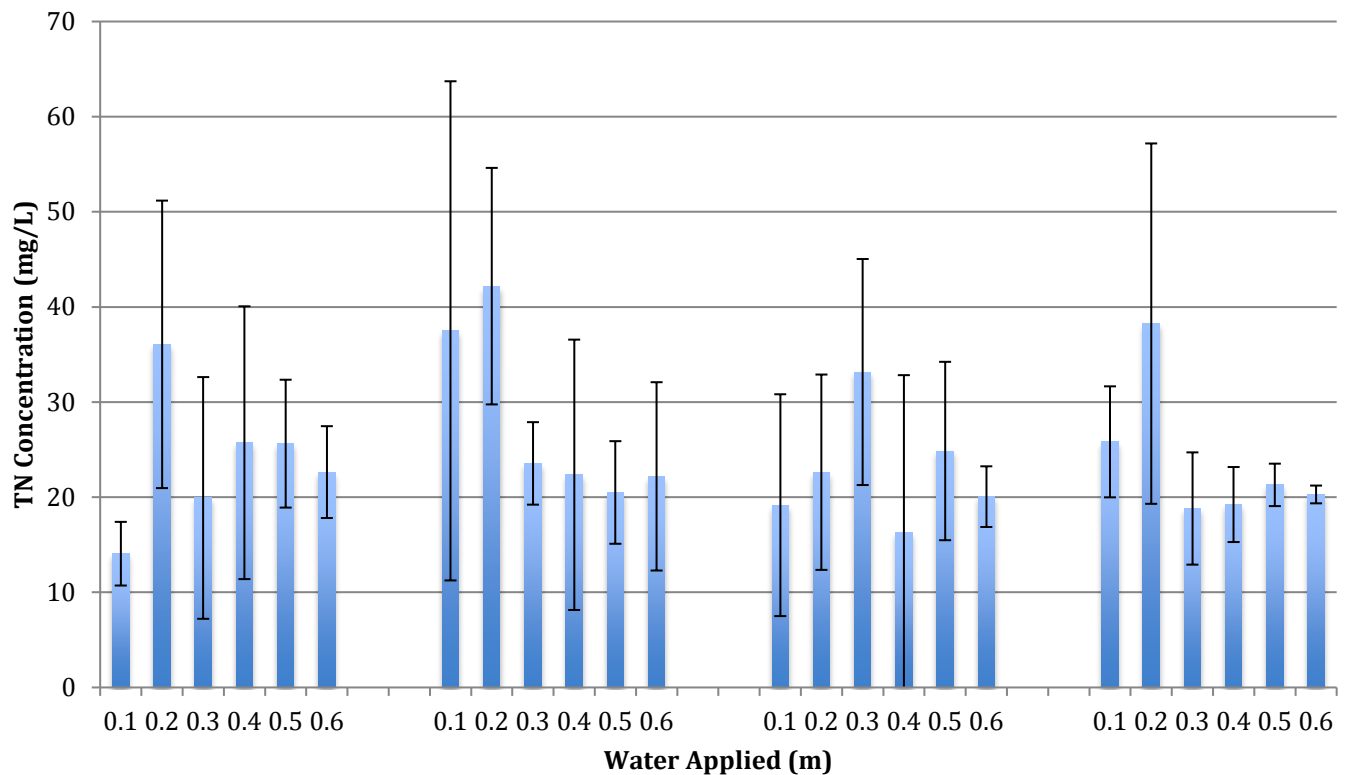


Figure 7- 3. The total nitrogen concentration data for the various sized CM pieces throughout the 6 week dry leach study. The error bars show one standard deviation within the triplicate samples for each size.

It is evident from Figure 7-3 that the TN concentrations for the dry leach study were on a lower order of magnitude than what was seen with the column studies and nutrient extractions. Concentrations in the column studies got to concentrations over 600 mg/L TN, whereas all of the concentrations in the dry leach study were below 70 mg/L TN.

It has to be taken into consideration that these experiments contained an average of 10.7 g of CM and the water influent was not pressurized through the material. The columns were a controlled, pressurized system that contained approximately 19 g of dry

(59 g wet) CM. Although these are two different systems, the results can be compared broadly.

Although the concentrations of TN coming from the dry CM in the leach study are much lower than that of the columns, the levels also do not decrease as drastically as they did in the column study. There was a more evident drop in TN concentration in both the column types that was not seen with the dry leach study. The material may have a longer- term slow leach availability with nitrogen as well if allowed to dry and re-wet. There is a slight decrease in TN concentrations for the samples taken at 0.5 and 0.6 meters applied water for each of the groups, however the majority of the samples were relatively stable at around 20 mg/L.

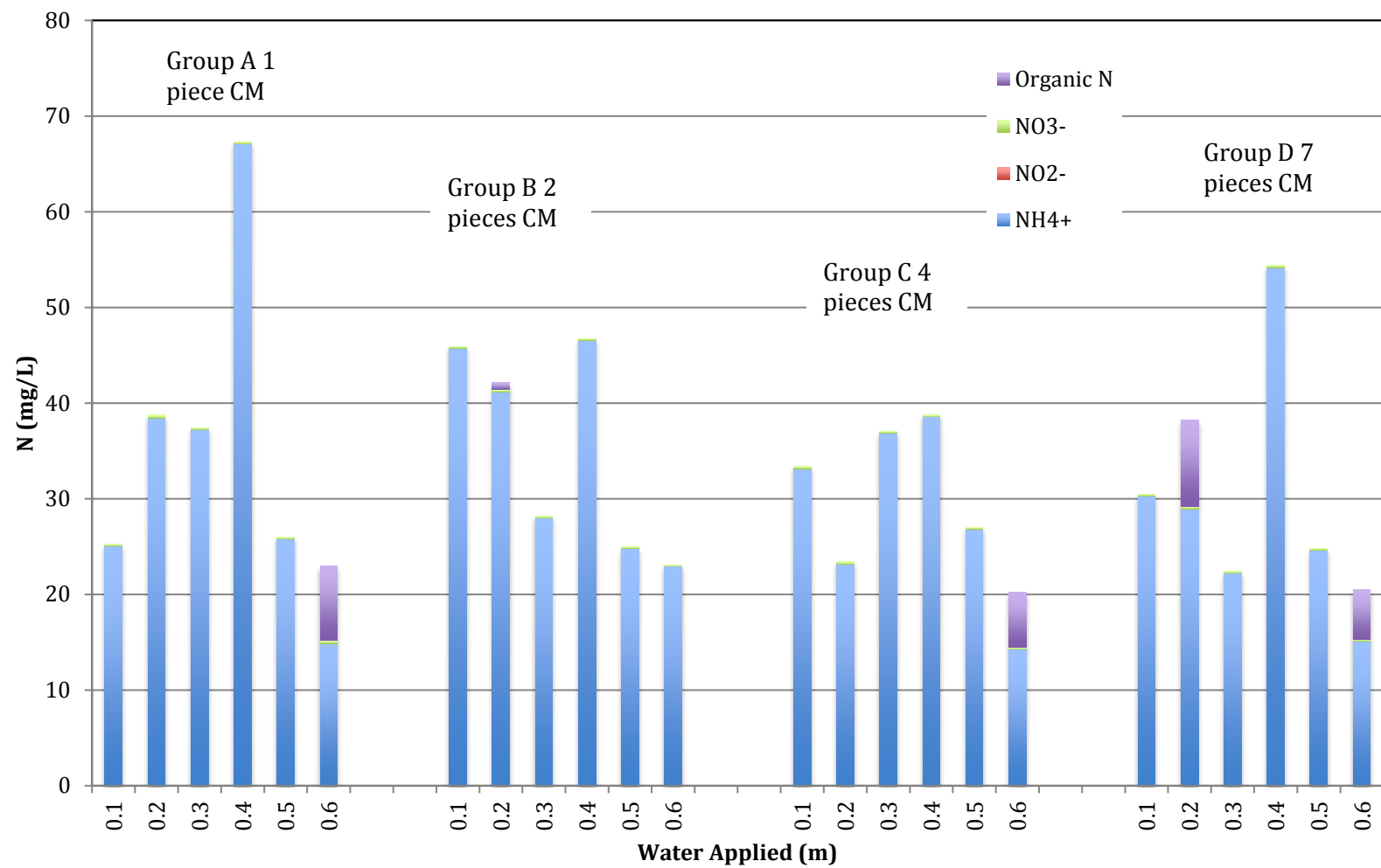


Figure 7- 4. The TN speciation for each group of CM throughout the duration of the dry leach study.

The total nitrogen speciation for the dry leach study is not surprising. The dominant form of nitrogen throughout the study was ammonium. Towards the end of the study and for Group D during the study, some organic N appears as well. Nitrate and nitrite are almost completely negligible. Similar to the phosphorus data for the dry CM leachate, a trend does not appear evident. It is predicted that if the study were to go on for more time the TN would continue to decrease and the ammonium would decrease as well.

Statistically, group A was significantly different ($\alpha=0.05$) from group D at 0.1 and 0.2 m water applied, and from group B at 0.2 m water applied. If group A were significantly different from all of the groups for other days, it could have shown the prediction that a smaller surface area produces a smaller amount of TN in leaching, however this was not the case, and it cannot be concluded that a smaller surface area produces smaller amounts of TN leaching. The average TN concentration data from the dry leach study is shown in Tables 7-4 and 7-5. Table 7-4 shows values to be significantly different between groups on sample days, and Table 7-5 shows values to be significantly different within groups throughout the study.

Table 7- 4. The average total nitrogen concentrations for each week and group size. Student t-tests were completed between columns for each week's information.

Group	Week and TN Concentration (mg/L)					
	1	2	3	4	5	6
A	14.1a	36.1a	19.9	25.7	25.6	22.6
B	37.5ab	42.2b	23.6	22.4	20.5	22.2
C	19.2ab	22.6ab	33.2	16.3	24.9	20.1
D	25.8b	38.2b	18.8	19.2	21.3	20.3

* Same letters within columns signify values that are not significantly different ($\alpha=0.05$).

Table 7- 5. The average total nitrogen concentrations for each week and group size. Student t-tests were completed between rows for individual groups TP concentration data.

Group	Week and TN Concentration (mg/L)					
	1	2	3	4	5	6
A	14.1a	36.1ab	19.9ab	25.7ab	25.6b	22.6b
B	37.5ab	42.2a	23.6ab	22.4ab	20.5b	22.2b
C	19.2	22.6	33.2	16.3	24.9	20.1
D	25.8	38.2	18.8	19.2	21.3	20.3

* Same letters within rows signify values that are not significantly different ($\alpha=0.05$).

7.3 Potassium Results

Throughout this project potassium, data were collected to gain a better perspective on the material from a fertilizer point of view. There are not many available studies on potassium, however, based on what had been seen with the project, it was predicted that the initial leachate from the dry CM would produce higher levels of TK and a decrease throughout the study. Figure 3 shows the TK data for the duration of the dry leach study.

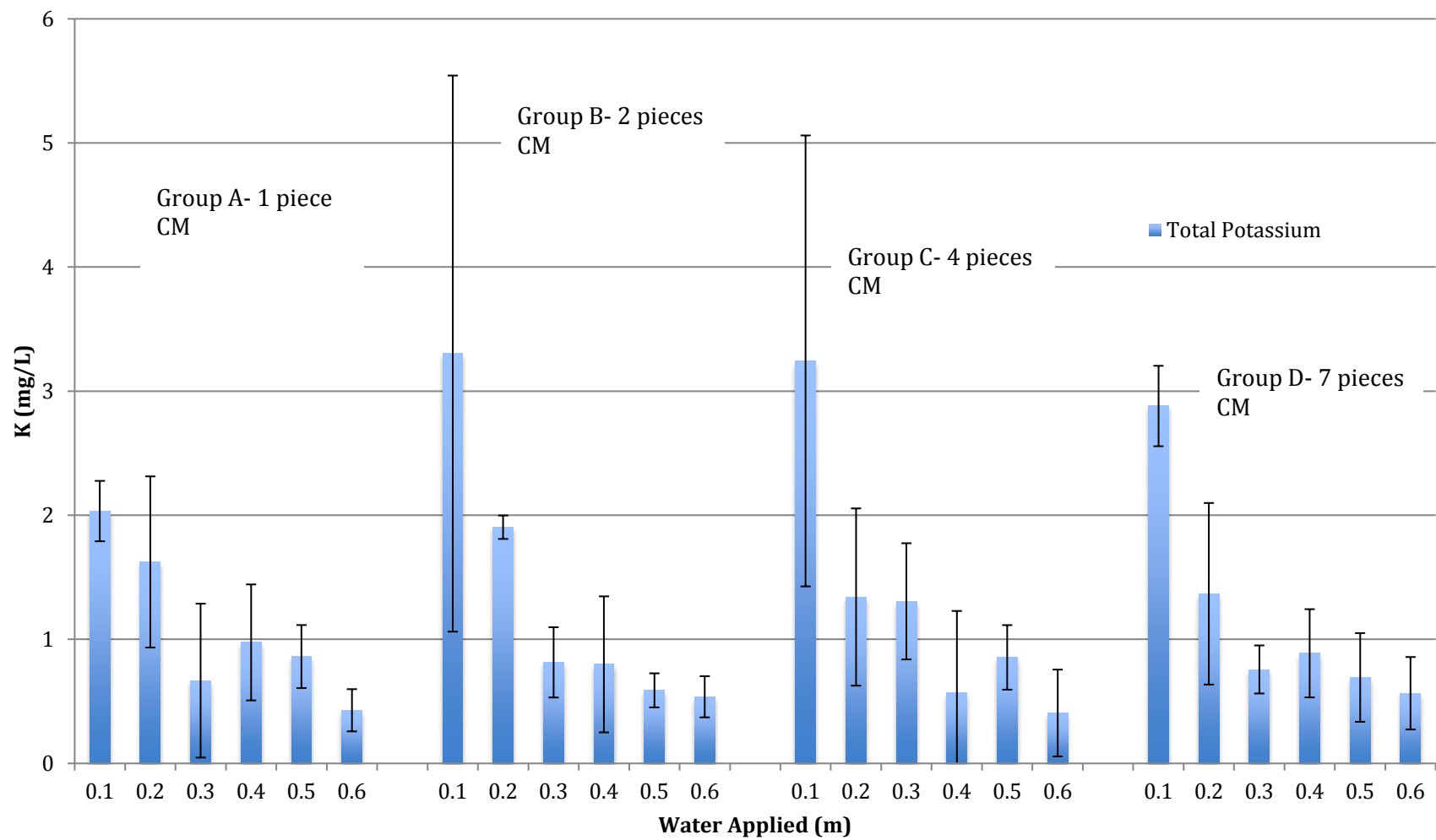


Figure 7- 5. The TK data for the leachate from the dry CM leach study. The error bars represent one standard deviation within the triplicate data that was completed for each size of CM.

Figure 7-5 shows the evident decrease in TK concentrations from each of the groups of CM. K showed a clearer and more drastic decrease throughout the study than both N and P did. This leads to the possible conclusion that K is highly soluble and can be washed off the material very easily. Also unlike N and P, there were more significant differences between samples for within groups for K concentrations. There were not however significant differences between groups.

Within group A, the samples at 0.1 m applied water were significantly greater than TK concentrations at 0.3, 0.4, 0.5 and 0.6 m applied water. For group B, the samples at 1.2 were significantly greater than the other five samples. In addition, within group B, C, and D (individually) TK concentrations at 0.2 m were significantly greater at 0.3, 0.4, 0.5, and 0.6 m applied water. Overall, almost all of the initial concentrations for all of the groups were significantly higher than the preceding sample TK concentrations. These statistics show the general decreasing trend that is evident for TK samples throughout the dry leach study and shows that the K within the CM is more soluble and likely to quickly leach out in comparison to the N and P. Tables 7-6 and 7-7 summarize the average TK concentration results and show values to be statistically different within groups and between groups.

Table 7- 6. The average total potassium concentrations for each week and group size. Student t-tests were completed between columns for each week's information.

Group	Week and TK Concentration (mg/L)					
	1	2	3	4	5	6
A	2.03a	1.62a	0.67	0.98	0.86	0.43
B	4.46ab	1.9b	0.81	0.80	0.59	0.54
C	4.22ab	1.34ab	1.31	0.57	0.85	0.41
D	2.88b	1.37b	0.76	0.89	0.69	0.57

* Same letters within columns signify values that are not significantly different ($\alpha=0.05$).

Table 7- 7. The average total potassium concentrations for each week and group size. Student t-tests were completed between rows for individual groups TP concentration data.

Group	Week and TK Concentration (mg/L)					
	1	2	3	4	5	6
A	2.03a	1.62ab	0.67bc	0.98bc	0.86b	0.43bc
B	4.46ab	1.9a	0.81b	0.80b	0.59b	0.54b
C	4.22a	1.34bc	1.31bc	0.57bc	0.85b	0.41bc
D	2.88a	1.37b	0.76b	0.89b	0.69b	0.57b

* Same letters within rows signify values that are not significantly different ($\alpha=0.05$).

The TK data for the leach study is comparable to what was observed in the column studies. TK is present in the CM, however it seems to leach out initially and continue to decrease in effluent concentrations.

Chapter 8: Conclusions and Recommendations

8.1 Conclusions

The nutrient leaching characteristics of DC Water's Cambi Material biosolids were studied from June 2015 to September 2016 at the University of Maryland. The goal of the project was to characterize the CM nutrient leaching behavior and develop CM amendments, processes and recommendations that would allow it to be used beneficially throughout the (sub)urban landscape.

From the various extractions and leaching studies completed on the CM, it was concluded that the material is rich in N, P and K. Due to the high amount of N, P, and K in the CM (KCl N-6420 \pm 2120 mg N/dry kg, M3P-1130 \pm 192 mg P/dry kg, M3K-1620 \pm 66.8 mg K, dry kg), the material would be very beneficial as a land application organic fertilizer. From the non-continuous column studies, aging study, and dry leach study, the material has been shown to have long-term slow leach abilities. Due to this, the material could be used for slow release, long-term fertilization. The leaching trends of the material over multiple years' worth of applied water make it a beneficial slow release material that could be used with the intent to last for more than one year. The column studies, both continuous and non-continuous, showed that the material does not need to be applied at amounts over 10% (dry mass basis) to get beneficial amounts of nutrient release/leaching.

For practical purposes, 15% (dry mass) CM could be high as a land application since column studies containing 15% CM with soils leached approximately 80 mg N, 0.55 mg P, and 5 mg K, respectively with 1 meter of applied water, which is comparable to one year of rainfall. The leached P was decreased by over 50% when a 75% WTR:

CM ratio was used. The P extraction concentrations from mixtures of WTR/CM show that the CM could be applied with higher or lower masses based on the P available to plants. Using the Mehlich III extractable P concentrations and certain plant P uptake values, the amounts of CM and CM/WTR mixtures needed for land application were calculated. The calculated application amounts are listed in Table 8-1.

Table 8- 1. The plant phosphorus uptake, and calculated CM and CM/WTR mixture application rates needed to supply the necessary plant available P. 1 dry ton =1000 kg.

Crop	Crop P Uptake (kg/ha)	Needed 100% CM Application (tons/ha)	Needed 35% WTR/CM Application (tons/ha)
Corn*	25	22.8	127
Barley**	20	18.3	101
Soybean**	29	26.5	147
Wheat**	24	21.9	121
Grass-Legume Hay**	30	27.4	152

* Crop uptake values (Shober and Sims 2003)

** Crop uptake values (A.L. Shober et al. 2012)

Table 1 shows that the CM alone provides adequate P amounts, with smaller amounts of CM if it is not mixed with WTR. As more WTR is added to the mix, a larger amount of CM can be applied and still supply the correct amount of P for crop uptake. Adding more WTR could be beneficial for land application mixtures since biosolids are typically applied based on N needs. With more available mixture mass, a higher amount of N can be applied, while still providing only the necessary P requirements.

Based on the P decrease with the WTR incorporation, it is concluded that WTR is a successful CM amendment for P mitigation, and the higher the ratio of WTR: CM, the more effective the P mitigation.

Biochar and zeolite proved to be effective in batch studies to have beneficial ammonium uptake effects, however were ineffective in column studies. The continuous

columns containing the biochar and zeolite at 20 and 30%: CM (dry mass) did not produce significant changes to the N leached from the columns. It is possible that higher percentages of these soil amendments would have been more successful at mitigating the N leached from the columns.

The dry leach study showed that smaller dried CM pieces with comparable masses, but larger total surface areas did not produce higher amounts of N, P and K leaching than larger sized CM pieces. The sizes did not make statistically significant differences. The study did show, however, that the material maintains its shape fairly well, and leaches consistently over time. The aging study also demonstrated the ability for the CM to maintain its nutrient content for over 2 months time. From these experiments it was concluded that the CM has a long-term leaching ability that could be beneficial as a fertilizer through slow release techniques.

8.2 Recommendations for DC Water

The possibility of the CM being incorporated into BMP systems such as bioretention cells was originally posed as a possible option for material reuse. However, this option does not appear practical. Due to the high amount of nutrients leaching from the material, only a very small amount of the material could be used to help fertilize the plants in the cell bed. The amount that would be beneficial would be much less than 10% (dry mass basis), as this amount allowed for excessive N and P leaching levels in column studies. With such a small amount of CM incorporation, installation costs would most likely be higher than the benefits of reusing the small amount of material.

The aging study showed how the material differs in both its physical, and nutrient leaching abilities, when it is dry vis-à-vis wet. When the material is initially

sampled, it is moist and pliable; the material is almost like “play dough” and difficult for water to penetrate through if it is in a compact shape or layer. When the material is dried for more than 48 hours it becomes extremely hard and difficult to crush; a mallet was used to crush the material for this project. The material typically dries into smaller pebble/rock like pieces. These different sized pieces exhibited little breakdown over time from the dry leach study and allowed small particulate pieces, of about 1 mm in diameter, to break off into the sampling bottles when water was applied. From these characteristics, it is predicted that after dried, the small pellets can be used as fertilizer applications that provide slow nutrient release to surrounding plants.

After testing the CM with the three-soil amendments, WTR, zeolite, and biochar, it was concluded that the WTR was effective at P mitigation, and zeolite and biochar were possibly effective, but needed to be studied further for N mitigation. The WTR was successful at mitigating P at the three ratios- 35%, 50%, and 75% (dry mass basis) in both batch study experiments and column studies. The 75% WTR: CM ratio was the most efficient at reducing P in the column leachate and on a volume basis would not be unrealistic. While the percentage sounds high, the amount is not impractical when the moisture content of the CM is taken into consideration. Due to this, the 75% WTR: CM (dry mass) ratio is recommended as the most efficient P mitigation from the CM if P mitigation is a goal for the material usage. The material will still leach P if mixed with the WTR, however it will be a reduced level, and more practical for land application in an area with high N and P runoff concentrations.

DC Water is interested in profitably marketing the CM. The material is very nutrient dense and would be beneficial for public use as a fertilizer and soil enrichment

amendment. The material packaging would need to be taken into consideration. As stated previously, the material acts very differently when it is wet compared to when it dries. For marketing purposes, it would be beneficial to dry out the material first, whether it is dried naturally, or from a heat application. Natural drying would probably be the most practical method, especially since the material is produced at such large daily quantities. A concern with the material from this project for manufacturing purposes is the odor it gives off. The CM may have to be mixed with another material(s) such as soil, woodchips or sand to help dilute the possibility of such strong scent. In addition, the material should be mixed with other materials since it is so nutrient dense; if it were to be added with soil at large amounts it could be releasing high amounts of N and P that could easily runoff and contribute to the negative environmental impacts of excess N and P.

8.3 Recommendations for Future Research

The work from this project showed that the CM has many beneficial nutrient leaching properties. However there are numerous opportunities for future research and development with the CM. The following research possibilities are recommended to further improve the knowledge on the leaching of the CM and how to use it beneficially throughout a (sub)urban landscape:

1. More long-term aging/leaching studies with mixtures of CM and WTR, zeolite and biochar should be completed to get an idea of how the mixtures age and leach when soil amendments and CM are allowed to age together. It is predicted that the soil amendments will mitigate a high amount of the nutrients that would otherwise leach

from the dried CM. In addition, studying the material over a longer time frame will allow for a better prediction of the material leaching lifespan.

2. Pot studies should be completed to compare plant growth using CM and commercial fertilizer to see which provides better fertilization to the plants. This study should be completed for at least 6 months to see short term and long-term effects.
3. Non-continuous column studies with a smaller percentage of CM mixed with the three soil amendments: WTR, biochar, and zeolite, should be completed to get a more realistic view of the material leaching with soil amendments. The non-continuous column studies that were completed in this study were done with 15% (dry mass) CM which gave insight into the material nutrient leaching, however was also not a realistic amount of CM to be used to relate to field applications. It is recommended that percentages such as 2.5-5% be used to get a more realistic, practical view of the material leaching.
4. A possible field study should be completed with the CM vis-à-vis a controlled plot to obtain a realistic idea of the land application benefits of the CM. It would be beneficial to use both the moist and dried CM pellets in different plots, along with mixtures of the CM and soil amendments to see if they provide different nutrient amounts to the land/vegetation.
5. An economic study should be completed to evaluate the benefits of manufacturing the CM as a public available fertilizer/organic soil amendment. Since the material is produced at such high mass quantities, it needs to make sense economically for DC

Water to take the extra steps to develop the material as a commercialized fertilizer rather than continuing to land apply it to agricultural lands and landfills.

Appendix A

Table A- 1. The nutrient extraction data for the CM biosolids throughout the project.

Date	M3P	CaCl ₂ P	Oxalate P	Oxalate Fe	Oxalate Al	PSI	KCl N	M3K
6/10/15							2131.3 ± 93.4	
6/24/15		8.11 ± 1.85						
6/30/16		3.35 ± 0.32						
7/10/15			23100 ± 23.1	87600 ± 87.6	2890 ± 2.89	0.446		
7/13/15	1327 ± 146	3.57 ± 0.25						
7/16/15			33100 ± 1210	88800 ± 3970	3920 ± 286	0.616 ± 0.015	6922.8 ± 144.6	
7/29/15			36300 ± 1010	92400 ± 3490	3950 ± 191	0.651 ± 0.010	7160.3 ± 46.6	
12/15/15	1095 ± 11	7.39 ± 0.14	44500 ± 1020	80700 ± 2490	2910 ± 47.8	0.926 ± 0.005	7679.9 ± 343.6	2132.4 ± 43.8
2/24/16	1370 ± 20							1620 ± 66.8
2/25/16			39800 ± 592	86100 ± 1470	2730 ± 31.1	0.781 ± 0.018		
4/14/16	894 ± 26	4.42 ± 0.23						
4/25/16							7250 ± 55.1	
4/26/16							7730.3 ± 55.1	
6/17/16	1020 ± 56.2	4.35 ± 0.23					7360 ± 344	

Table A- 2. The pH, moisture content, and LOI data for the CM.

Date	pH (2:1)	pH (3:1)	Moisture Content (%)	LOI (%)
6/10/15			67.9 ± 0.3	
6/29/15	7.99 ± 0.02	8.04 ± 0.01		56 ± 0.06

Appendix B

Table B- 1. Nutrient extraction data for 100% CM, and the various ratios of WTR:CM (dry mass basis). All extraction data is in mg per dry kg of mixture. Stdev is the standard deviation for the triplicate samples that were completed.

Sample	WEP (mg/kg)	stdev	M3P (mg/kg)	stdev	KCl N (mg/kg)	stdev	M3K(mg/kg)	stdev
100% CM	7.39	0.14	1094.49	10.50	7679.88	343.56	2132.38	43.85
7.5% WTR	7.08	0.29	625.40	16.17	7672.86	245.95	2148.91	11.73
15% WTR	6.60	0.60	555.86	11.97	6712.93	329.25	2322.39	17.06
25% WTR	4.82	0.48	417.04	64.06	7137.96	344.18	2347.17	7.80
35% WTR	4.08	0.54	198.44	56.31	6164.39	160.86	2517.55	347.87
50% WTR	3.62	0.27	82.54	18.74	7258.98	1197.24	2336.84	21.09
60% WTR	3.94	0.46	86.86	17.26			329.51	27.78
75% WTR	2.14	0.40	94.01	2.84			306.69	10.74

Table B- 2. Ammonium oxalate extraction data for 100% CM, and the various ratios of WTR:CM (dry mass basis). All extraction data is in mg per dry kg of mixture. Stdev is the standard deviation for the triplicate samples that were completed.

Sample	OX Al(mg/kg)	stdev	OX Fe(mg/kg)	stdev	OX P(mg/kg)	stdev	PSI	stdev
100% CM	116.43	1.91	3226.97	99.58	1781.29	40.98	0.926	0.005
7.5% WTR	9125.88	1439.22	86870.10	15462.01	49308.14	6973.84	0.844	0.058
15% WTR	11926.89	4298.33	88160.88	16467.05	48404.58	8492.76	0.773	0.072
25% WTR	16457.56	704.52	74607.60	4619.87	41305.25	2201.92	0.685	0.015
35% WTR	21627.17	212.98	72026.02	4758.44	41434.32	1024.53	0.641	0.045
50% WTR	39743.39	4609.63	61570.63	3694.00	36387.34	821.76	0.457	0.030
60% WTR	39485.23	1795.68	63377.74	1746.15	39227.08	905.67	0.487	0.010

75% WTR	51760.64	3247.56	54987.61	2156.04	35806.48	1210.59	0.399	0.025
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Appendix C

Table C- 1. Total Column effluent TP and TN concentration data for the continuous columns containing the sandy loam and loamy sand soils.

Date	Meters Applied	Sandy Loam Soil		Loamy Sand Soil	
		TP (mg/L)	TN (mg/L)	TP (mg/L)	TN (mg/L)
9/9/15	0.10	0.06	4.554	0.06	4.333
9/9/15	0.30	0.05	0.702	0.05	0.763
9/9/15	0.61	0.06	0.902	0.06	0.454
9/9/15	0.91	0.05	1.504	0.06	0.671
9/9/15	1.22	0.06	1.178	0.06	1.122
9/9/15	1.52	0.06	0.823	0.07	0.902
9/9/15	1.83	0.10	0.651	0.08	0.535
9/9/15	2.13	0.07	0.200	0.09	0.721
9/10/15	7.92	0.16	0.741	0.14	0.593
9/11/15	14.63	0.15	0.9885	0.12	0.6335
9/14/15	37.19	0.25	0.257	0.11	0.6325
9/16/15	52.73	0.16	0.07361	0.06	0.1293
9/18/15	66.75	0.06	0.1155	0.04	0.056
9/21/15	87.63		0.2404	0.04	0.02718
9/23/15	96.32	0.14	0.2779	0.03	0.08612
9/25/15	105.00		0.1183	0.03	0.03224
9/28/15	113.69			0.02	0.03956
9/30/15	122.39			0.03	0.02611

Table C- 2. Total Column effluent TP and TN concentration data for the continuous column containing 10% CM (dry mass basis) and Sand.

Date	Meters Applied	10% CM/Sand	
		TP (mg/L)	TN (mg/L)
7/23/15	0.10	4.13	561.25
7/23/15	0.30	0.70	130.23
7/23/15	0.61	0.40	65.93
7/23/15	0.91	0.29	27.24
7/23/15	1.22	0.24	21.56
7/23/15	1.52	0.24	17.96
7/23/15	1.83	0.27	11.06
7/23/15	2.13	0.24	9.02
7/24/15	7.92	0.56	2.05
7/26/15	14.63	0.90	1.88
7/27/15	37.19	1.12	1.73
7/28/15	52.73	1.21	1.75
8/3/15	66.75	1.11	1.08
8/5/15	87.63	0.80	1.24
8/7/15	96.32		0.36
8/10/15	105.00	0.71	0.13
8/13/15	113.69	0.64	0.08

Table C- 3. Total Column effluent TP and TN concentration data for the continuous columns containing 10% CM (dry mass basis) and loamy sand and sandy loam soil.

		10% CM/Loamy Sand	10% CM/Sandy Loam
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Date	Meters Applied	TP (mg/L)	TN (mg/L)	TP (mg/L)	TN (mg/L)
10/28/15	0.10	1.93	279.8	2.19	275.9
10/28/15	0.30	0.36	111.0	0.28	101.9
10/28/15	0.61	0.26	66.2	0.24	59.0
10/28/15	0.91	0.17	41.7	0.17	36.6
10/28/15	1.22	0.23	33.5	0.57	37.6
10/28/15	1.52	0.21	21.0	1.38	23.2
10/28/15	1.83	0.21	17.0	0.41	20.8
10/28/15	2.13	0.33	11.7	0.38	14.0
10/29/15	7.92	0.97	2.4	1.00	2.4
10/30/15	14.63	1.64	0.9	1.74	1.1
11/2/15	37.19	1.98	0.6	2.29	0.6
11/4/15	52.73	1.87	0.3	2.72	0.2
11/6/15	66.75	2.48	0.3	2.40	0.4
11/9/15	87.63	1.41	0.1	1.87	0.2
11/11/15	96.32	1.80	0.3	1.86	0.2
11/13/15	105.00	0.75	0.5	0.50	0.7
11/16/15	113.69	0.65	0.4	1.23	0.4
11/18/15	122.39	0.72		0.70	

Table C- 4. Total Column effluent TP and TN concentration data for the continuous column containing 15% CM (dry mass basis) and Sand.

Date	Meters	15% CM/Sand	
		TP (mg/L)	TN(mg/L)
7/23/15	0.10	6.61	772.00
7/23/15	0.30	1.46	166.63

7/23/15	0.61	1.01	75.20
7/23/15	0.91	0.74	45.46
7/23/15	1.22	0.69	36.28
7/23/15	1.52	0.54	28.02
7/23/15	1.83	0.69	21.90
7/23/15	2.13	0.58	14.01
7/24/15	7.92	0.76	3.47
7/26/15	14.63	1.31	1.01
7/27/15	37.19	1.56	2.79
7/28/15	52.73	1.70	1.13
8/3/15	66.75	1.29	1.39
8/5/15	87.63	1.10	3.08
8/7/15	96.32	0.93	1.23
8/10/15	105.00	1.15	0.22
8/13/15	113.69	1.00	0.17

Table C- 5. Total Column effluent TP, TN and TK concentration data for the continuous columns containing 15% CM (dry mass basis) and loamy sand and sandy loam soil.

Date	Meters	15% CM/ Loamy Sand Soil			15% CM/ Sandy Loam Soil		
		TP (mg/L)	TN (mg/L)	TK (mg/L)	TP (mg/L)	TN (mg/L)	TK (mg/L)
9/30/15	0.10	1.71	588.50	31.90	1.78	480.50	30.30
9/30/15	0.30	0.55	194.20	15.80	0.86	175.85	14.00
9/30/15	0.61	0.46	14.44	10.40	1.00	97.35	10.50
9/30/15	0.91	0.72	36.69	8.19	0.97	44.38	7.06
9/30/15	1.22	0.74	53.85	5.51	0.67	39.43	4.30
9/30/15	1.52	0.58	5.28	3.32	0.86	27.43	4.91

9/30/15	1.83	0.72	21.84	6.63	0.97	22.47	8.93
9/30/15	2.13	0.74	20.23	4.27	1.02	16.42	3.87
10/2/15	7.92	1.76	1.21	0.42	0.86	6.02	0.76
10/5/15	14.63	1.78	0.35	0.87	2.00	0.23	6.66
10/7/15	37.19	2.01	0.37	0.52	2.47	0.56	0.52
10/9/15	52.73	1.61	0.47		1.04	0.51	
10/12/15	66.75	1.35	0.53	0.28	0.84	0.73	0.63
10/14/15	87.63	0.82	0.57		1.11	1.61	0.53
10/16/15	96.32	1.25	0.54	0.16	1.97	0.57	0.49
10/19/15	105.00	0.98	0.40		0.81	0.46	0.42
10/21/15	113.69	1.87	0.32	0.11	1.82	0.42	0.43
10/23/15	122.39		0.41	0.11		0.36	0.43

Table C- 6. Total Column effluent TP, TN and TK concentration data for the continuous columns containing 15% CM (dry mass basis), various ratios of WTR (dry mass basis to CM) and loamy sand soil.

Date	Meters	Loamy Sand +15% CM+ 35% WTR			Loamy Sand + 15% CM+ 50% WTR			Loamy Sand + 15% CM+ 75% WTR		
		TP (mg/L)	TN (mg/L)	TK (mg/L)	TP (mg/L)	TN (mg/L)	TK (mg/L)	TP (mg/L)	TN (mg/L)	TK (mg/L)
1/6/16	0.10	1.50	624.600	37.80	1.65	546.000	34.100	1.41	502.600	41.300
1/6/16	0.30	0.50	164.500	16.60	0.47	151.500	14.900	0.40	166.800	17.200
1/6/16	0.61	0.33	107.800	11.50	0.26	91.270	9.630	0.21	82.400	10.700
1/6/16	0.91	0.26	95.050	8.04	0.20	53.070	7.620	0.23	49.370	7.470
1/6/16	1.22	0.18	44.505	6.13	0.23	39.630	2.670	0.26	35.950	9.930
1/6/16	1.52	0.20	34.825	5.64	0.23	11.015	3.240	0.20	36.625	4.870
1/6/16	1.83	0.23	23.770	5.00	0.23	16.245	4.450	0.14	27.750	4.500
1/6/16	2.13	0.23	19.345	4.67	0.14	19.570	3.880	1.10	30.350	4.120
1/7/16	7.92	0.35	2.792	1.39	0.25	2.286	1.200	0.35	1.727	0.858

1/8/16	14.63	0.30	1.475	0.933	0.27	1.615	0.927	0.20	1.337	0.869
1/11/16	37.19	0.61	0.742	0.732	0.57	0.687	0.838	0.40	1.578	0.789
1/13/16	52.73	0.81	0.443	0.698	0.65	0.467	0.727	0.30	0.616	0.763
1/15/16	66.75	0.775	0.678	0.674	0.324	0.393	0.735	0.607	0.554	0.781
1/18/16	87.63	0.822	0.473	0.678	0.802	0.309	0.726	0.371	0.306	0.691
1/21/16	96.32	1.200	0.269	0.667	1.065	0.362	0.699	0.647	0.303	2.970
1/25/16	105.00	0.744	0.3148	0.674	0.698	0.3126	0.677	0.458	0.2895	0.698
1/27/16	113.69	1.442	0.3575	0.664	1.364	0.3602	0.695	0.597	0.3558	0.648
1/28/16	122.39	1.178	0.3606	0.639	0.853	0.4214	0.666	0.396	0.266	0.673

Table C- 7. Total Column effluent TP, TN and TK concentration data for the continuous columns containing 15% CM (dry mass basis), various ratios of zeolite (dry mass basis to CM) and loamy sand soil.

Date	Meters	30% Zeo:15%CM/Soil			10% Zeo:15%CM/Soil		
		TP (mg/L)	TN (mg/L)	TK (mg/L)	TP (mg/L)	TN (mg/L)	TK (mg/L)
6/29/16	0.1	0.51	365.40	115.00	3.02	473.40	71.90
6/29/16	0.3	1.52	212.73	83.70	1.13	252.25	23.80
6/29/16	0.6	3.05	109.70	73.90	0.68	121.50	14.40
6/29/16	0.9	4.57	108.08	21.00	0.44	74.80	11.10
6/29/16	1.2	6.10	63.15	13.90	0.35	64.50	14.90
6/29/16	1.5	7.62	60.40	20.50	0.37	49.00	8.33
6/29/16	1.8	9.14	50.33	14.30	0.48	37.27	19.00
6/30/16	2.1	10.67	6.06	13.70	0.44	9.36	2.88
7/1/16	7.9	39.62	2.41	5.03	0.79	2.64	2.84
7/5/16	14.6	73.15	0.98	3.21	2.41	1.75	1.15
7/6/16	37.2	185.93	2.19	1.51	3.32	2.89	1.07

7/7/16	52.7	263.65	1.58	1.48	2.38	1.73	0.61
7/8/16	66.8	333.76	1.59	4.21	2.52	2.35	0.56
7/11/16	87.6	438.15	4.21	0.86	1.66	10.16	0.59
7/13/16	96.3	481.58	1.31	0.63			

Table C- 8. Total Column effluent TP, TN and TK concentration data for the continuous columns containing 15% CM (dry mass basis), various ratios of biochar (dry mass basis to CM) and loamy sand soil.

Date	Meters	30% Biochar:15%CM/Soil			10% Biochar:15%CM/Soil		
		TP(mg/L)	TN(mg/L)	TK(mg/L)	TP(mg/L)	TN(mg/L)	TK(mg/L)
7/6/16	0.1	0.5	538.6	87.8	6.8	303.6	49.6
7/6/16	0.3	1.5	264.8	40.7	1.1	209.0	20.2
7/6/16	0.6	3.0	144.5	22.5	0.6	80.5	18.2
7/6/16	0.9	4.6	80.3	17.3	0.5	55.8	10.0
7/6/16	1.2	6.1	59.8	12.6	0.5	42.5	8.0
7/6/16	1.5	7.6	41.1	6.0	0.3	48.3	8.8
7/6/16	1.8	9.1	32.1	7.9	0.4	37.8	7.6
7/6/16	2.1	10.7	32.5	5.7	0.3	22.2	4.9
7/7/16	7.9	39.6	3.4	1.4	0.6	3.4	1.3
7/8/16	14.6	73.2	2.8	0.7	1.1	2.8	2.6
7/11/16	37.2	185.9	6.3	0.9			
7/13/16	52.7	263.7	0.8	0.6	0.4	0.9	0.4
7/15/16	66.8	333.8	1.0	0.4	1.4	1.8	0.5
7/18/16	87.6	438.2	1.5	0.4	1.5	0.6	0.4
7/20/16	96.3	481.6	0.6	0.4	0.8	0.5	0.4
7/22/16	113.7	611.9	0.5		1.0	0.4	0.5
7/25/16	122.4	0.0	1.0	0.4	1.1	0.4	0.4

Table C- 9. Total Column effluent TP, TN and TK concentration data for the continuous columns containing 15% CM (dry mass basis) with Sand and Loamy Sand Soil.

Date	Meters	Duplicate 15%CM/Loamy Sand			Duplicate 15%CM/ Sand		
		TP (mg/L)	TN (mg/L)	TK (mg/L)	TP (mg/L)	TN (mg/L)	TK (mg/L)
3/2/16	0.1	3.85	482.7	31.90	2.39	504.80	43.3
3/2/16	0.3	0.73	51.75	15.80	0.83	86.20	14.6
3/2/16	0.6	0.37	77.81	10.40	0.73	31.82	14.1
3/2/16	0.9	0.32	53.95	8.19	0.73	26.85	6.46
3/2/16	1.2	0.29	32.395	5.51	0.58	13.71	4.36
3/2/16	1.5	0.25	27.075	3.32	0.48	31.78	4.53
3/2/16	1.8	0.30	14.845	6.63	0.68	21.68	6.62
3/2/16	2.1	0.34	12.63	4.27	0.65	8.56	4.18
3/3/16	7.9	0.53	2.408	0.42	0.94	1.39	4.81
3/4/16	14.6	0.63	2.181	0.870	1.12	1.01	1.63
3/7/16	37.2	1.32	1.146	0.517	0.83	0.46	1.74
3/9/16	52.7	2.07	1.102		1.37	0.45	1.5
3/11/16	66.8	5.02	0.4653	0.280	1.27	0.25	1.94
3/14/16	87.6	1.47	0.4287		2.06	0.47	1.7
3/17/16	96.3	7.27	2.953	0.157	1.35	0.30	1.37
3/18/16	105.0	1.78	0.5494		1.50	0.27	1.53
3/21/16	113.7	7.31	4.735	0.111	1.02	1.39	1.6
3/23/16	122.4	5.55	1.69	0.110	0.78	0.22	1.13

Table C- 10. Total phosphorus speciation data for various continuous column studies.

Date	Water Treated	TP	Phosphate	Organic P	Particulate P
	(Meters)	mg/L	mg/L	mg/L	mg/L
Loamy Sand					
9/9/15	0.914	0.055	0.0295	0.000	0.044
9/9/15	1.219	0.059	0.0358	0.000	0.035
Sandy Loam					
9/9/15	0.9144	0.054	0.0310	0.000	0.033
9/9/15	1.2192	0.059	0.0340	0.000	0.041
15% CM & Sand Column					
7/23/15	0.30	1.458	0.142	0.333	0.98
7/23/15	0.91	0.738	0.227	0.133	0.378
7/24/15	7.62	1.292	0.945	0.000	0.397
8/10/15	131.67	1.151	0.083	0.000	1.083
15% CM & Loamy Sand					
9/30/15	0.10	1.706	0.196	1.009	0.50
9/30/15	0.61	0.738	0.052	0.196	0.490
10/14/15	87.63	0.820	0.540	0.273	0.007
15% CM & Sandy Loam					
9/30/15	0.10	1.777	0.148	1.084	0.545
9/30/15	0.61	1.004	0.165	0.259	0.580
10/7/15	37.19	2.470	1.472	0.884	0.114
10/16/15	96.32	1.966	1.346	0.081	0.539
35% WTR: 15%CM/Loamy Sand Soil					
1/6/16	0.10	0.10	0.130	0.864	0.50
1/6/16	0.30	0.30	0.054	0.300	0.15

1/13/16	52.7	52.7	0.787	0.000	0.05
1/21/16	96.3	96.3	1.215	0.000	0.05
50% WTR: 15%CM/Loamy Sand Soil					
1/6/16	0.10	0.10	0.172	0.940	0.54
1/6/16	0.30	0.30	0.046	0.275	0.15
1/13/16	52.7	52.7	0.638	0.000	0.03
1/21/16	96.3	96.3	0.885	0.113	0.07
75% WTR: 15%CM/Loamy Sand Soil					
1/6/16	0.10	0.10	0.113	0.747	0.55
1/6/16	0.30	0.30	0.020	0.249	0.13
1/13/16	52.7	52.7	0.177	0.012	0.11
1/21/16	96.3	96.3	0.547	0.079	0.02
Duplicated 15% CM & Loamy Sand Soil					
3/2/16	0.10	3.85	0.328	1.795	1.72
3/11/16	96.3	5.02	0.701	0.091	4.23
Duplicated 15% CM & Sand					
3/2/16	0.10	2.50	0.50	1.98	0.02
3/2/16	0.30	0.73	0.20	0.47	0.05
3/11/16	96.3	1.27	0.78	0.00	0.45
30% Biochar: 15%CM/Loamy Sand Soil					
7/6/16	0.10	4.48	1.15	2.735	0.59
7/6/16	0.30	0.90	0.23	0.441	0.23
7/11/16	52.7	1.59	1.48	0.000	0.69
10% Biochar: 15%CM/Loamy Sand Soil					
7/6/16	0.10	6.84	0.328	0.218	6.29
7/6/16	0.30	0.55	0.137	0.348	0.07
7/18/16	96.3	1.50	0.701	0.322	0.48

30% Zeolite : 15%CM/Loamy Sand Soil					
6/29/16	0.1	3.32	0.14	2.049	1.13
6/29/16	0.6	0.68	0.10	0.419	0.17
7/11/16	87.6	0.93	0.80	0.041	0.09
10% Zeolite : 15%CM/Loamy Sand Soil					
6/29/16	0.1	6.84	0.328	0.218	6.29
6/29/16	0.6	0.55	0.137	0.348	0.07
7/11/16	87.6	1.50	0.701	0.000	0.48

Table C- 11. Total phosphorus speciation data for various continuous column studies.

Date	Water Treated	TP	Phosphate	Organic P	Particulate P
	(Meters)	mg/L	mg/L	mg/L	mg/L
10% CM & Sand					
7/23/15	0.30	0.704	0.126	0.180	0.398
7/23/15	0.91	0.287	0.028	0.072	0.186
7/24/15	7.62	0.558	0.543	0.000	0.016
8/10/15	131.67	0.707	0.064	0.000	0.668
10% CM & Loamy Sand					
10/28/15	0.10	1.929	0.085	0.908	0.936
10/28/15	0.30	0.363	0.038	0.137	0.189
11/16/15	113.69	0.65	0.133	0.058	0.459
11/18/15	122.39	0.71	0.083	0.074	0.561
10% CM & Sandy Loam					
10/28/15	0.10	2.187	0.094	0.917	1.176

10/28/15	0.30	0.277	0.044	0.130	0.103
11/16/15	113.69	8	0.075	0.048	1.106
11/18/15	122.39	1	0.058	0.098	0.544

Table C- 12. Total phosphorus speciation data for various non-continuous column studies

	Meters	TP	Phosphate	Organic P	Particulate P
Date		mg/L	mg/L	mg/L	mg/L
Sandy Loam Soil					
4/18/16	22.3	0.29	0.07	0.020	0.19
5/2/16	44.2	0.10	0.06	0.007	0.030
6/6/16	95.4	0.05	0.03	0.018	0.001
6/16/16	117.3	0.063	0.02	0.024	0.017
15% CM & Sandy Loam Soil					
4/18/16	22.3	6.00	1.80	1.945	2.25
5/2/16	44.2	4.52	1.56	1.680	1.280
6/6/16	95.4	5.19	1.94	2.101	1.148
6/16/16	117.3	5.714	2.31	2.729	0.672
15% CM and Sand					
4/18/16	22.3	7.44	1.74	1.881	3.82
5/2/16	44.2	4.43	0.46	0.462	3.506
6/6/16	95.4	3.10	1.64	1.771	0.000
6/16/16	117.3	4.495	1.43	2.986	0.080

Table C- 13. Total nitrogen speciation data for various continuous column studies.

Date	Meters	TN	NH4	Nitrite	Nitrate	Organic N
		mg/L	mg/L	mg/L	mg/L	mg/L
Loamy Sand Soil						
9/9/16	0.1	4.333	0.35	0.05	3.043	0.89
9/9/16	0.3	0.763	0.52	0.01	0.149	0.08
Sandy Loam Soil						
9/9/16	0.1	4.554	0.41	0.15	2.979	1.02
9/9/16	0.3	0.702	0.29	0.10	0.119	0.20
15% CM + Sand						
7/23/15	0.1	772.00	465.15	0.050	0.0	306.80
7/23/15	0.61	75.20	72.65	0.010	0.0	2.54
7/24/15	7.62	3.47	0.99	0.006	0.0	2.48
8/10/15	131.67	1.23	0.18	0.003	0.0	1.05
15% CM & Loamy Sand Soil						
9/30/15	1.22	53.85	16.753	0.006	0.0	37.09
9/30/15	1.83	21.835	16.176	0.000	0.0	5.66
10/9/15	52.7	0.4738	0.082	0.000	0.0	0.39
10/16/15	96.3	0.542	0.270	0.000	0.0	0.27
15% CM & Sandy Loam Soil						
9/30/15	1.22	39.43	25.216	0.006	0.0	14.21
9/30/15	1.83	22.465	8.098	0.001	0.0	14.37
10/9/15	52.7	0.5106	0.150	0.000	0.0	0.36
10/16/15	96.3	0.5732	0.241	0.000	0.0	0.33
30% zeolite: 15% CM & Loamy Sand Soil						
7/6/16	0.3	212.725	180.30	0.001	0.0	32.42
7/6/16	0.9	77.2	90.16	0.000	0.0	0.00

7/7/16	7.9	2.41	3.41	0.002	0.0	0.00
7/15/16	66.8	1.592	0.67	0.000	0.0	0.92
10% zeolite: 15% CM & Loamy Sand Soil						
7/6/16	0.3	252.25	201.46	0.000	0.0	50.79
7/6/16	0.9	74.8	99.52	0.003	0.0	0.00
7/7/16	7.9	2.638	2.66	0.002	0.0	0.00
7/15/16	66.8	2.352	0.53	0.000	0.0	1.83
30% Biochar: 15% CM & Loamy Sand Soil						
7/6/16	0.3	264.8	315.08	0.002	0.0	0.00
7/6/16	0.9	80.31	121.91	0.000	0.0	0.00
7/7/16	7.9	3.444	2.91	0.001	0.0	0.53
7/15/16	66.8	0.9948	0.52	0.000	0.0	0.47
10% Biochar: 15% CM & Loamy Sand Soil						
7/6/16	0.3	252.25	244.63	0.000	0.0	7.62
7/6/16	0.9	74.8	89.05	0.001	0.0	0.00
7/7/16	7.9	2.638	2.71	0.004	0.0	0.00
7/15/16	66.8	2.352	0.53	0.000	0.0	1.82
Duplicated 15% CM + Sand						
3/2/16	0.1	109.3	109.28	0.0	0.0	0.0
3/2/16	0.6	42.6	42.56	0.0	0.0	0.0
3/3/16	7.6	25.3	25.27	0.0	0.0	0.0
3/11/16	131.7	0.2	0.19	0.0	0.0	0.05
Duplicated 15% CM + Loamy Sand Soil						
3/2/16	0.1	86.2	114.7302	0.021	0.0	0.0
3/2/16	0.6	26.85	50.9021	0.009	0.0	0.0
3/3/16	7.6	1.3902	6.4309	0.002	0.0	0.0
3/11/16	131.7	0.2459	0.4582	0.003	0.0	0.0

35% WTR: 15% CM + Loamy Sand Soil						
1/6/16	0.61	107.800	59.337	0.017	0.0	48.45
1/6/16	0.91	95.050	54.322	0.007	0.0	40.72
1/13/16	52.7	0.443	0.230	0.002	0.0	0.21
1/25/16	105	0.3148	0.261	0.001	0.0	0.05
50% WTR: 15% CM + Loamy Sand Soil						
1/6/16	0.61	91.270	62.979	0.007	0.0	28.28
1/6/16	0.91	53.070	41.128	0.007	0.0	11.93
1/13/16	52.7	0.467	0.181	0.002	0.0	0.28
1/25/16	105	0.3126	0.123	0.001	0.0	0.19
50% WTR: 15% CM + Loamy Sand Soil						
1/6/16	0.61	82.400	66.143	0.012	0.0	16.24
1/6/16	0.91	49.370	48.352	0.005	0.0	1.01
1/13/16	52.7	0.616	0.263	0.002	0.0	0.35
1/25/16	105	0.2895	0.149	0.002	0.0	0.14

Table C- 14. Total nitrogen speciation data for various 10% CM continuous columns.

Date	Meters	TN	NH4	Nitrite	Nitrate	Organic N
		mg/L	mg/L	mg/L	mg/L	mg/L
10% CM + Sand						
7/23/15	0.1	561.25	341.80	0.054	0.00	154.85
7/23/15	0.61	65.93	60.69	0.009	0.00	5.23
7/24/15	7.62	2.05	0.67	0.005	0.00	1.37
8/10/15	131.67	0.36	0.06	0.003	0.00	0.30
10% CM & Loamy Sand Soil						
10/28/15	0.1	279.800		0.000	0.000	279.80
10/28/15	0.61	111.000	90.23	0.000	0.000	20.77

10/30/15	14.63	0.865	1.02	0.000	0	-0.15
11/2/15	37.19	0.602	0.33	0.000	0	0.28
10% CM & Sandy Loam Soil						
10/28/15	0.1	275.900	35.002	0.000	0.000	240.90
10/28/15	0.61	101.900		0.000	0.000	101.90
10/30/15	14.63	1.118	1.300	0.000	0	-0.18
11/2/15	37.19	0.568	0.458	0.000	0	0.11

Table C- 15. Total nitrogen speciation data for various non-continuous column studies.

Date	Meters	TN	NH4	Nitrite	Nitrate	Organic N
		mg/L	mg/L	mg/L	mg/L	mg/L
Sandy Loam Soil						
4/18/16	22.25	0.38	0.00	0.00	0.00	0.38
5/2/16	44.20	0.17	0.01	0.00	0.00	0.16
6/6/16	95.40	0.13	0.01	0.00	0.00	0.12
6/16/16	117.35	0.07	0.00	0.00	0.00	0.07
15% CM & Sandy Loam Soil						
4/18/16	22.25	29.45	5.92	0.00	0.00	23.52
5/2/16	44.20	15.09	0.03	0.00	0.00	15.06
6/6/16	95.40	2.32	0.18	0.00	0.00	2.14
6/16/16	117.35	2.48	0.26	0.00	0.00	2.23
15% CM and Sand						
4/18/16	22.25	14.08	3.50	0.00	0.00	10.58
5/2/16	44.20	12.69	8.39	0.00	0.00	4.30
6/6/16	95.40	3.89	1.77	0.00	0.00	2.11
6/16/16	117.35	3.84	0.99	0.00	0.00	2.85

Table C- 16. Cumulative masses of TP and TN for the continuous columns containing the loamy sand and sandy loam soils.

		Sandy Loam		Loamy Sand	
		Cumulative Mass		Cumulative Mass	
Date	Meters	TP (mg)	TN (mg)	TP (mg)	TN (mg)
9/9/15	0.10	0.00	0.23	0.00	0.22
9/9/15	0.30	0.01	0.30	0.01	0.29
9/9/15	0.61	0.02	0.43	0.02	0.36
9/9/15	0.91	0.02	0.66	0.03	0.46
9/9/15	1.22	0.03	0.84	0.03	0.63
9/9/15	1.52	0.04	0.96	0.05	0.77
9/9/15	1.83	0.07	1.15	0.07	0.93
9/9/15	2.13	0.09	1.18	0.08	1.03
9/10/15	7.92	0.49	3.07	0.43	2.54
9/11/15	14.63	1.53	10.12	1.28	7.06
9/14/15	37.19	2.42	11.02	1.67	9.29
9/16/15	52.73	3.09	11.33	1.91	9.83
9/18/15	66.75	4.34	13.76	2.80	11.01
9/21/15	87.63	4.34	15.43	3.07	11.20
9/23/15	96.32	5.37	17.54	3.29	11.85
9/25/15	105.00		18.80	3.60	12.19
9/28/15	113.69			3.83	12.62

Table C- 17. Cumulative masses of TP and TN for various continuous columns.

		15% CM & Sand	10% CM & Sand
		Cumulative Mass	Cumulative Mass

Date	Meters	TP (mg)	TN (mg)	TP (mg)	TN (mg)
7/23/15	0.10	0.330697706	38.6	0.21	28.06
7/23/15	0.30	0.476449029	55.2625	0.28	41.09
7/23/15	0.61	0.627884485	66.5425	0.34	50.97
7/23/15	0.91	0.738597801	73.36075	0.38	55.06
7/23/15	1.22	0.841675716	78.802375	0.42	58.29
7/23/15	1.52	0.923374508	83.00575	0.45	60.99
7/23/15	1.83	1.130548391	89.57575	0.53	64.30
7/23/15	2.13	1.217846478	91.677625	0.57	65.66
7/24/15	7.92	3.151893579	100.526125	1.99	70.88
7/26/15	14.63	12.46515642	107.7526563	8.42	84.30
7/27/15	37.19	17.9607745	117.5874063	12.38	90.40
7/28/15	52.73	25.0899258	122.3239563	17.48	97.76
8/3/15	66.75	52.21169701	151.6116063	40.84	120.38
8/5/15	87.63	59.87832692	173.0806563	46.41	129.02
8/7/15	96.32	66.90887701	182.4168438	46.41	131.75
8/10/15	105.00	79.1714839	184.7076588	53.94	133.15
8/13/15	113.69	89.84625823	186.5191113	60.80	134.02

Table C- 18. Cumulative masses of TP and TN for various continuous columns.

		10% CM & Loamy Sand		10% CM & Sandy Loam	
		Cumulative Mass		Cumulative Mass	
Date	Meters	TP (mg)	TN (mg)	TP (mg)	TN (mg)
10/28/15	0.10	0.10	13.99	0.11	13.80
10/28/15	0.30	0.13	25.09	0.14	23.99
10/28/15	0.61	0.17	35.02	0.17	32.83

10/28/15	0.91	0.20	41.27	0.20	38.32
10/28/15	1.22	0.23	46.29	0.28	43.96
10/28/15	1.52	0.26	49.44	0.49	47.44
10/28/15	1.83	0.33	54.53	0.62	53.67
10/28/15	2.13	0.37	56.29	0.67	55.77
10/29/15	7.92	2.84	62.37	3.22	61.98
10/30/15	14.63	14.51	68.53	15.63	69.94
11/2/15	37.19	21.50	70.65	23.71	71.94
11/4/15	52.73	22.63	71.77	24.72	72.95
11/6/15	66.75	28.17	77.31	32.92	81.14
11/9/15	87.63	29.07	78.22	34.28	82.51
11/11/15	96.32	31.18	80.32	36.12	84.35
11/13/15	105.00	36.22	85.37	43.18	91.40
11/16/15	113.69	40.47	89.62	47.02	95.24

Table C- 19. Cumulative masses of TP, TN, and TK for various continuous columns.

		15% CM & Sandy Loam			15% CM & Loamy Sand		
		Cumulative Mass			Cumulative Mass		
Date	Meters	TP (mg)	TN (mg)	TK (mg)	TP (mg)	TN (mg)	TK (mg)
9/30/15	0.10	0.09	24.03	1.52	0.09	29.43	1.60
9/30/15	0.30	0.18	41.61	2.92	0.14	48.85	3.18
9/30/15	0.61	0.33	56.21	4.49	0.21	51.01	4.74
9/30/15	0.91	0.47	62.87	5.55	0.32	56.51	5.96
9/30/15	1.22	0.57	68.78	6.19	0.43	64.59	6.79
9/30/15	1.52	0.70	72.90	6.93	0.52	65.38	7.29
9/30/15	1.83	0.99	79.64	9.61	0.73	71.93	9.28

9/30/15	2.13	1.15	82.10	10.19	0.84	74.97	9.92
10/2/15	7.92	3.35	97.45	12.12	5.33	78.06	10.99
10/5/15	14.63	17.62	99.09	59.57	18.02	80.59	17.19
10/7/15	37.19	26.32	101.06	61.41	25.11	81.89	19.01
10/9/15	52.73	30.70	103.20		31.89	83.88	19.01
10/12/15	66.75	48.29	118.44	74.64	60.20	95.04	24.89
10/14/15	87.63	56.05	129.65	78.34	65.93	99.00	24.89
10/16/15	96.32	70.94	134.00	82.05	75.36	103.10	26.08
10/19/15	105.00	79.61	138.85	86.51	85.82	107.38	26.08
10/21/15	113.69	99.11	143.37	91.10	105.89	110.85	27.27
10/23/15	122.39	121.73	147.30	95.71	121.25	115.26	28.46

Table C- 20. Cumulative masses of TP, TN, and TK for various continuous columns with WTR amendments.

		Loamy Sand +15% CM+ 35% WTR			Loamy Sand + 15% CM+ 50% WTR		
		Cumulative Mass			Cumulative Mass		
Date	Meters	TP (mg)	TN (mg)	TK (mg)	TP (mg)	TN (mg)	TK (mg)
1/6/16	0.10	0.07	31.23	1.89	0.08	27.30	1.705
1/6/16	0.30	0.13	47.68	3.55	0.13	42.45	3.195
1/6/16	0.61	0.18	63.85	5.28	0.17	56.14	4.6395
1/6/16	0.91	0.21	78.11	6.48	0.20	64.10	5.7825
1/6/16	1.22	0.24	84.78	7.40	0.23	70.05	6.183
1/6/16	1.52	0.27	90.01	8.25	0.27	71.70	6.669
1/6/16	1.83	0.34	97.14	9.75	0.34	76.57	8.004
1/6/16	2.13	0.37	100.04	10.45	0.36	79.51	8.586
1/7/16	7.92	1.27	107.16	13.99	0.99	85.34	11.646

1/8/16	14.63	3.42	117.67	20.64	2.90	96.84	18.250875
1/11/16	37.19	5.58	120.29	23.22	4.90	99.26	21.204825
1/13/16	52.73	8.97	122.14	26.15	7.65	101.23	24.258225
1/15/16	66.75	25.25	136.39	40.31	14.45	109.48	39.693225
1/18/16	87.63	30.99	139.69	45.03	20.04	111.63	44.757075
1/21/16	96.32	40.08	141.72	50.09	28.11	114.37	50.052
1/25/16	105.00	48.00	145.08	57.26	35.54	117.70	57.26205
1/27/16	113.69	63.46	148.91	64.39	50.17	121.56	64.715925
1/28/16	122.39	76.16	152.80	71.27	59.36	126.10	71.892075

Table C- 21. Cumulative masses of TP, TN, and TK the continuous column containing loamy sand soil, 15% CM (dry mass) and 75% WTR:CM.

		Loamy Sand +15% CM+ 75% WTR		
		Cumulative Mass		
Date	Meters	TP (mg)	TN (mg)	TK (mg)
1/6/16	0.10	0.07	25.13	2.07
1/6/16	0.30	0.11	41.81	3.79
1/6/16	0.61	0.14	54.17	5.39
1/6/16	0.91	0.18	61.58	6.51
1/6/16	1.22	0.22	66.97	8.00
1/6/16	1.52	0.25	72.46	8.73
1/6/16	1.83	0.29	80.79	10.08
1/6/16	2.13	0.45	85.34	10.70
1/7/16	7.92	1.35	89.74	12.89
1/8/16	14.63	2.79	99.27	19.08

1/11/16	37.19	4.18	104.83	21.86
1/13/16	52.73	5.45	107.42	25.06
1/15/16	66.75	18.19	119.05	41.46
1/18/16	87.63	20.78	121.18	46.28
1/21/16	96.32	25.68	123.48	68.78
1/25/16	105.00	30.55	126.56	76.22
1/27/16	113.69	36.96	130.38	83.17
1/28/16	122.39	41.22	133.24	90.42

Table C- 22. Cumulative masses of TP, TN, and TK for various continuous columns with zeolite amendments.

		Loamy Sand + 15% CM+ 10% zeolite			Loamy Sand + 15% CM+ 30% zeolite		
		Cumulative Mass			Cumulative Mass		
Date	Meters	TP (mg)	TN (mg)	TK (mg)	TP (mg)	TN (mg)	TK (mg)
6/29/16	0.10	0.15	23.67	3.60	0.17	18.27	5.75
6/29/16	0.30	0.26	43.85	5.98	0.27	35.29	14.12
6/29/16	0.61	0.37	62.08	8.14	0.37	51.74	25.21
6/29/16	0.91	0.43	73.30	9.80	0.45	63.32	28.36
6/29/16	1.22	0.49	82.97	12.04	0.52	72.80	30.44
6/29/16	1.52	0.54	90.32	13.28	0.57	81.86	33.52
6/29/16	1.83	0.69	101.50	18.98	0.67	96.95	37.81
6/30/16	2.13	0.75	102.90	19.42	0.74	97.86	39.86
7/1/16	7.92	2.77	109.63	26.66	2.10	104.01	52.69
7/5/16	14.63	19.96	122.07	34.85	14.56	110.97	75.56
7/6/16	37.19	31.64	132.26	38.62	30.14	118.68	80.88
7/7/16	52.73	41.62	139.53	41.19	42.52	125.29	87.10

Table C- 23. Cumulative masses of TP, TN, and TK for various continuous columns with biochar amendments.

		Loamy Sand + 15% CM+ 10% biochar			Loamy Sand + 15% CM+ 30% biochar		
		Cumulative Mass			Cumulative Mass		
Date	Meters	TP (mg)	TN (mg)	TK (mg)	TP (mg)	TN (mg)	TK (mg)
7/6/16	0.10	0.34	15.18	2.48	0.22	26.93	4.39
7/6/16	0.30	0.45	36.08	4.50	0.36	53.41	8.46
7/6/16	0.61	0.53	48.15	7.23	0.50	75.09	11.84
7/6/16	0.91	0.61	56.52	8.73	0.60	87.13	14.43
7/6/16	1.22	0.69	62.90	9.93	0.70	96.09	16.32
7/6/16	1.52	0.74	70.14	11.25	0.78	102.26	17.23
7/6/16	1.83	0.85	81.50	13.52	0.93	111.88	19.59
7/6/16	2.13	0.90	84.83	14.25	1.02	116.75	20.44
7/7/16	7.92	2.40	93.62	17.47	2.80	125.54	24.06
7/8/16	14.63	10.27	113.85	35.71	13.03	145.73	29.39
7/11/16	37.19	10.27	113.85	37.00	18.64	168.00	32.41
7/13/16	52.73	11.85	117.64	39.27	26.87	171.49	35.10
7/15/16	66.75	40.76	155.00	47.10	56.10	192.38	44.53
7/18/16	87.63	51.22	159.44	49.98	61.20	202.78	47.41
7/20/16	96.32	57.10	163.27	53.82	68.95	207.09	50.53
7/21/16	105.00	57.10	163.27	58.34	68.95	207.09	55.17
7/22/16	113.69	67.41	167.90		80.57	212.84	
7/25/16	122.39	78.76	172.62		95.07	223.29	

Table C- 24. Cumulative masses of TP, TN, and TK for the duplicated continuous columns.

		Duplicated Loamy Sand + 15% CM			Duplicated Sand + 15% CM		
		Cumulative Mass			Cumulative Mass		
Date	Meters	TP (mg)	TN (mg)	TK (mg)	TP (mg)	TN (mg)	TK (mg)
3/2/16	0.10	0.19	24.14	1.60	0.12	25.24	2.17
3/2/16	0.30	0.27	29.31	3.18	0.20	33.86	3.63
3/2/16	0.61	0.32	40.98	4.74	0.31	38.63	5.74
3/2/16	0.91	0.37	49.07	5.96	0.42	42.66	6.71
3/2/16	1.22	0.41	53.93	6.79	0.51	44.72	7.36
3/2/16	1.52	0.45	57.99	7.29	0.58	49.48	8.04
3/2/16	1.83	0.54	62.45	9.28	0.79	55.99	10.03
3/2/16	2.13	0.59	64.34	9.92	0.88	57.27	10.66
3/3/16	7.92	1.95	70.48	10.99	3.29	60.82	22.92
3/4/16	14.63	6.45	86.02	17.19	11.29	68.01	34.53
3/7/16	37.19	11.10	90.06	19.01	14.21	69.64	40.67
3/9/16	52.73	19.81	94.69	19.01	19.96	71.52	46.97
3/11/16	66.75	125.25	104.46	24.89	46.65	76.68	87.71
3/14/16	87.63	135.49	107.45	24.89	61.00	79.99	99.57
3/17/16	96.32	190.58	129.82	26.08	71.25	82.26	109.94
3/18/16	105.00	209.53	135.67	26.08	87.23	85.12	126.24
3/21/16	113.69	287.88	186.46	27.27	98.23	100.06	143.40
3/23/16	122.39	347.69	204.66	28.46	106.62	102.39	155.57

Table C- 25. Cumulative masses of TP, TN, and TK for various non-continuous columns.

	Sandy Loam Soil	15% CM & Sandy Loam Soil
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Meters Applied	TP (mg)	TN (mg)	TK (mg)	TP (mg)	TN (mg)	TK (mg)
0.30	0.01	0.18	0.29	0.15	30.87	0.94
0.61	0.01	0.24	1.04	0.21	57.44	2.40
7.32	0.48	0.98	10.78	2.17	71.52	11.31
7.62	0.49	1.01	10.92	2.29	74.45	11.54
7.92	0.50	1.05	11.22	2.54	76.39	11.85
14.63	0.79	1.24	19.27	9.61	80.82	20.00
14.94	0.81	1.27	19.40	9.88	82.02	20.16
15.24	0.83	1.29	19.66	10.34	82.76	20.45
21.95	1.18	1.51	27.39	19.19	86.51	28.04
22.25	1.19	1.53	27.54	19.49	87.21	28.18
22.56	1.21	1.55	27.79	19.84	87.90	28.38
29.26	1.52	2.01	36.67	29.15	91.08	35.97
29.57	1.53	2.02	36.79	29.47	91.28	36.13
29.87	1.55	2.03	37.02	30.01	91.49	36.42
36.58	1.83	2.19	44.45	38.74	92.43	42.16
36.88	1.84	2.20	44.47	39.15	92.70	42.17
37.19	1.85	2.21	44.51	39.81	92.91	42.27
43.89	2.19	2.33	45.68	49.00	94.66	42.76
44.20	2.20	2.34	45.70	49.23	95.30	42.78
44.50	2.21	2.34	45.73	49.51	95.63	42.83
51.21	2.50	2.42	46.78	57.61	96.58	43.19
51.5112	2.51	2.43	46.80	58.01	96.86	43.20
51.816	2.52	2.43	46.83	58.54	97.04	43.21
58.5216	2.74	2.55	47.73	61.65	97.67	43.59
58.8264	2.74	2.56	47.75	62.01	98.45	43.67

59.1312	2.75	2.57	47.81	62.58	98.99	43.73
65.8368	2.98	2.74	48.83	68.85	99.85	44.12
66.1416	2.98	2.74	48.86	69.00	100.58	44.16
66.4464	2.98	2.75	48.92	69.34	101.13	44.18
73.152	3.03	2.87	50.16	72.13	102.08	44.36
73.4568	3.04	2.88	50.21	72.33	102.78	44.37
73.7616	3.04	2.88	50.28	72.73	103.29	44.40
80.4672	3.25	3.24	51.14	74.36	104.32	44.81
80.772	3.25	3.25	51.15	74.90	104.79	44.85
81.0768	3.26	3.26	51.19	75.66	105.35	44.85
87.7824	3.46	3.52	52.30	77.80	106.65	45.17
88.0872	3.47	3.56	52.34	78.13	108.33	45.24
88.392	3.47	3.57	52.38	78.60	109.52	45.30
95.0976	3.60	3.62	53.38	79.23	110.61	45.62
95.4024	3.60	3.63	53.40	79.49	110.81	45.63
95.7072	3.61	3.64	53.44	79.51	111.01	45.67
102.4128	4.10	4.50	55.67	81.97	111.79	45.90
102.7176	4.11	4.52	55.68	82.26	112.08	45.90
103.0224	4.11	4.53	55.72	82.53	112.35	45.91
109.728	4.30	4.72	56.19	83.91	113.16	46.15
110.0328	4.31	4.73	56.21	84.27	113.88	46.16
110.3376	4.31	4.73	56.24	84.75	114.47	46.17
117.0432	4.48	4.73	56.90	86.74	115.12	46.34
117.348	4.48	4.73	56.91	87.03	115.31	46.34
117.6528	4.49	4.73	56.94	87.27	115.47	46.36
124.3584	4.65	4.73	57.76	92.34	115.47	46.56

Table C- 26. Cumulative masses of TP, TN, and TK for various non-continuous columns.

Meters Applied	15% CM & Sand			15% CM & Loamy Sand & 75% WTR:CM			
	TP (mg)	TN (mg)	TK (mg)	TP (mg)	TN (mg)	TK (mg)	Linear TP Est. (mg)
0.30	0.29	30.87	0.77	0.15	19.66	0.49	0.11
0.61	0.43	57.44	1.81	0.19	31.71	1.07	0.22
7.32	4.72	71.52	10.68	0.92	61.05	9.05	2.62
7.62	4.88	74.45	10.86	0.95	64.74	9.44	2.73
7.92	5.13	76.39	11.21	1.00	66.42	9.74	2.84
14.63	12.74	80.82	20.02	4.65	73.32	10.95	5.24
14.94	12.97	82.02	20.16	4.69	74.95	11.12	5.35
15.24	13.36	82.76	20.45	4.72	75.48	11.20	5.46
21.95	19.93	86.51	28.24	8.38	78.47	11.91	7.87
22.25	20.31	87.21	28.41	8.48	79.15	11.96	7.98
22.56	20.93	87.90	28.65	8.62	79.63	12.01	8.09
29.26	30.88	91.08	36.74	10.91	81.82	12.39	10.49
29.57	31.11	91.28	36.87	11.00	83.10	12.44	10.60
29.87	31.43	91.49	37.12	11.12	84.57	12.52	10.71
36.58	41.20	92.43	43.19				13.11
36.88	41.50	92.70	43.22				13.22
37.19	42.00	92.91	43.28				13.33
43.89	51.49	94.66	43.87				15.73
44.20	51.71	95.30	43.92				15.84
44.50	52.13	95.63	43.97				15.95
51.21	60.00	96.58	44.38				18.36
51.5112	60.34	96.86	44.40				18.47

51.816	60.71	97.04	44.43				18.58
58.5216	64.41	97.67	44.79				20.98
58.8264	64.83	98.45	44.88				21.09
59.1312	65.43	98.99	44.99				21.20
65.8368	71.23	99.85	45.51				23.60
66.1416	71.66	100.58	45.57				23.71
66.4464	72.00	101.13	45.63				23.82
73.152	75.21	102.08	46.01				26.22
73.4568	75.45	102.78	46.04				26.33
73.7616	75.96	103.29	46.10				26.44
80.4672	78.39	104.32	46.45				28.85
80.772	78.85	104.79	46.47				28.96
81.0768	79.49	105.35	46.49				29.07
87.7824	82.76	106.65	46.94				31.47
88.0872	83.22	108.33	46.97				31.58
88.392	83.86	109.52	46.99				31.69
95.0976	90.48	110.61	47.29				34.09
95.4024	90.64	110.81	47.29				34.20
95.7072	90.86	111.01	47.30				34.31
102.4128	94.03	111.79	47.48				36.71
102.7176	94.29	112.08	47.49				36.82
103.0224	94.49	112.35	47.51				36.93
109.728	96.64	113.16	47.96				39.34
110.0328	97.06	113.88	47.98				39.45
110.3376	97.53	114.47	47.98				39.56
117.0432	100.73	115.12	48.29				41.96
117.348	100.95	115.31	48.30				42.07

117.6528	101.12	115.47	48.31				42.18
124.3584	103.43	115.47	48.55				44.58

Appendix D

Table D- 1. Nitrogen speciation from CaCl₂ extractions for each sample taken throughout the aging study.

Weeks	Date	AVG CaCl ₂ TN	STDEV	CaCl ₂ NH ₄ ⁺	STDEV	CaCl ₂ NO ₂ ⁻	STDEV	CaCl ₂ ORG N	CaCl ₂ NO ₃ ⁻
		mg/kg		mg/kg		mg/kg		mg/kg	mg/kg
0	13-Jun	2754.03	33.88	3388.22	492.23	0.78	0.61	0.00	0.00
1	20-Jun	3465.00	331.51	3071.24	355.35	0.62	0.36	393.14	0.00
2	27-Jun	2227.08	203.89	2026.15	917.51	0.70	0.68	200.24	0.00
3	5-Jul	1529.92	81.32	1064.81	446.34	0.33	0.07	464.78	0.00
4	11-Jul	1759.00	371.15	1827.51	723.46	0.45	0.18	0.00	0.00
5	18-Jul	1443.17	110.78	1668.43	142.16	0.00	0.00	0.00	0.00
6	25-Jul	1793.25	337.22	1168.09	212.14	0.00	0.00	625.16	0.00
8	8-Aug	1577.92	345.00	1304.45	139.38	0.00	0.00	273.47	0.00
10	22-Aug	1607.67	231.11	1292.73	309.92	0.00	0.00	314.93	0.00
12	6-Sep	1456.25	420.91	1068.89	274.02	0.00	0.00	387.36	0.00

Table D- 2. Various nutrient extraction data for each sample taken throughout the aging study.

Weeks	Date	CaCl ₂ P	STDEV	M3P	STDEV	M3K	STDEV	KCl N	STDEV
		mg/kg		mg/kg		mg/kg		mg/kg	
0	13-Jun	4.87	0.09	372.61	20.63	297.71	8.06	7622.37	34.69
1	20-Jun	6.18	3.03	158.51	17.03	207.33	23.35	4309.87	358.06
2	27-Jun	4.03	1.27	122.27	13.50	244.00	22.91	3329.87	315.03
3	5-Jul	2.64	1.04	256.40	96.66	302.33	147.51	3053.87	139.96
4	11-Jul	3.84	1.00	260.15	67.80	278.00	79.23	3501.60	394.70

5	18-Jul			105.21	11.59	871.67	447.50	3331.67	186.02
6	25-Jul	2.38	0.24	152.62	12.99	248.00	24.43	3576.33	440.21
8	8-Aug	2.24	0.78	169.87	10.16	220.08	18.32	2978.00	134.28
10	22-Aug	2.15	0.27	164.91	14.23	247.67	51.52	2745.00	105.00
12	6-Sep	2.52	0.11	166.11	8.39	335.00	19.00	2954.00	651.99

Appendix E

Table E- 1. Total nitrogen speciation data for the dry leach study.

Meters	Group	Date	TN	STDEV	NH ₄ ⁺	STDEV	NO ₂ ⁻	STDEV	NO ₃ ⁻	STDEV	Org. N	STDEV
0.1	A	8/8/16	14.060	3.346	25.085	0.166	0.002	0.002	0.138	0.020	-11.165	3.340
0.2	A	8/15/16	36.065	15.110	38.412	17.801	0.000	0.002	0.323	0.015	-2.664	10.963
0.3	A	8/22/16	19.920	12.705	37.226	11.310	0.001	0.001	0.192	0.050	-17.499	15.506
0.4	A	8/29/16	25.727	14.338	67.071	55.260	0.001	0.001	0.254	0.010	-41.599	42.306
0.5	A	9/6/16	25.627	6.721	25.789	8.586	0.001	0.001	0.208	0.046	-0.371	1.906
0.6	A	9/12/16	22.637	4.827	14.862	3.208	0.001	0.001	0.296	0.010	7.774	3.872
0.1	B	8/8/16	37.485	26.236	45.727	28.151	0.004	0.004	0.212	0.061	-8.457	11.761
0.2	B	8/15/16	42.183	12.431	41.178	10.507	0.000	0.008	0.202	0.053	0.806	4.800
0.3	B	8/22/16	23.552	4.335	27.951	5.910	0.001	0.001	0.257	0.024	-4.656	2.110
0.4	B	8/29/16	22.353	14.209	46.574	24.479	0.001	0.001	0.185	0.024	-24.407	26.813
0.5	B	9/6/16	20.497	5.393	24.813	3.359	0.001	0.001	0.156	0.056	-4.421	4.750
0.6	B	9/12/16	22.189	9.897	22.927	7.640	0.001	0.001	0.156	0.015	-0.738	2.814
0.1	C	8/8/16	19.160	11.659	33.143	22.351	0.002	0.002	0.233	0.058	-14.217	10.756
0.2	C	8/15/16	22.625	10.267	23.138	7.484	0.000	0.003	0.269	0.067	-0.775	3.654
0.3	C	8/22/16	33.157	11.880	36.867	13.273	0.004	0.003	0.182	0.028	-3.896	2.869
0.4	C	8/29/16	16.265	16.563	38.557	55.288	0.004	0.003	0.208	0.028	-22.504	38.735
0.5	C	9/6/16	24.853	9.376	26.829	8.142	0.004	0.003	0.177	0.053	-1.980	15.734
0.6	C	9/12/16	20.057	3.189	14.276	5.871	0.004	0.003	0.156	0.024	5.777	7.172
0.1	D	8/8/16	25.818	5.834	30.273	12.701	0.002	0.001	0.227	0.078	-4.684	7.503
0.2	D	8/15/16	38.243	18.940	28.921	12.305	0.000	0.002	0.249	0.062	9.082	7.680
0.3	D	8/22/16	18.810	5.901	22.206	7.471	0.002	0.002	0.225	0.054	-3.623	2.857

0.4	D	8/29/16	19.228	3.943	54.170	35.454	0.002	0.002	0.227	0.044	-35.170	33.196
0.5	D	9/6/16	21.293	2.231	24.618	9.562	0.002	0.002	0.209	0.050	-3.326	11.021
0.6	D	9/12/16	20.288	0.934	15.122	4.074	0.002	0.002	0.179	0.010	5.164	3.188

Table E- 2. The total phosphorus speciation data for the dry leach study.

Meters	Date	Group	AVG TP	STDEV	AVG SRP	STDEV	AVG Org	STDEV	AVG PP	STDEV
0.1	8-Aug	A	0.192	0.042	0.056	0.025	0.039	0.023	0.097	0.041
0.2	15-Aug	A	0.486	0.271	0.006	0.005	0.171	0.073	0.309	0.194
0.3	22-Aug	A	0.376	0.227	0.035	0.027	0.137	0.095	0.204	0.163
0.4	29-Aug	A	0.445	0.143	0.112	0.046	0.170	0.061	0.163	0.045
0.5	6-Sep	A	1.868	2.459	0.136	0.098	0.137	0.177	1.595	2.504
0.6	12-Sep	A	0.603	0.317	0.076	0.025	0.129	0.131	0.398	0.204
0.1	8-Aug	B	0.253	0.147	0.093	0.028	0.059	0.071	0.100	0.050
0.2	15-Aug	B	0.869	0.245	0.014	0.006	0.295	0.189	0.560	0.296
0.3	22-Aug	B	0.452	0.223	0.025	0.004	0.172	0.040	0.255	0.182
0.4	29-Aug	B	0.327	0.173	0.084	0.038	0.125	0.071	0.118	0.079
0.5	6-Sep	B	3.097	2.498	0.252	0.226	0.000	0.204	2.850	2.431
0.6	12-Sep	B	0.446	0.168	0.088	0.031	0.102	0.088	0.256	0.217
0.1	8-Aug	C	0.204	0.118	0.090	0.029	0.053	0.032	0.060	0.074
0.2	15-Aug	C	0.494	0.242	0.013	0.006	0.090	0.038	0.391	0.258
0.3	22-Aug	C	0.611	0.130	0.061	0.033	0.219	0.046	0.332	0.127
0.4	29-Aug	C	0.247	0.217	0.063	0.064	0.084	0.051	0.100	0.104
0.5	6-Sep	C	0.358	0.126	0.095	0.048	0.206	0.092	0.058	0.039
0.6	12-Sep	C	0.947	0.792	0.040	0.019	0.079	0.055	0.828	0.723
0.1	8-Aug	D	0.187	0.056	0.093	0.018	0.056	0.015	0.037	0.026
0.2	15-Aug	D	0.601	0.108	0.014	0.004	0.240	0.108	0.347	0.125

0.3	22-Aug	D	0.343	0.092	0.040	0.024	0.177	0.032	0.126	0.041
0.4	29-Aug	D	0.384	0.045	0.102	0.022	0.131	0.009	0.151	0.059
0.5	6-Sep	D	2.390	2.318	0.066	0.028	1.814	1.570	0.511	0.850
0.6	12-Sep	D	0.211	0.126	0.059	0.036	0.108	0.094	0.044	0.006

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