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Pilot-Scale Study of Low-Tech Methods of Sustainable Class A Biosolids Production: A Trial Run

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PILOT-SCALE STUDY OF LOW-TECH METHODS OF SUSTAINABLE CLASS A
BIOSOLIDS PRODUCTION: A TRIAL RUN

By

Rebecca L. Green

A REPORT

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Abstract

Water resource recovery facilities can improve sustainability by producing Class A biosolids. However, many conventional methods for achieving Class A biosolids are expensive, with high maintenance and energy costs. However, low-cost, low-tech (LCLT) treatment methods are also available. The overall goal of the larger project of which this study was a part is to develop a rational and universal approach for design of LCLT processes. To achieve this goal, pilot-scale studies of LCLT processes will be performed. The goal of this portion of the project was to perform a trial run pilot-scale study. During the trial run, the pilot-scale biosolids storage boxes were constructed. Methods were also developed for monitoring the environmental conditions, and the analytical methods and associated quality control were developed. Data collected during the trial run demonstrated reasonable trends and were also used to improve the methods for application to the full pilot-scale study.

Chapter 1 Introduction

1.1 Background

Land-applied biosolids in the U.S. are classified as Class A or B. Class B biosolids are allowed to contain detectable levels of pathogens and indicator organisms (PIOs). As a result, a number of restrictions are in place on land applications of Class B biosolids to limit public exposure to these pathogens. Class A biosolids, on the other hand, must contain essentially non-detectable concentrations of PIOs and, therefore, can be land-applied or distributed without restriction. The Federal Part 503 rule specifies six alternative methods that can be used to achieve Class A biosolids, as summarized in Table 1.1. Several of these, including Alternative 5, the use of Processes to Further Reduce Pathogens (PFRPs), must be met by using technologies that are generally expensive, have high maintenance costs, and/or are energy intensive. Alternative 6, the use of processes equivalent to a PFRP, requires extensive monitoring and characterization of the biosolids to determine PFRP equivalency, which is generally evaluated and granted on a site-by-site basis. Unfortunately, many water resource recovery facilities (WRRFs), especially small WRRFs, lack the resources to implement PFRPs, or negotiate the PFRP equivalency process. Therefore, there is a great need for low-cost low-tech (LCLT) and sustainable processes for Class A biosolids production.

Table 1.1 Part 503 rule alternatives for achieving Class A Biosolids (1)

Alternative	Description
1	Thermally treated biosolids are subjected to one of four time-temperature regimes
2	Biosolids are treated in a high pH-high temperature process, and meet specific pH, temperature, and air-drying requirements.
3	Biosolids are treated in other known processes that can reduce enteric viruses and viable helminth ova.
4	Biosolids treated via unknown processes, with each batch tested for pathogen abundance.
5	Biosolids are treated in one of the Processes to Further Reduce Pathogens (PFRPs)
6	Biosolids are treated in a process equivalent to a PFRP.

Fortunately, sustainable, LCLT processes for achieving Class A status do exist. These processes include long-term lagoon storage, air drying, and cake storage (2). For example, the Metropolitan Water Reclamation District of Greater Chicago has successfully demonstrated the application of a combined long-term sludge storage/air drying process for achieving Class A biosolids (3). Despite the potential of these LCLT processes, wide spread implementation has been prevented because little is currently

known regarding the rates of inactivation of PIOs in LCLT biosolids treatment systems. Specifically, there is a lack of understanding of the impact of environmental factors and biosolids properties on PIO inactivation (2). Thus, WRRFs must use trail-and-error approaches to develop site-specific LCLT treatment processes, and demonstrate PFRP equivalency.

1.2   Scope and Objectives of Study

The overall goal of the larger project of which this research was a part is to develop a rational and universal approach to the design of LCLT Class A biosolids treatment processes. The specific objectives of the larger project include: (1) a systematic laboratory evaluation of the impact of biosolids storage temperature, organic matter and ammonia content, pH, and moisture on the inactivation of PIOs as a function of time; (2) fitting of inactivation constants to the data obtained in Objective 1 for each PIO of interest, coupled with development of a model of inactivation that can be used to determine the treatment criteria needed to achieve Class A biosolids pathogen requirements; and (3) performance of pilot-scale studies of four LCLT processes to validate the predictions based on the laboratory studies and mathematical modeling of Objectives 1 and 2, respectively.

The goal of the portion of the ongoing overall project that was covered in this study was to perform a soft-start or trial run of the planned full pilot-scale study of long-term lagoon storage to be performed as part of Objective 3 of the overall project. The purpose of performing the trial-run start to the project was to test the study set-up, sampling procedures, laboratory methods and materials, and timing. Specifically, the objectives of this study were as follows:

1. Build and test a subset of the pilot system boxes,

2. Develop and test sensor stacks,

3. Test analytical methods and perform quality control for those methods,

4. Collect trial run data and analyze it, and

5. Make suggestions for improvement before starting the full pilot-scale study.

Chapter 2   Materials and Methods

## 2.1   Pilot Study Location and Approach

The trial-run pilot-study was performed at Portage Lake Water and Sewer Authority (PLWSA) in Houghton, MI. Secondary treatment at PLWSA is provided by a conventional activated sludge system with a design wastewater flow of 3.1 million gallons per day (mgd). PLWSA currently treats the primary and secondary solids produced during treatment using mesophilic anaerobic digestion for vector attraction reduction, producing 270 dry tons Class B biosolids per year. The solids are conditioned using a cationic polymer and dewatered with a belt filter press to 16% total solids (TS). The dewatered cake is land applied and stored in an uninsulated closed shed during the winter months.

The pilot-scale study at PLWSA is focused on simulating a two stage sequential long-term lagoon storage/air drying process, with the trial-run study focused on the long-term lagoon storage stage. Long-term lagoon storage is being simulated using pilot-scale box reactors, which are described further below. To compare the effects of long-term indoor and outdoor storage, the pilot-scale tests are being performed indoors in the un-insulated storage shed at PLWSA (Treatment 1), and outdoors at PLWSA (Treatment 2). For the trial-run, one pilot box was used for each treatment, while for the full pilot-scale project, triplicate boxes will be used for each treatment. Efficacy of the treatments is largely a function of the environmental conditions in the treatment systems, and how they vary with time. Environmental factors to be monitored during the trial run included the ambient environmental conditions (temperature, precipitation, solar radiation), and the conditions known to impact PIO inactivation during long-term lagoon storage, specifically temperature, moisture content (as TS), organic content (using volatile solids (VS)), pH, volatile fatty acids (VFA), and ammonia. In addition, the levels of PIOs were monitored using fecal coliforms (FC), and coliphage.

2.2   Pilot Study Box Construction

Many criteria were considered when designing the pilot study boxes. One, each box had to be able to hold roughly two tons of biosolids (plus any precipitation that may occur), and maintain structural integrity through the full freeze/thaw cycle. Two, the boxes had to allow for drainage of water from the biosolids, as occurs in full-scale storage. Three, they also had to stay water-tight for up to two years to ensure no leachate seeps out from places other than the drainage pipe. Finally, the boxes had to be able to withstand the local environmental conditions, e.g. snow, rain, winds, and ambient temperatures ranging from -20°F to 95°F. Taking these criteria into account, the boxes were built out of exterior-grade plywood with exterior dimensions of 4' high, 4' wide, and 8' long. In addition, 2' x 4''s were used to brace the top and bottom of the boxes

(Figure 2.1). The corners were reinforced on the interior and exterior with wooden and metal chamfers, respectively. To prevent weathering, the boxes were painted with a primer and top coat on the interior and exterior (see Figure 2.1). Once the boxes were fully constructed, two wooden cross-beams were added across the tops of each box to decrease the possibility of bowing over time and to provide an attachment point for the sensor stacks so that they would stay in place as the biosolids were added and during treatment.
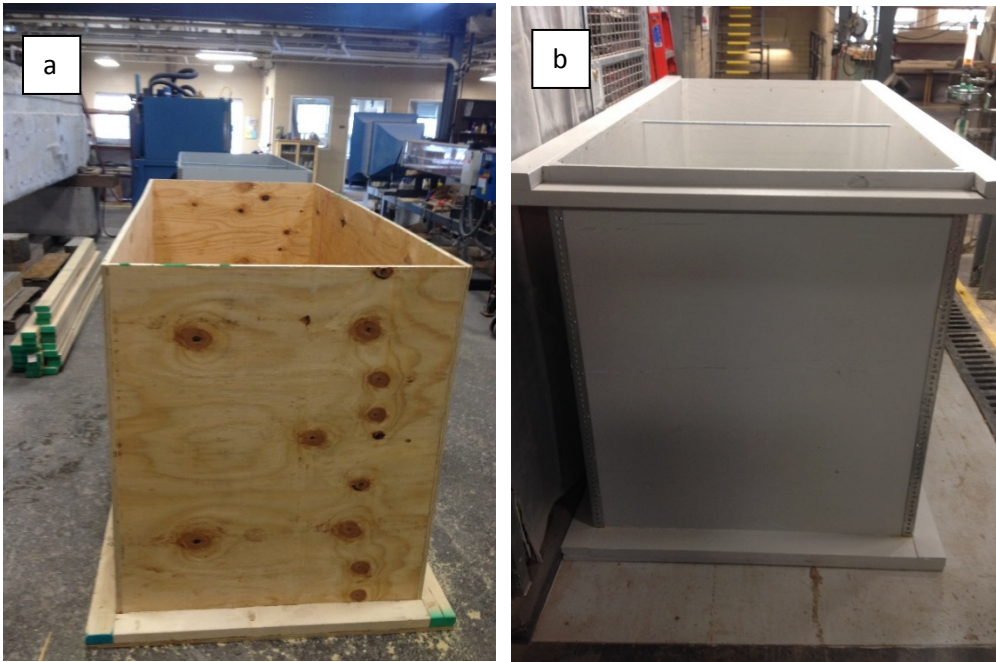
Figure 2.1. Boxes were built of (a) exterior grade plywood, and (b) then painted to protect the box from weathering, with the addition of 2x4 braces at the top and bottom to maintain structure shape

Once the boxes were built and painted, the next step was to install the liners, drainage systems, and temperature sensor stacks. First, a 1.5-2" sand layer was added to the bottom of each box before the liners were put in to ensure no tearing or catching of the liner on the bottom of the box (Figure 2.2). Next, custom, potable-grade vinyl liners (Tri-City Vinyl, Saginaw, MI) were stretched and inserted into the boxes. The liners were subsequently attached to the box frame with metal strips along the full top interior of the box (Figure 2.3a), and secured on the top of the box frame with wooden cleats. A hole was cut in the bottom of the box and liner for the drainage pipe (4 in. diameter perforated PVC drainage pipe, ASTM No. D2729 specifications), which was then installed across the bottom of the box. The drainage pipes were attached to the liners using a witch's hat attachment, vinyl glue, and hose clamps (Figure 2.3b). Once the drainage pipe was glued and secured to the liner, a layer of size 6A was added below the drainage pipe, followed by a second layer of size 6A washed gravel to roughly 1-2" above the top of the drainage pipe (Figure 2.4). Filter fabric was next added on top of the gravel to ensure only the leachate permeated through to drain out of the box (Figure 2.5). Finally, sensor stacks

were also added to support the sensors used for monitoring the temperature in the biosolids over time (see the next section for more details) (Figure 2.5).
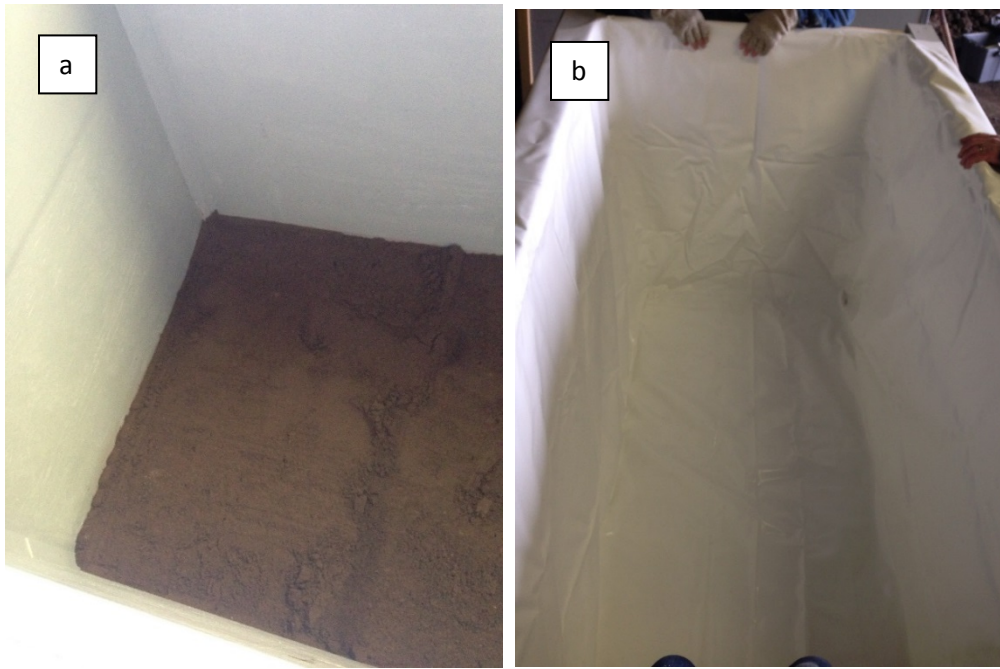
Figure 2.2. (a) A sand layer was added to the bottom of the box to prevent catching of the (b) liner on any roughness in the plywood.
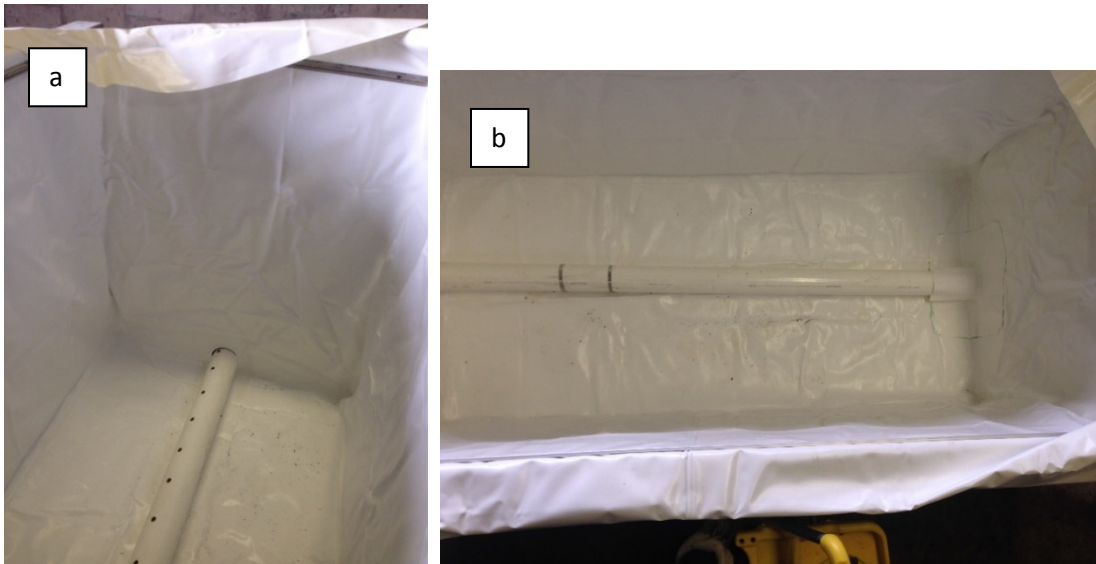
Figure 2.3. During liner installation,(a) the liner is held in position by metal strips, and (b) the drainage pipe is placed in the bottom center of the box.

Figure 2.4. To construct the drainage system, first (a) Small gravel was added below the drainage pipe, followed by (b) size 6A gravel were added for drainage.

Figure 2.5. Landscaping cloth is layered on the grave and held in place by sensor stacks.

When it was time to initiate the trial run study, the inside and outside boxes were loaded with one-week old dewatered biosolids. The bisolids were first carefully packed around the edge of the box to make sure the landscaping cloth stayed in place (Figure

2.6a). Biosolids were then shoveled into the boxes to a height of 3 ft, taking care not to damage the liners and sensor stacks (Figure 2.6b,c).

Figure 2.6. Filling the boxes. Biosolids were shoveled in to make sure the sensor stacks and liners were not harmed in the loading process.

2.3   Monitoring Methods
2.3.1 Environmental Conditions
2.3.1.1   Ambient Environmental Conditions

To aid in comparing the results from the boxes stored indoor and outdoor and resolving differences between the two treatments, the ambient environmental conditions were monitored and recorded. To do this, a Davis Wireless Vantage Pro2 Plus weather station was placed at each test location at PLWSA (i.e., one inside the uninsulated storage barn, and one outside). The conditions monitored using these stations via the included sensor suite are rainfall, temperature, humidity, wind speed, solar radiation (wavelengths of 300 to 1100 nm), and UV radiation (wavelengths of 290 to 390 nm).

2.3.1.2   In situ Biosolids Temperature

In addition to the ambient environmental conditions, the temperature in the biosolids over the course of the study is also expected to be an important factor impacting PIO inactivation. To monitor the temperature in the biosolids, two sensor stacks were fabricated for each box (Figure 2.5), with one sensor stack placed approximately at the center of the box, and the other stack near the ouside edge of the box. The stacks were designed to hold Thermochron iButtons (Embedded Data Systems, Cat. No. DS1921G-F5#), which were set to measure and record the temperature every two hours. These specific iButton sensors have an accuracy of $\pm 1.0°C$ and an operating range of -40°C to 85°C. Each sensor stack was made of a 4 ft length of rectangular cross section PVC tubing (2.5 in. width), and supported at the top and bottom of the box with wood and PVC cross braces, respectively.The iButtons were placed at 1 ft increments starting 0.5 ft above the fabric filter cloth, for a total of 3 iButtons per stack, and 6 iButtons per box. The iButtons were mounted in a clip holder (Embedded Data Systems, Cat. No. DS9094F+) that was then soldered to waterproof wire cabling (Embedded Data Systems, Cat. No. P3011). Eac iButton assembly was installed in a hole in sensor stack tube, with the sensor wires running up the center of the tube. The iButtons can hold 2048 temperature values and, thus, the data are downloaded periodically using an iButton probe cable reader (Embedded Data Systems, Cat. No. DS1402-RP3+). The ends of the iButton and reader cables are fitted into female Ethernet ends that connect using a female-female Ethernet reader to facilitate the data downloading. The probe cable reader is used in conjuction with a USB to iButton Adaptor (Embedded Data Systems, Cat. No. DS9490R#), which can be connected to a notebook or lap top computer for the data collection.

2.3.2 Physical/Chemical Parameters
2.3.2.1 Sampling Techniques

The boxes were sampled following the EPA guide for sampling dewatered sludge drying beds (4). To perform the sampling, each box was initially divided into 4 quadrants. Then, a sample location was selected in each quarter. At that location, a sanitized hand scoop was used to remove about 6 inches of biosolids, which were put aside (5). Next, a grab sample was taken at selected location in each quadrant by coring across the depth of the bed of biosolids by using a 4 ft. long, 2 cm diameter, plastic tube that had been washed and disinfected with ethanol before use. To be able to use the plastic tube to obtain a representative sample for the full depth of the box during the course of this study, the tube was used to take sequential 6 inch samples starting 6 inches below the surface of the biosolids until the tube hit the bottom of the biosolids portion of the box. All core samples were added into an autoclaved stainless steel bucket. The sampling locations were backfilled with biosolids and marked with a flag to ensure the spot was used again and avoid any pooling of water (6). The one exception to this procedure was on day zero, for which grab samples were removed from the bucket of the front-end loader used to fill the boxes by using ethanol-rinsed stainless steel scoops, with the samples then transferred to an autoclaved stainless steel bucket.

Once all of the samples from the quadrants were collected, a clean, ethanol-rinsed stainless steel hand trowel was used to mix the core material together to form a composite representative sample for the box. This was accomplished by folding the sample back onto itself several times (4, 5). A clean, autoclaved plastic scoop was then used to randomly remove composited biosolids from the bucket and fill 3 autoclaved ½ pint mason jars for laboratory testing. One of the jars was filled completely, with as little air space as possible available to minimize the impact of volatilization on the measurement of volatile acids and pH. This jar was also used for fecal coliforms and TS/VS. A second jar was filled with 20 to 30 grams of biosolids for use in the coliphage analysis. The last jar was filled with 6 grams of biosolids and immediately mixed with 35 grams of sulfuric acid to acidify the sample to a pH less than 2, and preserve the sample for analysis of ammonia.

2.2.2.2 Total and Volatile Solids

TS and VS were determined using EPA Method 1684 (7). To perform the test, approximately 25 grams (wet weight) of biosolids were placed into duplicate crucibles for each sampling location. As per EPA Method 1684 (7), quality control (QC) tests were conducted for TS and VS throughout the trial-run testing by performing an initial demonstration of laboratory capability, conducting analysis of on-going precision and recovery samples, running blanks, and analyzing duplicate samples. The MDL was also

determined by analyzing seven 30 mL aliquots of a 200 mg/L potassium hydrogen phthalate and sodium chloride solution.

2.3.2.3 pH

The pH of the biosolids was measured by mixing 10 grams (wet weight) of biosolids with 20 mL of DI water (8). The pH of the resulting solution was measured using a Thermo Scientific Orion Dual Star pH/ISE Benchtop meter and Thermo Scientific Orion 9156DJWP Double Junction Low Maintenance pH electrode. For QC, the pH meter/electrode were calibrated using a 3 point calibration, and duplicate samples were analyzed. Note that for the full-scale pilot study, the pH will be measured following EPA Method 9045D (9), which uses a 20 g (wet weight) of biosolids per 20 mL DI water ratio.

2.3.2.4 Volatile Fatty Acids and Alkalinity

Total Volatile Fatty Acids (VFA), bicarbonate alkalinity, and total alkalinity of the biosolids were determined following the two-point titration method as described by Anderson and Yang (10). To perform the test, the biosolid sample was first mixed with DI water in a 1:50 (g:mL) ratio (2 grams wet weight of sample were mixed with 100 mL of DI water) on a stir plate for 1 hour to extract the water soluble fraction (11,12). The mixture was then centrifuged for 15 minutes at 6000 rpm to separate the aqueous extract from the solids (13). A 50 mL sample of the aqueous extract was then pipetted into a 250 mL beaker containing a magnetic stir bar and capped using a rubber stopper with holes for the titration pipet and pH probe. The initial pH was then measured and the extract titrated sequentially to a pH of 5.1 and 3.5, with 0.1 N H_2SO_4. The volume of titrant needed to achieve these pHs was used in calculating the bicarbonate and total VFA concentrations using a matlab program that simultaneously solving Equations (2) and (3) in Anderson and Yang (10). This analysis was performed in duplicate for QC purposes. In addition, QC was also ensured by titrating varying known concentrations of sodium bicarbonate and sodium acetate, as well as a DI water sample.

2.3.2.5 Ammonia

Ammonia-nitrogen for the biosolids was determined colorimetrically using the Hach Company TNTplus 832 Ammonia Method 10205 (range 2-47 mg NH_3-N/L), which is equivalent to USEPA Method 350.1 for regulatory reporting. To perform the test, 5 mL of the acidified sample solution (described above in 2.3.2.1 Sampling Techniques) was diluted to 100 mL using distilled water to ensure the concentration did not exceed the range of the TNTplus 832 vials (2-47 mg NH_3-N/L). Then 6 mL subsamples of the diluted solution were prepared for analysis via distillation using Standard Method 4500-NH_3 (14). Next, 0.5 mL of the distillate was then added to each of the TNTplus 832 vials,

and the solution mixed. Subsequently, the sample absorbance was measured using a Hach DR6000 spectrophotometer via the barcode method because the spectrophotometer has a built in calibration curve for Hach vials. For QC, an initial demonstration of laboratory capability, analyses of on-going precision and recovery samples, and duplicate and blank analyses were performed. Note that after the trial run study it was learned that the 0.5 mL sample size used with the TNTplus 832 vials was incorrect, and that 0.2 mL samples should have been used.

2.3.3 Microbiological Methods
2.3.3.1   Fecal coliforms
Fecal coliforms were analyzed because they must be quantified to demonstrate compliance with the Class A biosolids pathogen requirements. Following EPA Method 1681 (15), fecal coliform concentrations were determined using the most probable number (MPN) method. A 30 gram (wet weight) sample was homogenized in 270 mL of buffered dilution water using a blender. That solution was then used to prepare 4 serial dilutions. Each dilution, was used to inoculate a set of 5 sterile tubes (10 mL of sample per tube), with each tube containing an inverted gas collection tube and roughly 2 mL of sterile A-1 media. After inoculation, the tubes were incubated at 35°C ± 0.5°C for 3 hours then flipped to get rid of any bubbles that formed during the initial incubation period. Subsequently, the tubes were incubated an additional 21 hours at 44.5°C ± 0.5°C. The number of vials with a positive result (bubbles in the inverted gas collection tube) for each dilution was recorded and used to determine the MPN. Positive and negative control samples along with spiked samples and blanks should be analyzed for QC, but this was not completed during the trial run study.

2.3.3.2   Coliphage
Coliphage are viruses that infect fecal coliforms, like *Escherichia coli*. These viruses were quantified in this study because several studies have shown that their concentration is correlated with the abundance of enteric viruses (16, 17). The coliphage preparation and analysis for this study were performed by Christa Meingast, Zhimin Song, and Tanner Keyzers. Male-specific and somatic coliphage were enumerated using the single agar layer procedure, according to EPA Method 1602 (18). The male-specific coliphage (MS2, ATCC #15597-B1) and its host (*E. coli* HS (pFamp)R, ATCC #700891) and the somatic coliphage (phi-X 174, ATCC #13706-B1) and its host (*E. coli* CN-13, ATCC #700609) were obtained from the ATCC. Frozen host cultures were prepared using the *E. coli* cultures and were used to inoculate overnight host stock cultures. Each biosolids sample was divided into two aliquots and amended with $MgCl_2$. Log-phase *E. coli* HS (pFamp)R was added to one aliquot (for quantification of male-specific coliphage), and log-phase *E. coli* CN-13 was added to the other aliquot (for quantification

of somatic coliphage). The *E. coli*-sample mixtures were warmed to 43℃ ± 1℃, mixed with tryptic soy agar containing the appropriate antibiotics for the host strain, and poured into five 150-mm petri dishes. After the agar hardened, the petri dishes were covered, inverted, and incubated for 16 - 24 hours at 36°C ± 1.0°C. The lysis zones or plaques on each of the five dishes were counted and summed to obtain the total coliphage abundance (plaque-forming units [PFU]) in a sample. Positive and negative controls were assayed with each batch of samples to determine the effect of the matrix on the method's coliphage recovery and ensure that media was free from contamination, respectively. (19)

Chapter 3 Trial Run Results and Discussion

3.1 Environmental Conditions

3.1.1 Weather Station Data

Due to software and communication problems between the weather stations and their respective consoles, it has not yet been possible to download data from the weather stations. Efforts to resolve these problems are ongoing.

3.1.2 Biosolids Temperature Data

The temperature within the biosolids was recorded every 2 hours by the iButton® sensors and downloaded every few weeks for analysis. Due to incorrect waterproofing techniques, all but two of the iButtons® stopped working during the course of the trial run. Figure 6 shows the results obtained using the two sensors that did not fail. As expected, the temperature inside the boxes increased over time consistent with the increasing ambient temperatures during this time frame (data not shown). Also as expected, the temperature recorded by the upper iButton® was consistently higher than that recorded by the deeper button.
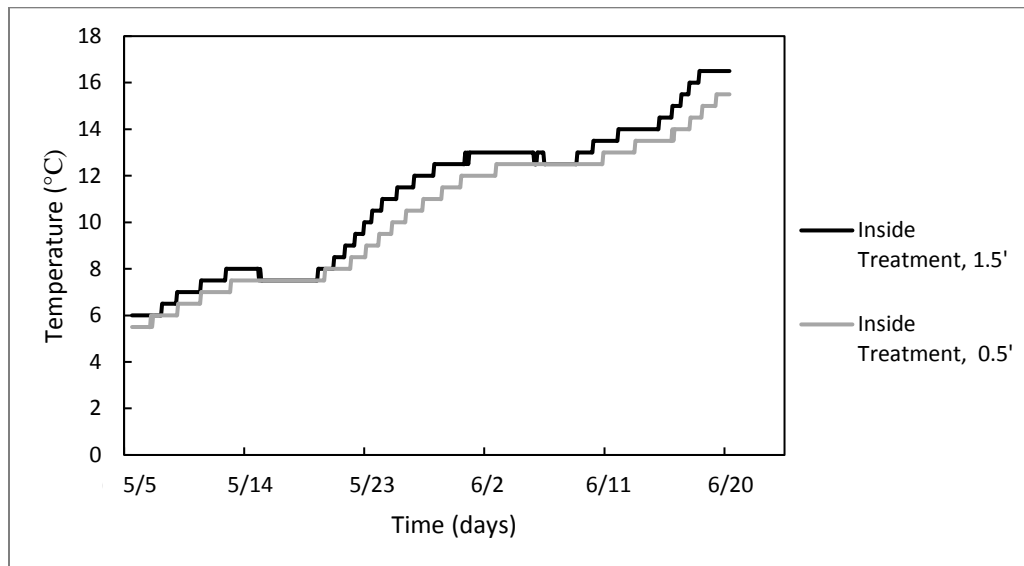
Figure 3.1. IButton results from iButtons that worked throughout testing time.

3.2 Total and Volatile Solids

TS and VS were measured in duplicate on each sampling day, as shown in Figure 3.2. As noted above, according to plant personnel and our own previous measurements, the biosolids at PLWSA are dewatered to approximately 16% TS. The initial TS levels measured in both the inside and outside boxes were 15.7% and therefore are consistent with past observations. Interestingly, no clear trend was observed in the TS levels over

time, suggesting that substantial amounts of water did not drain from the boxes and, therefore, that the remaining water is tightly bound vicinal water of hydration (20). The sampling event on day 58 followed a period of high precipitation. Therefore, it is not surprising that the TS levels decreased in the outside test bed on that day was found to be lower than average. The average TS values measured were 14.8% and 14.6% for inside and outside boxes, respectively. The average VS values measured were 65.9% and 60.7% for inside and outside boxes, respectively. The VS values, rather than diverging with time as would be expected, actually appear to converge after initial differences.
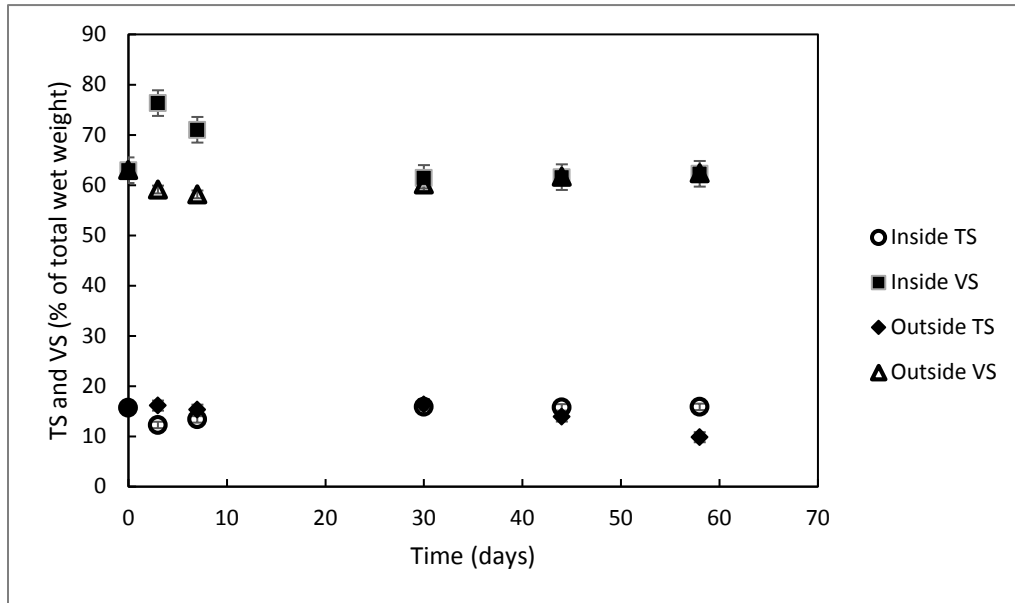

Figure 3.2.Total solids (TS) and volatile solids (VS) measured in the inside and outside test beds. Each data point represents the average concentration in duplicate samples. Error bars represent ± one standard deviation.

Following the quality control guidelines specified in EPA method 1684, duplicate sample analyses, analysis of blanks, and on-going precision and recovery (OPR) tests were performed. These quality control measures were implemented on day 30. The TS values measured in all but one set of the duplicate samples were within 10% of each other. The TS and VS concentrations measured in the blanks were consistently below their MDLs, which were 26 mg/L and 18.6 mg/L, respectively. OPR values were calculated for day 58, resulting in +42% recovery for TS and +4% for VS. The TS percent recovery is outside the acceptable criteria given by the EPA (-20% to +10% of theoretical values). Further work will be done on OPR to develop new recovery criteria.

3.3   pH

The pH was measured in duplicate samples at each sampling event. As Figure 3.3 shows, the pH measured in the inside and outside test beds started to diverge after day 30. The lower pH in the outside test bed could be due, at least in part, to the rainfall because

rain water is slightly acidic (pH ≈ 5.6). It is also conceivable that the saturation of biosolids in the outside test bed by the rainfall caused anaerobic conditions to develop to a greater extent in the outside test bed than in the inside test bed. This could lead to the occurrence of fermentation reactions in the outside test bed. Fermentation reactions consume alkalinity and therefore it is common for the pH to drop in fermentation systems.

The quality control measures for the pH assay consisted of performing analyses on duplicate samples. For the most part, the pH values measured in duplicate samples were within 10% of each other. A comparison was also made between the pH values obtained using EPA method 9045D for the analysis of pH and the method described above. The primary difference between the two methods is that 20 g of biosolids cake is mixed with 20 mL of water in the EPA method, whereas 10 g of cake is mixed with 20 mL of water in the method used in this study. The pH was determined using each method and values measured were 7.77 and 7.65, respectively. The difference in measured values was determined not to be statistically significant. Therefore, the current method will continue to be used for future testing.
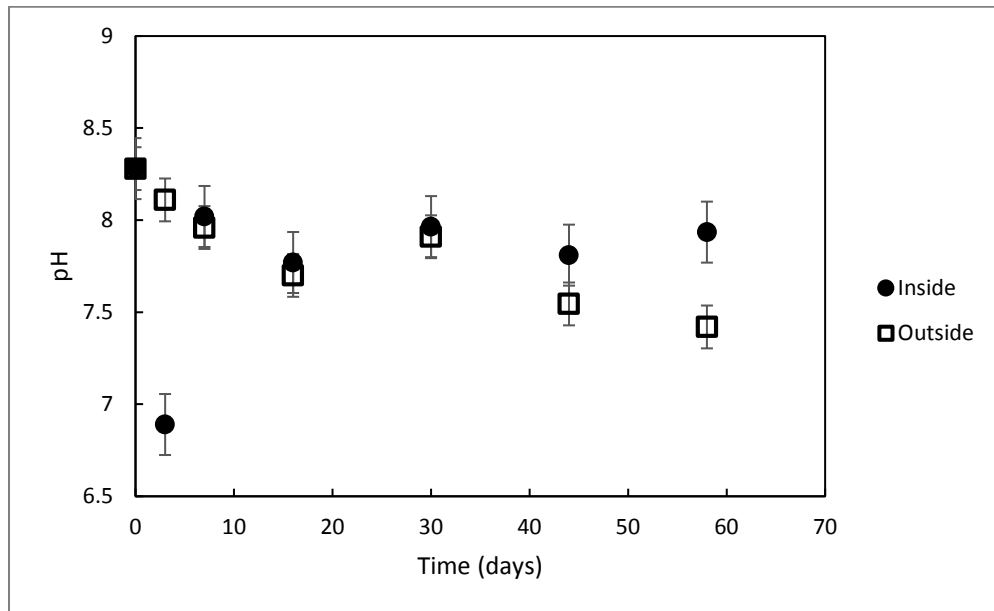

Figure 3.3. pH results for indoor and outdoor samples. Each data point represents the average concentration in duplicate samples. Error bars represent ± one standard deviation.

3.4   Volatile Fatty Acids

VFAs and the bicarbonate ion concentration were quantified in duplicate samples. In the initial assays, negative and low values were obtained for the VFA and bicarbonate measurements, respectively. Several steps were taken to minimize errors in the analytical method, including covering the beaker containing the sample to minimize VFA loss

during titration. All of the concentrations measured after day 44 were greater than zero. Therefore, all data collected before day 44 were omitted from Fig. 3.4. The day 0 VFA and bicarbonate ion concentrations were estimated by collecting and analyzing a sample from a fresh biosolids pile outside of the pilot boxes. It is difficult to draw any significant conclusions based on the small number of available VFA and bicarbonate ion data points; however, the decrease in alkalinity (Fig. 3.4) is consistent with the observed decrease in pH (Fig. 3.3) in the outside test bed. In general, it is expected that the concentration of VFAs will increase in all of the test beds over time due to the fermentation of biosolids, as illustrated by the acetogenic fermentation of biomass ($C_5H_7O_2N$), according to:

$$C5H_7O_2N + 3H_2O \rightarrow 2.5\ CH_3COO^- + 1.5H^+ + NH_4^+ \qquad \text{(Eqn 1)}$$
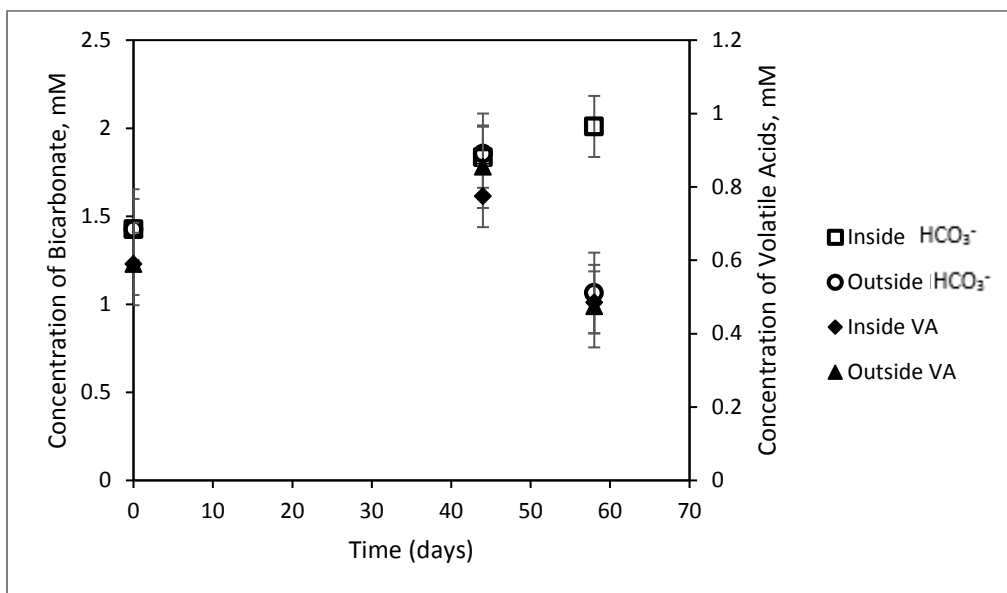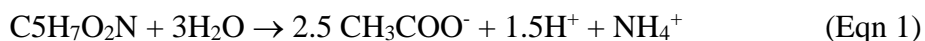

Figure 3.4. Bicarbonate and Volatile Acid results using current titration setup for indoor and outdoor samples. Each data point represents the average concentration in duplicate samples. Error bars represent ± one standard deviation.

The quality control analysis of the VFA analytical method included measuring the VFA and bicarbonate ion concentrations in a series of standard solutions containing known concentrations of these constituents, before and after improvements to the analytical method were implemented. As shown in Fig. 3.5, the method improvements had little impact on the measured HCO_3^- concentrations, which were consistently lower than the known concentrations. In contrast, the measured VFA concentrations increased significantly after the improvements to the method were implemented (as noted above);

however, the measured concentrations still deviated significantly from the known concentrations (Fig. 3.6).
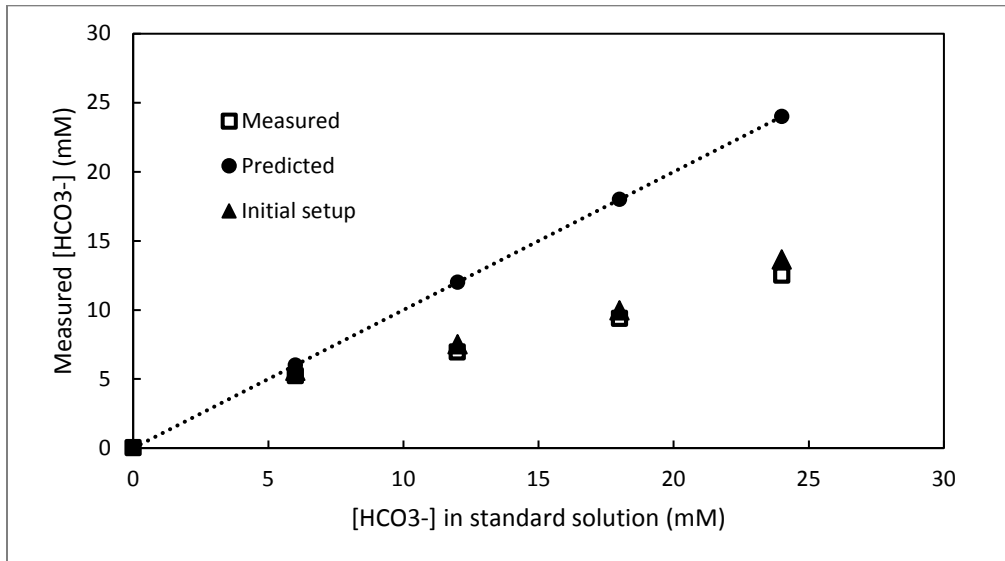
Figure 3.5. Measured HCO3- concentrations, before and after improvements to the VFA method were implemented, as a function of the known HCO3- concentration in standards.

Figure 3.6. Measured VA concentrations, before and after improvements to the VFA method were implemented, as a function of the known VA concentration in standards.

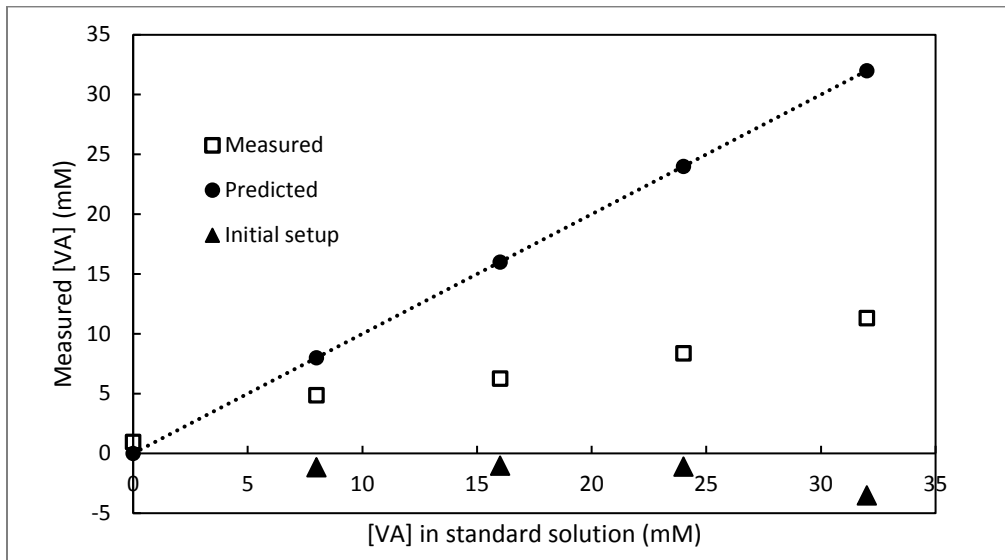   Work continues to be done to improve upon the methods for measuring VFAs and bicarbonate in the biosolids. Because of all the issues encountered with this test, future work may not include the measurement of VFAs unless significant progress is made with quality control.

3.5 Ammonia

The ammonia concentrations measured in the biosolids on days 0, 30, 44 and 58 are shown in Figure 3.7. With the exception of the concentrations measured on day 30, which were less than 3000 mg NH_3-N/kg biosolids, the concentrations ranged from 5000 to ~7500 mg NH_3-N/kg biosolids. These values are consistent with the typical ammonia concentrations measured in the biosolids by PLWSA personnel (4,000 to 6,000 mg NH_3-N/kg biosolids). Ammonia concentrations measured on other sampling days were not included in Figure 3.7 because those samples were not preserved correctly and thus the data are not reliable.
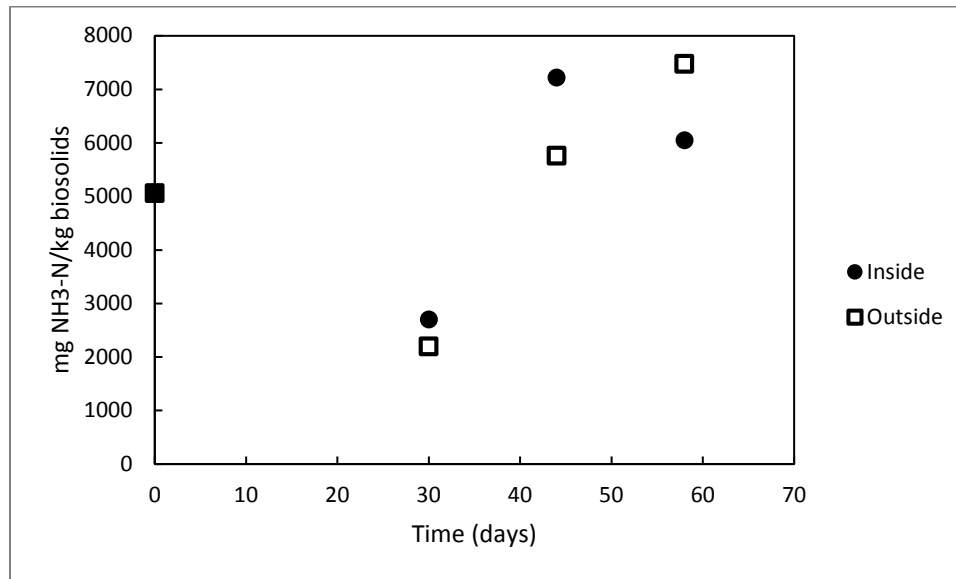

Figure 3.7. Biosolids ammonia concentrations measured in the inside and outside test beds. Concentrations were measured by reacting 0.2 mL of distilled samples in the TNT832 vials, and then converted to the correct concentrations using the correlation.

As part of the quality control measures, seven aliquots of a 10 mg/L NH_4Cl standard solution were treated using the full distillation and reaction process and then analyzed spectrophotometrically. The resulting concentrations were two times higher than the known concentration of the standard solution. It was then discovered that the volume of distilled sample that had been added to the TNT832 vials (0.5 mL) was incorrect. When the correct sample volume (0.2 mL) was used, the correct concentrations were measured.

Therefore, a series of standards was prepared and both 0.2 and 0.5 mL of each standard was reacted in the TNT832 vials. The concentration measured in the 0.2 mL aliquots is plotted as a function of the concentration measured in the 0.5 mL aliquots in Figure 3.8. The two sets of concentrations appear to be correlated. The correlation was used to calculate the ammonia concentrations shown in Figure 3.7.
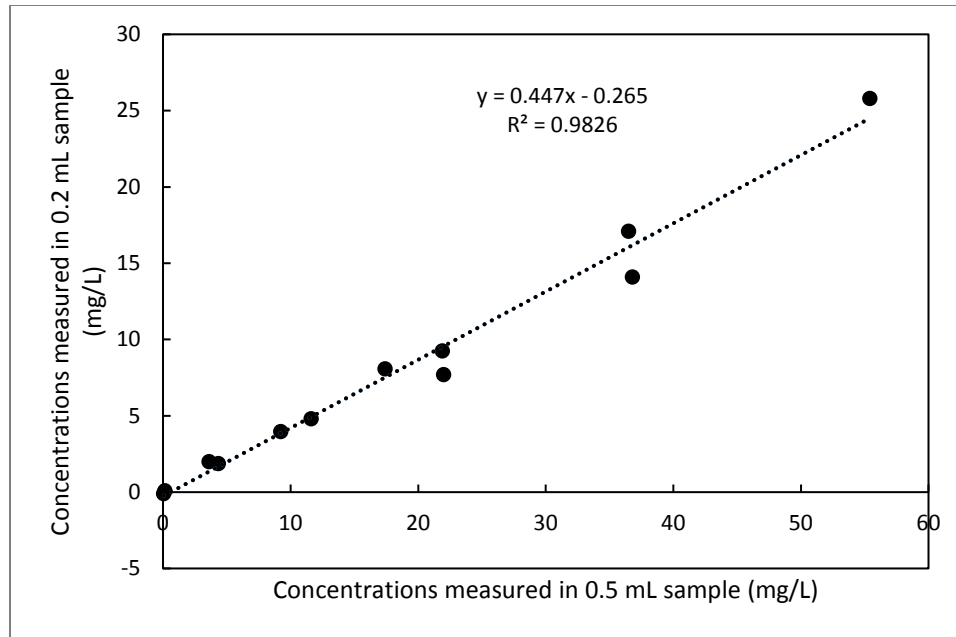
23

Figure 3.8. Conversion graph for ammonia concentrations.

Future work will use the correct volume of sample in the TNT vials and may show a trend in time. Blanks and standards will continue to be run for each set of samples.

3.6   Fecal coliforms

Fecal coliform concentrations measured in the inside and outside test beds on days 0 and 58 are shown in Figure 3.9. Fecal coliform concentrations are not available for the other sampling days because after the analyses were performed, it was discovered that the required A-1 media had not been added to the vials on those days. Based on previous fecal coliform measurements made on PLWSA biosolids stored inside the shed (cite references, i.e., one or more of our poster presentations), fecal coliforms are expected to regrow, or increase in abundance, as ambient temperatures rise during the,summer. Although no conclusions can be drawn based on the limited data set presented in Figure 3.9, the relatively high fecal coliform concentration measured on day 58 in the inside test bed is consistent with a regrowth phenomenon. Previous data show that when winter begins and the freeze-thaw cycle commences, the abundance of fecal coliforms drops rapidly.
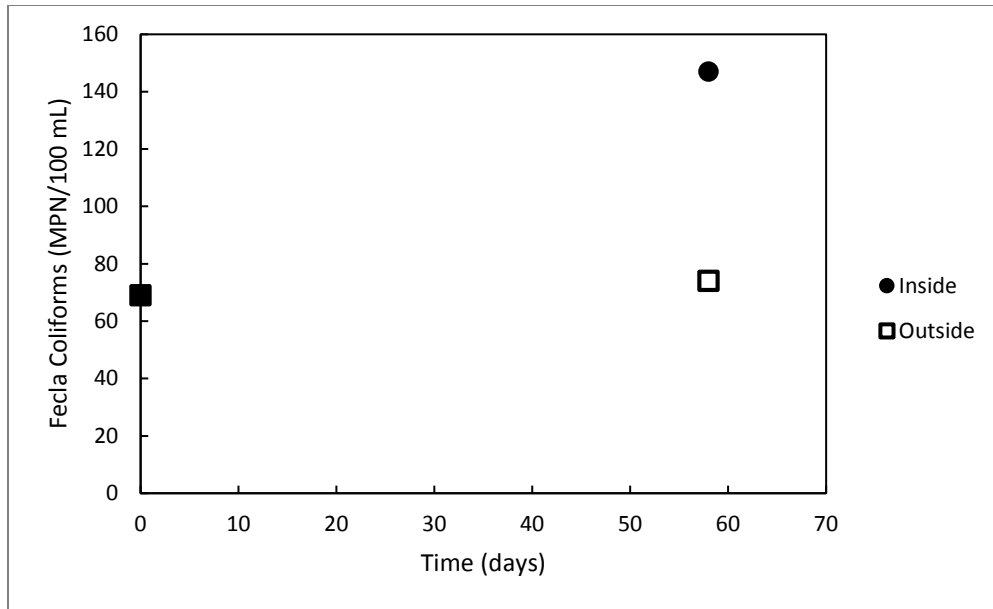
Figure 3.9. Fecal coliforms results for inside and ouside samples.

3.7 Coliphage

The concentrations of somatic and male-specific (MS) coliphages in the inside and outside test beds are presented in Figure 3.10. Values for day 0 may be incorrect because the sample was frozen before measuring.

Recall, somatic coliphages infect coliforms via the cell wall. Male-specific (MS) or F^+ coliphages infect coliforms via the pili (or fimbrae) produced by coliforms that possess the F plasmid. Two key trends can be observed in the MS and somatic coliphage data. First, because only a fraction of fecal coliforms produce the appendages known as pili or fimbrae, whereas all fecal coliforms have cell walls, the abundance of male-specific (or F^+) coliphages is expected to be lower than the abundance of somatic coliphages. In fact, that is what was observed. The abundance of MS coliphages were lower than the somatic coliphages in both the inside and outside test beds. Second, the somatic coliphage in the outside test bed appear to be trending upward at the end of the study, which is consistent with both the increasing ambient conditions and the regrowth of coliforms observed in previous studies. In the future, it is expected that the concentrations of coliphages and the fecal coliform bacteria that they infect will follow similar trends.
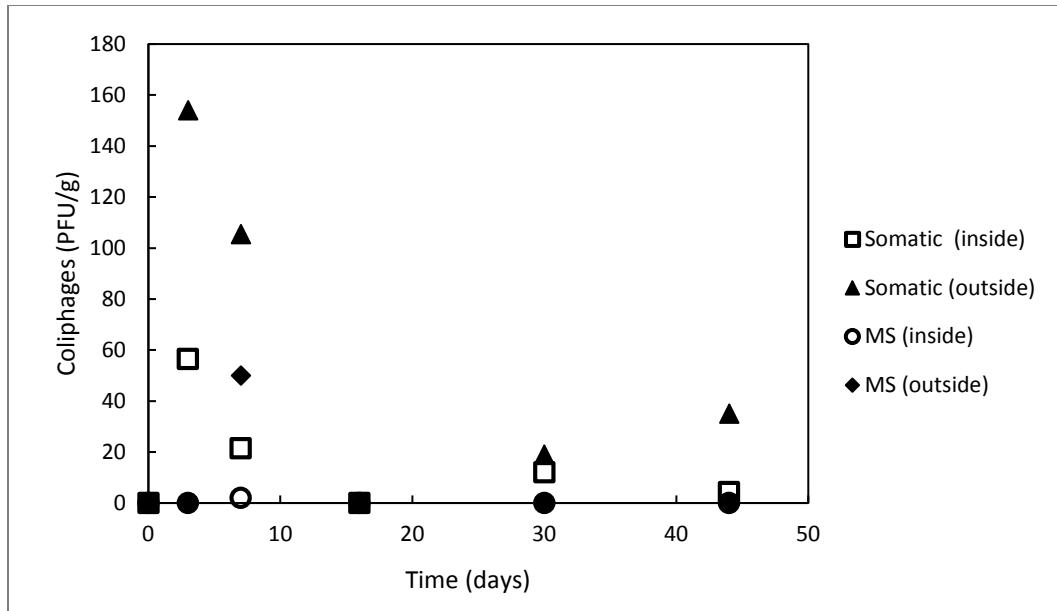
Figure 3.10. Coliphage results for inside and outside samples.

Chapter 4   Summary and Conclusions

The overall goal of the larger project of which this study was a part is to develop a rational and universal approach to the design of LCLT Class A biosolids treatment processes. Specific objectives of the larger project include: (1) a systematic laboratory evaluation of PIO inactivation; (2) fitting of inactivation constants and development of a model of PIO inactivation; and (3) performance of pilot-scale studies of LCLT processes to validate the laboratory-based predictions. The goal of the particular portion of the overall project represented by this study was to perform a soft-start of the LCLT pilot-scale study. As part of this trial run, the materials and methodologies were developed for the construction of the boxes for the pilot-scale storage of the biosolids. Methods were also developed and equipment obtained for monitoring the environmental conditions within the boxes as well as the ambient conditions. In addition, the analytical methods and associated QC were developed for TS/VS, pH, VFAs, ammonia, and fecal coliforms. Data collected using these monitoring tools and analytical methods demonstrated reasaonable trends when there are sufficient data. The trial run results were also used to improve the methods for future application. Nevertheless, further study is still required to optimize several of the testing methods (e.g., TS/VS, VFAs),moving forward.

Chapter 5   Suggestions for Future Study

Moving forward with the overall project, the boxes in used for this trial run will be emptied and refilled at the same time as the remaining 4 boxes for a uniform start time. Before doing so, a number of modifications will be made based on the results of this trial run study:

1. Pilot study box construction: By the end of the trial run study, the sides of the boxes had begun to bow out. To prevent this in the full pilot study, the sides of the boxes will be reinforced with 2"x4" studs, and a ratchet strap.

2. In situ biosolids temperature: The waterproofing technique will be changed to ensure the iButtons will stay dry and able to record data throughout the full duration of testing. Several improvements will be implemented. First, to keep the interior of the iButton sensor stacks dry and free of biosolids, caps will be added to each end of the sensor stacks. Second, silicone caulk will be used to waterproof the exterior of the iButtons.

3. Analytical methods: TS/VS measurements have exhibited reasonable results, and generally acceptable attainment of QC requirements. However, the OPR results for percent recovery of standard solutions have been outside of the acceptable range, and the cause needs to be resolved. The titrimetric method for VFAs, while improved, is still not giving acceptable recoveries for standard solutions. If this issue cannot be resolved, an alternative method may need to be found, or the measured dropped. Issues with the ammonia analysis have been resolved, but QC analyses need to be completed for the revised method. Similarly, the errors in the fecal coliform method have been corrected, but QC analyses have not been completed. Finally, for winter testing, the sampling methods will have to be changed to account for the frozen biosolids.

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