THESIS

ENHANCING HYDRAULIC PERFORMANCE OF A MULTI-STAGE ANAEROBIC DIGESTER FOR HIGH SOLIDS CATTLE MANURE

Submitted by

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ABSTRACT

ENHANCING HYDRAULIC PERFORMANCE OF A MULTI-STAGE ANAEROBIC DIGESTER FOR HIGH SOLIDS CATTLE MANURE

Anaerobic digestion is an attractive technology for waste handling because it converts low value waste material into energy and other useful products while performing necessary treatment for proper waste disposal. Conventional anaerobic digestion technology, however, has been met with many economic challenges when being applied to high solids substrate such as dry-lot cattle manure. In Colorado and the rest of the arid west, feedlot practices and dry climate combine to form a waste product that is very high in total solids (TS) content, from 50% up to 90% TS. Since the most common conventional digestion practices typically can only treat wastes up to a maximum of 15% TS, other options must be considered to digest this abundant waste product and convert it to a valuable resource.

Research at Colorado State University has led to the development of an innovative multi-stage anaerobic digester (MSAD) technology capable of digesting high solids content waste with very low water addition. The CSU MSAD has demonstrated the ability to successfully digest high solids content waste like that found at the many Colorado feedlots. This system differs from conventional technology in that hydrolysis takes place in one reactor and methane generation takes place in a separate high rate digester.

The development of the MSAD for digestion of high solids cattle manure leads to the promising opportunity for valorization of a prevalent waste product in Colorado to create valuable products including methane biogas, compost, and fertilizers. The present research aims to advance

the technology by assessing the performance of the MSAD running in a fully linked configuration including each of its individual components: the Upflow Solid-State Hydrolysis Reactor (USSHR), the Leachate Feed Tank (LFT), and the Fixed Film Reactor (FFR).

A fully functional Central Leachate Processing System (CLPS) was constructed to demonstrate the technology, to facilitate column scale studies, and to link with the prototype USSHR (P-USSHR) to enable the evaluation of an improved liquid distribution system. The MSAD was constructed at column scale to evaluate the impact on organic leaching potential of varying hydraulic loading rate (HLR) through the USSHR using HLRs of 20, 41, and 75 cm/day for 16-day cycles. This experiment was the first successful demonstration of a fully linked MSAD system using cattle manure as feedstock. It was found that the higher HLR of 75 cm/day yields 25% more COD leached over the 16-day operating period than the two lower loading rates. Additionally, it was found that the P-USSHR achieved notable improvements over the previous operation in hydraulic distribution through the reactor and therefore improved digestion performance and volatile solids destruction, though areas for further improvement were noted.

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Most of all, I would like to express my thankfulness to God for creating this incredible world and for giving us the charge to explore, question, study, and steward our beautiful Earth.

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AD	Anaerobic digestion
CC	Calibration column
CEU	Column experiment unit
CEU1	Column experiment unit #1 (left side of cart, looking at USSHRs)
CEU2	Column experiment unit #2 (right side of cart, looking at USSHRs)
C-FFR	Central fixed film reactor
C-LFT	Central leachate feed tank
COD	Chemical oxygen demand
CSU	Colorado State University
C-USSHR	Central upflow solid-state hydrolysis reactor
E-FFR	Experimental fixed film reactor
E-LFT	Experimental leachate feed tank
E-USSHR	Experimental upflow solid-state hydrolysis reactor
FFR	Fixed film reactor
HFT	Hydrolysis feed tank
HLR	Hydraulic loading rate
HMI	Human-machine interface
LFT	Leachate feed tank
MSAD	Multi-Stage Anaerobic Digester
OLR	Organic leaching rate
PLC	Programmable logic controller
P-USSHR	Prototype upflow solid-state hydrolysis reactor
TS	Total solids
USSHR	Upflow solid-state hydrolysis reactor
VS	Volatile solids

CHAPTER 1. Introduction

1.1 Research Motivation

Anaerobic digestion (AD) is an attractive and increasingly popular technology for treating organic wastes due to its effectiveness and the ability to recover resources and offset operational costs of waste treatment and management. In AD, organic wastes undergo four sequential biochemical processes, namely hydrolysis, acidogenesis, acetogenesis, and methanogenesis. Through the AD process, organic carbon is converted into biogas, a gaseous mixture predominantly consisting of methane and carbon dioxide. Biogas can be burned in a boiler or cogenerator to recover energy in the form of heat, electricity, or both, or it can be purified to supply natural gas pipelines or used as a vehicle fuel (Metcalf & Eddy, 2013). Processed solids can be safely applied to land and are useful as a fertilizer (Nelson, 2002).

Anaerobic digesters are most commonly used in municipal wastewater treatment plants to treat a mixture of sludge settled out of the primary clarifier and waste activated sludge from the mainstream treatment train. This application typically uses a large volume complete-mix digester with long retention time (30-60 days) and low total solids (TS) content (<10% TS). A completely mixed digester has the benefit of increased stability and low risk of upset. The large footprint, long retention time, and low solids-handling capabilities, however, render it ill-equipped to treat many different types of organic wastes such as food waste, municipal solid waste, or livestock manure. Substantial dilution water and/or mixing would be necessary, so these feedstocks usually require some form of dry digestion technology, or a digester capable of handling high solids content generally defined as ≥15-20% (Dinh Pham Van, 2020). Some plug flow reactors have been

operated to treat organic solid waste with as high as 40% TS content using process adaptations like rotating impellers (Oh, 2011).

Feedlot livestock manure is a ubiquitous organic waste product in Colorado which poses a unique challenge for digestion, as the arid western climate and local feedlot practices combine to produce a product that is often as high as 90% TS content which is heavily contaminated with inorganic materials like rocks and sand (Hanif, 2013). Considering the low availability of water in the area, conventional digester technologies are not a pragmatic solution to treat livestock manure waste in Colorado. These challenges motivated a research effort to assess alternative methods to implement AD for treatment of livestock manure in the arid west.

To limit necessary water addition, an AD process was developed with separate stages, the Multi-Stage Anaerobic Digester (MSAD). The initial high solids cattle manure (HSCM) feedstock was loaded into a separate reactor where organics could be leached out by passing water through the substrate bed. A method was developed following the model of multiple stage digesters to recycle water in the system by recirculating leachate, or nutrient rich water flowing out of the initial feedstock, through the separated processes.

In the MSAD system, the initial solid substrate is retained in the first stage, referred to as the Upflow Solid State Hydrolysis Reactor (USSHR), which is separately controlled to maximize hydrolysis. Liquid is pumped into this reactor from the leachate feed tank (LFT). The leachate from the USSHR flows back into the LFT, which recirculates continuously with both the USSHR and a high-rate fixed film reactor (FFR) simultaneously (Figure 1). This allows for much shorter hydraulic retention times as the active methanogens are retained on floating plastic media within the FFR.

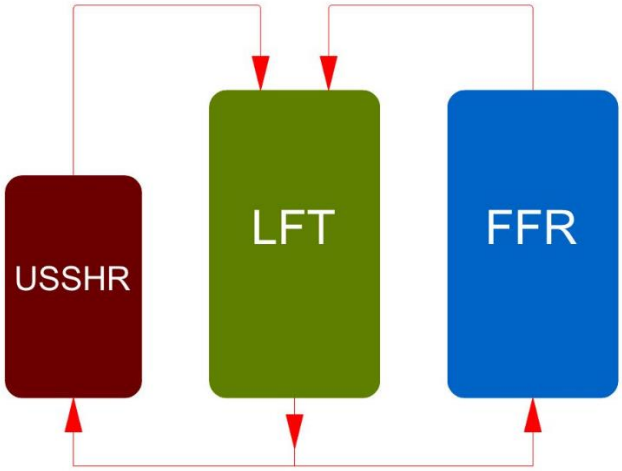

Figure 1 – General MSAD process flow

The biodegradable carbon that is leached out of the initial feedstock is continually broken down in the FFR to produce biogas, a methane-rich mix of gases also including carbon dioxide, hydrogen, and hydrogen sulfide. Then, the processed leachate with low methane production potential is pumped back through the USSHR again where organic matter is once more leached into the liquid, increasing methane production potential. As the leached organic carbon is converted into biogas, other byproducts begin to accumulate, namely nitrogen in the form of ammonium as well as inorganic salts which are predominantly potassium, sodium, and calcium. The makeup of these inorganic salts was determined by analysis of leachate from the system operated in Lewis (2018). When either of these reach concentrations too high, methanogenesis begins to be inhibited and the process decreases in efficiency until reaching complete inhibition (Griffin, 2012). To avoid this, dilution water is necessary to reduce concentrations of inhibitory constituents or, ideally, the nitrogen and salts are recovered as valuable resources. This process allows for effective digestion of the HSCM commonly found at Colorado feedlots.

To date, research on the novel CSU MSAD technology treating cattle manure has focused on one component at a time rather than the fully linked system. A previous MSAD study using food waste included the FFR, but data collection was focused on the hydrolysis portion (Griffin, 2012). The motivation for this research is to assess the performance of the fully linked system at both column scale and a larger demonstration scale to inform future research direction to be aimed at optimization of full-scale operation. Specific objectives were to:

1. Understand the impact of varying flow rate through the hydrolysis reactor on organic leaching potential in a fully linked system, measured by COD leaching rate
2. Assess the hydraulic distribution of liquid flow through the prototype USSHR with an improved liquid injection system

To accomplish these objectives, experiments were run at both column and prototype scale. Two identical column systems with high process control were designed and built on a cart-mounted skid and placed inside the custom-built research lab. In addition, a prototype-scale MSAD system was designed and built to demonstrate the technology, to study the relevant parameters, and to assess the MSAD performance with an improved liquid distribution system compared to previous experiments. Using these systems, it was possible to investigate and characterize the performance of the MSAD system and inform future work direction and a path forward for the MSAD technology.

CHAPTER 2. Background and Literature Review

2.1 Anaerobic Digestion

Anaerobic digestion (AD) is a commonly-used technology with many applications around the world. AD is used to treat waste products that are otherwise harmful to the environment, meanwhile producing as a byproduct valuable methane gas. As a naturally occurring microbiological process, engineered anaerobic digesters work to create an optimal environment for bacteria to work, harnessing and effectively controlling the natural process. In the absence of oxygen, mutualistic and symbiotic groups of bacteria break down and consume organic materials, reducing organic carbon to the simple and stable gas forms of methane and carbon dioxide (Figure 2). The lack of oxygen is important, as other species of bacteria that utilize oxygen reproduce much faster and outcompete the methane-producing anaerobes for substrate consumption (Metcalf & Eddy, 2013). There are four primary groups of microorganisms responsible for the anaerobic digestion process: hydrolytic, acidogenic, acetogenic, and

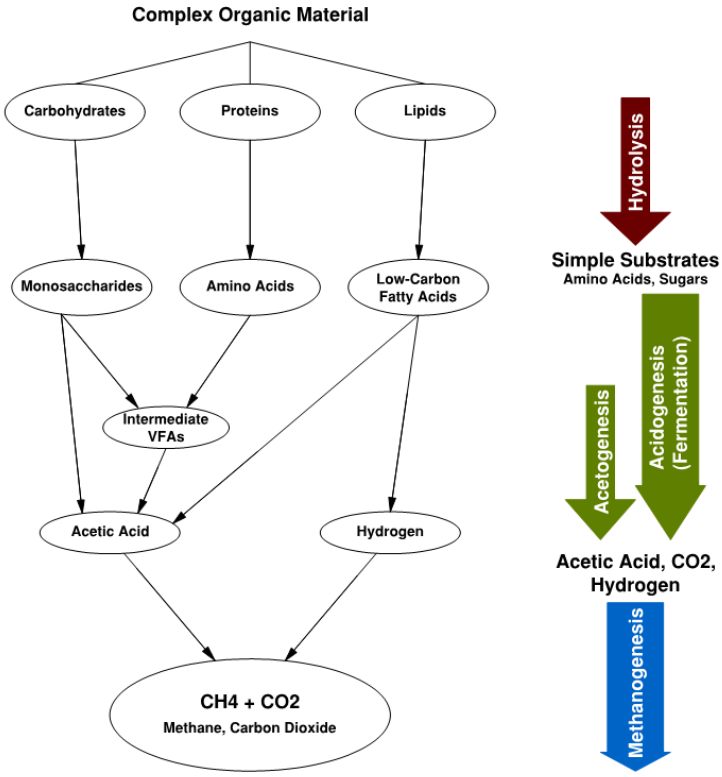

Figure 2 – Anaerobic digestion process

methanogenic bacteria. Each of these groups of bacteria play a major role in AD. Hydrolysis is the process by which complex and insoluble organic materials are broken down to simpler compounds readily available for other bacteria to consume. Hydrolyzed products then go through the acidogenic phase, where they are broken down further into volatile fatty acids and alcohols. Next, during acetogenesis, these compounds form acetate, which is available for methanogens to perform the final step and produce methane gas.

Many different organic substrates are commonly used in anaerobic digesters. Treating waste for safe disposal and simultaneously producing energy in the form of biogas makes AD an attractive option. Wastewater from food and beverage processing industries (such as dairies and breweries) are commonly treated in anaerobic digesters, as well as animal waste and domestic wastewater (United States EPA, 2015). Many digesters reuse produced biogas for heat or energy on site. Much, if not all, energy required for heating and pumping can be recovered from produced biogas. Methane is the predominant component in biogas, usually making up 55%-65% by volume (Metcalf & Eddy, 2013). The remaining portion is mostly carbon dioxide, with a very small percentage going to hydrogen sulfide and hydrogen gas (Metcalf & Eddy, 2013). Biogas can be burned as produced in boilers and cogenerators, usually for use on site, or it can be purified and sent to a municipal natural gas line or compressed for transport and use offsite. During biogas purification, sulfides are especially important to remove by scrubbing to avoid corrosion of equipment and gas lines. Residual solids from digesters can also be valuable byproducts. The processed solids are often applied as soil amendments containing nutrients that serve as effective fertilizers. Land application is particularly beneficial when agricultural land is nearby the digester that produces the biosolids since transportation costs can be high. Some digesters are located very

far from the croplands they are applied to, however, with hauling distances as many as 2,000 miles (United States EPA, 2000).

Digesters have been used for over a century, with the first applications being municipal wastewater sludge in the late 1800s to the early 1900s (Metcalf & Eddy, 2013), but modern advances have stemmed from studies showing how differing environmental and physio-chemical characteristics can substantially alter digester performance. Everything from altering temperature and pH to retention time and the presence of background micro-nutrients show effects on rate of digestion and even on the types of microorganisms that develop (Schnürer, 2019). Hydraulics of reactors become a major design parameter, as there are many different ways to move substrate and active biomass. Examples of common reactor designs include complete mix and plug flow reactors as well as fixed-film technologies such as Upflow Anaerobic Sludge Blankets and Membrane Bioreactors. The former have solids retention times (SRTs) equal to hydraulic retention times (HRTs), while the latter retain solids and therefore have an essentially infinite mean cell residence time, allowing higher concentrations of healthier bacteria which can lead to decreased volume requirements.

2.2 Complete Mix Digesters

Conventional digestion technology typically consists of large, completely mixed digesters with continuous flow. These reactors must be built with very large volumes to accommodate biomass growth, since biomass leaves the reactor along with flow and solids retention time (SRT) is equal to hydraulic retention time. As such, retention times can be in the range of 10-25 days (Neibling, 2014). The large volume and complete-mix scheme does, however, provide stability and resistance to system shocks from unexpected influent (Loetscher, 2018). Solids can be settled out and recycled back to the process to increase SRT and thereby decrease required volume.

Complete mix digesters can only accommodate low-solids content waste, typically in the 3%-10% range (Neibling, 2014). Higher solids wastes must be diluted to acceptably low levels.

Most digesters at conventional wastewater treatment plants are complete mix reactors. They are typically used as a tertiary treatment alongside conventional activated sludge treatment processes. Primary settled sludge from the clarifier is sent to the digester along with wasted activated sludge from the secondary clarifier for further treatment and stabilization. Inside the digester, the sludge is degraded by anaerobes and biogas is produced, which can be burned on site for heat and power generation. Plants with flows greater than 5 MGD are typically good candidates for anaerobic digestion as they can benefit greatly from the reduced sludge volume as compared to aerobic stabilization. Aerobic digestion, by contrast, is often employed by wastewater treatment plants treating less than 5 MGD flow to save on capital costs (Metcalf & Eddy, 2013). Even when using anaerobic digestion, small plants often will not use the produced biogas due to the high capital expense of equipment necessary to capture, purify, and/or convert biogas to usable energy. After digestion, solids can be applied to land to enhance soil organic matter and nutrient content.

2.3 Plug Flow Reactors

The other most common reactor design used in AD is plug flow. Plug flow reactors work to increase rates by modifying hydraulic conditions so that, in a long and narrow flow regime, mixed conditions only occur within differential elements along the flow path. As opposed to the complete mix design where equal concentrations of substrates and microorganisms are present at all locations within the reactor, in a plug flow reactor the concentrations of each vary with position through the reactor. It is not possible to achieve true plug flow characteristics as mixing along the direction of flow is impossible to prevent. Plug flow can, however, be approximated by very long and narrow reactor designs, by using baffled walls, or by putting many separated reactors in series

(McCarty, 2001). Plug flow anaerobic reactors are typically unmixed rectangular-shaped basins treating moderately high solids content waste, usually 10-18% TS, with retention times between 20-30 days (Metcalf & Eddy, 2013).

2.4 High Solids Digestion

Of the anaerobic digesters operating in agricultural applications in the United States, dry digesters (reactors treating waste products with >20% TS) make up a very small fraction (less than 1%) of those in operation. The majority are of the conventional complete mix or plug flow type, processing low solids waste typically produced by dairies and hog farms (Figure 3).

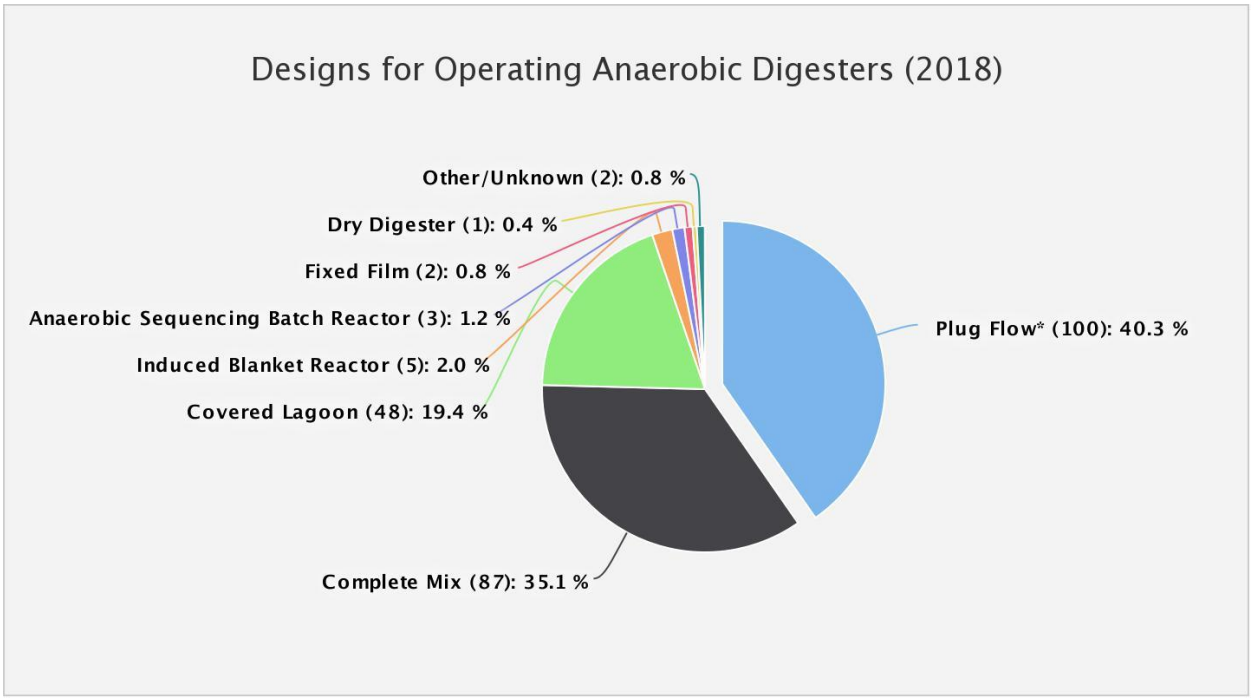

Figure 3 – Agricultural anaerobic digester designs pie chart (United States EPA)

Although conventional digesters are a very mature technology with high stability, there are many biodegradable substances that are not conducive to digestion in these reactors. Other reactor configurations have been developed to digest substrates higher in solids content, such as municipal solid waste and certain types of animal waste, food waste and other industrial waste. These

configurations include variations of plug flow reactors in both vertical and horizontal flow schemes which have been shown able to treat wastes containing up to 40% total solids (Oh, 2011). Existing high solids reactor designs are effective for treating moderately thick sludges, but hydraulic conditions present at higher solids content make it very challenging to move substrate through the reactor. Enough water content must be present to maintain flow of substrate through the reactor. Batch operation of digesters, a method with both low and high solids applications, can help mitigate this issue because hydraulic flow may not be required during all phases of operation. Instead of a continuous flow through the reactor, batch processes operate by loading all substrate initially and then completing the digestion process to the desired level before emptying the reactor for the next batch to begin. Treatment occurs by biochemical processes with no new additions and can simply be stopped when the reaction is complete (McCarty, 2001). Batch reactor design is highly tailorable to suit specific applications, offering excellent process control. Since the substrate does not travel in and out of the reactor continuously, hydraulic flow is not as critical, and batch reactors in various schemes have been designed to handle up to 45% solids content (Metcalf & Eddy, 2013).

2.5 Multiple Stage Digesters

With the motivation to effectively treat higher solids content waste like that found at dry feedlots in Colorado, innovations in AD have been considered attempting to overcome the inherent challenges. A possible approach to effective high-solids waste digestion involves separating the process into multiple stages. The initial substrate can be partially digested in one reactor where the first steps of anaerobic digestion take place, after which the effluent is sent to another reactor designed specifically for methanogenesis to complete the digestion process. This process

configuration opens up the possibility of completely different flow regimes, such as leaching organics from solids and then processing the leached liquid to produce methane.

Research into multi-stage anaerobic digestion has gained momentum more recently as engineers and researchers seek to optimize digestion processes. Since the microorganisms responsible for the different steps involved in AD have differing optimal environmental conditions (Zoetemeyer, 1982), separating the stages allows a more optimal environment for each step to take place. Each stage has a different limiting factor and control can be taken to optimize each separately (Chaudhary, 2008). Multi-stage digesters have been designed to aid in improving stability of the process caused by organic loading rate (OLR) fluctuations, heterogeneous wastes, or excessive levels of inhibitors (Ward, 2008).

The attractive benefits of the AD of waste has led to attempts to expand digestion capabilities to more substrates. Grass silage is an example of one such product. Silage is a common feedstock for digesters, but it requires very long detention times in conventional digesters (typically at least 60 days). Taking a multi-stage approach to digesting this feedstock has been shown to effectively reduce the required detention time by 33%, to 40 days, by leaching the organic content from the silage in a "dry batch leaching" stage before treating the leachate in an upflow anaerobic sludge blanket reactor (Nizami, 2010).

Multiple-stage digestion has also been used to process waste higher in solids content. A commercialized technology was developed to treat high solids food waste that operates very similarly to the MSAD developed in this study (GICON Group, n.d.). Process water is applied to a feedstock mound and the percolate is sent to a separate reactor to produce methane. This process

differs from the CSU MSAD in that the process flow is a downward percolation system rather than the upflow configuration of the MSAD, further elucidated in Section 2.7.1 below.

Livestock manure presents another opportunity for potential application of a multi-stage digestion process. A two-phase AD process, which enabled selection and enrichment of different bacteria in each phase, was used to increase biogas yield while decreasing overall retention time by half in the digestion of unscreened dairy manure (Chen, 2005). Separating AD into two stages allowed the design of different operating conditions to favor growth of different bacteria in each stage. The first stage was designed to favor acidogenic bacteria growth while the second stage was tailored to suit the slower growing methanogens. The two-phase operation produced over 50% more biogas than the conventional one-stage control at a retention time of just 10 days (2 days in the acidogenic phase followed by 8 in the methanogenic phase) compared to 20 days for the conventional process.

2.6 Livestock Manure as Feedstock

Digestion of commercial feedlot cattle manure, being ubiquitous in Colorado and throughout the Rocky Mountain west, is another attractive feedstock for focus of research efforts. Manure is a nutrient-rich substance with high potential for methane production (S.D. Kalamaras, 2014). As a high solids content waste, however, it is difficult to digest in typical reactor designs. There are very few digesters operating for feedlot waste in this region. The map in Figure 4 below displays nationwide density of on-farm anaerobic digesters by type. Of particular note is the lack of any digester on a beef feedlot in the central region of the United States, where there are many feedlots.

A research effort into the noted phenomenon of limited adoption of AD in the arid west region led to an economic viability study of anaerobic digestion of feedlot manure in Colorado (Keske, 2009). It was found that due to feedlot manure collection practices in the arid western region, the resulting product is very high in solids content, ranging from 50% to as high as 90% total solids, and often contains lots of inorganic materials such as rocks and dirt (Loetscher, 2018). This “dry scrape” manure management practice severely limits the possibilities of digestion in a conventional anaerobic digester. Significant water addition would be necessary to achieve a lower solids content like that seen in a typical complete-mix digester. The high costs and scarcity of water in the arid west renders this an impractical solution

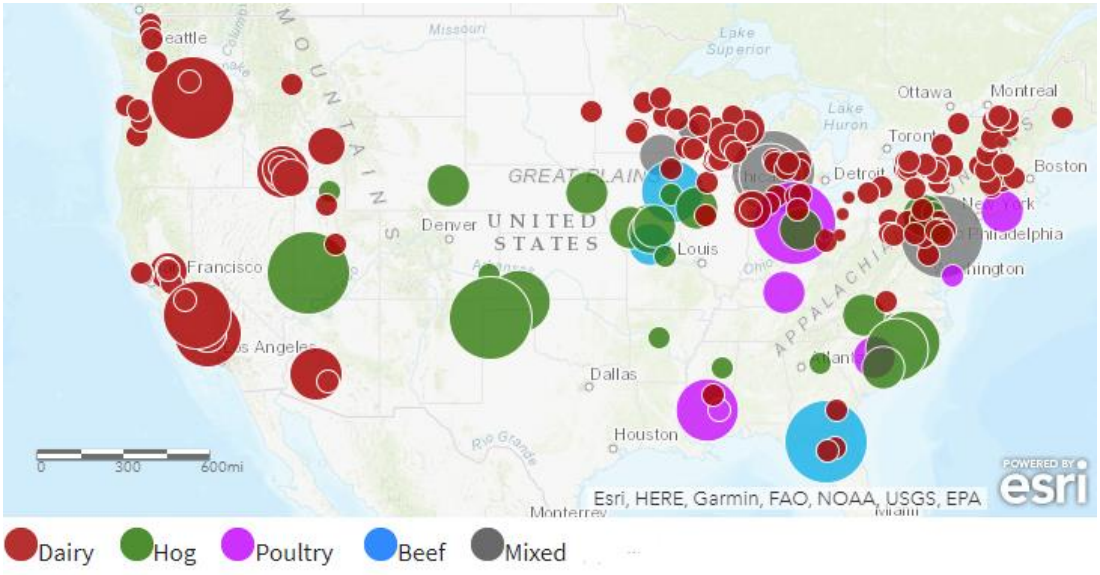

Figure 4 - On-Farm Digesters in the United States (United States EPA)

2.7 Present Research

2.7.1 MSAD Configuration

The novel multi-stage approach taken by the CSU MSAD to digesting HSCM and other high solids waste works by utilizing a separate reactor for solids where water can flow through in

an upflow configuration and hydrolyze the organics into dissolved and suspended particles, carrying them out with the effluent liquid while the solids remain in the reactor. This leached fluid can then be sent to a high-rate fixed film anaerobic digester. These separated steps allow the opportunity to individually optimize each step, providing high potential for faster kinetics. The initial concept operated the solids reactor as a leach bed reactor (LBR) design. Water was dispersed atop the substrate bed and collected as it flowed out the bottom. A significant and persistent problem continued to arise; leaching channels would eventually clog, leading to hydraulic failure of the reactor. Many different techniques including organic and inorganic bulking materials and dispersion media atop the LBR were tried before it was discovered that an upflow configuration was able to sustain flow (Wu, 2017).

The MSAD developed in this study works by separating the AD process into 3 components. The initial high-solids substrate to be digested is placed in the first reactor tank, the USSHR (Figure 5). The tank is equipped with an inlet port on the bottom and an outlet at the top to enable an upflow configuration. The outlet passes through a phase separation filter to allow the liquid to leave while retaining the solid portion in the USSHR. The liquid effluent, referred to as “leachate”, contains organics (and inorganics) leached from the substrate. This leachate is deposited back into the LFT, which acts as a central hub of the system. Leachate is drawn out of the LFT to feed the FFR, which houses the methanogens on attached growth media. The organics previously leached from the USSHR are readily degraded in the FFR to produce biogas, and the digested leachate is then discharged back into the LFT where it can be fed back into the USSHR again to replenish the biochemical methane potential (BMP), a measure of leached organic content available for digestion.

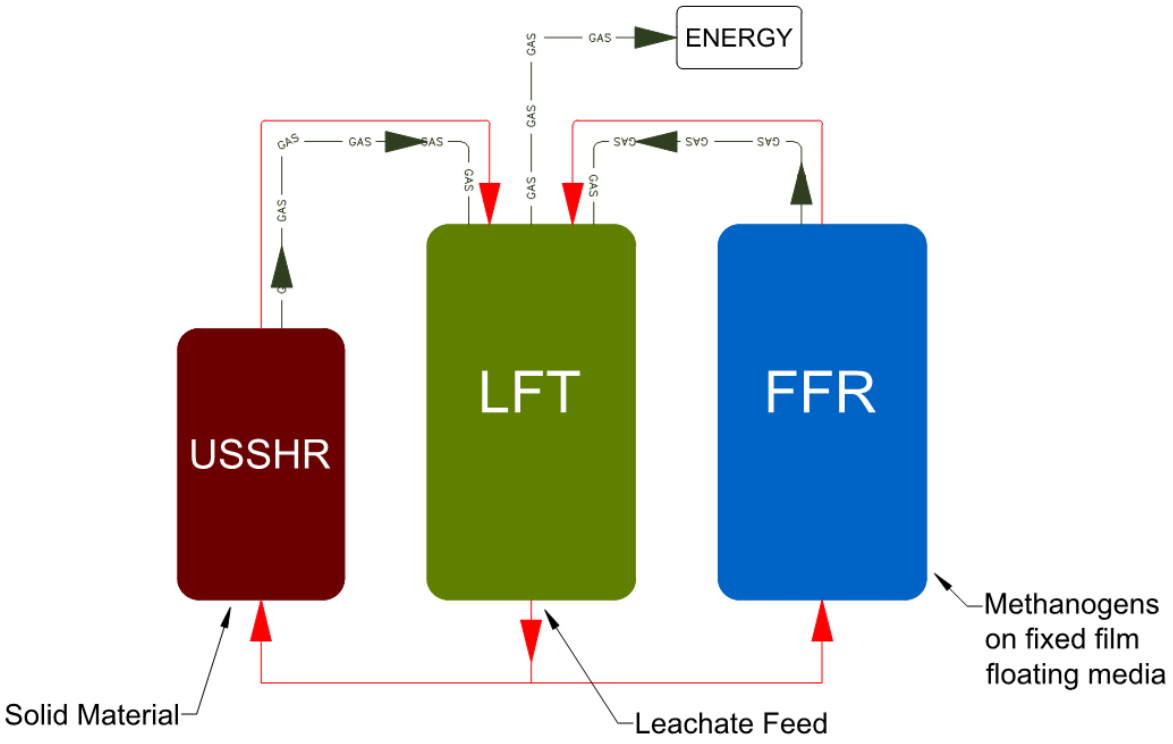

Figure 5 – MSAD process schematic

Both of these processes run continuously and side by side out of the same LFT. The FFR recirculation process can be thought of as a continuous flow, while the USSHR operates more closely to a batch process. The USSHR can be taken offline, emptied, and refilled with new substrate once the desired leaching potential is reached. This approach allows for the MSAD to be configured in a modular setup with multiple “batch operated” USSHRs running at once and taken online, offline, and replaced independently (Figure 6).

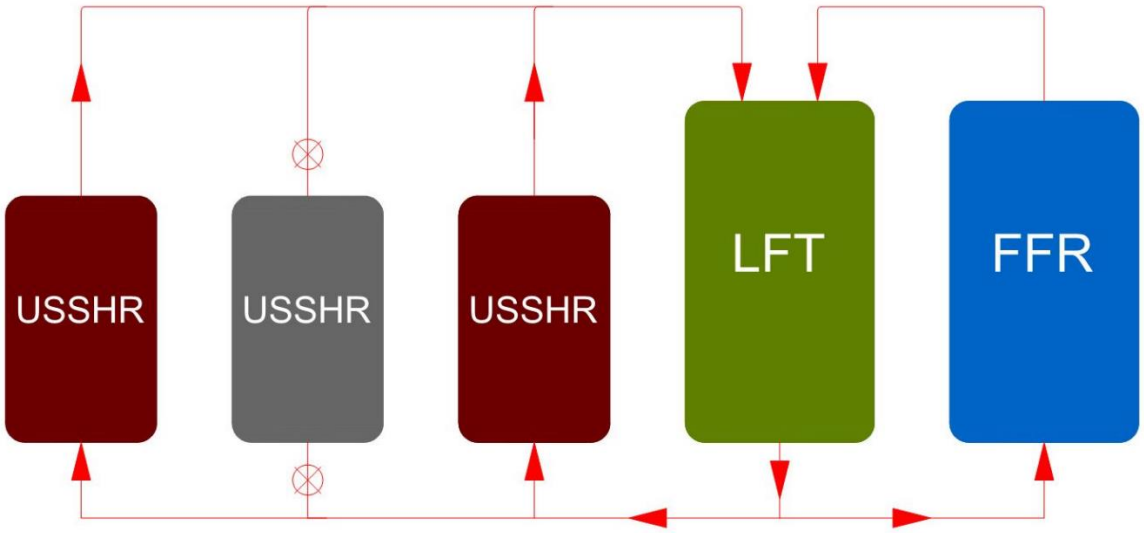

Figure 6 - MSAD process flow schematic demonstrating modular capability with separate batch-operated USSHRs

2.7.2 Research Background and Past Contributions

Colorado State University's innovative MSAD was developed in an effort to overcome the challenges posed by such high solids content waste by recycling water through the system. If water could be reused, it would take considerably less water addition to operate a digester with high-solids cattle manure feedlot waste. The CSU MSAD was originally conceived as part of Lucas Loetscher's graduate studies under Dr. Sybil Sharvelle (Loetscher, 2018). Further advancements of the MSAD technology were made by subsequent studies seeking to improve the reliability and performance of the hydrolytic leaching stage (Hanif, 2013) (Wu, 2017) (Lewis, 2018). Process refinements and target optimal loading rates and leachate concentrations in hydrolytic and methanogenic stages were investigated (Griffin, 2012) (Arita, 2013). Post-processing of residual solids for beneficial use after digestion was also explored, finding that digested manure from the CSU MSAD system can serve as an effective soil amendment (Sandefur, 2016) (Surendran, 2018) (Larson, 2019).

2.8 Motivation for Research

The CSU MSAD system presents the opportunity to implement effective AD to treat a common waste product found in Colorado and throughout the arid western region, opening the possibility to harness the many noted benefits of AD in a new application. Research to date has proven the MSAD concept but has not evaluated the process operating in a fully linked configuration. Understanding the impact of varying flow rates through the hydrolysis reactor in a fully linked system will enable future research to optimize the operation of the USSHR stage. Additionally, evaluation of the performance of the prototype USSHR with improved liquid distribution will aid in future commercialization of the MSAD technology.

CHAPTER 3. Materials and Methods

3.1 Equipment Setup

To accomplish the objectives of this research, it was necessary to construct a cart-mounted skid with a complete column-scale MSAD system, a prototype-scale USSHR, and a Central Leachate Processing System (CLPS) to link with each of the experimental components (Figure 7). The cart-mounted skid is referred to as the Column Experiment Unit (CEU). The prototype USSHR reactor is referred to as the P-USSHR. The CLPS and the CEU were housed in the built-to-suit pilot lab (Section 3.2). These systems were comprised of the following:

- CLPS – A fully functioning MSAD at demonstration scale
    - The CLPS consisted of a central FFR (C-FFR), a central LFT (C-LFT), a central USSHR (C-USSHR), and all the process equipment necessary to run the CLPS as a fully linked MSAD. This included pumps, storage tanks, gas collection, sample probes, a sump tank, and all other related components. It was designed with flexible linkages to use in conjunction with the CEU and P-USSHR.
    - The purpose of the CLPS was to develop a microbiota in the C-FFR specifically suited to manure leachate degradation, ensuring the experiments started with an inoculum representative of full-scale operation with mature leachate. Leachate from the CLPS was supplied to the CEU to start each experiment and was circulated through the CEU experimental FFRs (E-FFRs) when not in use to maintain healthy microbiota. Additionally, the CLPS served to process the leachate produced in the P-USSHR so that it could be recycled in that system. During the prototype experiment, the P-USSHR replaced the C-USSHR in the CLPS.

- CEU – A cart-mounted skid containing two twin MSAD systems (CEU1 and CEU2) at column scale, each consisting of experimental MSAD reactors (E-FFR, E-LFT, and E-USSHR)
    - The CEU was used for column experiments investigating the impact of varying flow rates in the hydrolysis reactors. It was connected with the CLPS to drain and fill leachate between experimental runs.
    - CEU1 and CEU2 each contained three E-USSHRs for a total of six E-USSHRs on the CEU cart. The two MSAD systems were run in parallel so that it was possible to compare results from all six E-USSHRs in real time with replication.
- P-USSHR – An 800-gallon prototype of the USSHR
    - The P-USSHR was fed leachate from the C-LFT, which was subsequently digested in the C-FFR. Together, they formed a completely linked MSAD system.

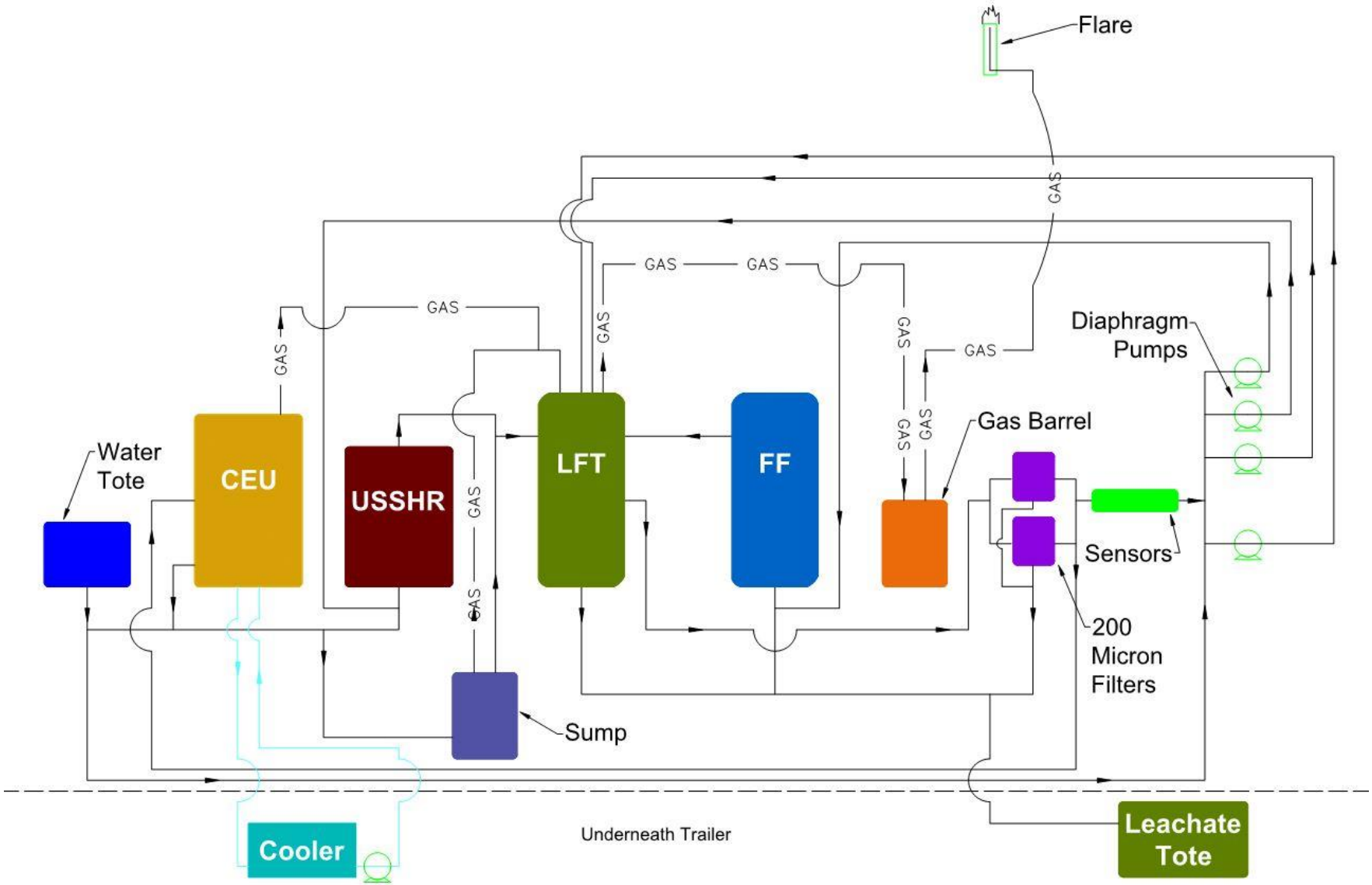

Figure 7 - CLPS layout and flow configuration

3.2 Pilot Lab Configuration

The custom-built pilot lab was constructed in a converted 48’ refrigerated semi-trailer to house each of the components of the CLPS and CEU to facilitate experiments for the MSAD technology. Additional insulation was provided inside the trailer by plywood walls lined with fiberglass insulation. Foam insulation was placed atop a false roof inside for added insulation. A sealed plywood wall with door was installed at the front of the trailer to separate the reactor room from the control room, which housed all the electronics for safety in an explosion-potential environment. The electrical system and components were described in detail in Loetscher (2018) and remained unchanged for this experiment.

3.2.1 Programmable Logic Controller System

The CLPS was configured with a Beckhoff programmable logic controller (PLC) to automate the operation and monitoring of the CLPS. Using all the various electronic measures, probes, and control points throughout the CLPS, the PLC was equipped for monitoring important parameters such as pH, temperature, liquid and gas flow, pressure, liquid level, and safety functions to detect explosive gas concentration in the atmosphere. Using these inputs, the PLC was programmed to respond appropriately to maintain functions within the operational range of the CLPS (Table 1).

Table 1 - PLC Control Response

Parameter	Acceptable Range	PLC Response to Control CLPS
pH	7.0 – 8.0	Turn on chemical pump feed into C-LFT for caustic or acidic solution to bring pH back to acceptable level. Stop C-FFR pump until leachate conditions are acceptable
Ambient Temperature	34 – 36°C	When temperature falls to 34°C, turn on heat circulation pump until temperature reaches 36°C
Pressure	1" to 12" water column	If pressure falls below 1" water column, open solenoid valve to external CO_2 gas tank to bring system back to 6" water column. If pressure rises above 12", shut down entire pilot lab. Dangerous conditions possible.
Leachate Temperature	34 – 36°C	When leachate temperature falls to 34°C, turn on heat circulation pump to leachate heat exchanger until temperature reaches 36°C

The PLC was also programmed with run commands and timing for the operation of the CLPS. To accomplish adequate mixing in the C-FFR, the PLC would call for the biogas recirculation compressor to run for 20 minutes every hour, pushing gas through the diffusers inside the bottom of the C-FFR and mixing the tank. Additional liquid leachate recirculation back to the C-LFT was also accomplished using the PLC in various circumstances including when heating, adjusting pH, or any other time more flow was desirable. In these circumstances, a separate pump in the manifold would take leachate from the C-LFT and discharge directly back to the C-LFT for recirculation.

The PLC output a Human Machine Interface (HMI) on a touchscreen monitor in the control room to keep track of system parameters and control functions. The HMI included graphs and histograms of crucial system parameters over the most recent hour, 12-hour, and 24-hour time periods to monitor system performance. Reactor room temperature and gas safety were monitored,

as well as liquid reactor temperature, biogas production, pH, and liquid flow. This data was manually checked daily as well as stored continuously on an external server

3.2.2 Heating

The pilot lab was equipped with a heat recovery ventilation (HRV) air exchanger. Air in the pilot lab was continuously recirculated with air from outside and the HRV transferred heat from the air leaving the reactor room to the incoming air. A hydronic heating system was installed in the pilot lab to heat the reactor space to 35°C to facilitate mesophilic temperatures. A Takagi T-D2-OS-LP tankless water heater, powered by propane from a large temporary tank located outside the trailer, heated a glycol mixture that was circulated through 64 feet of Slant/Fin Baseline 2000 baseboard heat tubing using a Grundfos Alpha 15-55FC circulation pump. Using a thermocouple located in the HRV ducting, temperature was controlled by the PLC as described in Table 1.

3.3 CLPS Configuration

The CLPS reactor system was constructed within the pilot lab. Two three-hundred-gallon polypropylene tanks were repurposed from a previous Colorado State University study. Banjo bulkhead fittings were used to provide gas-tight access to the tanks. One tank was set up as the C-LFT and acted as the primary leachate storage and central hub of the system. The other tank was set up to be the C-FFR and was loaded 50% by volume with Kaldnes K-1 floating plastic media commonly used in Moving Bed Bio Reactor (MBBR) applications due to its high specific surface area (500 m^2/m^3) for attached growth organisms. Two 20” Pentair EPDM Membrane Air Diffusers were installed at the bottom of the tank and plumbed to a California Air Tools SP-9413 1.0HP Air Compressor Motor, which was mounted with strut underneath the trailer to recirculate the produced biogas through the C-FFR and provide mixing. A sidearm sample port was installed in the C-FFR to access sludge and media using a 2” Valterra 2203X PVC Unibody Gate Valve. The

tank was connected at the effluent side to the C-LFT using 3” PVC pipe. A large pore outlet filter constructed of PVC pipe perforated with ¼” holes and wrapped in a coarse synthetic nonwoven geotextile fabric was installed inside the C-FFR to keep the media inside the tank while allowing liquid to flow through to the C-LFT. The same coarse synthetic geotextile fabric was used in many applications throughout the CLPS, CEU, and P-USSHR components, and is referred to as “ERC filter fabric”. The ERC filter fabric (Figure 8) was of unknown manufacturer, as it was repurposed from an unknown Colorado State University Engineering Research Center Hydraulics Lab legacy project.

Figure 8 – Coarse nonwoven synthetic geotextile fabric used for filter construction

An existing bulkhead port on the C-LFT tank was used to feed the reactors through 1.5” PVC pipe connected to the bulkhead fitting. Leachate was conveyed through this line to the filters, which provided protection against clogging the leachate feed pumps. Two interchangeable Pur Flo

200 Micron filters were installed for this purpose, allowing one to be in operation while the other is serviced. A Brazetek stainless steel tube in shell heat exchanger was installed to heat the flowing leachate to 35°C. The heat exchanger was plumbed to another Grundfos Alpha pump branched from the same heater line as the reactor room hydronic heat system and called to heat by a Pro Sense temperature transmitter in the leachate pump feed line. An inline sensor array was built to house a secondary (after heat exchanger) identical temperature transmitter and a pH probe. A pump manifold to feed four channels, each with a Northern Tools 2.2 GPM Diaphragm Pump, was constructed using PVC. Inline 60 mesh stainless steel strainers were installed before each pump to prevent clogging. Each pump was plumbed to its destination using ½" Gates Adaptaflex rubber hose after passing through a flow meter for PLC control.

Prior to beginning controlled experiments with the CEU, the C-FFR was inoculated by being fed manure leachate representative of the feedstock to be used during experimental runs for one month. A 58-gallon plastic drum was repurposed from a cucumber pickling barrel purchased from a surplus website to serve as the C-USSHR. The barrel was set on a drum dolly to wheel in and out of the pilot lab when filled with manure. The inlet was attached to the leachate delivery line with a union fitting. Inside, the inlet was fitted with a bidirectional filter apparatus for both injection and draining. The filter consisted of 1 /2" perforated PVC pipe wrapped in ERC filter fabric.

The CLPS was designed with a sump basin located underneath the trailer to collect flow and pump it back up to the C-LFT to remain in circulation. The sump was configured so multiple CLPS-connected processes could discharge to it via gravity feed. The C-USSHR effluent was routed to the sump drain manifold to mitigate back pressure problems encountered when connecting effluent straight to the inlet port on top of the C-LFT. The CEU, when connected, both

drained and discharged flow to the sump using the drain manifold. Additional ports were installed on this manifold to be available for future additions and experimental needs such as additional CEUs. Underneath the trailer, a sump basin access provided connection to the P-USSHR from heat-traced hose lines routed between the adjacent building and the pilot lab.

Biogas was collected as it was produced in each reactor and exited along with the leachate before liquid-gas separation was accomplished in line. Gas was allowed to fill the headspace of the C-LFT and excess was purged through a venting line at the top of the tank as more biogas was produced. The gas traveled into the gas collection tank, which for the purposes of this study was of fixed volume and pressure, and effluent was vented to a flare mounted on the roof of the pilot lab rather than being reused for power or heat production, which would be implemented in a commercial scale system. A 55-gallon plastic barrel tank was used to collect biogas. A 2" PVC line was fed from the C-LFT tank into the gas barrel and submerged 12" deep in water for back pressure. Provision was made for measuring and recording biogas produced within the CLPS. A smaller ¾" PVC preferential flow path was submerged through another port in the gas barrel 6" deep to direct gas flow after it was routed through an EKM Metering gas flow meter equipped with electronic feedback. A signal was sent to the PLC to record each cubic foot of biogas produced. Biogas was then vented through a Spears ball check valve (to prevent backflow of oxygenated air) and a spark arrestor (to protect the system from explosion if the gas were to ignite after mixing with the atmospheric air upon discharge) before releasing to the flare mounted on the pilot lab roof (Figure 9).

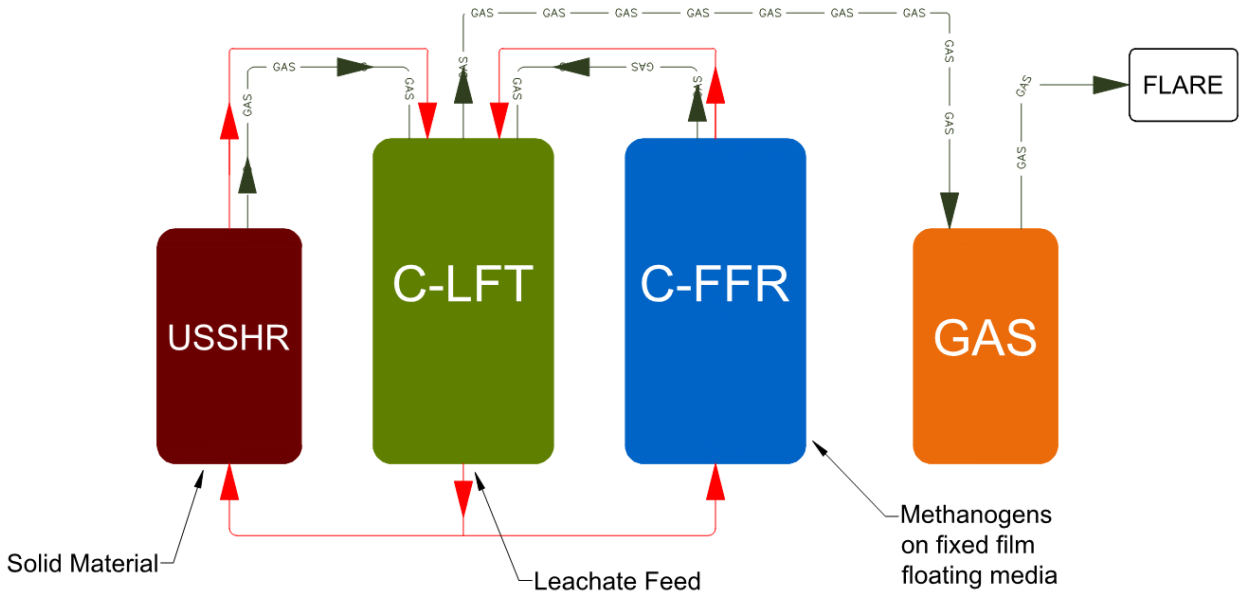

Figure 9 - CLPS process flow schematic

3.4 CLPS Operation

The CLPS was operated as a self-contained MSAD system for a period of six weeks prior to beginning CEU experiments. This period of time allowed the leachate to develop mature characteristics representative of full-scale operation. Additionally, it offered the C-FFR the ability to develop a microbiota specifically tailored to the manure used as feedstock for the column experiments.

The same feedstock source was used for the C-USSHR that would be used for column experiments. Manure was collected from JBS Five Rivers Cattle in Kersey, CO. Five Rivers maintains a consistent waste management practice and has been the source for most MSAD research to date, lending additional value to compare data with past research efforts. The manure used in this study was collected July 31st, 2018 from the Five Rivers feedlot. The manure was

brought immediately to the pilot lab location at CSU's foothills campus and the C-USSHR was loaded to begin CLPS operation.

3.5 Column Experiment Unit

To investigate the performance of the MSAD system, two identical MSAD reactors at column scale were constructed on a mobile cart-mounted skid, named the CEU. Each MSAD (CEU1 and CEU2) consisted of the following components:

- Three identical E-USSHRs
    - E-USSHRs were upflow reactors where solid feedstock (i.e. manure) was loaded at start of each experiment.
- Dedicated calibration columns (CCs) for each E-USSHR to capture daily flow
    - CCs functioned to individually collect and store each E-USSHR's entire daily flow until it was manually pumped out and back into the main flow of the system. This enabled measurement of total flow volume through each reactor each day as well as sample collection before pumping back to the system.
- One main E-LFT
    - The E-LFT is the central "hub" of the system, circulating continuously by feeding both the E-USSHRs and the E-FFR as well as receiving the effluent from each.
- A secondary Hydrolysis Feed Tank (E-HFT) for enhanced data control
    - A secondary leachate feed tank was added to the column setup in between the E-LFT and the E-USSHRs so that the same concentration leachate was pumped into the hydrolysis reactors during the entire day (as opposed to being mixed with E-FFR effluent continuously, enabling more consistent operation of systems to simplify data analysis).

- Two identical E-FFRs to alternate between experimental runs
    - One E-FFR was kept isolated from the rest of the CEU by circulating with previously digested stock leachate from the CLPS. Each new experimental run started with a “fresh” E-FFR not impacted by the previous column study. Since the E-LFT was initially filled from the same CLPS leachate, this also ensured the E-FFR had been fed with the same concentration leachate as used for experiment startup and was given time to adapt naturally.
- A variable volume biogas collector to collect and measure produced biogas.

CEU1 and CEU2 were run in parallel so that results could be compared in real time. The CEU was designed with high process control to track the leachate throughout the MSAD experiments (Figure 10).

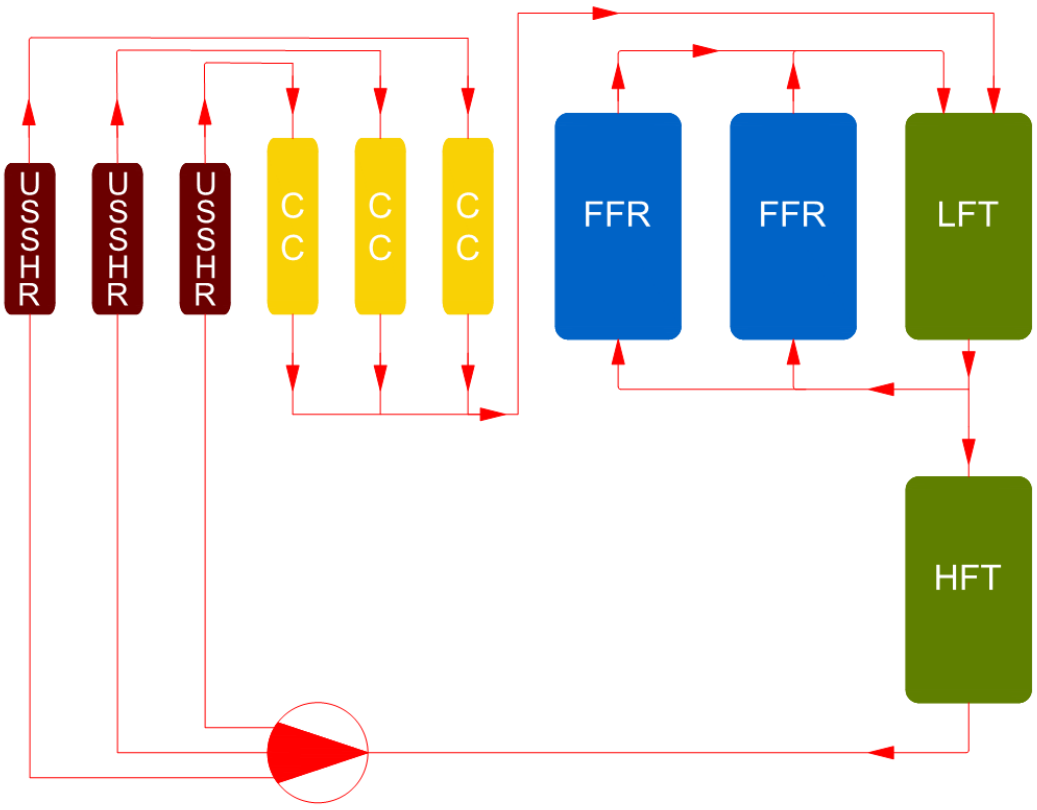

Figure 10 - CEU MSAD process flow

The CEU cart was mounted with two large metal enclosures. One served as the electrical cabinet and housed all electronics on the system for safety in a Class 1 Division 2 environment (Figure 11).

Figure 11 - Photo of electrical cabinet

The electrical cabinet was affixed with a rapid purge device to manage the air delivered from a compressor in the adjacent CSU building and maintain positive pressure in the event of a gas leak in the lab. A vent was installed atop the cabinet to vent the enclosure when purging air. Banjo bulkhead fittings were used for each in/out of the enclosure, including liquid and gas pumps,

sensors, probes, and electrical connections. Another smaller electrical enclosure was installed internal to the larger enclosure and built to house raw electrical components and control items.

The other enclosure (Figure 12) was modified to serve as a refrigerated housing for the CCs, maintaining the composited leachate at <10°C while collecting the full days' effluent. The cabinet was insulated and then mounted with a Superstrut frame inside to attach the CCs. Banjo bulkheads were used to convey refrigerated liquid in and out of the enclosure. Glycol was cooled to 0°C in a Penguin brand chiller bath set up outside underneath the trailer and a diaphragm pump was used to pump the liquid up through a port in the floor and into the enclosure. The glycol was circulated through ¼" copper tubing which was soldered to copper sheets wrapped around the back of each CC. This served as an effective way to minimize further degradation of the collected leachate from each E-USSHR before a sample was drawn from the composited liquid of the day.

Figure 12 - Refrigerated enclosure with Calibration Columns

The E-LFT, E-HFT, and the E-FFRs were all made from home brewing carboys (Figure 13). The Strange Brew FerMonster 7 Gallon Carboy was chosen as an effective and economical option. Although the carboys had gasket seals on the lids, they were not intended for pressure and had to be modified to use silicon caulk as a sealant to prevent the caps from breaking. Banjo bulkheads of various sizes were used for plumbing connections to the carboys.

Figure 13 – Carboy plastic tank used as FFR

The E-HFT was designed to isolate the feed into the E-USSHR columns to provide a consistent feed concentration throughout each day, allowing for accurate analysis of organic leaching rate from the columns. The E-HFT was connected to an inline strainer and fed a Cole Parmer peristaltic pump with a multiple-channel head fitted with 1.14mm ID Ismatec 3-Stop E-3603 Lab Tubing located inside the electrical enclosure. The pump plumbing exited the enclosure and entered the E-USSHRs, each through a Spears Sch 80 PVC valve-union-valve assembly for detachment and isolation. The E-USSHRs were constructed with clear 2" Sch 40 PVC pipe mounted vertically on one end of the CEU cart and fitted with a Spears union on each side to detach and clean (Figure 14).

Figure 14 – USSHRs mounted on end of column cart

The inlets and outlets each were designed with a coarse filter made from PVC pipe and ERC filter fabric (Figure 15).

Figure 15 – Solids separation filter made from ERC filter fabric

The CCs, located inside the cooled cabinet, were also built out of 2” clear PVC so the contents could clearly be seen. A millimeter scale was affixed to each column to read depth of composite collected and calculate daily flow volume (Figure 16). A cross-shaped magnetic stir bar was placed inside each CC to mix liquid using a neodymium magnet before samples were drawn. The columns were each attached to one dedicated E-USSHR at the inlet. A common gas line connected the three CCs to the rest of the CEU MSAD for variations in liquid volume throughout the day and to vent produced biogas from the hydrolysis portion of the reactor. The liquid outlets passed through a PVC tee for sample access from the sidearm using Qosina luer lock fittings for syringe-drawn samples. A hand valve controlled liquid capture and drained the columns into a diaphragm pump which emptied the day’s flow back into the E-LFT.

Figure 16 – Calibration columns mounted inside the refrigerated enclosure

The E-LFT was fitted at the lid with various inlets and gas ports as a central hub of the system. The outlet passed through an inline stainless steel 60 mesh strainer and then into the electrical cabinet where a sensor array manifold was located. ProSense temperature probes were installed and the manifold was built to accompany other inline probes for future experimentation including pH, conductivity, and ion selective probes. The flow was then directed outside the cabinet once more to a switching manifold which controlled the inlet to each E-FFR feed pump line.

CEU1 and CEU2 were each designed with two identical E-FFRs which would alternate each 16-day experimental run. While one FFR was running in conjunction with the active USSHR

columns, the other was “at rest” circulating leachate from the CLPS. This design feature was important to ensure that each experiment was initiated with a microbiota representative of that expected in a large-scale system and that was not impacted by the previous experiment. This ensured a microbiota acclimated to a particular experimental condition was not being created. The valved switching manifold (Figure 17) allowed for the switching between feed source from the CLPS system for baseline operation and the local CEU MSAD connected with the USSHRs. After this manifold, the leachate fed through a Cole Parmer peristaltic pump in the electrical cabinet with MasterFlex 18 tubing to feed the FFRs.

Figure 17 – CEU cart with valved switching manifolds (bottom right) for E-FFR control

The carboys used as the E-FFRs included large pore filters constructed from PVC pipe perforated with ¼" holes and wrapped in ERC filter fabric on inlets and outlets to retain floating fixed film media. The floating media used was the same Kaldnes K-1 media used in the C-FFR, commonly used in Moving Bed Bio Reactor (MBBR) applications due to its high specific surface area (500 m^2/m^3) for attached growth organisms. Each E-FFR was inoculated identically using media and sludge drawn from the 300-gallon C-FFR. A Pentair sintered glass fine bubble diffuser was introduced through a bulkhead into the bottom of the E-FFR to mix the reactor using recirculated biogas (Figure 18). Apollo Pumps VP5054 aquarium pumps with a dedicated inlet were installed inside the electrical cabinet and plumbed to the E-FFRs as well as a switching manifold similar to the leachate switching manifold discussed earlier (the gas switching manifold can also be seen in Figure 16). When an E-FFR was isolated and "at rest" it needed to be mixed using biogas from the CLPS instead of the CEU MSAD since its effluent was carried to the CLPS and the CEU MSAD carboys would collapse in vacuum if the biogas was pumped out. Each pump had 5 watts power and could push between 1.9- and 2.5-liters biogas per minute depending on system pressure. Another switching manifold was located above the E-FFR at the outlet to control effluent flow to either the integrated CEU system or to the CLPS sump, which pumped liquid back into the C-LFT. The effluent flow switching manifold was upsized in pipe size from ½" to ¾" PVC to convey the liquid-gas mixture before gas separation occurred in the E-LFT or C-LFT.

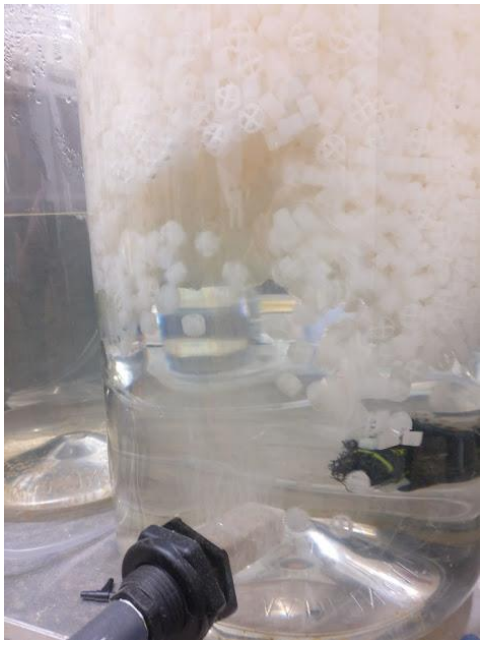

Figure 18 – Sintered glass diffuser mixing the E-FFR

Biogas collection was measured using variable volume gas storage reservoirs located in the pilot lab (Figure 19). Hoses connected the CEU to a collector built from a repurposed 5-gallon water jug with the bottom cut off. The jug was sealed at the top with an inlet created using PVC fittings and a Fernco flexible coupler and allowed to float on top of water contained in a 5-gallon Home Depot Homer bucket. To measure biogas produced each day, the collectors were vented into a gas calibration column powered by a vacuum pump located underneath the pilot lab. Vacuum was drawn in a 2" clear PVC column up to a specified height of water out of a separate water bucket, then biogas was vented from the collector into the pipe and allowed to return to atmospheric pressure to read depth and calculate gas volume (Figure 20). This process was repeated until all produced gas was measured from the day and sampled using a luer lock syringe port.

Figure 19 – Gas collectors and calibration column

Figure 20 – Gas calibration column under vacuum

3.6 Column Experimental Design

Five experiments were carried out to meet the stated research objectives. Previous MSAD studies (Section 2.7.2) used a hydraulic loading rate of ~40 cm/day. To assess the MSAD's varying hydrolysis performance, three different HLRs were tested ranging from 19 – 75 cm/day (above, below, and near the previously utilized flow rate) (Table 2). Experiments 1-3 were each carried out in one of CEU1 or CEU2 using a different HLR for each of the three E-USSHRs. Experiment 4 was conducted using CEU1 and the higher end flow rates (H: 75 +/- 0.9 cm/day) were tested in triplicate. Experiment 5 was conducted using CEU2 and a mid range flow rate was tested (M: 41 +/- 0.3 cm/day). The HLR of low end flow rates (L) was 20 +/- 1 cm/day.

Table 2 – Hydraulic loading rates tested in column experiments

Experiment #	Hydraulic Loading Rate (cm/day)			Experiment Description
	USSHR-1	USSHR-2	USSHR-3	
1	20.2	38	65.2	Low, Medium , and High HLR tested in parallel with one another
2	18.8	37.4	74.1	Low, Medium , and High HLR tested in parallel with one another
3	21.2	41.1	74.9	Low, Medium , and High HLR tested in parallel with one another
4	74.9	76.3	74.6	Triplicate test in parallel with High HLR
5	41.4	41	40.9	Triplicate test in parallel with Medium HLR

The column experiments were carried out over a duration of 16 days each. The time period of 16 days was selected based on previous MSAD leaching studies (Wu, 2017) (Hanif, 2013) that demonstrated the majority of methane yield was observed during the first two weeks of system operation. The extra two days provided a buffer to ensure data was not lost if a particular experiment experienced slower kinetics. Previous studies using manure had only focused on specific individual processes and not the entire system as a whole, therefore this experiment was designed to assess and characterize the overall performance of the MSAD over the 16-day operational period.

Fifteen experimental runs were conducted using CEU1 and CEU2 over the course of four months, with the initial five runs serving as trials to refine design and stabilize operation. encountered various instances of hydraulic failure through the USSHR columns. Because of the geometry of the E-USSHRs using tall and skinny pipes, these five initial experiments failed due to clogging and hydraulic failure. It was determined that fine particulate matter was shifting in the column and agglomerating together to prevent hydraulic flow. Thus, it was necessary to place

manure feedstock inside a synthetic non-degradable mesh bag (SumDirect 4”x6” synthetic organza bags were used) and load the E-USSHRs in layers separated by ERC filter fabric disks to aid hydraulics and prevent clogging (Figure 21). In the large-scale P-USSHR system with a typical geometry, this proved not to be an issue as the height to surface area ratio is much smaller. This reduces chance of clogging and hydraulic failure considerably, as evidenced by the successful operation of the P-USSHR (Section 3.11).

Figure 21 – Synthetic mesh bag to hold manure layers separate in E-USSHRs

Once consistent flow had been achieved, a set of two experimental runs was conducted to ensure proper operational and analytical protocol. Once processes and protocols were established and consistent results across experimental runs were realized, the five experiments were conducted for in-depth study to assess the performance of varying flow rates in USSHR columns.

3.7 Column Experiment Feedstock

The feedstock used as substrate for the column experiments in the CEU was the same manure collected for the CLPS from JBS Five Rivers Cattle in Kersey, CO (Section 3.4). The manure was brought immediately to the pilot lab location at CSU’s foothills campus and the

portion to be used for CEU experiments was stored in individual buckets in the refrigerator to preserve as-collected characteristics by minimizing degradation.

3.8 Column Experiment Startup Process

Three days prior to beginning the experiment, leachate from the C-LFT was injected into the E-LFT using the diaphragm pump in the control room. As previously stated, the C-LFT was used to provide a mature leachate representative of a large scale operation and to avoid use of leachate processed in prior experiments that may not be not be representative of typical leachate. The delivery line, originally routed to the C-USSHR, was disconnected from its termination point at the C-USSHR inlet and, using a 4-foot hose equipped with fittings allowing tie-in directly into the CEU anaerobically, was connected to the CEU using the E-LFT isolation fitting union.

Leachate was filled to the top of the E-LFT tank in this manner and the tank was reconnected to the CEU MSAD. The valve positioning for the E-FFR influent and effluent manifolds were carefully selected to route flow to and from the chosen E-FFR, while the other E-FFR would cycle leachate from the CLPS. The peristaltic pump was set to pump leachate through the E-FFRs at 20mL/min. The E-FFR was allowed three days cycling period with the E-LFT to finish digesting residual BMP from the CLPS before beginning the experiment.

At experiment start, 400g initial feedstock (manure) was loaded into each of the three E-USSHRs. The manure was pre-processed to achieve consistent homogeneity and therefore accurate replicates. First, all the manure was processed in a mechanical grinder [JWC Environmental Model 10000 Muffin Monster]. The resulting material was carefully dispersed evenly by volume using a 1-quart bucket between each of the individual buckets that would be used for an experimental run to ensure homogeneity (Figure 22). Next, each bucket's contents

were manually forced through a small pore (roughly 1/2") stainless screen to break apart the chunks before repeating this process with an even smaller screen (roughly 1/4") to produce a highly homogeneous consistency (Figure 23).

Figure 22 – Initial substrate processing method

Figure 23 – Loading columns with initial substrate

The manure was then loaded into each column in 10 separate layers (referred to as lifts) of 40g each. The layered loading enabled the use of the mesh bag for hydraulic enhancement (Section 3.6). The six E-USSHR columns (3 for each of CEU1 and CEU2) were loaded simultaneously, measuring and then loading the lifts one at a time across each column (i.e. the first lift in Column

1, then the first lift in each subsequent column before beginning to load the second lift in each column).

Once loaded, the E-USSHR columns were placed in their dedicated racks on the CEU and connected to CEU1 and CEU2 using the union fittings. Leachate was drawn from the E-LFT tank using the luer lock valve sample port and a 140 mL syringe. One at a time, syringes were injected into the luer port on the E-USSHR influent line and liquid was discharged until each E-USSHR column was saturated and the liquid level reached the top union cap. The total volume leachate injected was recorded for initial mass estimation.

To begin the experiment, leachate was filled into the E-HFT up to the 120 mm line to provide four days flow into the E-USSHRs. Initial samples were drawn from all locations to analyze the chemical makeup of all liquid beginning in the system. This included the E-FFR, the E-LFT, and the E-HFT. This analysis revealed that for Experiments 4 and 5 the initial leachate COD concentration was notably higher than for experiments 1 – 3 (Table 3). The cause for this sudden increase is unknown. There were no known events such as irregular additions or water loss that may have potentially led to higher concentrations in the leachate, so it may be that the CLPS was building up non-biodegradable COD over the experimental period and these experiments, performed last, saw the effect. This is uncertain, however, because during the previous three months of operation of the CLPS and CEU leachate concentrations were consistently around 2,500 - 3,000 mg COD/L. Experiments 1-3 were in this range before the sudden spike for both Experiments 4 and 5, which were performed simultaneously using CEU1 and CEU2. Analysis for leached COD was performed taking into account initial COD concentrations (Section 3.10.1). Using this method overcame issues with differences in initial concentrations by measuring total

mass COD leached out of the E-USSHRs, allowing directly comparable leaching rates regardless of initial concentration. Potential effects on hydrolysis are discussed in Section 4.1.1.

Table 3 – COD concentration of leachate initially fed to CEU at beginning of each experiment

	Initial Leachate COD at Experiment Start [mg/L]
Ex 1	3102
Ex 2	3288
Ex 3	2486
Ex 4	6470
Ex 5	6650

After measurement of initial concentration, flow was then started from the E-USSHR feed pump, spinning at 3.1 RPM to produce the prescribed HLR for each E-USSHR. CEU1 and CEU2 each had a dedicated pump head with 7 channels to distribute flow proportionally to the three E-USSHR columns. Column 1 received one channel, Column 2 received flow from two channels, and Column 3 received the flow from 4 channels to load the three E-USSHRs with low, mid, and high HLRs respectively. The multi-channel feeds combined into the same outlet hose to route to the column.

3.9 Column Experiment Sample Collection

3.9.1 Liquid Sample Collection and Preparation

Leachate was continuously circulated through both the E-USSHRs and the E-FFRs for 16 days. Each day, operation of the CEU involved first checking and recording environmental characteristics including ambient temperature in the pilot lab. Twenty-four-hour flow through E-USSHR columns was recorded by taking height measurements of CCs. Height in the E-LFT and

E-HFT were also recorded to track volume of CEU leachate. Samples were then drawn from the E-LFTand E-HFT and preserved for analysis.

Then, using a neodymium magnet, each CC was mixed with the internal magnetic stir bar by raising the stir bar to the top and letting it fall all the way through the collected leachate sample four times. To ensure complete mixing, the stir bar was then vigorously pulled through the leachate up and down before another round of controlled dropping of the stir bar. Samples were then collected from the inline luer sample port. To purge the sample line of stagnant leachate, 100mL was drawn into each of the dedicated 140 mL syringes (one at each CC). The sample syringe (5mL or 10mL volume) was attached to the side arm of the luer lock fitting and was then used to draw a full volume liquid out. Then, holding the syringe vertically upside down to discharge up and purge entrained air, the contents of the syringe were emptied back into the CC. An additional 30 mL was then drawn into the 140 mL syringe (now filled with a volume up to 130 mL) to finish purging the line before finally drawing the 5mL or 10 mL sample into the sample syringe. All three CCs for each system could then be opened at bottom to drain into the pump-out manifold.

The diaphragm pump connecting the CCs with the E-LFT was then powered on after verifying safe operation and the absence of explosive gas from the reactor room atmosphere. Control for the pumps was located on the touchscreen interface in the Pilot Lab control room. Biogas was measured by turning on a vacuum air pump located underneath the pilot lab (after verifying the absence of explosive condition in the space underneath the lab) and connected at the inlet to the gas collector manifold with valve control. The pump discharged to atmosphere. To measure biogas volume produced, the valve was opened to allow the Gas Calibration Column to vent gas and build vacuum pressure by drawing water up into the column until measurement 0 mm (at the top). The floating gas collector was then opened to the vacuum column to vent gas down to

atmospheric pressure, where total volume gas was recorded by means of height on the column. This process was repeated until less than one full column remained. The column was then filled only with water to the approximate volume remaining in the collector, ensuring gas volume was not expanded nor contracted due to vacuum pressure remaining (i.e. liquid level was even to outside the column). Samples collected during the experimental runs were preserved anaerobically at 4°C in the syringes they were drawn in until analysis.

Samples were analyzed individually at first for higher resolution during the highly active first few days. After the initial spike, composites were used for simpler analysis. Initial values and days 1 and 2 were analyzed for COD individually. Days 3 and 4 were composited in a two-day sample and then the rest of the experiment was carried out with 4-day composites. Composites were prepared from each day's preserved samples by normalizing proportions volumetrically by flow collected each day. To illustrate in an exaggerated example (flows were fairly consistent each day in each reactor), a sample might have been prepared from day 3 and 4 samples which saw flow of 400 and 600 mL, respectively. This composite sample would therefore include, in a 10 mL sample, 4 mL from Day 3 and 6 mL from Day 4. To create accurate proportions, a purpose-built work station was constructed by building a suspended plywood platform and drilling exact-sized holes (the outer diameter of the syringes) to hold clean syringe bases attached to luer fittings where the sample syringes could attach. The samples were forced into the open syringe base where a pipette could access from the top and precisely draw the specified volume. Prepared composites were labelled and stored separately for individual analysis.

At shutdown on Day 16, all final height measurements were recorded and samples were taken at all locations including CCs, E-LFT, E-HFT, and E-FFR. The E-USSHR columns were taken off the cart after shutting the isolation valves and brought to the lab for post-run analysis.

Ending mass was measured, then columns were emptied to sample post-digested manure from each lift as well as remaining leachate (the liquid portion left in the columns). The pilot cart was then returned to idle state, pumping all E-FFRs from the CLPS leachate contained in the C-LST to prepare for the next experiment set.

3.9.2 Solids Sample Collection and Preparation

Solid samples of initial manure substrate were sampled while loading the columns (Section 3.8). To ensure a representative sample of initial substrate was collected, a bucket at the end of the line was filled identically to the six columns as a pseudo "7^{th} column". Each time the six columns were loaded with a 40g lift of manure, another 40g was loaded into the sample bucket. The resulting sample was then stored in the refrigerator to keep preserved until total solids and volatile solids were analyzed (Section 3.10.3 below).

Final solids were sampled by draining all remaining leachate in the E-USSHR, then removing each lift and measuring the final mass (m_f) on a mass balance before taking a sample and preserving it in the refrigerator alongside the initial sample.

3.9.3 Gas Sample Collection

Biogas was sampled from the gas collection manifold using the luer lock port and the collector was opened back to the system to continue collecting the next day's production. Before leaving the system to run for the next day, all system valves were double-checked to ensure proper positioning. It was imperative to open the gas collector valve back to the system in order to allow produced gas a place to collect without over-pressurizing the system. It was also extremely important to shut the CC bottom valves to collect each USSHR effluent individually, as the columns were hydraulically connected with the valves open.

3.10 Column Experiment Sample Analysis

3.10.1 COD Analysis

COD analysis was performed according to Hach Method 8000 using Hach COD Digestion Vials High Range Plus reagent vials. Vials were measured colorimetrically using Hach program 435 COD HR. Each vial was measured twice to ensure reproducibility and the average was reported. COD leaching rate was chosen as the primary source of data so that results could be directly compared regardless of leachate makeup and concentrations, since influent COD mass is accounted for in the calculation. COD leached from each E-USSHR column daily was estimated using the COD concentration of both the influent (COD_I; from E-HFT sample) and effluent (COD_E; from CC sample) and the flow volume over a time period of analysis (V_{dr}; Section 3.9.1).

$$Mass\ COD\ Leached = (COD_E - COD_I) \times V_{dr}$$

3.10.2 Estimation of Hydraulic Loading Rate

The flow rate to each of the USSHR columns was regulated by the peristaltic pump. Back pressure against the pump caused a lower actual flow than indicated by RPM and tube diameter, so hydraulic loading rate of each of the columns was determined by measuring the flow rate through the reactor. Volume collected in each CC each day was divided by time during initial startup experimental runs to determine actual flow and HLR. The peristaltic pump speed was adjusted to match the desired flow and target HLR for each column. Reported observed HLRs are the total volume of water collected in the CC divided by the number of days of operation (Table 2, Section 3.6).

3.10.3 Total Solids and Volatile Solids Analysis

Volatile solids (VS) content of the initial substrate mass was used to normalize all column COD data per gram initial VS added to columns at experiment start. The initial mass sample was collected as described in Section 3.9.2. Using the careful column loading procedure described in Section 3.8, it was assumed that each of the six E-USSHR columns loaded together (three each from CEU1 and CEU2) were replicates and that the VS and total solids (TS) content of the initial mass sample applied to all six experimental columns. TS and VS were measured according to EPA Method 1684. The analysis was begun by blending each of the full samples separately in a Vitamix blender before drawing small representative samples in trays. The mass of each tray was measured, then the mass with substrate. The trays were baked at 105°C until water content was gone and then weighed again. After it was confirmed water sufficiently evaporated (mass was remeasured by removing from the oven and weighing in 10-minute increments until no change was detectable) the trays were placed in a muffle furnace at 550°C to volatilize organic content. TS was determined by subtracting empty tray weight from measurements, then dividing the post-105°C mass by the original mass. VS was determined by dividing the post-550°C mass by the post-105°C mass.

Initial mass (m_i) was estimated by using mass measurements taken of all components of E-USSHR columns before and after loading with manure. Before loading each column, the "dry" weight of the column without filter cap was measured, followed by the filter cap alone, the 10 mesh bags together, and the 10 ERC filter fabric disks. After loading, the entire column was weighed and the difference was recorded as the initial manure mass. Final mass (m_f) was estimated from weight of entire column after digestion, weight of contents (manure and leachate) emptied from column, and original "dry weight" column and components from measurements taken at experiment start.

Total solids and volatile solids destructions were calculated using the TS and VS results with estimates of initial (m_i) and final (m_f) mass.

$$TS\ destruction = \frac{m_i \times \%TS_i - m_f \times \%TS_f}{m_i \times \%TS_i}$$

$$VS\ destruction = \frac{m_i \times \%VS_i - m_f \times \%VS_f}{m_i \times \%VS_i}$$

3.11 Prototype Scale USSHR

The P-USSHR was constructed following successful operation of MSAD experiments demonstrated at column scale to address design concerns of scale up and to assess performance in terms of solids reduction and leachate organic content. Using the C-FFR and C-LFT (300 gallons each), a two-week experimental run was completed with an 800-gallon USSHR. A four-cubic-yard construction dumpster had been converted to run as the P-USSHR for a previous experiment (Lewis, 2018). The dumpster was mounted on a steel frame with an axle about the center so it could rotate fully upside down to dump processed material between runs (Figure 24).

Figure 24 – Rotated dumpster about central axis displaying ability to dump out USSHR between runs

Leachate inlet and drain ports were installed at the bottom using Banjo bulkhead fittings underneath the reactor. Inside, a plastic pallet served as the base to support the solid substrate. A strut-mounted injector array with 12 ports diffused leachate flow up into the substrate bed in an evenly-distributed grid (Figure 25). The 12-port diffuser aimed to improve the leachate flow distribution in the reactor, which was noted with challenges in previous experiments (Lewis, 2018). Specifically, difficulties were encountered with heating and differences in volatile solid destruction throughout the reactor indicated uneven liquid distribution. The assembly was bracket-mounted to strut channels fixed to the inside of the reactor and held the pallet base in place, using

2x4s as spacers. Underneath the pallet base “false floor,” a drain port wrapped in ERC filter fabric was used to drain leachate at the end of runs.

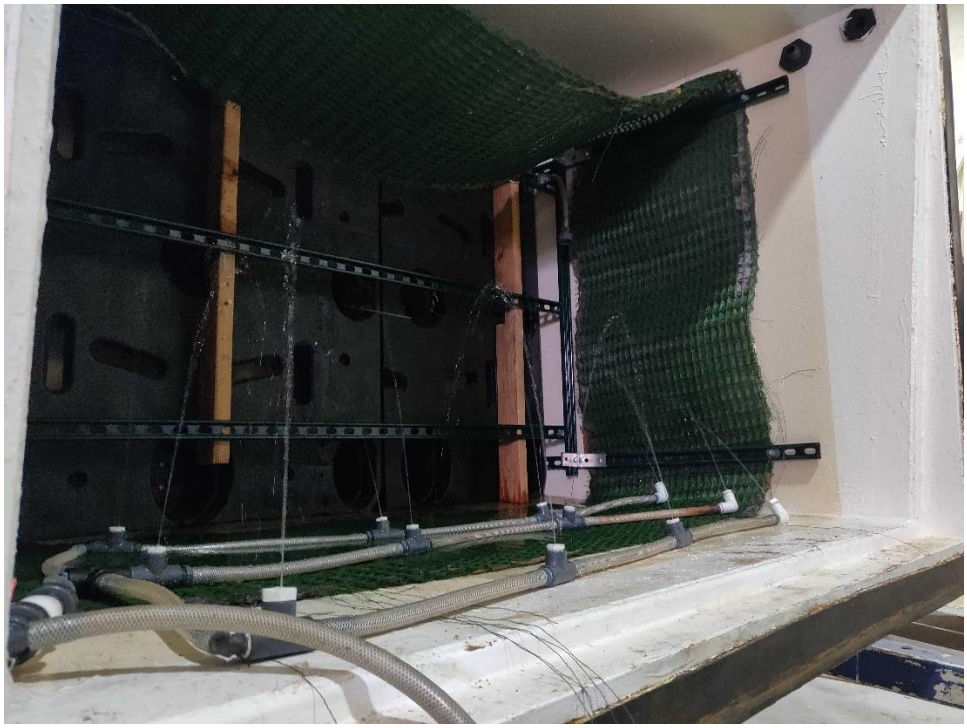

Figure 25 - Leachate injectors demonstrating equal flow

The effluent was routed through a PVC pipe assembly mounted to the removable lid of the reactor and attached by hose to the outlet port on the side. Seven cross pipes attached by flexible couplers were fixed to the pipe frame and collected leachate across the reactor (Figure 26). The cross pipes were configured similarly to other outlet filters used in the MSAD experiments, wrapping ERC filter fabric around perforated PVC pipe. Liquid flowed down into the outlet and gas was separated off and flowed up into the gas outlet port (Figure 27), joining the P-USSHR gas collection system constructed outside the reactor in the lab space.

Figure 26 - Outlet collection pipes suspended from lid of P-USSHR.
ERC filter fabric wrapped around perforated PVC pipe cross sections allowed liquid effluent to collect and flow out of reactor.

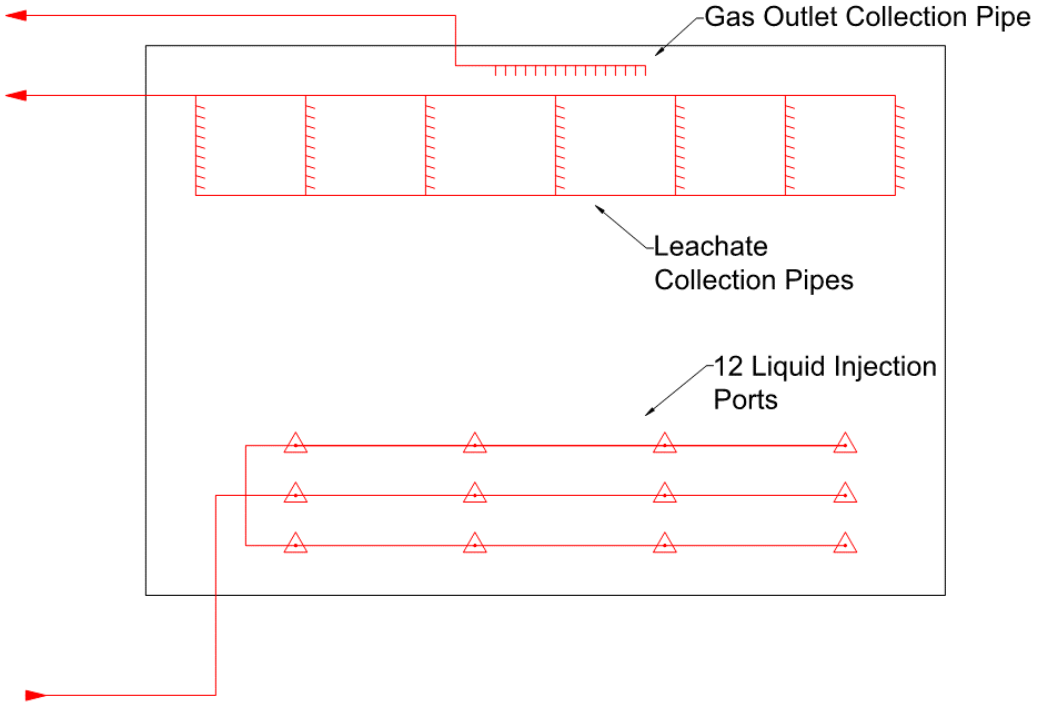

Figure 27 – P-USSHR flow schematic

Biogas produced in the hydrolysis process and/or released inside the P-USSHR was collected through an outlet filter located above the liquid outlet. Gas flow was routed into a variable volume gas collector constructed from a 48-gallon plastic barrel floating upside down over water in a larger 58-gallon barrel. To pressurize the system, ensuring anaerobic conditions would sustain during periodic effluent pump operation, the collector was restricted from floating higher (expanding volume) by ratchet straps. This allowed sufficient biogas storage within the P-USSHR system to maintain positive gas pressure when the pump kicked on and emptied the sump of collected leachate. When the floating gas barrel reached the restricted maximum volume, excess produced gas then flowed out of the collector, through a gas meter, and up into a pressure regulating tank, where it was ultimately vented out of the system. The pressure was regulated to 6" water column by the outlet hose submerged in a water tank.

3.12 Prototype Experimental Design

The P-USSHR experiment was conducted in the following way:

The lid was removed with a forklift and the P-USSHR was loaded using a skid steer with 1 yd^3 loader bucket. Four level buckets full were loaded into the reactor, taking care to ensure load was representative of the manure as received (including the average proportions of inorganic dirt and rock materials as well as grasses present). The lid was replaced on the top of the reactor and sealed by applying a layer of synthetic grease and tightening by using C-clamps every foot around the rim of the entire lid to ensure a gas-tight seal.

The reactor was then filled with tap water to fully saturate the internal volume with liquid. A hose running from the lab building supply was connected to a float valve attached to an open top 58-gallon barrel on a drum dolly beside the P-USSHR. A 2.2 gpm diaphragm pump identical

to the pilot lab feed pumps was used to draw water out of this barrel, through a 60-micron Pur-Flo canister filter, to an electric heat exchanger, and into the bottom of the P-USSHR dispersion platform. Once the P-USSHR was fully saturated with liquid, effluent would begin to flow through the PVC pipe assembly and out of the reactor. The outlet hose was temporarily routed directly to the open top barrel during the startup procedure. Once the 58-gallon barrel was full, the float valve would automatically stop the flow of building tap water. The initial start-up water was allowed to recirculate for 24 hours to come to the design temperature of 35 C. After the desired temperature was reached, the 58-gallon barrel was disconnected and leachate from the Pilot Lab LFT was pumped into the P-USSHR to begin the experiment. The effluent hose was returned to its position flowing into the outlet sump, which was controlled by an internal float switch to turn on the utility pump, sending flow from the P-USSHR effluent back to the C-LFT by way of the sump located underneath the pilot lab.

Autosamplers were connected to both the inlet and outlet of the P-USSHR to collect samples every hour. The samples were preserved and stored for future analysis to be included in future research. The experiment was carried out for two weeks at the high HLR used in the CEU studies (76 cm/day). This amounted to a flow of approximately 1.1 liters per minute.

3.13 Prototype USSHR Feedstock

The cattle manure used as feedstock for the P-USSHR was delivered to the pilot lab site at CSU's foothills campus by Horton Feedlot in Wellington, CO. The manure was delivered December 18th, 2018 and was stored outside for one month before the P-USSHR experiment began. Outdoor storage was appropriate in this case since it reflected the same storage used on-site at the feedlot and the manure experienced minimal biological degradation due to the cold temperatures outside in December.

3.14 Prototype Experiment Analysis

3.14.1 Sample Collection

The volume of the P-USSHR was divided into six sections for the purpose of analysis. Looking down at the top of the reactor, the sections were divided as shown in Figure 28. Each section was a full column extending to the bottom of the reactor.

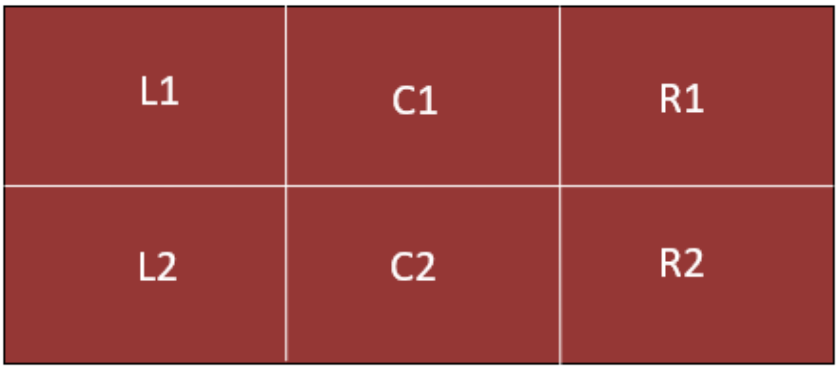

Figure 28 – P-USSHR volumetric divisions (seen from the top of the reactor looking down)

Initial solid substrate sample (manure) was taken by collecting a representative sample from each loader bucket and mixing thoroughly in a 5-gallon bucket. It was assumed the method of loading the initial substrate using a loader bucket produced a reliably consistent TS/VS content across the entire reactor, though it was not possible to accurately sample and measure the initial solids from each of the six sections individually. The assumption was made that this large volume bucket was representative of field conditions without mixing, and care was taken when scooping bucket loads not to disturb the pile but to take a large portion without adding any mixing.

At the end of the two-week (14-day) digestion period, solid samples were taken from the P-USSHR from each section (Figure 28) by taking a full core of post-digested mass using a sample device similar to a sludge judge crafted from 2-inch diameter PVC pipe (Figure 29). The core went from top to bottom of substrate bed where possible (in the sections at the front of the reactor where

it was accessible: L2, C2, and R2). The back sections, L1, C1, and R1, could only be accessed for sampling in the top portion. Each sample core was deposited in a clean bucket and mixed thoroughly before drawing three smaller samples from each for TS/VS analysis performed according to the same procedure as the column study detailed earlier in Section 3.10.3.

Figure 29 – "Sludge judge" device used to sample solid cores from P-USSHR

3.14.2 Estimation of TS and VS destruction

To assess hydraulic distribution, percent volatile solids remaining after the digestion period was analyzed, along with volatile solids destruction. To enable volatile solid destruction calculations, initial (m_i) and final (m_f) mass had to be determined. It was not possible with the available equipment to measure the total initial or final mass in the P-USSHR, so a procedure was developed to estimate m_i and m_f using bulk density and volume. For the m_i , the bulk density of each loader bucket full was estimated by filling a large plastic bucket to the height of the loader bucket and measuring bulk density by dividing the mass by volume. This bulk density was then

multiplied by the volume of the level loader bucket (one cubic yard) to determine mass added to the reactor. A +/- 5% error was assigned to m_i to account for uncertainty in measurement and in manufactured bucket size.

Bulk density for m_f was measured separately for each of the six sections. The volume of each sample core drawn from each section was measured by height in the two-inch pipe before being deposited into the sample collection bucket. The bulk density was determined for each core by measuring mass of sample and dividing by the volume of the core it was taken from. The bulk density was then multiplied by 1/6 the volume of the post-digested manure to find the total m_f in each of the six sections. Since a full depth core was not possible to access for sampling from the three back sections (R1, C1, and L1), a 'bulk density depth factor' was determined for each from the corresponding section next to it that was accessible (i.e. R2 depth factor assumed to be applicable to R1, C2 to C1, and L2 to L1). An 8" deep core was taken from each of R2, C2, and L2 and bulk density was measured the same way as the full depth core. The bulk density depth factor was determined by dividing the calculated bulk density of the full depth core by the 8" core. This gave a ratio of the bulk density of the top 8 inches to the full depth. The resulting depth factor was multiplied by the calculated bulk density from the top 8" of the corresponding inaccessible section to estimate overall bulk density in that section.

Final volume (V_f) at the end of the digestion period was measured by calculating volume of the USSHR up to the level of the manure bed. Using each of the 4 corner sections, the depth to the rim was measured and the average was taken as the final height for volume calculations. The 6-inch pallet floor beneath the manure bed was subtracted from this height and then the result was multiplied by length and width to get volume of final substrate. The average of the bulk densities estimated from each section was multiplied by V_f to estimate m_f .

Percent TS and VS as well as TS and VS destruction were estimated using the equations in Section 3.10.3. The measured values of TS and VS were obtained for each of the six divided P-USSHR sections individually. The average of all six sections was used to estimate TS and VS destruction for the entire P-USSHR.

CHAPTER 4. Results

4.1 Column Experiment Results

Results of the experiment yielded successful operation of the fully-linked MSAD for 16-day runs. Demonstrating successful operation of the fully-linked system using cattle manure as feedstock proved the MSAD concept and opened the door for future detailed analysis to enhance processes and improve performance. Recommendations derived from this experiment for future research direction will be discussed later in Section 5. Additionally, many samples were chosen from this study to preserve for further analysis of other constituents of interest, which will enable the creation of a mass balance for the system that will track, in addition to carbon, valuable nutrients such as nitrogen, phosphorus, and salt ions (namely potassium and sodium).

4.1.1 COD Leaching Rate

The COD leaching rate was plotted by day (Figure 30) and cumulatively over the entire 16-day experiment (Figure 31) for the Low HLR, tested as replicates in one E-USSHR column in each of Experiments 1, 2 and 3.

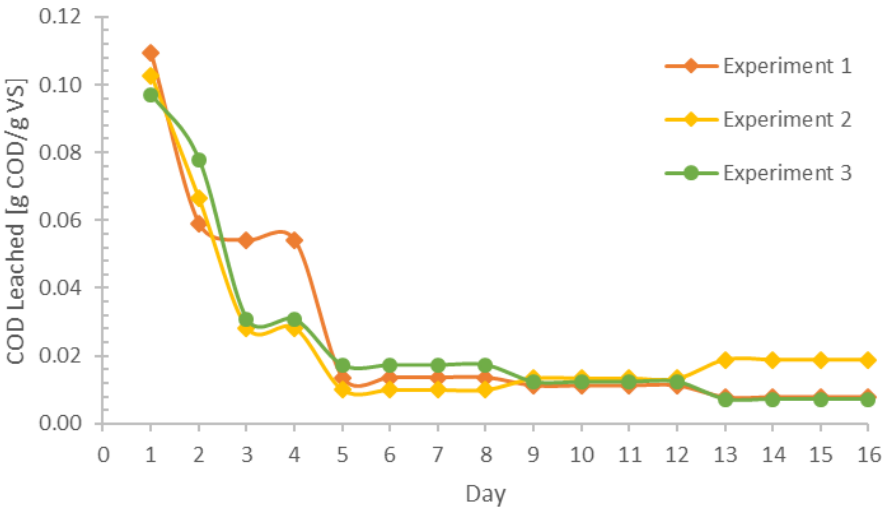

Figure 30 – Low flow [HLR=19cm/day] COD leached over time

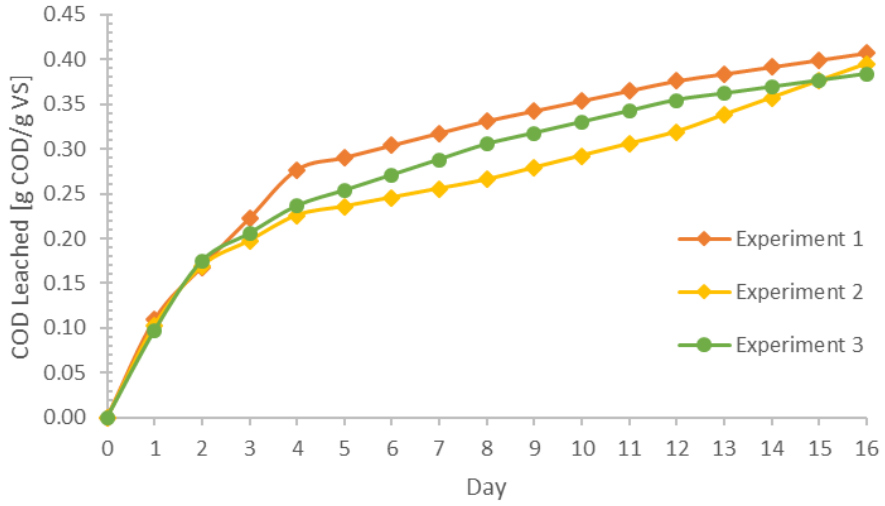

Figure 31 - Low flow [HLR=19cm/day] cumulative COD leached over time

It is evident to see that an initial slug of the more easily leachable COD comes out quickly (in the first two to four days). The rest of the COD comes out much more slowly, with days 5 through 16 averaging just 12.4 mg COD/g VS initially loaded per day compared to the first four days averaging 61.6 mg COD/g VS each day, about five times the rate. Similar trends can be seen at higher flow rates. The daily and cumulative COD leaching rates at Medium HLR were plotted

(Figure 32 and Figure 33), tested in one USSHR column in each of Experiments 1, 2 and 3, and in all three USSHRs in Experiment 5.

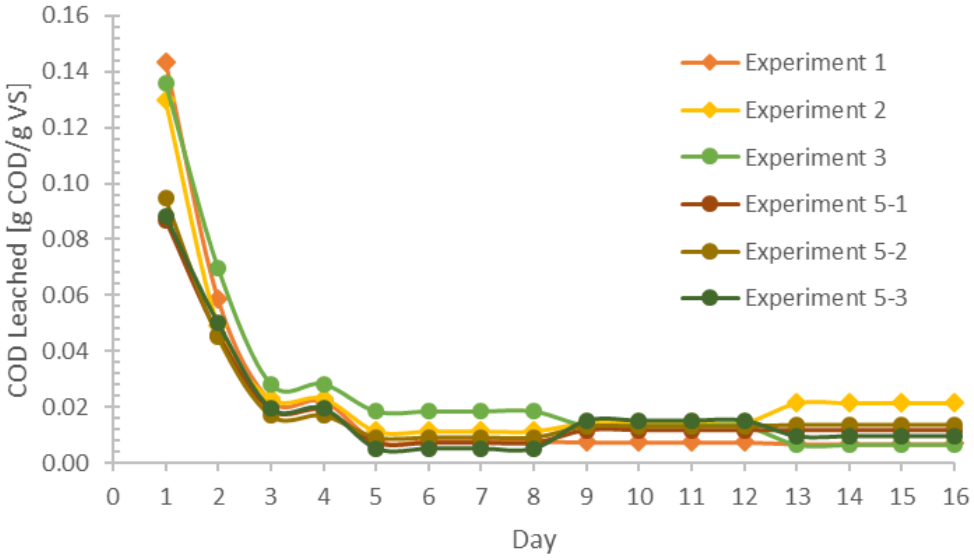

Figure 32 - Medium flow [HLR=38cm/day] COD leached over time

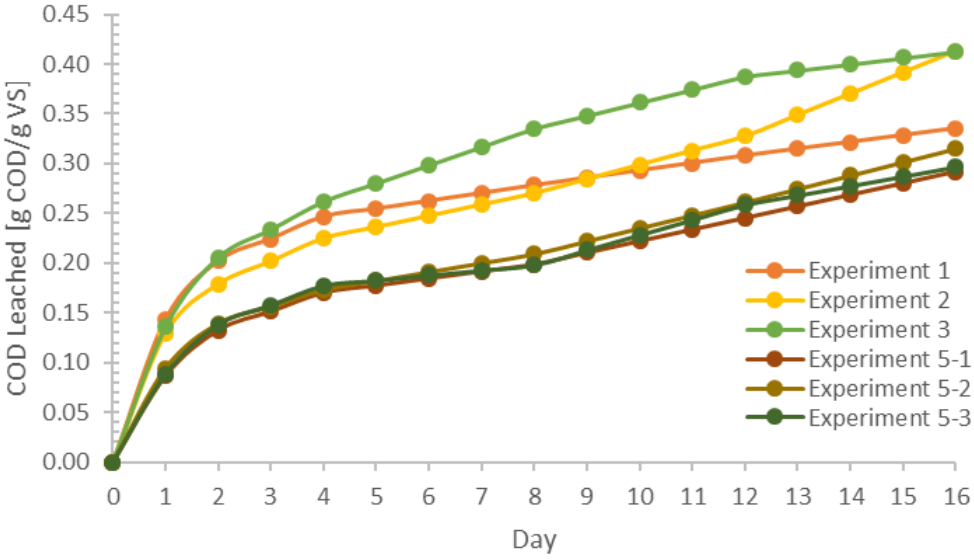

Figure 33 - Medium flow [HLR=38cm/day] cumulative COD leached over time

The same trend appears here, with days 1-4 averaging 52.3 mg COD/g VS, more than 4.6 times the rate seen over the last 12 days (11.3 mg COD/g VS). It is interesting to note how

consistent the leaching rate remained across all three columns in Experiment 5 compared with slightly more variance between the three other columns each tested during a different experiment. This may indicate that another influencing factor may have been present in the system during Experiment 5. Though it was attempted to keep all experiments under identical conditions, environmental factors could have played a more significant role than expected, such as slight variations in temperature in the pilot lab. Another possible explanation is that, as the experiment went on and the "sludge age" of the bacteria contained within the leachate increased, the more mature bacteria began to produce more consistent results.

The High HLR experiments also showed substantial early production, but the initial slug of organic content came out even faster (Figures 34 and 35).

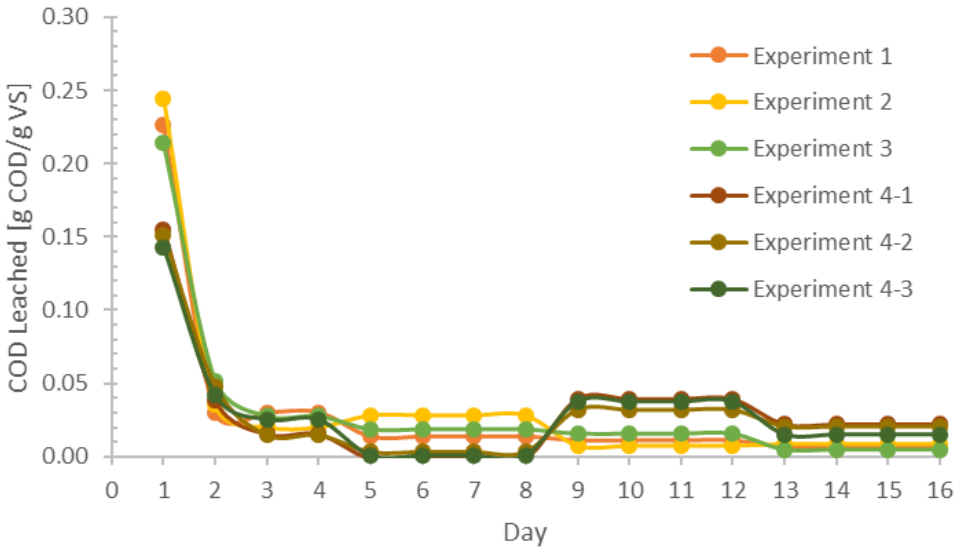

Figure 34 - High flow [HLR=76cm/day] COD leached over time

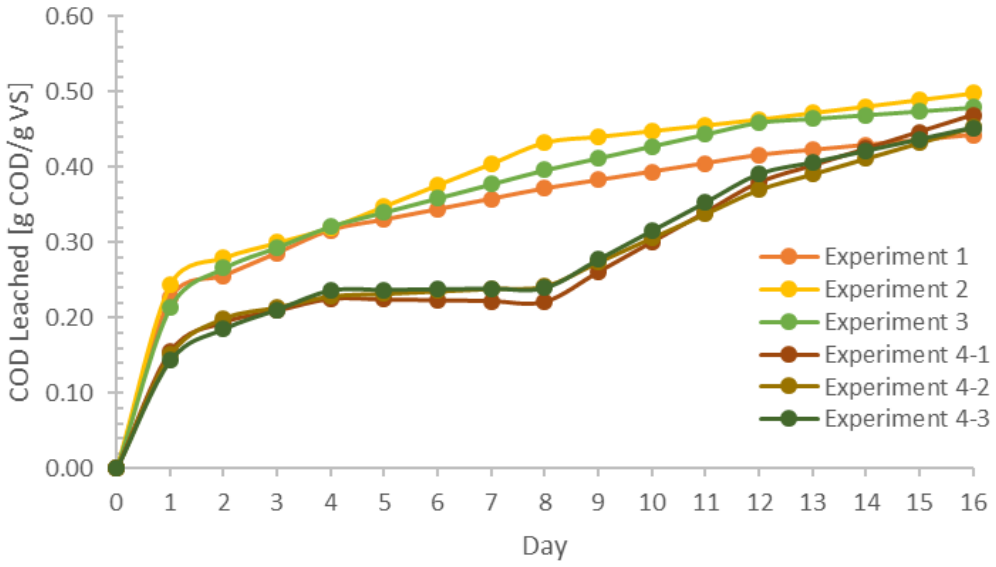

Figure 35 - High flow [HLR=76cm/day] cumulative COD leached over time

Similarly to Medium HLR results, all three columns from Experiment 4 very closely matched in leaching rate at the High HLR. In addition to the above mentioned possibilities, this may be linked to the noted peculiarity that for both Experiments 4 and 5 the initial leachate COD concentration was notably higher than for other experiments (Table 3). To understand this phenomenon and to acquire more data to confirm if it is repeatable or an anomaly, future research should perform more replicates of Experiments 4 and 5 using mature leachate. Whatever the cause of the higher background COD concentrations, it appears hydrolysis rates were affected, particularly in the first few days of operation. In both the medium and high HLRs, the three E-USSHRs seeing the higher initial COD concentration yielded less COD leached than the other three USSHR columns until the cumulative begins to catch up towards the end. It is possible the high concentrations of influent COD impact the solubility of hydrolyzed particles and COD takes longer MSAD run time to leach out.

At the highest HLR tested, the first day realizes over 40% of the COD leached during the entire 16-day experimental run (189 mg COD/g VS on day one). In total, after 16 days, the experiments averaged 465.8 mg COD/g VS at the High HLR. Days two through four in the High HLR experiments then yielded lower leaching rates than Medium and Low HLRs did, demonstrating this initial slug of the more easily leachable organic content coming out much faster at higher flow rates, which is reasonable to expect. It is important to note that the lower flows did not "catch up" by day 16, however, yielding a total of just 395.0 mg COD/g VS and 344.2 mg COD/g VS for the Low and Medium flows, respectively (Figures 36 and 37; error bars represent one standard deviation).

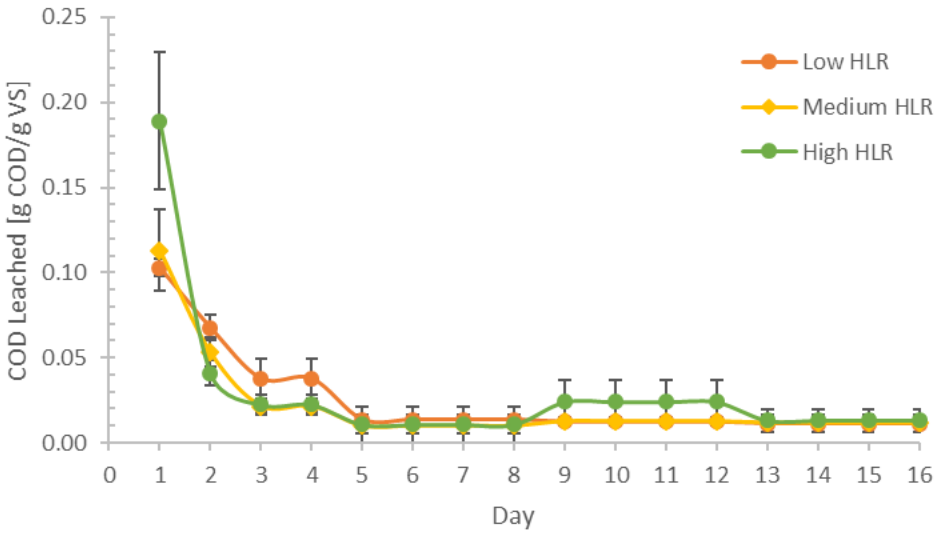

Figure 36 - COD leached over time by hydraulic loading rate

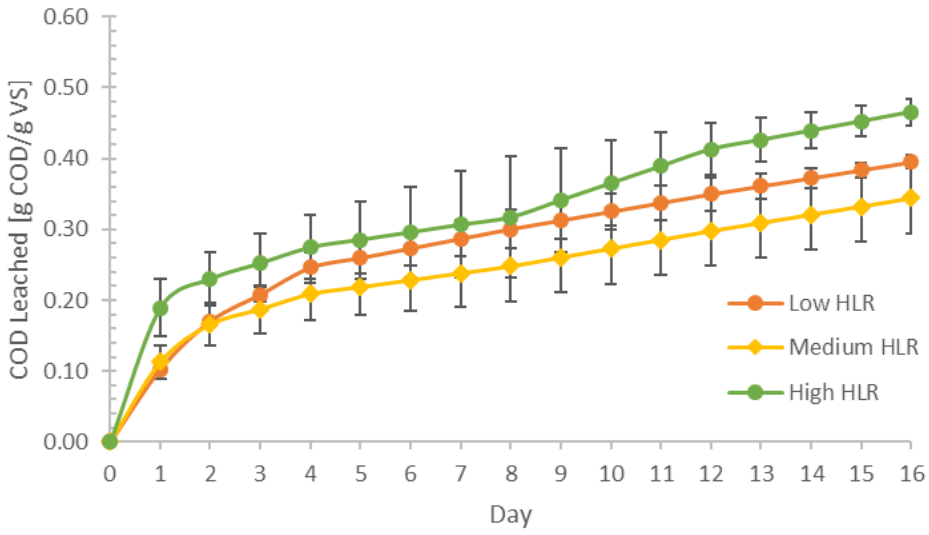

Figure 37 – Cumulative COD leached over time by hydraulic loading rate

This trend seems appropriate that easily leached COD would come out more quickly in higher flow rates, but it is peculiar that the lower flow rates do not appear to catch up over time. It could be expected that if hydrolysis was carried out over a much longer duration, surpassing that which would be economically viable for the MSAD system to run, this "missing" portion would eventually be leached out. Another possible explanation is that at lower flow rates, hydrolysis byproducts build up at too great a rate to fully push through the reactor and out, leading to reaction inhibition preventing further chemical degradation and leaching. These results show that a better yield of organics (represented as COD) can be leached from the substrate in the USSHR with a higher hydraulic loading rate. The optimal HLR would need to be determined through further research replicating this experiment at higher flow rates. This study has already begun, as the CEU was easily modified to accommodate higher flows by adding a larger variable volume collector to each CC in the refrigerated enclosure. Recycled IV bags from Colorado State University's Veterinary Teaching Hospital were used for this purpose, and the results will be analyzed in a future study.

4.1.2 Solids Destruction

The reduction of total and volatile solids mass within the reactor is an important performance measure for anaerobic digestors. Typical expected VS destruction in a complete mix mesophilic digester digesting manure ranges anywhere from 45% - 65% depending on detention time (Metcalf & Eddy, 2013). The VS destruction observed in the column studies was much lower than typically reported values (Figure 38). The TS destruction is shown in Figure 39.

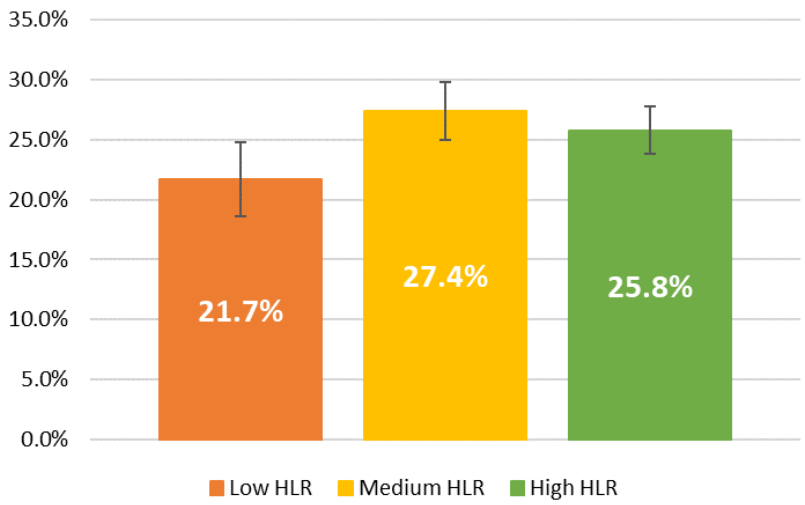

Figure 38 – Volatile solids destruction by flow rate. Error bars represent one standard deviation.

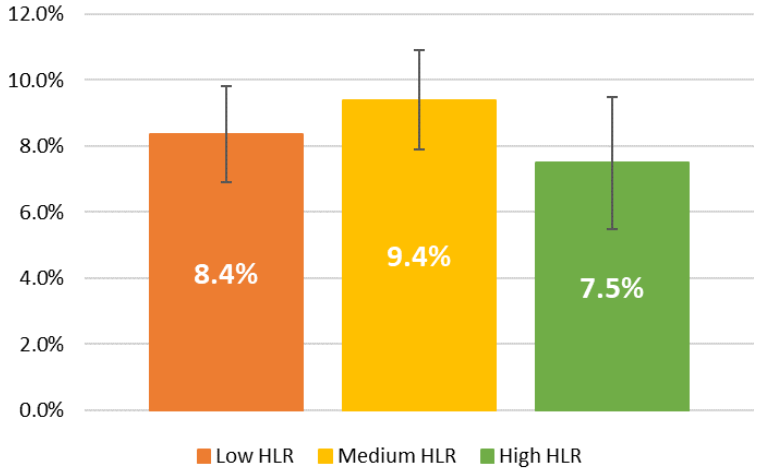

Figure 39 – Total solids destruction by flow rate. Error bars represent one standard deviation.

It can be seen that the lowest VS destruction was realized in the columns fed with the Low HLR. There does not appear to be a significant difference between the Medium HLR and High HLR columns, however. This is somewhat surprising given the much higher COD leaching rates seen at the High HLR over the Medium HLR, but all columns regardless of hydraulic loading measured very low VS destruction compared to typically expected rates. It is likely the high final VS content is in part due to the mesh bags used to maintain hydraulic flow in the columns. While serving the intended purpose of holding solids in place to avoid clogging, the mesh also likely created significant preferential pathways which would limit the amount of contact flowing leachate has with all portions of the substrate bed. The innermost portions of each layer may not have received flowing leachate throughout the operational period. If this phenomenon had not taken place and VS destruction was more typical of expected, it would likely be seen that the High HLR columns would experience more VS destruction than at the Medium and Low HLRs, leading to the increased organic leaching rate. The larger scale prototype USSHR had much more typical VS destruction results, as described in Section 4.2.3 below. Particulate solids in leachate were not accounted for, however, meaning some of the solids destruction may have come from being removed physically rather than being “destroyed”.

4.2 Prototype Results

4.2.1 Overview

The Prototype USSHR experiment was successful in demonstrating larger-scale operation of the MSAD system. Additionally, though there are still opportunities for improvement, hydraulic distribution was significantly improved over the observed deficiencies encountered in the previous experiment using this reactor. The previous experiment lacked comparative data, but significant

improvement was visually observed (Figure 40). The previous experiment post-digestion solids did not appear homogenous and undisturbed chunks were visible after dumping out the substrate.

Figure 40 – Consistency of post-digested manure from the P-USSHR

An additional issue encountered in previous experiments is post-digestion dewatering. The resultant material consisted of a thick slurry that was very difficult to manage. This experiment aimed to improve dewatering capability by attaching an inorganic mesh fabric to the interior walls of the reactor space to add lateral drainage pathways down the sidewalls (Figure 41). This, coupled with a larger drain filter put in place of the original bottom drain, produced a much more manageable substance with solids content up to 66%. This is a significant advancement, as the resultant material previously would have been unable to be handled with conventional solids handling equipment and would have posed a significant additional operational cost for full scale development.

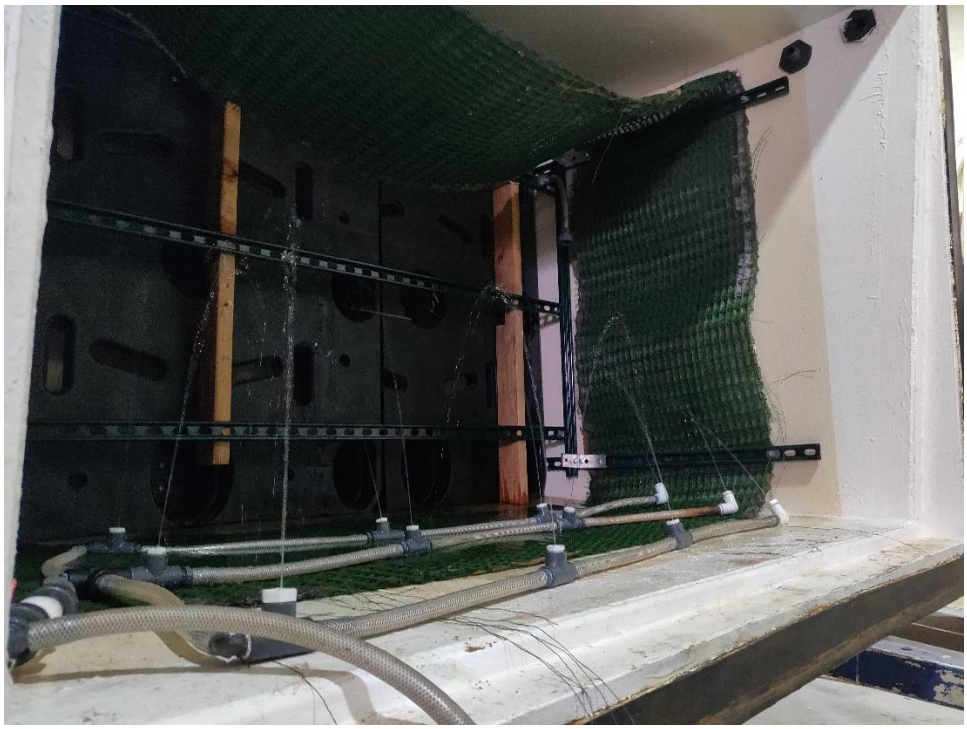

Figure 41 – Inorganic mesh fabric installed to assist with post-digestion dewatering

4.2.2 Percent Volatile Solids

Total Solids and Volatile Solids were analyzed in each of the six sections to determine remaining percent volatile solids after digestion (Figure 42).

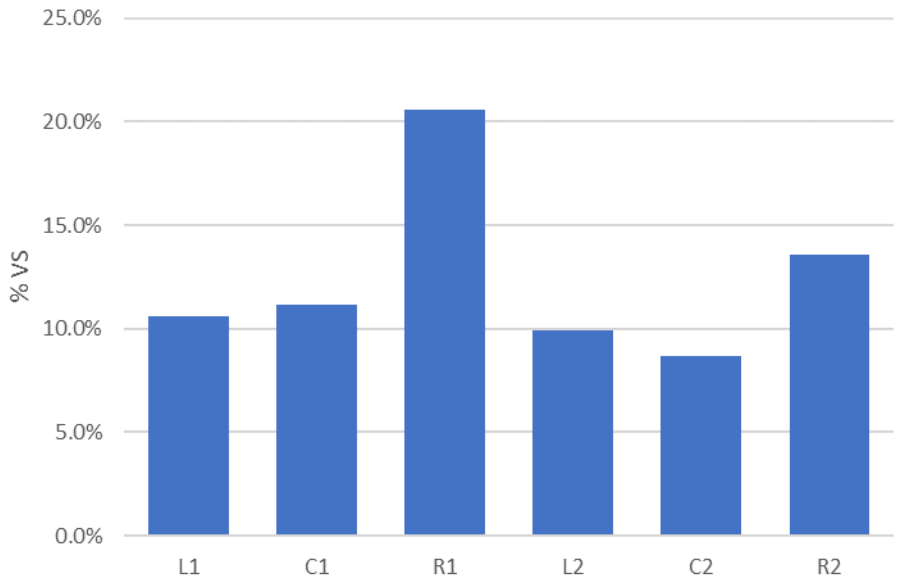

Figure 42 – Post-digested substrate percent volatile solids by section in Prototype USSHR

It can be seen that the right side of the USSHR did not experience as much volatile solid degradation, indicating unequal flow patterns favoring the rest of the reactor. A heat map image of each side of the USSHR was taken during operation which confirmed this observation (Figure 43). As influent leachate is pumped into the reactor, it passes through a heat exchanger to bring the temperature up to >35 C. The cooler portions of the reactor do not receive as much of the heated influent.

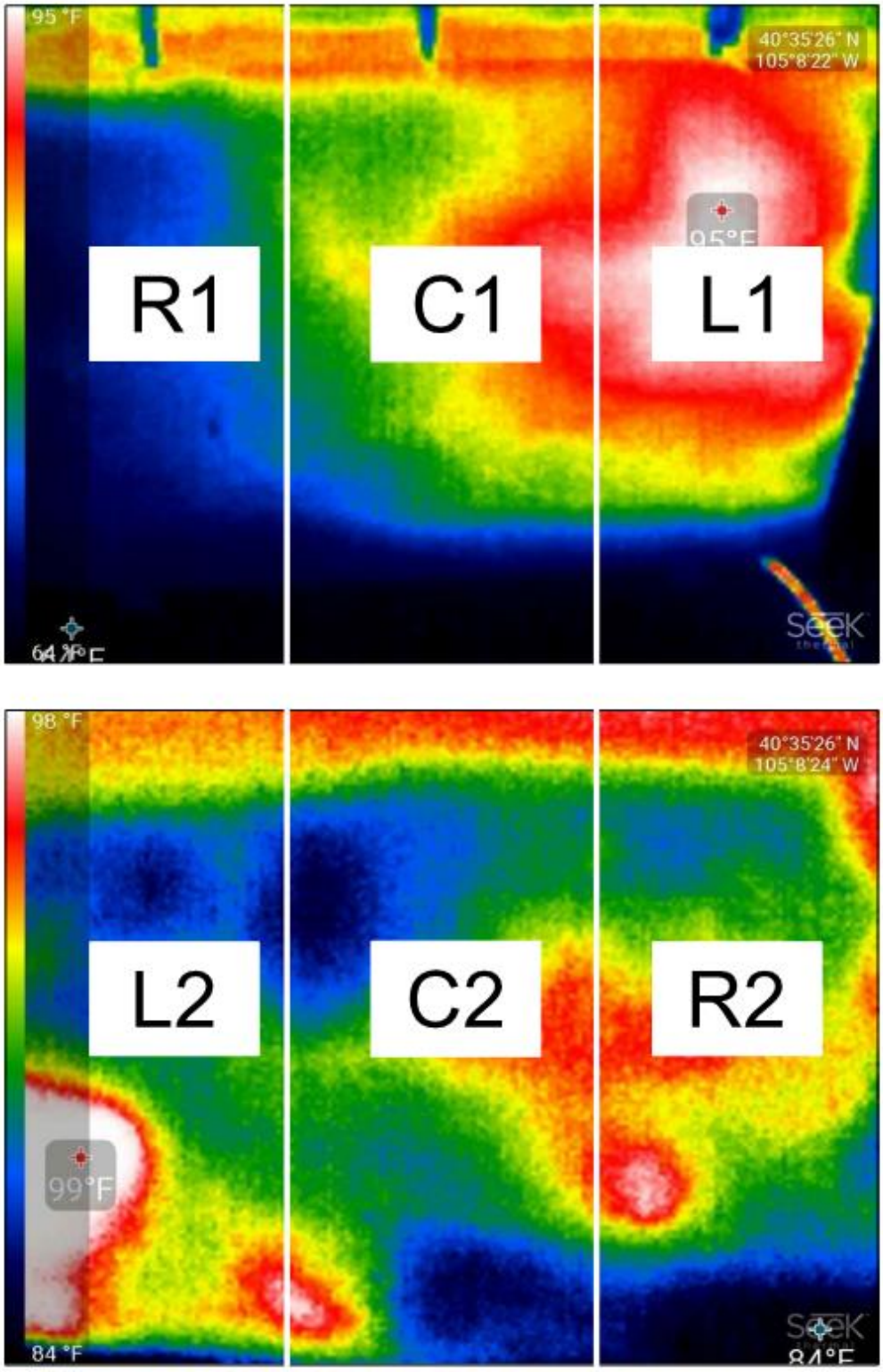

Figure 43 - Heat map images of the front and back of the USSHR. Labelled divisions of the reactor are shown

It can be seen that cooler portions of the reactor correspond well with the sections that achieved less volatile solids degradation. Better liquid flow through these regions would have improved degradation as well as temperature distribution. The highest remaining volatile solids content section (R1) was the coldest section within the reactor, as clearly seen.

4.2.3 Volatile Solids Destruction

The TS and VS destruction (Figure 44) were estimated as described in Section 3.14.2.

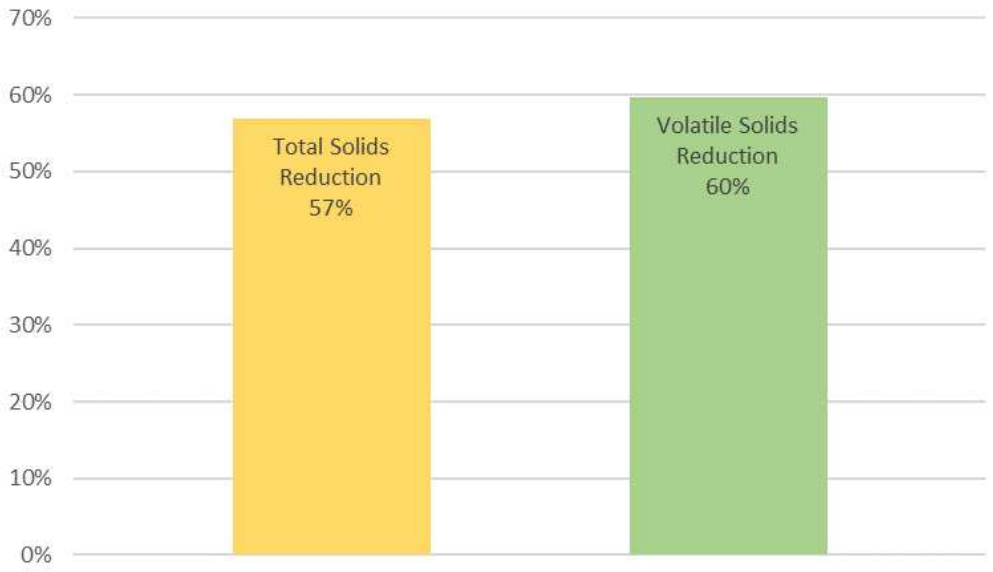

Figure 44 – TS reduction and VS reduction seen in the P-USSHR operation

The VS destruction seen in the prototype system is much more typical of what would be expected from a properly working anaerobic digester than what was seen at the column scale, indicating better hydraulic flow without the preferential pathways brought about by the mesh bags in the column experiments.

CHAPTER 5. Summary and Recommendations

The successful operation of each of the column Experiments 1-5 and the P-USSHR with the CLPS offers tremendous insight to the future development of the MSAD technology. The data gathered from this experiment set, including the COD leaching rates and TS/VS analyzed as part of this study, provide important information about the performance of the reactors. Of note is that samples collected from this experiment are currently undergoing analysis for biogas production by volume, biochemical methane potential, ammonia content and leaching rates, phosphorus content and leaching rates, sodium, potassium, and column experiment TS/VS. These data will be reported by Lucas Loetscher in his PhD dissertation. It was clear from CEU experiments that a higher leaching rate and therefore better performance was realized from the higher hydraulic loading rates into the E-USSHRs

Future column experiments utilizing the CLPS and CEU have already been underway, focusing on improvement of the process given data from this experiment set. The highest leaching rates occurred at the highest flow rates, which begs the question if that trend would continue at even higher flow rates. Future experiments that investigate higher HLRs than those investigated here may be of value to identify tradeoffs between increased HLR energy requirements and MSAD overall performance. Modifications to the CEU will allow for flow rates as high as 200 cm/day to be tested and results compared with the data from these experiments.

The P-USSHR was improved, but further adjustments may have an even greater impact on increasing performance of the reactor. The leachate injection platform worked well to spread out flow, but limited resources led to crude flow regulation which could be improved upon. Higher flow rates into the existing injectors will likely help with this immensely, which will correspond

well with the increased HLR experiments at column scale. The flow was throttled down substantially from the influent pump using a hand valve to match the 76 cm/day target high HLR. A simple and highly valuable experiment would be to increase flow by double, quadruple, and/or even higher and compare percent VS content and heat map images to the data from this study.

Further research should also focus on a peculiar observation noted from this experiment set. Supplementary analysis performed included biochemical methane potential (BMP), and it was noticed there was a decreasing ratio of BMP to COD leaching from the columns as the experiment went on over time. One possible cause for this is methanogenic colonization of the E-USSHR columns. This hypothesis is further supported by observations from the P-USSHR experiment. The demonstration system design and layout led to biogas being collected and measured separately between the hydrolysis and methanogenesis portions of the system. Significant biogas volume was produced directly from the P-USSHR – roughly at a 1:1 ratio to biogas from the C-FFR. Some gas is expected from the hydrolysis step, but it should be a small portion of the gas produced overall. Additionally, samples of the gas collected from the P-USSHR proved to be extremely flammable, indicating a likely high methane percentage. Difficulties were encountered with the storage method for preserving biogas samples for concentration analysis. These issues will be resolved for future experiments allowing data collection for a clearer picture of the observed phenomenon. A focus of future experiments should be to measure biogas volume and concentration accurately from each reactor component of the MSAD to quantify methane production from each component. It is theorized that hydrolysis should be taking place at a rate rapid enough that pH would stay acidic, inhibiting methanogens from effectively producing within the USSHR. Monitoring of this possible phenomenon should take place by measuring pH throughout the reactor over the duration of the experiment. Sample ports should be introduced in various locations to draw during experiment

runs or inline probes should be installed to understand what is taking place within the reactor. If pH begins to rise in an area, methanogenesis is likely taking place there. It could be expected that pH will rise as the liquid passes up through the USSHR as opposed to falling.

Exploration into the possibility of methanogenic colonization of the hydrolysis reactor is important for several reasons. It is unknown how this may impact overall performance of the entire system in terms of efficient leaching and degradation of initial substrate as well as on resource recovery by methane production. If it is determined that this is an overall detractor, which is possible since the MSAD was designed to separate the steps and separately optimize each, increasing flow rates may help alleviate the issue. If hydraulic residence time within the USSHR is too long, leached organics may take too long to leave the reactor and enter the FFR. It is possible this leads to inhibition of further hydrolysis, and the leached organics would need to be fully degraded within the hydrolysis reactor before further hydrolysis could effectively take place.

Another interesting nuance that is valuable to explore is the impact of separating hydrolysis feed from methanogenesis leachate feed tanks. The original concept did not consider this, feeding both processes out of one LFT. The P-USSHR was set up with the CLPS in this way, but the CEU was designed with the separate HFT to ensure a consistent feed was introduced to the E-USSHR columns throughout the day. This was done strictly for analytical purposes, as the effluent was collected and stored each day and therefore the feed leachate would degrade and become more dilute throughout the day before the new day's leachate was pumped back to the feed tank again. Maintaining a consistent feed allowed the COD of influent leachate to be subtracted (by mass) from effluent COD measured, thereby determining "new" COD leached from each reactor each day. Observing this process led to the question if this process separation is actually ideal for optimal performance. If the same leachate line is used to pump into the FFR for degradation and

the USSHR for replenishment of organics, some of the leachate being pumped into the hydrolysis reactor already contains organics and may be saturated or close to it. If separated, but hydraulically linked tanks (linked to address possible varying flow rates between the FFR and one or more USSHRs) are used, it could be controlled so that the FFR is only fed with leachate from the USSHRs and the USSHRs are fed with dilute leachate from the FFR effluent. A small diameter connection pipe could be used as a sort of a two-way pressure reducing check valve. The connection could be designed to provide enough head loss so that flow only crosses if one tank is a certain level above the other. If one tank was being drawn from faster than the other, this connection would maintain hydraulic connectivity and prevent process failure.

These observations represent opportunities for further improvement of the MSAD technology. The research goals of understanding the MSAD process and determining a detailed direction for future research to optimize the system were met by constructing a working system, the successful operation of which was an important accomplishment, and by the impactful findings related to flow rate and process performance. The findings resulting from this study provide a clear set of research questions to answer with future research efforts. The highly flexible and adaptable CLPS and CEU designed and constructed for this experiment will enable these future research tasks to be carried out efficiently with limited modifications. The MSAD technology has taken a notable step forward with this research effort and is poised to be optimized for larger and even commercial scale applications once these detailed process questions are answered.

REFERENCES

Arita, C. E. (2013). *Anaerobic Digestion Comparison of Manure Leachate by High-Rate Anaerobic Reactors*. Fort Collins: Colorado State University.

Chaudhary, B. (2008). Dry Continuous Anaerobic Digestion of Municipal Solid Waste in Thermophilic Conditions. *MS Thesis*. Asian Institute of Technology.

Chen, G. N. (2005, November). Two-phase anaerobic digestion of unscreened dairy manure. *Process Biochemistry, 40*(11), 3542-3549.

Dinh Pham Van, T. F. (2020). A review of anaerobic digestion systems for biodegradable waste: Configurations, operating parameters, and current trends. *Environmental Engineering Research*.

Griffin, L. P. (2012). *Anaerobic Digestion of Organic Wastes: The Impact of Operating Conditions on Hydrolysis Efficiency and Microbial Community Composition*. Fort Collins: Colorado State University.

Hanif, A. (2013). *Evaluation of a trickle flow leach bed reactor for anaerobic digestion of high solids cattle manure*. Fort Collins: Colorado State University.

Keske, C. (2009). *Economic Feasibility Study of Colorado Anaerobic Digester Projects: Colorado Governor's Energy Office.* Fort Collins, CO: Colorado State University, Department of Soil and Crop Sciences.

Larson, V. (2019). *Impacts of Operating Conditions on Aerobic Removal of Emerging Contaminants from Animal Waste: Composting Post Anaerobic Digestion*. Colorado State University.

Lewis, M. (2018). *Maintaining Leachate Flow Through a Leach Bed Reactor During Anaerobic Digestion of High-Solids Cattle Manure*. Fort Collins: Colorado State University.

Loetscher, L. (2018). *The Development and demonstration of a multiple stage anaerobic digester for the treatment of high solids wastes*. Fort Collins: Colorado State University.

McCarty, B. E. (2001). *Environmental Biotechnology: Principles and Applications.* McGraw Hill.

Metcalf & Eddy. (2013). *Wastewater Engineering: Treatment and Resource Recovery.* Boston: McGraw-Hill.

Neibling, L. C. (2014, June). *Anaerobic Digestion Basics.* Retrieved from University of Idaho Extension: https://www.extension.uidaho.edu/publishing/pdf/CIS/CIS1215.pdf

Nelson, C. (2002). *Haubenschild Farms Anaerobic Digester Updated.* St. Paul: The Minnesota Project.

Nizami, A. a. (2010). What type of digester configurations should be employed to produce biomethane from grass silage? *Renewable and Sustainable Energy Reviews*.

Oh, D.-H. K.-E. (2011). Continuous high-solids anaerobic co-digestion of organic solid wastes under mesophilic conditions. *Waste Management, 31*(9-10).

P. E. Poh, D. G. (2016, June). Optimization of Wastewater Anaerobic Digestion Using Mechanistic and Meta-heuristic Methods: Current Limitations and Future Opportunities. *Water Conservation Science and Engineering*, 1-20.

S.D. Kalamaras, T. K. (2014). Anaerobic co-digestion of cattle manure and alternative crops for the substitution of maize in South Europe. *Bioresource Technology*(172), 68-75.

Sandefur, J. (2016). *Aerobic Post-Processing of Digestate From a Multi-Stage Anaerobic Digester*. Fort Collins: Colorado State University.

Schnürer, M. W. (2019, February). *Microbial Responses to Different Operating Practices for Biogas Production Systems.* Retrieved from IntechOpen.com: https://www.intechopen.com/books/anaerobic-digestion/microbial-responses-to-different-operating-practices-for-biogas-production-systems

Surendran, A. N. (2018). *Evaluation of Cow Peat as a Plant Growth Media*. Fort Collins: Colorado State University.

United States EPA. (2000, September). *Biosolids Technology Fact Sheet - Land Application of Biosolids.* Retrieved from epa.gov: https://www3.epa.gov/npdes/pubs/land_application.pdf

United States EPA. (2015, October). *EPA/600/R-15/304 Anaerobic Digestion and its Applications.* Retrieved from epa.gov: https://www.epa.gov/sites/production/files/2016-07/documents/ad_and_applications-final_0.pdf

United States EPA. (n.d.). *AgSTAR: Biogas Recovery in the Agriculture Sector.* Retrieved from EPA AgSTAR: https://www.epa.gov/agstar

United States EPA. (n.d.). *Basic Information about Anaerobic Digestion*. Retrieved from epa.gov: https://www.epa.gov/anaerobic-digestion/basic-information-about-anaerobic-digestion-ad

Ward, A. J. (2008). Optimisation of the anaerobic digestion of agricultural resources. *Bioresource Technology*, 7928–7940.

Wu, R. (2017). *Enhancement of liquid flow through a leach bed reactor for anaerobic digestion of high solids cattle manure*. Fort Collins: Colorado State University.

Zoetemeyer, R. V. (1982). pH influence on acidogenic dissimilation of glucose in an anaerobic digester. *Water Research* , 303-311.