THESIS

ENHANCEMENT OF LIQUID FLOW THROUGH A LEACH BED REACTOR FOR ANAEROBIC DIGESTION OF HIGH SOLIDS CATTLE MANURE

Submitted by

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ABSTRACT

ENHANCEMENT OF LIQUID FLOW THROUGH A LEACH BED REACTOR FOR ANAEROBICDIGESTION OF HIGH SOLIDS CATTLE MANURE

Due to animal production waste increases in Colorado, anaerobic digestion (AD) has become increasingly considered as a technology to convert organic solid waste (OSW) into renewable energy. The arid climate with water resource limitation in Colorado results in high solids cattle manure (HSCM) production, containing between 50% and 90% total solids (TS). Conventional AD for animal manure is best option to treat manure with less than 20% TS, but limited feasibility for conventional anaerobic digesters treats manure in Colorado. The multi-stage anaerobic digester (MSAD) investigated in this study can digest HSCM. An integral part of the MSAD is the Leach Bed Reactor (LBR), which is loaded with HSCM (up to 90% TS). A small quantity of water percolates into the LBR and is recirculated through the LBR where hydrolysis occurs until a large amount of organic material is solubilized into the leachate. A review of the literature has indicated that clogging can be an issue in operation of manure LBRs. Since sustaining liquid flow through LBRs can be a challenge, research was conducted to better understand how to use this technology to treat HSCM. The objectives of this research were to 1) assess the performance of the LBR component of the MSAD technology with different top layer materials and flow regimes to enhance duration of sustained flow, 2) assess the ability of varying top layer materials and flow regimes to enhance hydraulic conductivity of the manure bed in the LBR to maximize hydrolysis in the LBR.

For this study, downward flow and upward flow LBR configuration experiments were conducted. The combination of a sand layer on top of the manure beds and an improved top filter for the LBR was added in the upward flow LBR configuration. HSCM samples from each stage of the experiment were analyzed for TS, fixed solids (FS), and volatile solids (VS), and the leachate samples were analyzed for chemical oxygen demand (COD). The leachate outflow rate and column pressure head were also measured daily.

Due to failure of all downward flow experiments, the upward flow LBR configuration was evaluated. The clogging issues and leachate flow through the LBR improved by changing to the upward flow LBR configuration. The average operation time of the upward flow experiment was prolonged to 21 days comparing with downward flow experiment, which operated for an average of only 7 days. The percentage reduction of VS in upward flow experiments was on average above 40% indicating successful hydrolysis in the LBRs, comparable to VS reduction observed by other researchers (Uke and Stentiford, 2012). The COD concentration of the upward flow experiments started at an average of 45 g COD/ L and approached the LSTs COD concentration of 10 g COD/L at day 10. This indicates that the MSAD was effectively degrading the HSCM throughout the batch digestion period. The constant pressure head of upward flow experiments indicated that no pressure built up inside the LBRs resulting in improved flow through the manure in these systems. In summary, this research showed that the upward flow LBR configuration with the combination of a sand layer on top of the manure bed and improved top cap filter can sustain leachate flow through the LBR for 21 days of operation.

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TABLE OF CONTENTS

CHAPTER 1: INTRODUCTION

1.1 Research Motivation

Population growth along with rising standards of living has resulted in a global increase in energy demand. The United States is one of the biggest energy consumers in the world. The major energy resources used in the United States are derived from fossil fuels. Currently, AD has become a potential renewable energy technology, and interest in biomass-to-energy technology is increasing worldwide. AD technology can directly convert the OSW into biogas, which typically contains methane (near 60%) and carbon dioxide (near 40%).

Fossil fuels are non-renewable sources of energy, and because of high demands on these resources that are continually increasing, fossil fuel availability is becoming limited. In the future, fossil fuel scarcity may lead to a considerable increase in energy prices (Rubin, Chen, and Rao, 2007). Additionally, burning excessive fossil fuel for energy use results in greenhouse gases (GHG) accumulation in the atmosphere. This phenomenon will be main driver to the severity of climate change effects (Wuebbles and Jain, 2001). The negative consequences associated with fossil fuel utilization are driving the need to develop clean, cost-effective renewable energy.
In the United States, approximately 10% of total energy consumption in 2015 was from renewable energy, which includes 49% biomass, 25% hydroelectric, 19% wind, and 6% solar (Fig.1). This proportion has been growing since the 1950s.

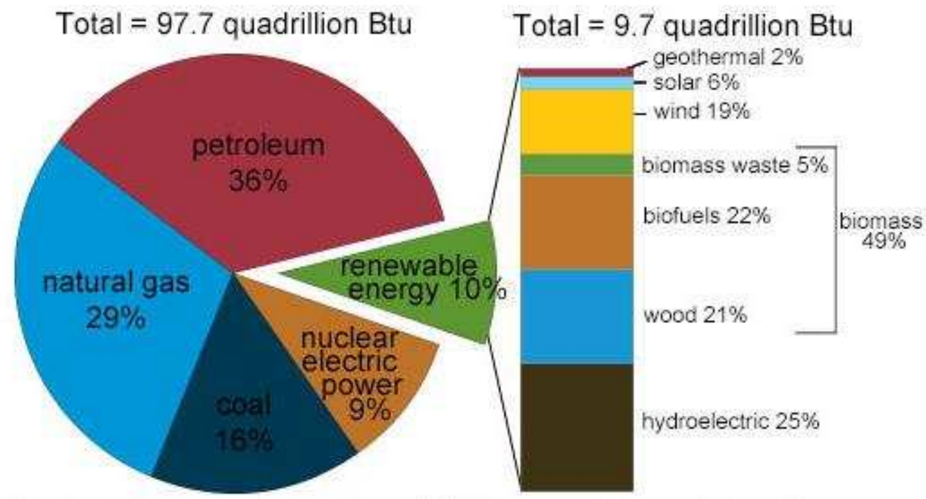

Fig. 1 U.S. energy consumption by energy source, 2015Source: U.S. Energy Information Administration

Biomass fuels have been a potential renewable source of energy in the last few decades due to a large amount of OSW generation. U.S. Energy Information Administration (EIA) estimates "biomass energy will increase by 4.4% per year, the third largest increase in renewables behind solar (7.5%) and geothermal (5.4%) annual growth rates" (EIA, 2014). Biomass technology uses organic matter including scrap lumber, forest debris, certain crops, manure, food waste, landscaping (green) waste, and some types of waste residues to generate heat or power. Previous studies indicated that Colorado has a fair biomass potential at 5.2 billion kilowatt hours (kWh) of electricity per year (Burnell et al., 2007)

An EPA study (2011) mentioned that Colorado ranks the top ten states in nation for potential electricity production on dairy operations. The expanding of dairy sector already happened in Rocky Mountain Region. Currently, the total population approximately 143,000 of milk and heifer cattle in the state Colorado represents 27% increasing from the last four years when there were 116,000 (Colorado Energy Office). This has led to an increase in the production animal waste. Accumulation of large amounts of animal waste can contribute to air quality problems, additional greenhouse gas emissions, and pollution of groundwater. Managing this manure can be challenging for farms; however, the waste can potentially provide a valuable source of biomass.

AD has become a renewable energy option for the agricultural sector to reduce greenhouse gas emissions and generate renewable energy. AD systems break down the organic materials to generate biogas. The biogas could use as energy resources which include electricity generation, heating, transportation energy or upgradating to inject into natural gas piplines. Agricultural operations can potentially benefit from using biogas for energy production, which can generate an additional revenue source for farms (Colorado Energy Office). In 2000, approximately 10 million KWh/yr equivalent energy was generated by the livestock- related AD technology in the US, and by 2013, 700 million KWh/yr were produced in the US (fig.2) (Colorado Energy Office).

However, due to the arid climate and limited water resources in Colorado, the manure collection process used is generally scraping instead of flushing (Sharvelle et al., 2011). This results in the solids content of manure being from 50% to 90% (Sharvelle et al., 2011). This kind of manure is referred to as HSCM, defined here as manure with greater than 30% TS. Conventional AD for animal manure is best suited to treat manure with less than 20% solids content rendering limited feasibility for conventional anaerobic digesters to treat manure in Colorado (Sharvelle et al., 2011).

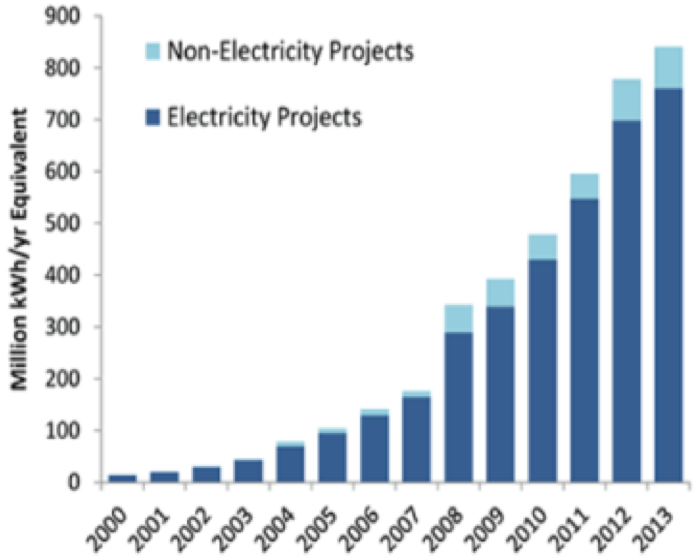

Fig. 2 U.S. Equivalent energy generation in kWh generated by livestock-related AD systems from 2000-2014Source: Colorado Energy Office

The MSAD was developed by Dr. Sharvelle's group at Colorado State University (CSU). The advantage of this technology is its ability to digest HSCM waste from dry lot animal feeding operations. An integral part of the MSAD is the LBR (LBR; Fig. 3), which percolates a small quantity of water through the high solids waste. Water is recirculated through the LBR where hydrolysis took place in the first place to degradate the organic waste until organic waste content is dissolved into the liquid. The high organic content liquid leachate is stored in a leachate storage tank (LST) and fed at a constant rate to a high rate anaerobic digester (HRAD) to produce biogas. The process of recirculating the leachate (water containing an amount of dissolved organic materials) helps to minimize water use for digestion. The MSAD technology flow diagram is shown below (fig. 3).

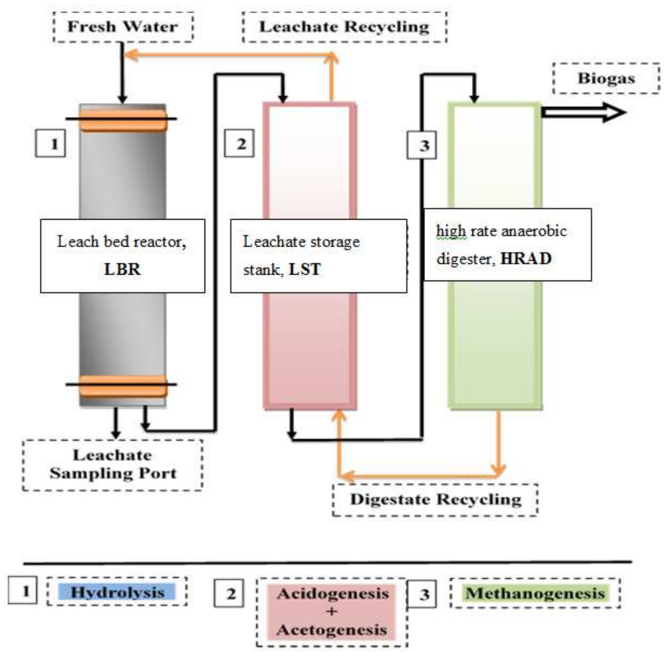

Fig. 3 General MSAD process flow diagram(Karim, 2013)

A major challenge to successful operation of the MSAD technology is sustaining liquid flow through the LBR for periods long enough to maximize capture of organic material into the liquid leachate. The high density of HSCM can cause clogging of the LBR resulting in failure to maintain leachate flow through the HSCM bed over the needed time period, 2-4 weeks. In previous research, sand was added on top of the HSCM bed as dispersion media to help sustain leachate flow through the reactor (Karim, 2013). However, the additional step of adding sand could increase operation and maintenance costs. Therefore, in this research, adding geo-synthetic materials as dispersion media on top of the HSCM bed was evaluated as an alternative to sand.

1.2 Objective Research

The objectives of this research were to1) Assess the performance of the LBR component of the MSAD technology with different top layer materials (geosynthetic materials, wood chips, gravels, sands, and manure with particle sizes) and flow regimes to increase duration of sustained flow, 2) Assess the ability of varying top layer materials (geosynthetic materials, wood chips, gravels, sands, and manure with particle sizes) and flow regimes to enhance liquid flow through the LBR to maximize hydrolysis in LBR.

CHAPTER 2: BACKGROUND AND LITERATURE REVIEW

2.1 Anaerobic Digestion as a Waste Management Tool

OSW management is intended to reduce environmental pollution and GHG emissions. Major technologies for OSW management include landfilling, thermal treatment, aerobic composting, and AD. AD has advantages because active anaerobes can break down OSW to produce biogas and other by-products. The biogas produced from AD systems generally has a composition of near 60% methane and 40% carbon dioxide (Chynoweth et al., 2001).

Biogas can be used to generate energy in the form of heat and electricity. The generated electricity can potentially help recover the AD system energy input and reduce energy expenditures. The valuable by-products of AD systems include high quality stabilized compost and nutrient rich liquid fertilizers that can be used in agricultural applications. “Additional intermediary by-products include solvents and volatile fatty acids (VFAs), which can be extracted from the system and converted to products such as methyl or ethyl esters” (Brummeler, Horbach, and Koster 1991). These can be made into commercial products. In addition, the benefits of AD processes include waste stabilization, odor control, and pathogen reduction. During the AD process, the mass of OSW is reduced, which cuts down the cost of waste transportation. Since methane is harmful to the environment and can be used as a valuable source of energy, biogas is collected during the AD process to avoid gas emissions into the atmosphere. Methane in the biogas can then be used as an energy source. As a result, the two major benefits of AD systems, environmental pollution control and energy generation has resulted in increased implementation in some parts of the world.

2.2 AD Process

The AD process has four major steps (fig 4). The steps include hydrolysis, acidogenesis, acetogenesis, and methanogenesis. Each step has its own microbial community, but each community also is dependent on the others.

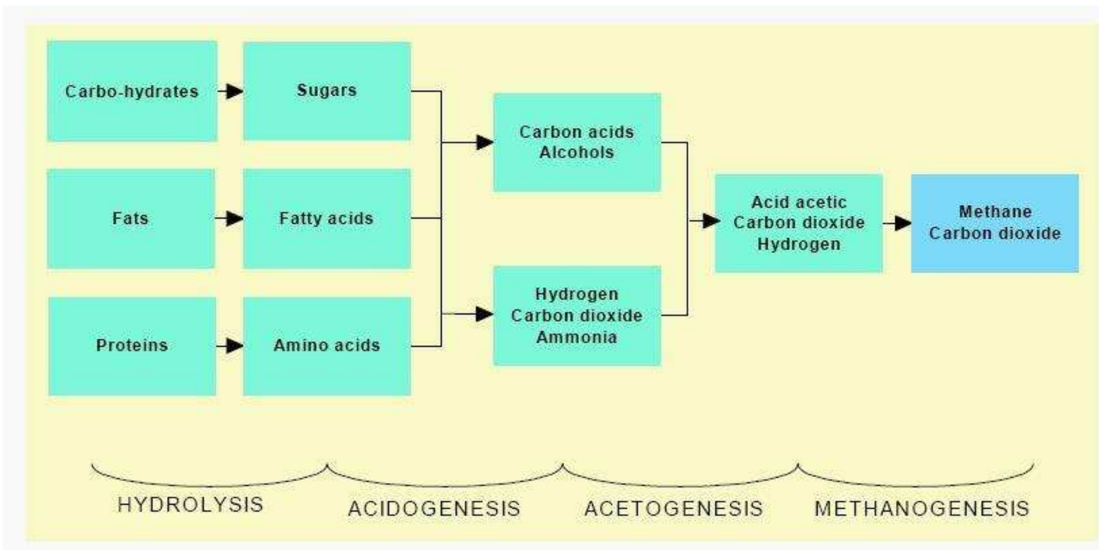

Fig. 4 Biochemical process of AD Source: Biogas Energy Overview

The hydrolysis process breaks down complex organic material such as carbohydrates, proteins, lipids, and fat into simple soluble organic compounds like sugars, amino acids, and fatty acids by hydrolytic bacteria (Chaudhary 2008). The process of hydrolysis depends on reaction conditions such as pH, temperature, the concentration of hydrolytic microorganisms, as well as the properties of the OSW feedstocks.

"The generalized molecular formula for organic wastes is approximated to be $C_6H_{10}O_4$. Equation (1.1) represents a hydrolysis reaction where complex organic compounds are broken down to simple sugars" (Chaudhary 2008).

$$C_6H_{10}O_4 + 2H_2O \rightarrow C_6H_{12}O_6 + 2H_2 \qquad (1.1)$$

The acidogenesis process continually utilizes simple soluble organic compounds, which are fermented to produce volatile fatty acids (VFAs) such as acetic, propionic, butyric, valeric acids etc., and neutral compounds such as ethanol, methanol, and ammonia by acidogenic bacteria (Chaudhary 2008). The production of those simple molecules generated in this stage depends on the classes of acidogenic bacteria and reactor conditions such as pH and temperature. As the simple molecules accumulate in this stage, the pH level decreases. "Equations (1.2) and (1.3) represent the reactions that take place in the acidogenic stage" (Chaudhary 2008).

$C_6H_{12}O_6$ (glucose) $\leftarrow \rightarrow 2CH_3CH_2OH$ (ethanol) + $2CO_2$ (1.2)

$C_6H_{12}O_6$ (glucose) + $2H_2 \leftarrow\rightarrow 2CH_3CH_2COOH$ (propionate) + $2H_2O$ (1.3)

In the acetogenesis process, acetogenic bacteria further digest the simple molecules from the acidogenesis step into acetic acid, carbon dioxide, and hydrogen. "The reaction only proceeds if the hydrogen partial pressure is low enough to thermodynamically allow the conversion. Equation (1.4) represents the reaction that takes place in the acetogenic stage" (Chaudhary 2008).

$CH_3CH_2COO^- + 3H_2O \leftarrow\rightarrow CH_3COO^- + H^+ + HCO_3^- + 3\ H_2$ (1.4)

(Propionate) (Acetate)

The last step is the methanogenesis process, which uses methanogenic bacteria to generate insoluble CH_4 as the final product, and other by-products such as carbon dioxide and hydrogen from previous intermediate products. In the process, two major types of microorganisms participate in methane generation: acetoclastic methanogens and hydrogenotrophic methanogens. The acetoclastic methanogens are defined as microorganisms that utilize acetic acid to produce methane. The concentration of methane from acetoclastic methanogens generated is around 75% (Chaudhary 2008). The hydrogenotrophic methanogens are defined as microorganisms that consume carbon dioxide and hydrogen to generate methane. Two thirds of methane production are derived from acetate conversion (Eq. 1.5 and 1.60) or the fermentation of an alcohol (Eq. 1.7). One third of methane production is from carbon dioxide reduction by hydrogen (Eq. 1.8). “Equations (1.5 - 1.8) represent the reactions that take place in the methanogenic stage” (Chaudhary 2008)

$$2\ CH_3CH_3OH + CO_2 \leftrightarrow 2\ CH_3\ COOH + CH_4 \quad (1.5)$$

$$CH_3COOH \leftrightarrow CH_4 + CO_2 \quad (1.6)$$

$$CH_3OH + H_2 \leftrightarrow CH4 + H_2O \quad (1.7)$$

$$CO_2 + 4H \leftrightarrow 2\ CH_4 + 2H_2O \quad (1.8)$$

2.3 AD Technology

In the last few decades, AD technology has been deployed around the world. Major types of AD technologies are fixed film reactors, covered lagoons, upward flow sludge blanket reactors, plug flow reactors, and continuously stirred tank reactors (Sharvelle et al., 2011). Those major types of AD technologies are shown in the fig.5. Selection of AD systems depends on several elements including “the type of OSW to be treated, the solids content of the waste, the size of the facility, economic feasibility, the location of implementation, and water availability in the area” (Sharvelle et al., 2011). Covered lagoon systems have lower capital cost for initial construction, and this system can effectively limit odors spreading into the atmosphere. The advantage of this system includes longer retention time for the reactor. The plug flow system also has lower capital cost for construction and can handle the higher solid content of OSW. However, due to the sensitivity of the system to heterogeneous OSW, it has variable gas production. In addition, the system needs a larger footprint. Upward flow sludge blanket reactors have a higher VS reduction in order to generate higher methane production. The disadvantages of this system include longer reactor start up time and the limitation of only being able to process lower solid content of OSW. The fixed film only needs a small footprint and can generate higher methane production due to high microbial community concentration. However, this type of reactor can easily clog when digesting certain feedstocks. The CSTR can easily mix heterogeneous OSW with existing nutrients and microbes within the reactor volume; this gives the CSTR the ability to handle variable solid content OSW. However, the system requires a higher capital investment and needs more energy input to maintain the reactor's daily operation compared to other reactors. In conclusion, each AD technology has its advantages and disadvantages. However, none of those systems can handle high solid content OSW which is collected from the dry cattle lots without adding a huge amount of water.

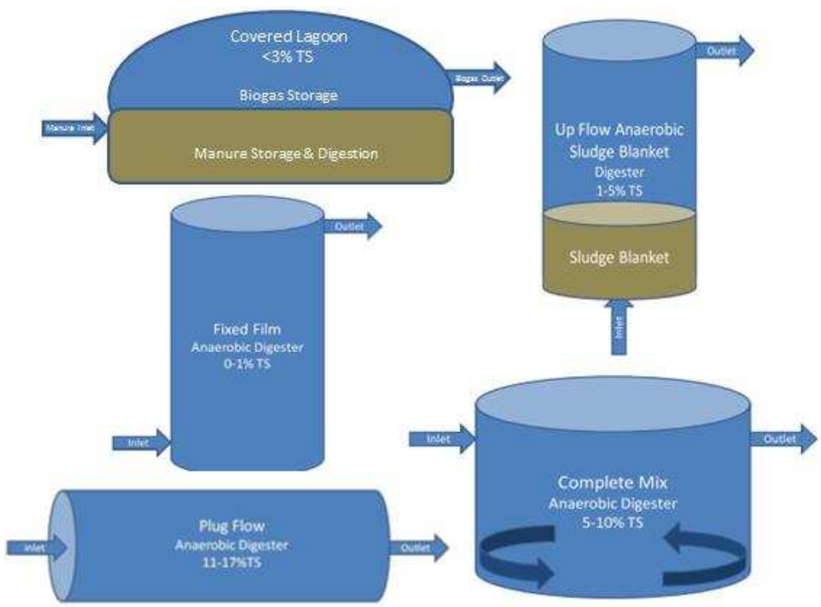

Fig. 5 Current Low Solids AD Technology Diagrams and Recommended Waste Solids Content(Sharvelle et al., 2011)

2.4. Current Agricultural Solid Waste Management in Colorado

Due to the arid climate and water resource limitations, the agricultural solid waste management practices in Colorado differ from other humid areas in the U.S. The manure collection method in dry cattle lots is to scrape manure from the beds and load it into large piles onsite. Many facilities compost the collected manure (Sharvelle et al., 2011). This collection process combined with the arid climate in CO results in manure solids contents between 50% - 90% (Sharvelle et. al, 2011), referred to here as HCSM. In non-arid regions, manure collection included slurry manure (5 to 15% solids) or liquid manure (0 to 5% solids) collection and handling systems (Fulhage and Harner, 2012). The formation of liquid manure is due to adding wash water to manure. Flushing water to remove the manure from a dairy freestall barn is one of example of manure collection resulting in a slurried manure. “The slurry manure results from systems where little or no bedding is added to the excreted manure and urine” (Fulhage and Harner, 2012). Conventional AD technologies require water dilution until the manure is diluted to less than 15% TS (Vavilin, Vasily A., et al. 2003). Therefore, limited water resources and high TS content of HSCM render challenging conditions for implementation of conventional AD technologies in Colorado (Sharvelle et al., 2011). In addition, the scraping collection method of manure in Colorado can mix additional inorganic material into the manure such as gravel and sand. Additional sand and gravel takes up space in the digester, reducing the residence time of manure and can decrease biogas production rates or even damage the system and its pumps (Sharvelle et al., 2011). Additional infrastructure is needed to remove undesirable inorganic materials, which would increase reactor capital and operation cost. Therefore, conventional AD technologies are rarely implemented at feeding operations in areas with arid climates where manure is collected on dry lots.

2.5 MSAD Technology

Multi-stage anaerobic digestion (MSAD) is one promising option to process dry HSCM. The major feature of MSAD technology is to separate reactors into three stages instead of a single reactor. The three stages of reactors include LBR, leachate storage tank (LST), and high rate anaerobic digester (HRAD). Fig. 3 (Chapter 2) shows the general MSAD components and process, and liquid flow configuration. Dry HSCM is loaded into the LBR, and water trickles at a constant flow rate through the manure bed while hydrolysis occurs. The leachate discharges from the LBR and is stored in the LST. In this stage, acidogenic and acetogenic bacteria further break down the dissolved organic materials. Then, the high organic content leachate is further pumped into the HRAD where methanogens continually degrade organic materials to generate methane. The dissolved carbon in the leachate is pumped back to the LBR to serve as inoculum and hydraulic medium to maximum the contact time between the HSCM and the anaerobes microorganic. The MSAD becomes a recycling system, and fresh water is added into the system to dilute the leachate concentration in order to prevent salt or ammonia toxicity inside the TFLBR (Novella, Ekama, and Blight 1997). Compared with conventional AD technology, the method reduces the amount of water required for solid waste AD.

2.6 Advantage of a Multi-Stage

Due to acidogenic and methanogenic microbial communities having different physical and chemical properties including nutritional needs, growth kinetics, optimum pH-value and sensitivity to environmental conditions, the multi-stage reactors can provide more optimal environmental conditions for those two groups of microorganisms than a single reactor. The major advantage of the multi-stage is to improve the stability and control over the whole AD process (Demirel and Orhan 2002). In the LST reactor, volatile fatty acids (VFAs) are generated, while in the HRAD reactor, methane and carbon dioxide are converted from VFAs by methanogenic microorganisms. The reactor separation allows an improvement with regard to process stability by installing a ideal pH-values and temperatures for each reactor. According to the Lehtomäki and Björnsson (2006), a two-stage pilot-scale reactor has a 5% higher methane production than a laboratory batch system digesting grass (Lindner, Jonas, et al 2015). Another major advantage of the multi-stage reactor is the fractionation of the gases during AD. This allows the HRAD reactor to produce high concentration methane biogas around 85% (Lindner, Jonas et al., 2015).

2.7 Advantage of Leachate Recirculation

Because HSCM has especially high solids content, it can be difficult to handle and mix; recirculation of leachate becomes necessary to achieve maximum degradation and enhance biogas production during the AD process. According to previous research, leachate recirculation results in three advantages: “(i) additional methanogenic activity for manure, (ii) an ideal pH and a buffered environment for the biological reactions of AD and (iii) nutrients for efficient acetogenesis and methanogenesis” (Degueurce et al., 2006).

Hamed M.El-Mashad (2006) showed that degradation rate and biogas production can be effectively improved by having leachate recirculation in the AD process. Leachate contains abundant inoculum. When the leachate recirculates back to LBRs, the inoculum can mix well with freshly added manure to increase the rate of degradation. During the solid manure digestion, the reintroduction of leachate can affect the substrate degradation rate. Because leachate recirculation can enhance the contact between biomass and substrate, it has the potential to improve system performance. (El-Mashad, Hamed M., et al., 2006)

One of the most important parameters to promote AD efficiency is moisture content of substrates during anaerobic degradation. In landfills, using the process of leachate recirculation to control the moisture content of substrates has been used for more than 40 years (Degueurce et al., 2016). In recent years, leachate recirculation has been incorporated into the AD process, particularly to handle HSCM (Degueurce et al., 2016). In addition, leachate recirculation can decrease water demand for the AD process, thus reducing pressure on water resources in arid regions where water limitation can make implementation of AD more challenging.

2.8 MSAD Design Considerations

The technology of MSAD combines multiple stages and leachate recirculation, resulting in a reactor configuration that can more easily handle HSCM compared to conventional AD processes. Advantages of MSAD include less extensive mixing requirements, lower water consumption, smaller energy inputs to heat the AD system, and limited needs for effluent dewatering (El-Mashad, Hamed M., et al., 2006).

However, in certain situations, the multiple stages and leachate recirculation processes present operational challenges. Most problems can be overcome by the advantages of the combined system. For example, one problem relates to accumulated volatile fatty acids (VFA) due to leachate recirculation. The accelerated leachate recirculation can increase the process of acidogenesis and result into an accumulation of VFAs, which can toxic the methanogens bacterial (Degueurce et al., 2016). VFAs can inhibit methanogenesis during the AD process. However, in the MSAD technology, the acidogenesis and methanogenesis processes happen in separate reactors, which reduce VFAs toxicity for methanogens during the AD process.Another issue to be overcome in the MSAD technology is that the hydrolysis processes in the LBR is the rate-limiting step in the MSAD process (Veeken, Adrie, et al., 2000). One possible reasons are high biomass concentration. Due to the high biomass concentration in the LBR, the substrate surface to contact with anaerobes became limitation. In addition, the variables of manure particle sizes may possible cause the limitation of substrate surface. This substrate surface limitation lead to the mass-transfer limitation, which may impede the enzymes and leachates transportation within the LBR. The mass-transfer limitation resulted in reduction of the hydrolysis rate in the LBR (Myint and Nirmalakhandan, 2006). In addition, HSCM with the low wet shear strength property had high tendency to collapse under weight. This property of HSCM may cause "leachate channeling inside the LBR thus leading to preferential pathways" (Lissens et al., 2001). Accordingly, limited flow through the manure beds was observed over time in the LBRs in this research experiments. This phenomenon was likely due to the preferential flow pathway in the manure beds. As a result, an inefficient leaching process and difficulty with HSCM degradation in the LBR resulted in limited biomass hydrolyzed and transferred into the next reactor in the AD process.

An option to overcome limited liquid flow through HSCM in LBRs is to add bulking agents or additional material as dispersion media on top of the waste bed. The bulking agents can enhance the void spaces inside the waste bed and evenly distribute the leachate flowed through the waste bed in the LBR (Nguyen, Kuruparan, and Visvanathan 2007). However, because adding bulking agents requires additional expenditure and displaces active reactor volume, it may not be the best option. The addition of sand as dispersion media on top of the waste bed in the LBR was used in the experiments conducted by Karim (2013) and became a better alternative. However, pre- and post- treatment is required for addition of sand to the LBR. Therefore, in this study, one of the major tasks was to investigate the use of new materials in place of sand or bulking agents.

2.9 Enhancing liquid flow though HSCM in LBRs

Hydraulic conductivity is a parameter that measures the ability of a liquid to flow through porous media. It is a property which is affected by the physical, chemical, and biological conditions in the system. Some major factors can directly influence hydraulic conductivity including particle size, void ratio, substrate composition, fiber content, the degree of saturation, pore geometry, and properties of the test fluid (Chen and Chynoweth 1995). For the MSAD system to function properly, hydraulic conductivity must be sustained for the entire duration of the batch digestion. A critical parameter for successful operation of the LBR is maintaining hydraulic conductivity through the reactor. This possibly overcomes issues such as preferential pathway in the reactor (Lissens et al., 2001), mass-transfer limitation (Myint and Nirmalakhandan, 2006), and clogging resulting in column failure (Daniel and Bouma, 1974). In anaerobic hydrolysis of HSCM containing fibrous material, mass transfer limitations in the LBR may hinder the transport of enzymes and leachates within the leach bed (Myint and Nirmalakhandan, 2006). These issues can be caused by the degree of saturation in the waste bed, low porosity of substrates in the waste bed, microbial growth resulting in clogging in the waste beds, and formation of a crust layer on the surface of waste bed.

2.9.1 The relationship between hydraulic conductivity and the degree of saturation of OSW beds

A factor that can negatively affect hydraulic conductivity through a LBR is related to the degree of saturation of OSW bed. According to Chen and Chynoweth (1995), the hydraulic conductivity can be variable with the degree of saturation in the media. Results showed that hydraulic conductivity variation in the column can be caused by changing degrees of saturation. Various stages of hydraulic conductivity were observed in this study which was conducted by Chen and Chynoweth. The hydraulic conductivity decreased at the first stage as the gas generated and excluded water from pore spaces in the substrate matrix, even if the substrate was already submerged in the liquid for several days. The gas was carried by the liquid as flow continued, and the substrate matrix tended to become more saturated. This started the hydraulic conductivity increasing at the beginning of the second stage. Over time, the liquid flow squeezed the gas out of pore spaces and the substrate matrix became saturated at the end of the second stage. The hydraulic conductivity reached a peak and levelled-off (Chen and Chynoweth, 1995). The research conducted by Chen and Chynoweth concluded that hydraulic conductivity in the waste bed was related to the degree of the saturation of substrate matrix.

2.9.2 The relationship between media porosity and hydraulic conductivity in waste beds

Porosity of the waste bed is one parameter which can improve the performance of the columns. The enhancing of porosity of the bed may reduce the mass transfer limitations in the column. The porosity of waste bed can be controlled by the particle size and volume fraction in the columns (Myint and Nirmalakhandan, 2009).

One previous study indicated that, diffusion processes controls the liquid transport under unmixed conditions in columns, “which are strongly related to the porosity of the media and to the water content” (Abbassi-Guendouz, Amel, et al., 2012). In addition, the packing density can also change the porosity of the waste bed and finally affect the hydraulic conductivity in an LBR. According to the Custer et al. studies (1990), using chopped sorghum, the packing density is one of the significant factors to affect the hydraulic conductivity of the materials. As the depth increased in the LBR, the packing density increased due to the supported weight increased with increasing depth. Some researchers have increasing media porosity by adding packing materials to the waste bed to improve liquid flow through the bed (Custer et al., 1990).

2.9.3 The relationship between microbial growth and hydraulic conductivity in waste be

Due to biological growth, hydraulic conductivity through waste beds could decrease. There are two mechanisms by which biological activity can impact liquid flow through waste beds including breakdown of solid particulates to suspended particles and growth of biomass in pore space. One research study indicated that the infiltration may decrease due to the suspended particle from the effluent to clog the pores spaces. The data suggested that “clogging may be due to the production of gums derived from organics in the liquid waste and in the pores” (Daniel and Bouma, 1974).

In addition, microbial activity can clog pores spaces by microbial cells and results in reduce the hydraulic conductivity (Seki, 2013; Okubo and Matsumoto, 1983). Okubo and Matsumoto (1983) developed “a formula to calculate the effect of biological clogging to the reduction of hydraulic conductivity”. Due to microbial growth in the void pores, increasing alignment of cells in the direction of liquid flow in pore spaces progressed to decrease hydraulic conductivity (Fowlerand and Robertson, 1991). Another previous research indicated that the carbon load of the influent is one of the factors to affect the clogging issue in the column reactors. The study shows that “aerobic treatment of wastewater, which can reduce the carbon load could lengthen the operation time of the system and reduce clogging” (Daniel and Bouma, 1974). The reason that reducing carbon load to a system can reduce clogging is that microbial biofilm may grow more quickly in a liquid medium with a high level of dissolved organic carbon.

2.9.4 Crust layer formation to reduce hydraulic conductivity in waste beds

A factor that can negatively affect hydraulic conductivity through a LBR is related to the formation of a crust layer on the surface of waste bed. In the LBR reactor, the condition about leachate trickling on the surface of the waste bed is similar as raindrops precipitated on the soil surface. According to Hadas (2012), two mechanisms can form a surface crust by liquid drops. This reference discusses crust formation on soil surfaces, but findings are likely also applicable to manure columns. First, liquid drops impact on the surface and breaks down the surface aggregates into fine particles, and those particles can accumulate and fill the void spaces between surface aggregates. The second mechanism is that liquid drops directly strike on the soil surface and the compaction by the drops results in fragmentation of aggregates on the surface. This causes rapid formation of a seal on the surface (Hadas, 2012). These two mechanisms could impact manure in a LBR, resulting in formation of a crust layer on the surface of the waste bed. Surface crust formation can restrict leachate infiltration into the waste beds and reduce liquid flow through the manure. The restriction of leachate infiltration causes the reactor to become clogged, leading to reactor failure as leachate begins to pool. According to the experiments conducted by Karim (2013), a sand layer added on top of the manure bed in the LBRs was to restrict the impact by the leachate drops on the surface of manure beds. The purpose of a layer of sand was to prevent the crust layer formation on the surface of manure beds and clogging issue occurrence in the LBRs.

2.10 Addition of Top Layer Materials to the LBR

According to the experiments conducted by Karim (2013), a sand layer was added as dispersion media on top of the waste bed in the LBRs. This promoted good hydraulic flow through the reactor and reduced the clogging issues. Sand promoted water dispersion evenly through the reactor and may have prevented leachate channeling in the LBRs. This resulted in sustaining leachate flow through the reactor until maximum organic content leached into the liquid. However, addition of a sand layer could add extra cost for system maintenance and operation including pre- and post-treatment requirements. The pre-treatment includes cost to obtain sand material and prepare consistent amount of sand for each batch. The post-treatment required to separate the sand particles and manure, and collect and reuse sand for future batches. Because manure could turn to slurry condition after experiments, separation and collection of sand became difficult. Sand would be difficult to use operationally. Therefore, in this research, investigation of geosynthetic materials used as the top layer options instead of the layer of sand. The new top layer materials should be further enhanced liquid flow through the waste bed and can be reused for future experiments.

2.11 Use of Geosynthetic Materials to Improve Liquid Flow

According to the American Society for Testing and Materials (ASTM), a geosynthetic materials was defined as "a planar product manufactured from polymeric material used with soil, rock, earth, or other geotechnical engineering related material as an integral part of a man-made project, structure, or system" (Begr and Suits). The material includes a wide range of synthetic products, including geotextile geogrids, geonets, geomembranes, geofoams, and geocomposites (Shukla, 2012). These materials are used in different industries, and some main applications include geotechnical, environmental, hydraulic and transportation engineering (Shukla, 2012). The material always performs one or more functions when used in conjunction with soil, rock or other civil-engineering-related material including separation, reinforcement, filtration, drainage and fluid transmission, and acting as a fluid barrier (Shukla, 2012).

The drainage function of geosynthetic materials could be a key feature to implement as a dispersion media on top of the waste bed in the LBRs. Several geosynthetic materials such as geocomposites have the ability to control surface erosion (Shukla, 2012). Those types of medias on the top of the waste bed could evenly distribute the leachate flow through the waste beds resulting in the possibility of reducing leachate channeling, enhancing the leachate flow through the waste beds and increasing organic leaching potential of the LBRs.

2.12 Downward flow and Upward Flow Configuration in LBRs

LBR in both down flow and up flow configuration provide the advantages of being able to handle HSCM with less water consumption and energy input than conventional AD (Dogan, E., et al 2009). An additional benefit of the LBR technology is that it allows the HSCM feedstock to be directly loaded into the reactor with no need for pre-treatment (Brummeler, Horbach, and Koster 1991).

In the downward flow LBR configuration, leachate is trickled into the reactor from the top cap and collected from the bottom. Leachate is then "recycled back into the solid waste bed to provide inoculum and a hydraulic medium to optimize contact between the substrate and microbial cells" (Uke and Stentiford, 2012). A disadvantage of the down-flow LBR configuration is that less biogas is produced compared to a continuously fed one stage system (De Bere, 2000). This disadvantage is caused by limitations created by leachate preferential pathways and clogging issues in the LBR. These hydraulic problems can lead to an inefficient leaching process in the LBR. An additional observation from the Brummeler study is that due to their density, the leachate filled waste beds can become compacted, which limits hydraulic flow and increases digestion time (Brummeler, Horbach, and Koster 1991).

In the upward flow LBR configuration, leachate flows in the upward flow direction and recycles back into solid waste beds. Compared to the down-flow LBR configuration, the up-flow setup can avoid clogging issue and maintained a constant water level in the LBR reactor (Uke and Stentiford, 2012). According to the research conducted by Uke and Stentiford (2012), operation of LBRs under upward flow water direction and leachate recycle resulted in more leachate production compared to downward flow water addition and leachate recycle. This is due to the fact that leachate addition from the bottom of the reactor could unclog the screen at the base, and this enhanced leachate flow through the waste beds without clogging issues (Uke and Stentiford, 2012). In addition, the study recorded the upward flow configuration reactor with higher pH values, which could be due to more leachate released to achieve better dilution than downward flow reactor. The upward flow LBR configuration can be an alternative to address clogging in the LBR (Uke and Stentiford, 2012).

2.13 Summary

AD has notable benefits compared to other waste management including conversion of waste into renewable energy and the ability to reduce the environmental pollution from the waste. The MSAD technology may offer potential advantages for implementation in Colorado due to the arid climate and water resource limitations. One issue with the MSAD technology is maintaining liquid flow through the manure contained in the LBR. A layer added on top of the waste bed in the LBRs could be a dispersion media. A layer of sand was a successful dispersion media in a previous experiment conducted by Karim (2013). However, because sand is difficult to use operationally, such as separation and collection difficulties after each experiment, the investigation of new top layer materials was conducted in this research. The geosynthetic material was one of good options due to its reinforce drainage separator function and control surface erosion function. This dispersion media was expected to evenly distribute the leachate flow through the waste beds resulting in reducing leachate channeling and preferential flow paths, and enhancing the leachate flow through the waste beds. The dispersion media was expected to also possibly impede the crust layer formation on the surface of waste bed. In addition, increasing the contact surface between substrates and anaerobes can reduce the mass-transfer limitations in the LBRs. In addition, the upward flow LBR configuration may enhance the saturation degree in the waste bed and reduce preferential pathways through waste beds. This research evaluated multiple top layer materials for manure LBRs and the upward flow configuration.

CHAPTER 3: MATERIAL AND METHODS

3.1. Experiment Setup

The purpose of this study was to assess the ability of various top layer materials and flow regimes to improve liquid flow through a LBR to digest the HSCM. A system was constructed inside the Simlab building. The system included six LBRs which connected to MSAD system in the trailer where was located outside the Simlab (fig.6). The trailer included LST and HARD reactors. Downward and upward flow experiments were conducted into this research. Each upward flow experiment was conducted in triplicate to collect reliable results. Representative manure samples (section 3.2) were added into LBRs to digest. The study included downward flow and upward flow phase experiment to find out the best set up to enhance the liquid flow of LBR. The summary of the setup of downward flow and upward flow experiments details listed in table 1. U1 (a) was the alternating experiment from downward flow to upward flow phase. The representative manure before digestion in the LBR reaction was named "pre-digested' manure, and manure after digestion in the LBR reaction was named as 'post-digested' manure. All pre-digest, post-digest HSCM and leachate sampled from the LBR were subjected to several labs analyses. The tests conducted on HSCM samples included TS, FS, and VS. The test conducted on the leachate samples included COD. The LBR experiment inflow and outflow and pressure head were measured daily.

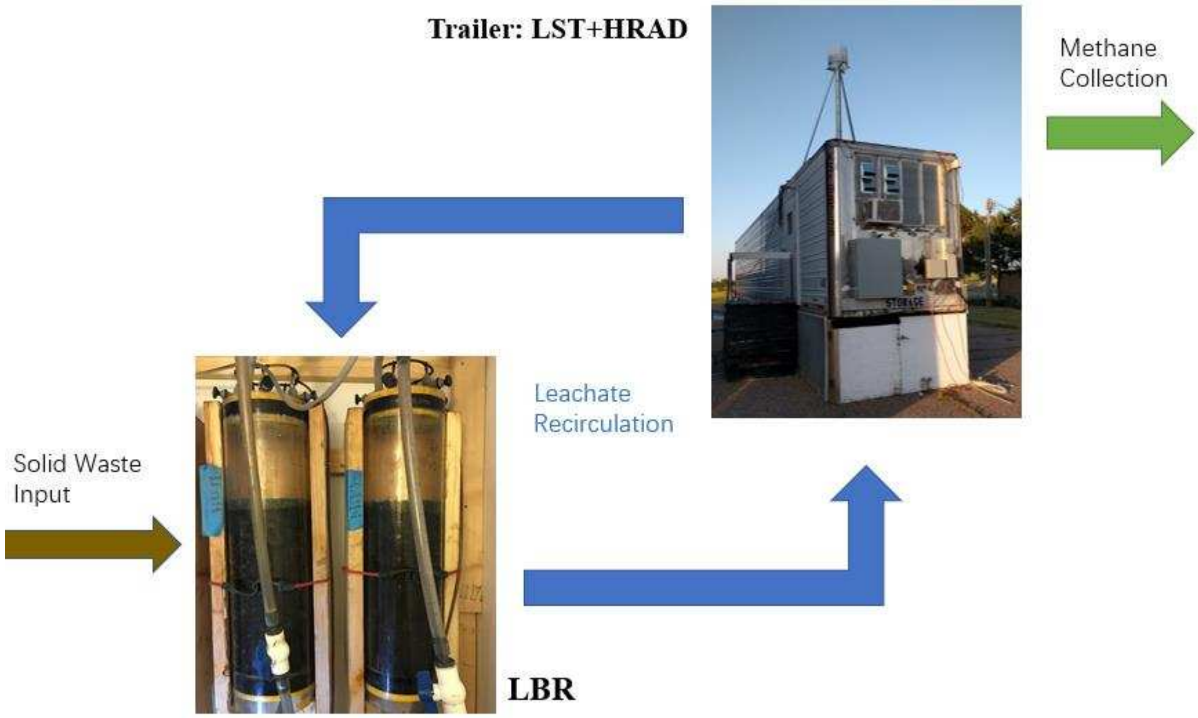

Fig.6 Flow Direction of Reactor System

Table 1 Summary of Downward flow and Upward flow Experiments

Experiment #	Experiment Summary
Downward flow Phase	
Downward Flow Experiment 1 (D1)	3 columns in downward flow configuration (no sieve, no compression) #1 12 in depth holes filled with sand #2 Sand on the top of manure (no hole) #3 4 in depth holes filled with sand
Downward Flow Experiment 2 (D2)	3 columns in downward flow configuration (no sieve, compressed manure (P.E.= 47.17 J)[1]) #1 12 in depth holes filled with sand #2 Sand on the top of manure (no hole) #3 4 in depth holes filled with sand

Downward Flow Experiment 3 (D3)	3 columns in downward flow configuration (no sieve, compressed manure (P.E.= 47.17 J)[1]) #1 Green waste on the top of manure #2 Wood chips on the top of manure #3 non-woven monofilament geosynthetic on the top of manure
Downward Flow Experiment 4 (D4)	6 columns in downward flow configuration (sieved manure, compressed manure (P.E.= 47.17 J)[1]) #1 Raw Manure (sieve lease than 2.2cm) #2 Gravel on top of manure #3 Sand on the top of manure (Home depot sand) #4 8 oz geotextile with ring #5 12 oz geotextile with sand inside sandbag on the top #6 large manure particle (1/2 to ¼ inch)
Downward Flow Experiment 5 (D5)	6 columns in downward flow configuration (sieved manure, compressed manure (P.E.=30.9 J)[2]) #1 Raw Manure (sieve lease than 0.85 in) #2 Gravel on top of manure #3 Sand on the top of manure (Home depot sand) #4 geonet material with ring #5 non-woven monofilament geonet composite with sand inside sandbag on the top #6 large manure particle (1/2 to ¼ inch)
Downward Flow Experiment 6 (D6)	6 columns in downward flow configuration (sieved manure, no compression) #1 Raw Manure (sieve lease than 0.85 in) #2 Gravel on top of manure #3 Sand on the top of manure (Home Depot sand) #4 geonet material with ring #5 non-woven monofilament geonet composite with sand inside sandbag on the top #6 large manure particle (1/2 to ¼ inch)
Downward Flow Experiment 7 (D7)	6 columns in downward flow configuration (no sieve, no compression, top manure layer) #1 Sand on the top of manure (Home depot sand) #3 Manure (> 0.25in) on the top of manure #4 Manure (0.25-0.20in) on the top of manure #5 Manure (0.20-0.10in) on the top of manure #6 Manure (< 0.10in) on the top of manure
Downward Flow Experiment 8 (D8)	Tested four different particle sizes mature as dispersion layer in downward flow configuration: #1 Sand on the top of manure (Home depot sand) #3 Sand (> 0.25in) on the top of manure #4 Sand (0.25-0.20in) on the top of manure #5 Sand (0.20-0.10in) on the top of manure #6 Sand (< 0.10in) on the top of manure

Upward flow phase	
Upward Flow Experiment 1 (U1a)	Previous 4 columns operated in an upward flow configuration;
Upward Flow Experiment 1 (U1b)	3 columns (U1-1, U1-2, U1-3) in upward flow configurations (no compression, no sieve, no top sand layer);
Upward Flow Experiment 2 (U2)	3 columns (U2-1, U2-2, U2-3) in upward flow configuration (no compression, no sieve, improved filter, sand layer on top)
Upward Flow Experiment 3 (U3)	3 columns (U3-1, U3-2, U3-3) in upward flow configuration (compressed manure (P.E.=47.17 J)[1], no sieve, improved filter, no top sand layer)
Upward Flow Experiment 4 (U4)	3 columns (U4-1, U4-2, U4-3) in upward flow configuration (no compression, no sieve, improved filter, sand layer on top)
Upward Flow Experiment 5 (U5)	3 columns (U5-1, U5-2, U5-3) in upward flow configuration (no compression, no sieve, improved filter, sand layer on top)

1: Compression was accounted in terms of applied potential energy, and the equation below calculates total potential energy to compress the column manure. Potential Energy Applied to the column=M*g*h*N*l where M is the mass of the weight dropped = 2.1kg; g is the gravitational force = 9.81 $\frac{m}{s^2}$; h is the distance from which the weights were dropped = 0.127m; N is the number of compressions per lift = 3; l is the number of lifts per TFLBR = 5

2: Compression was accounted in terms of applied potential energy, and the equation below calculates total potential energy to compress the column manure. Potential Energy Applied to the column=M*g*h*N*l where M is the mass of the weight dropped = 2.1kg; g is the gravitational force = 9.81 $\frac{m}{s^2}$; h is the distance from which the weights were dropped = 0.15m; N is the number of compressions per lift = 2; l is the number of lifts per TFLBR = 5

3.2. Substrate Collection and Preparation

The HSCM used in this study was cattle manure collected from JBS Five Rivers Feedlot on May 2015 and April 2016. The manure collected in May 2015 was used for downward flow experiment D1 to D6. The manure collected in April 2016 was used for downward flow experiment D7 and D8 and upward flow experiments U1 to U5. The HSCM was transferred into the lab building. Before HSCM was loaded into the LBR, the manure was sorted through a 0.85 in sieve (fig.7). The manure passed through the filter was mixed well again and redistributed into three homogenous and representative samples.

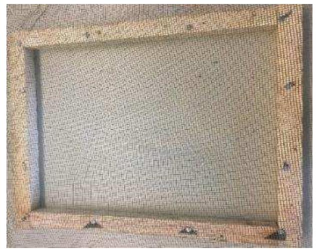

Fig. 7 Manure Sieve

3.2.1.Manure Processing Prior to Loading Columns

In Colorado feedlots, cattle manure is often collected from dry feedlots and dumped into huge piles. The bottom of the manure can be compressed by the heavy weight of the whole pile and resulting agglomeration of manure. The large mass of manure cannot be processed in the column scale experiments due to their size with respect to the columns. A process was developed to remove large masses of manure from the manure samples and attempt to load columns with homogeneous and representative samples.

A 0.85 inches filter was used to sort out big chunks and allow the small particles of manure to go pass through. Manure that passed through the filter was mixed well in a large tub. The manure samples were redistributed into three parts (fig.8), resulting in a homogenous distribution of samples in the columns.

Fig. 8 Three homogeneous samples in Black tub

3.3. System Construction and Set-Up

Six LBRs were operated in this research. The LBRs (fig.9 (a)) were made from a transparent acrylic cylindrical column. The total and working volume of each LBR were 30 L and 22.65 L approximately. The diameter and height of the LBRs were 20.32 cm (8 in) and 91.44 cm (3 feet) approximately. Each LBR contained one top and bottom caps (fig.9 (b)). The cap was composed of two pieces of yellow circular plastic plates, one rubber o-ring, 3/4"-10 carriage bolts, and wing nut. Silicon grease was smeared on the surface of o-ring before fitting caps onto the column to prevent the air going inside the column.

Fig. 9(a) (Left) Photograph of LBR Setup; Fig. 9(b) (Right) Photograph of top and bottom cap of the LBR

Six columns were stored inside a closed, insulated room (fig.10 (a)). A heater heated up the room temperate to $35^\circ C \pm 2^\circ C$. The room temperate was maintained in the mesophilic temperature range ($30^\circ C$ - $40^\circ C$) because AD process has a good performance in the mesophilic temperature range (Kim, Moonil and Young-Ho 2002). Six parallel columns were vertically mounted on the wall of the room with wooden frames. Each column was filled with two third of water and sat on the wooden frames for 12 hours to check that columns were leak-proof (fig.10 (b)).

Fig. 10(a) (Left) Photograph of the closed, insulated room;Fig. 10(b) (Right) Photograph of the LBR leak-proof test setup

3.3.1 Downward flow Setup

The system layout is shown in fig.11. The top cap connected with a leachate circulation inlet connected to the LST and a leachate distribution system. The top pressure head observation tube (fig.12(a)) connected with leachate inlet and mounted on the wall outside the incubator. The leachate was delivered by the MasterFlex peristaltic pump (fig.12 (b)) from the LST. The rate of pumping was maintained at 10 ml/min. Before the leachate flowed into the LBRs, the leachate went through the Rota-meters to make sure the flow rate was the same as the pump rate. The leachate distribution system can evenly and uninterruptedly trickle leachate into the columns. The bottom cap contained a leachate circulation outlet connected to the LST and the sampling port. The leachate circulated back to the LST by gravity. The bottom pressure head observation tube connected to the bottom cap and was mounted parallel with the top pressure head observation tube on the wall.

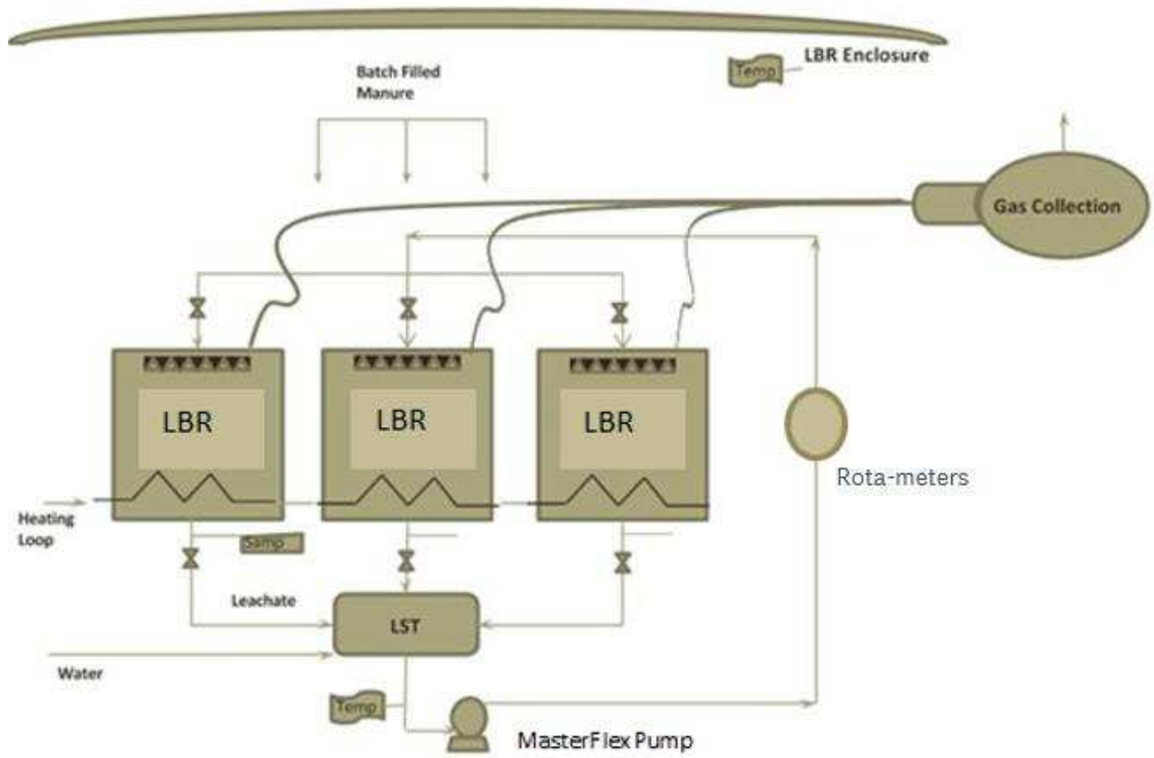

Fig.11 System Layout

Fig. 12(a) (Left) Photograph of pressure head observation tube; Fig. 10(b) (Right) Photograph of MasterFlex peristaltic pump

3.3.2 Upward flow Setup

The bottom cap connected with a leachate circulation inlet. The bottom pressure head observation tube connected with the bottom cap and was mounted on the wall outside the incubator. The leachate was delivered by a MasterFlex peristaltic pump from the LST. The rate of pumping was at 10 ml/min. The leachate went through the rota-meters before the leachate flowed into the LBRs. The purpose was to check the leachate flow rate was the same with the setup pump rate. The leachate was pumped into the bottom of the LBR and collected from the top cap. The top cap contained a leachate circulation outlet connected to the LST and sampling port. The leachate circulated back to the LST by gravity. The top pressure head observation tube connected to the top cap and was mounted parallel with the top pressure head observation tube on the wall.

3.4. Loading Reactors

The homogenous representative HSCM was loaded into LBRs equally. A layer of non-woven monofilament geonet composite (fig.11) was added at the bottom of the LBR to prevent manure from clogging the leachate outlet or inlet (depended on upward flow or downward flow LBR configuration). Then, loading the homogenous and representatively manure samples into the LBR. After that, a liquid distribution media was placed on top of the manure to 1) allow leachate to trickle through the manure evenly and 2) prevent leachate droplets to compact directly on the infiltration surface of manure beds.

Fig. 11 Photograph of Bottom layer of the LBR

3.4.1 Compression

Because the bottom part of the manure in LBRs is compressed due to the heavy weight of the top part of the manure, “the manure in the lab scale LBRs was subjected to manual compression to simulate full-scale operational conditions” (Karim, 2013). Manure was loaded into LBRs in 10cm layers each time which named as a “lift”. After a lift was loaded, a weight was dropped into the LBR to compress the manure. Each lift compressed 5 times. Compression was in term of potential energy which applied in the manure. The setup of manure compression is shown on fig. 12. The potential energy which apply to compress the manure in the LBR was calculated by the equation shown below based on the approach developed by Karim (2013):

$$\boldsymbol{Potential\ \ Energy\ \ Applied\ \ to\ \ the\ \ TFLBR = M * g * h * N * l}$$

Where:

M is the mass of the weight dropped =1.525 kg

g is the gravitational force =9.81 m/s^2

h is the height from which the weights were dropped =0.127 m

N is the number of compressions per lift=5, l is the number of lifts per TFLBR=5

As a result: $P.E. = 1.525 \;\; kg * 9.81 \frac{m}{s^2} * 0.127 \;\; m * 5 * 5$

$$\boldsymbol{P.E. = 47.47 \;\; J}$$

Fig. 12 Photograph of Manure Compression setup

3.4.2 Liquid Distribution Medium Selection

It was expected that the selected new dispersive top materials would have the ability to sustain liquid flow through the manure beds and maintain the LBR operation time over 2-4 weeks. The effect of the dispersive top materials was to reduce compaction on the surface of waste bed by leachate droplets. In addition, the top materials could prevent crusting on the manure's infiltration surface and clogging on the surface layer of the manure beds. One dispersive top material of particular interest was the use of sieved large diameter manure particles to provide an infiltrative surface that is less likely to fail hydraulically. The hypothesis is that the top layer is subject to additional shear forces caused by droplets impinging on it, so using larger diameter manure particles as the infiltrative surface may prevent premature failure at the top of the column; the rationale is that if larger particles are used in the high-shear environment, they are more likely to undergo a specific rate of size reduction comparable to that experienced by the smaller manure particles in the rest of the column. Thus, the top of the column may be less likely to fail before a substantial amount of COD can be captured in the leachate. The selection of dispersive top materials for downward flow experiments included geosynthetic materials (8 oz geotextile, 16 oz geotextile, non-woven monofilament geosynthetic, geonet material, non-woven monofilament geonet composite), wood chips, gravels, sands, and manure with particle sizes (>0.25 in, 0.25-0.20 in, 0.20-0.10 in, ,0.10 in). The geotextiles were from Colorado Lining International.

3.5. System Operation Sampling

The leachate samples were collected before flow through the LBRs and after flow through the LBRs. The leachate samples before the LBRs were collected from the rota-meters port (fig.13), and the samples after the LBRs were from the sampling port (fig.13). The first two leachate samples were collected at 12 and 24 hours after the experiment started. Then leachate samples were collected every three days until the experiment terminated. The pre-digested manure samples were collected from the well-mixed manure before loading to the LBR. The post-digested manure samples were collected from the LBR after the experiment ended 21 days approximately.

In the upward flow experiment, the manure was submerged within the leachate in the LBRs. In some columns, manure turned into slurry texture during the AD process which a large amount of leachate remained in the LBRs. So, the sampling port was opened to drain the leachate out of the LBR after experiment terminated. The drainage process dewatered the leachate out of the manure which turned to be drier than the condition before dewatering process. This process simplified the post-digested manure sample collection for TS, FS, and VS analysis. After the process completed, the post-digested manure poured out from the LBR within a black tub (fig.14). Manure separated into 3 nearly equal volumes representing the top, middle and bottom. For each layer, three samples were collected for later TS, FS, and VS lab analysis. The purpose of layer samples collection was to analyze how much % VS remained and reduced in each layer and how differences are % VS between each layer after AD process accomplishment.

Fig. 14 Discernible top, middle, and bottom layers of Post-Digested Manure

3.6. Evaluation of Leachate Flow through the HSCM in the LBR

The column experiment was to analysis the leachate flow through the LBR. The HSCM with good hydraulic conductivity in the LBR would maintain a longer operation time. Theoretically, it would enhance hydrolysis and acidification reaction in the LBR and generate acid metabolites for methanogenesis. Downward flow and upward flow column experiments were based on the research objectives to conduct. Each experiment was a further improvement based on the previous experimental results.

3.6.1.Reactor Experiment – Downward flow Phase

The downward flow experiments included experiments 1-8. Each experiment was loaded with HSCM in three or six LBR reactors. The flow rate of leachate to the LBRs was 10 mL/min pumped by the MasterFlex peristaltic pump. The leachate flow went through the rota-meter before entering the LBRs to maintain the flow rate.

One task of the research was to test different materials as liquid distribution media instead of sand. Although using a layer of sand as the liquid distribution medium maintained leachate flow through the manure bed, adding a sand layer could lead to additional cost. Each downward flow experiment column tested one kind of top layer material to maintain the even distribution in an effort to maintain hydraulic conductivity.. The additional liquid distribution media included geosynthetic materials (8 oz geotextile, 16 oz geotextile, non-woven monofilament geosynthetic, geonet material, non-woven monofilament geonet composite), wood chips, gravels, sands, and manure with particle sizes (>0.25 in, 0.25-0.20 in, 0.20-0.10 in, ,0.10 in). Unfortunately, during downward flow phase 1-8 we observed clogging issues inside the LBR resulting in reactor failure as leachate began to pool.

3.6.2.Reactor Experiment – Upward Flow Phase 1 (U1)

The U1 included U1 (a) and U1 (b). U1 (a) was the alternating experiment from downward flow to upward flow phase. The setup of this experiment was to change previous 4 columns operated in an upward flow configuration. The leachate was pumped by MasterFlex peristaltic pump into the LBRs at 10 mL/min, and the flow went through rota-meters to maintain the flow rate. The LBRs worked well until that experiment was terminated because one LBR was leaking from the bottom in day 11. This result showed that the experiment was a successful experiment and demonstrated that the upward flow configuration could be an alternative for future experiments.

U1 (b) was conducted with three reactors (triplicate). Due to the success of U1 (a), this experiment was designed to operate three new columns in the upward flow configuration. Each reactor was loaded with HSCM with compression. No additional liquid distribution media was added in this experiment. The leachate was pumped by MasterFlex peristaltic pump into the LBRs at 10 mL/min, and the flow went through rota-meters to maintain the flow rate. Due to the success of the previous experiment, a new experiment was conducted to further verify the utility of the upward flow LBR configuration.

The difficulty observed in U1 (b) was that manure turned into slurry texture instead of solid phase and dispersed throughout the column headspace. While U1 (a) added a layer of sand on the surface of manure bed as weight which may prevent manure turning into slurry texture, U1 (b) did not have such layer.

3.6.3.Reactor Experiment – Upward Flow Phase 2 (U2)

The U2 was conducted with three LBRs. The LBRs were loaded with manure without compression. Causes for the success of U1(a) and the subsequent failure of U1(b) were considered. The most important difference in these two experiments was the sand layer added at the top of the manure of column in U1(a). A layer of sand as weight may possible prevent the manure became slurry texture instead of solid phase in the LBR. So, additional top material sands were added on the top of manure. The sand was bought from Home Depot, and its diameter is 0.0025 in. An improvement top cap filter (fig.15) was also added on top of the manure bed. The new improved filter incorporated the non-woven monofilament geosynthetic that was used in the downward flow configuration by encapsulating it into a french drain cover bag (fig.15). The additional improved top cap filter was expected to prevent the manure particles from clogging the outlet on the top cap. The hypothesis was that the combination of a sand layer on top of the manure bed and the improved top cap filter could prevent the clogging issue inside the column and sustain leachate flow through the manure beds in the LBR. The leachate was pumped by MasterFlex peristaltic pump into the LBRs at 10 mL/min, and the flow went through rota-meters to maintain the flow rate. The manure samples for pre-digest and post-digest were collected before and after the experiment and tested in the lab for TS and VS. The leachate sampled from the sampling port was tested in the lab for COD.

Fig. 15 Non-woven monofilament geosynthetic and french drain cover bag attached with column's top cap

3.6.4. Reactor Experiment – Upward Flow Phase 3 (U3)

U3 was conducted with three LBRs. The LBRs were loaded with manure that was compressed (P.E. =47.17 J). Only the improved top cap filter was added to the reactor. The purpose of this experiment was to determine whether adding the new improved filter instead of adding a top sand layer onto the manure was a better approach to maintain leachate flow through the manure beds in the LBR. The hypothesis was only added improved top cap filter can also prevent clogging issue and maintain leachate flow inside the column. The leachate was pumped by MasterFlex peristaltic pump into the LBRs at 10 mL/min, and the flow went through rota-meters to maintain the flow rate. The manure samples including pre-digest and post-digest were collected before and after experiment and tested in the lab for TS and VS. The leachate sampled from the sampling port was tested in the lab for COD.

3.6.5.Reactor Experiment – Upward Flow Phase 4 (U4)

U4 was conducted with three LBRs. The same column setup as upward flow phase 2 was rerun in this phase.

3.6.6.Reactor Experiment – Upward Flow Phase 5 (U5)

U5 was conducted with three LBRs in the upward flow configuration. The setup was the same as U2.

3.7. Analytical Methods

Pre-digested and post-digested HSCM samples from each stage of the experiment were analyzed for TS, FS, and VS. The leachate samples were analyzed for COD.

3.7.1.Solid Waste Characterization

Waste characterization was to analyze the quantity of VS reduction from pre-digested to post-digested HSCM due to the hydrolysis reaction.

3.7.1.1. TS

The TS was defined as the total mass of solid material sample after evaporating all moisture storage inside the sample. Pre-digested and post-digested homogenized representative HSCM samples were added into each aluminum dish between 5g and 10g. The dishes with and without samples were weighed and recorded. The samples with dishes were placed in an electric oven to dry at 110°C until the mass stabilized. The oven duration was approximately 2 to 6 hours. The mass of the samples with dish were recorded again.

Mass of TS present in manure sample

$$= Weight\ \ of\ \ sample\ \ with\ \ dish\ \ after\ \ 110C - weight\ \ of\ \ empty\ \ dish$$

% TS present in manure sample

$$= \frac{Mass\ \ of\ \ TS\ \ present\ \ in\ \ manure\ \ sample}{Initial\ \ weight\ \ of\ \ sample\ \ in\ \ dish - weight\ \ of\ \ empty\ \ dish}$$

Mass of TS of manure in LBR

$$= \%\ TS\ \ present\ \ in\ \ manure\ \ sample * \ \ total\ \ mass\ \ of\ \ manure\ \ in\ \ LBR$$

3.7.1.2. FS

The FS was defined as the total mass of residual solid material after the samples were heated at 550°C for one hour. The samples dishes (section 3.7.1.1) remained in the furnace until mass stabilized. Once a stable mass was observed the mass was recorded.

Mass of FS present in manure sample

$$= Weight\ \ of\ \ sample\ \ with\ \ dish\ \ after\ \ 550C - weight\ \ of\ \ empty\ \ dish$$

% FS present in manure sample

$$= \frac{Mass\ \ of\ \ FS\ \ present\ \ in\ \ manure\ \ sample}{Initial\ \ weight\ \ of\ \ sample\ \ in\ \ dish - weight\ \ of\ \ empty\ \ dish}$$

3.7.1.3. VS

The VS is defined as the total mass of volatilized solid material sample after cooked at 550°C. The VS is the portion of organic manure sample. The difference in weight between TS sample (section3.7.1.1) and FS sample (section3.7.1.2) is the mass of VS of the manure sample.

Mass of VS present in manure sample

$$= \ Mass\ of\ TS\ present\ in\ manure\ sample\ \ — Mass\ of\ FS\ present\ in\ manure\ sample$$

% VS present in manure sample

$$= \frac{Mass\ \ of\ \ VS\ \ present\ \ in\ \ manure\ \ sample}{Mass\ \ of\ \ TS\ \ present\ \ in\ \ manure\ \ sample}$$

Initial Mass of VS of manure in LBR

$$= Inital\ \ Mass\ \ of\ \ TS\ \ \ of\ \ manure\ \ in\ \ LBR$$
$$* \ \ Initial\ \ \%\ \ VS\ \ present\ \ of\ \ manure\ \ in\ \ LBR$$

Final Mass of VS of manure in LBR $= Final\ \ Mass\ \ of\ \ TS\ \ of\ \ manure\ \ in\ \ LBR *$

$$= Final\ \ Mass\ \ of\ \ TS\ \ of\ \ manure\ \ in\ \ LBR$$

$$*\ \ Final\ \ \%\ \ VS\ \ present\ \ of\ \ manure\ \ in\ \ LBR$$

Avg % VS reduction of manure in LBR

$$= \frac{Initial\ \ Mass\ \ of\ \ VS\ \ of\ \ manure\ \ in\ \ LBR - Final\ \ Mass\ \ of\ \ VS\ \ of\ \ manure\ \ in\ \ LBR}{Initial\ \ Mass\ \ of\ \ VS\ \ of\ \ manure\ \ in\ \ LBR}$$

3.7.2. Leachate characterization

Leachate characterization was conducted to analyze the quality of leachate produced from the LBRs in terms of COD. The COD data represented the changes of organic content in the leachate samples during the reaction period.

3.7.2.1. Chemical Oxygen Demand

COD represents “the oxygen equivalent of the soluble organic matter that can be oxidized using a strong chemical oxidizing agent in an acidic medium” (Karim, 2013). The method to measure the leachate samples’ COD used a ‘Hach test N tube kit’ from Hach Company, a colorimetric test. Due to detection range (20-1,500 mg/L) of the Hach DR 3900 spectrophotometer, the leachate samples had to be diluted 1:10 or 1:20 in order to be inside the detection range. The undiluted samples usually had very high values of COD. The diluted samples were added to each COD vial in a specific amount. The D.I. water was also added into each COD vial in order to have each COD vial contains a total of 2ml liquid. Each performed run also included one control vial (only D.I. water) and a 1000mg/L COD standard (Hach COD standard solution, cat. 2253929). The COD vials were inverted a few times and then incubated in Hach COD heater for two hours. After two hours, all vials were placed into the wood frame to cool down to room temperature and then measured for COD using the Hach DR 3900 spectrophotometer. The control vial was used to zero the instrument before all other samples were measured. The dilution factors are used to calculate the final COD values for those leachate samples.

3.7.3 Column Physical Characterization

Column physical characterization was conducted to analyze the physical properties of the reactor and to understand how well leachate was flowing through the manure bed in the reactor. Column flow rate data represented the leachate flow rate through the reactor, and column pressure head represented the condition and pressure build up inside the reactor.

3.7.3.1 Column Experiment Flow rate

The flow rates of the leachate leaving the column were measured. Leachate was collected into a beaker from the sampling port over the course of one minute to determine the volumetric flow rate in ml/min. The column flow rate was recorded daily until the experiment ended.

3.7.3.2 Column Experiment Pressure Head

Column pressure heads were measured by the difference between pressure head on the top cap and bottom cap of the reactor. Both ¼ inch tubes were attached to the top and bottom cap and mounted on the wall in parallel. The top and bottom pressure heads were recorded every day, and column pressure heads were manually calculated and recorded.

CHAPTER 4: RESULTS

4.1. Reactor Experiment – Downward Flow Phase

In downward flow experiments (1-8), all the columns tested downward flow failed hydraulically in 4-6 days of operation time. Leachate could not easily permeate through the manure layer in the LBRs, and it caused leachate to build up on the top of LBRs. Fig. 16 is shown an example of a failed LBR. D1 through D6 experiments were used the manure collected on May 2015. And, D7 and D8 experiment tested the manure which was collected on April 2016. Further analysis of solid and liquid quality was not conducted for the downward flow phase experiments due to system failure. Due to no analysis data of solid sample, the differences of initial %VS between two manure sources could not be told. However, all 8 experiments which conducted both manure sources did not success at the end, and this mean that the failure of columns did not be mainly affected by the sources of manure. The downward flow experiments demonstrated that geo-synthetic top materials could not be substituted instead of addition of sand as a dispersion medium to the LBRs. In addition, the experiments showed that downward flow configuration was hard to maintain for 21 days of operation.

Major failure mechanisms were observed in these experiments. Clogged layers formed in the waste beds, but the location of clogged layers could not be affirmed. The possible options included either level of the manure beds, surface of manure beds due to crust formation, or both. One possible location of clogged layer was on the surface of manure bed as crust layer. The mechanism to form crust layer was that leachate droplets trickled to the surface manure and broke down manure into very fine particles. Those fine particles filled in the void pores between surface aggregates and finally clogged the pores resulting in crust layer (Hadas, 2012). Although top layer materials were added on the surface of the manure beds as dispersion media in the LBRs, the crust layer may still form due to the materials did not function as reinforce drainage separator and control surface erosion. Another possible location was inside any levels of manure bed. The mechanisms to form clogged layer inside the manure bed was due to biomass growth. The gums which derived from the organic in the leachate or substrate pores likely clogged the aggregate pores resulting in clogging layers in the manure beds. The clogged layers reduced the manure permeability (Daniel and Bouma, 1974), and the leachate became hard to flow through the manure beds. The less leachate flowed through the pores space may result in less gas squeezed out the pore spaces, and finally gas continually accumulated inside the pore spaces (Chen and Chynoweth, 1995). This would cause less saturation in the manure matrix and also built up the pressure inside the LBRs. According to the observation, many gas bubbles were present and escaped from the top part of manure beds by thumping the LBRs. Eventually, this buildup of gas pressure may have been the cause for inability of prevented leachate from draining to drain through the whole column, which caused leachate to pool on the manure surface (fig.16).

Fig. 16 Photograph of leachate pooling/ buildup on the surface of manure bed

4.2 Reactor Experiment – Upward Flow Phase 1 (U1)

Because none of the column experiments including sand or geotextile upper layers were successful, the column configurations were switched to upward flow. Thus, an upward flow configuration was further investigated. U1 (U1; See experiment definitions in Table 1) included U1 (a) and U1 (b). U1 (a) was the turning point experiment from downward flow to upward flow phase. In this experiment, the failed columns from experiment 8 (downward flow phase) were switched to an upward flow configuration, while keeping the same manure inside of each LBR. The LBRs worked well until that experiment was terminated after 11 operation days, except one LBR was terminated on day 11 due to leaking. This experiment indicated that the upward flow configuration could be an alternative for future experiments.

U1 (b) was the first new set of upward flow configuration experiments. U1-1 and U1-2 only operated for 6 days. Although U1-3 still worked after day 6, it was terminated because of U1-1 and U1-2 failure. It was observed that instead of maintaining a solid structure, the manure became slurry and dispersed throughout the column headspace. In addition, considerable amounts of bubbles were released from the manure when the column surface was gently knocked upon. It seemed that a high pressure built up inside the columns because of gas clogging inside the column. Due to the pressure buildup, the top caps of both columns were forced out of the column bodies, causing the experiment to fail.

4.3. Reactor Experiment – Upward Flow Phase 2 (U2)

Causes for the success of U1 (a) and the subsequent failure of U1 (b) were considered. The most important difference in these two experiments was the presence of the sand layer at the top of the columns in U1 (a). Thus, it was considered that a layer of sand as weight on the top of the manure could keep the manure at the bottom of the column. This could prevent manure tend to be slurry and dispersed throughout the column headspace. The improved top cap filter was also added in this experiment to enhance the top cap filter ability (fig. 14). For U2 (See experiment definitions in Table 1), the columns were loaded with the top sand layer and improved top cap filter.

U2-1, U2-2, and U2-3 operated for 23 days. The U2 experiment was the first successful experiment which all three columns operated for 3 weeks.

Total initial, final volatile solids and average percentage reduction of VS in U2 is shown in fig. 17. The reduction in VS was caused by HSCM solubilization between the initial stage and the post-digestion stage. The avg % VS reduction was between 48% and 53%. Compared with the overall reduction 45.92% and 39.31% for VS in downward flow and upward flow reactors in the Uke and Stentiford (2012) studies, VS reduction results were relatively comparable to other reported values. The high rate of VS reduction indicated that the hydrolysis function worked well and efficient in the LBRs.

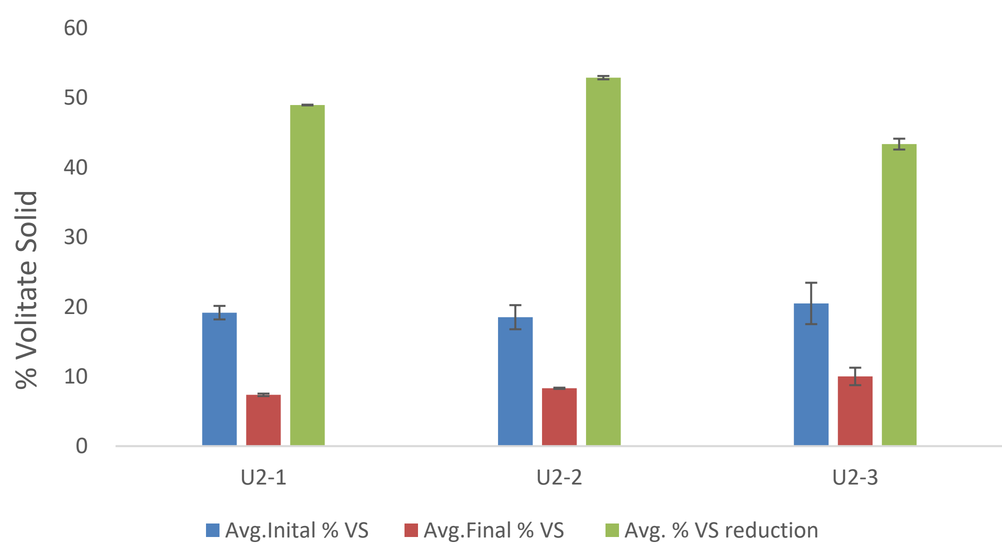

Fig. 17 Total Average Initial, Final Volatile Solid, and Average Percentage Reduction of VS in Upward Flow phase 2 (U2; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)). Error bars indicate +/- one standard deviation.

Average percentage VS in the top, middle, and bottom layers in U2-1 is shown in fig. 21. In one column (U2-1) among all three columns, after pouring out the manure from the column body, the post-digested manure had formed three obvious layers including top, middle, and bottom (fig.19). Because the drainage time was not long enough, the manure still remained the slurry texture in U2-2 and U2-3. After manure pouring out from the column body, the top, middle, and bottom layers could not be distinguished in those two columns (fig.18).

This distribution profile of %VS (fig.20) indicated a tendency for % VS increase with the flow direction. That indicated that in U2-2 experiment, more solubilization of HSCM happened in bottom layer than in the middle and top layers. VS degradation decreased with flow direction. Accordingly, the results from Uke and Stentiford (2012) study presented that the solids degradation decreasing with the water flow direction. The related mechanism is that hydraulic conductivity reduction in the waste bed is due to the fine particle movement to create a denser matrix and hence lower degradation (Uke and Stentiford, 2012). This experiment had the similar trend of VS degradation as the Uke and Stentiford (2012) study.

In addition, because the value of avg % VS standard deviation was large in the top layer of U2-1, it had a higher deviation within the data set. That meant the values of avg % VS in the top layer were more variable than in other portions of the column. The HSCM was not evenly degraded by the microorganisms in the top layer. This may be due to preferential flow pathway appeared in order to the leachate flow probably did not distribute uniformly in the top layer.

Fig.18 Slurry Texture of Post-Digested Manure

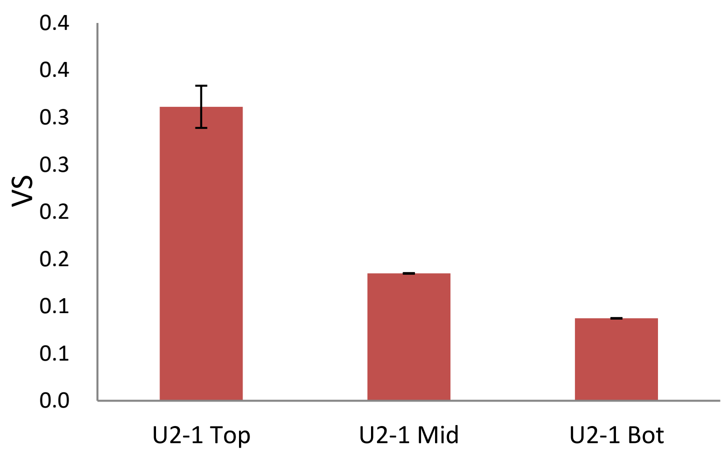

Fig. 19 Average percentage VS in top, mid, and bottom layers in on column Upward Flow phase 2 (U2; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)). Error bars indicate +/- one standard deviation

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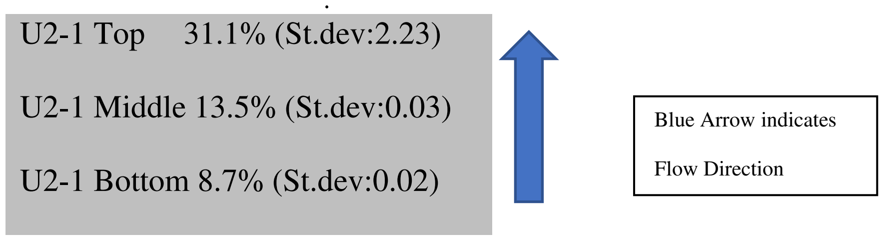

Fig. 20 Distribution of % VS in the top, mid, and bot layer in one column from Upward Flow phase 2(U2; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)).

The concentration of COD in leachate between U2-1, U2-2, and U2-3 is shown in fig. 21.

During the first five days of operation, COD concentration of leachate rapidly decreased and reached the LST COD concentration. The similar COD leachate tendency was observed from the previous studies with LBRs processing manure (Demirer and Chen, 2007). The data indicated that the hydrolysis process was almost finished during the first 10 days of operation. Only a small amount of degradable organic matter or extremely difficult to degrade organic compounds remained in the LBRs. At day 11, all three columns' leachate COD concentration approached to LST COD concentration and became constant. This demonstrated good leaching potential and hydrolysis in the LBRs in this experiment.

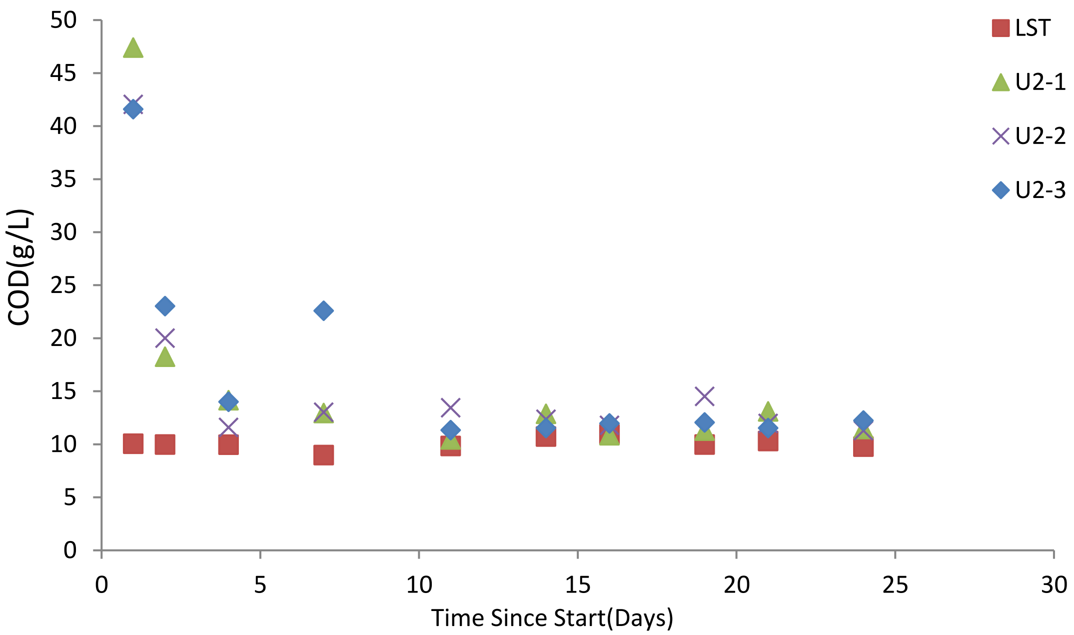

Fig. 21 Leachate COD Concentration in Upward Flow phase 2 (U2; See experiment definitions in Table 1).Error bars indicate +/- one standard deviation.

The column experiment flow rate in U2 is shown in fig. 22. The flow rate of U2-1, U2-2, and U2-3 started at 10ml/min and approached to 15ml/min at day 5 (expect U2-1). After day 5, all three columns' flow rates were constant at 15 ml/min approximately. In day 23, no leachate flowed out of the sampling port over the course of one minute and thus the flow rate was measured to be 0 ml/min at that day. The constant flow rate in U2 meant that the leachate flow through the manure bed sustained at a constant level during the operation time

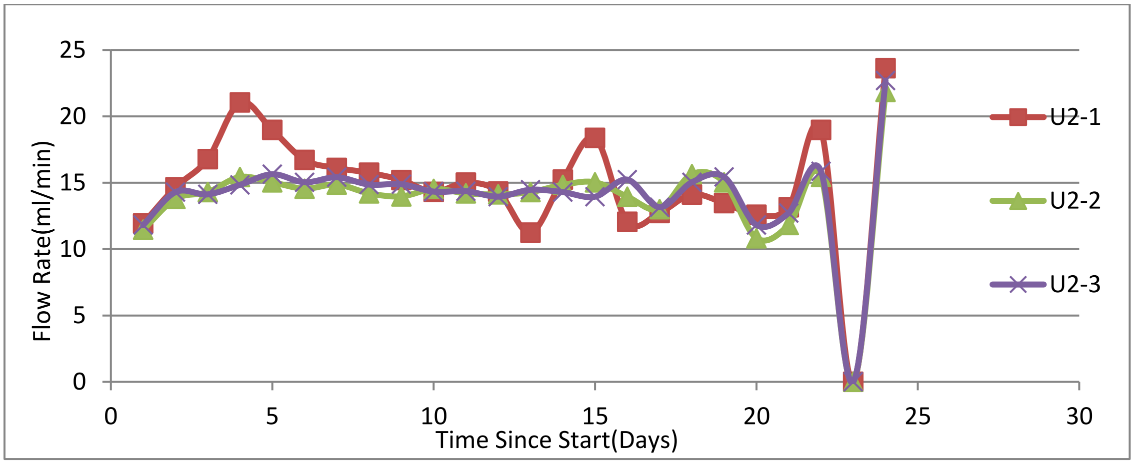

Fig. 22 Column Experiment Flow Rate in Upward Flow phase 2 (U2; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1))

The column experiment pressure head difference in U2 is shown in fig. 23. The variation in U2-1, U2-2, and U2-3 pressure heads was between 1 and 4 inches. At the end of the experiment, pressure head in the three columns' still maintained around 3 inches. These data indicated no pressure buildup inside the LBRs in U2, and it meant that clogging layer did not form inside the column.

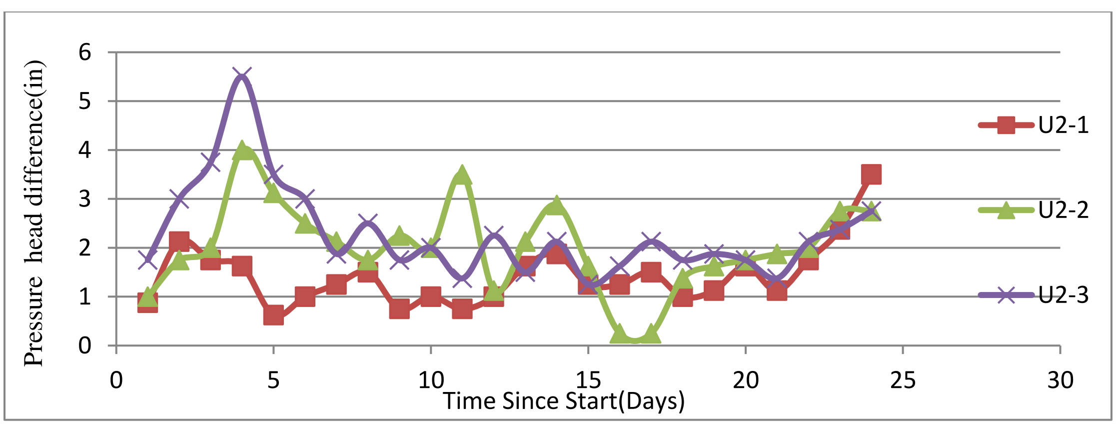

Fig. 23 Column Experiment Pressure head difference in Upward Flow phase 2 (U2; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1))

U2 was the successful upward flow experiment. The average percentage reduction of VS in this experiment was above 48% which indicated a successful hydrolysis process in the LBR. The similar leachate COD trend as the previous study illustrated a completed hydrolysis process in the LBR. The constant pressure head indicated that no pressure built up inside the LBR resulting in no clogging issue. All data illustrated that U2 demonstrated a successful upward flow experiment, and the experiment indicated that adding top material sand on manure and improved top cap filter could enhance flow in upward flow LBR columns.

4.4. Reactor Experiment – Upward Flow Phase 3 (U3)

For developing U3 experiments, causes for success of U2 experiment was considered. A combination of improved top cap filters and a sand layer on top of the manure bed was added in U2. However, when considering operational costs associated with the added sand layer, there was a need to determine whether that sand layers was truly needed for successful operation. Thus for U3, the columns were loaded to include only the top sand layer on the manure bed in the LBR.

In U3, U3-3 operated 23 days after that experiment was initiated. The U3-1 and U3-2 failed at day 7and 16 days respectively. This result indicated that only adding the new improved filter might not be a sufficient replacement for adding the combination of a sand layer and improved top cap filter.

After termination of phase 3 experiments, a simple test was done to indicate whether or not columns failure was due to the top cap clogging. Those caps were removed from the failed columns and were installed into new columns as bottom caps. Water was added to the new columns, and valves were opened on the tested cap. The result showed that the flow came out from each failed columns' caps and this demonstrated that top caps from the failed columns had not clogged. That meant there was a clog somewhere between the top and bottom of the manure.

The total average initial, final volatile solid and average percentage reduction of VS in U3 is shown in fig. 24. The values of avg % VS reduction stayed ranged between 21% and 43%. The avg % VS reduction in U3 was much lower than the U2 (between 48% and 53%), likely the results of U3-1 and U3-2 failure. Hydrolysis of the HSCM likely did not work efficiently in those two LBRs. In U3, only U3-3 demonstrated a high rate of % VS reduction (43.38%) which indicated successful hydrolysis of the HSCM in the LBR.

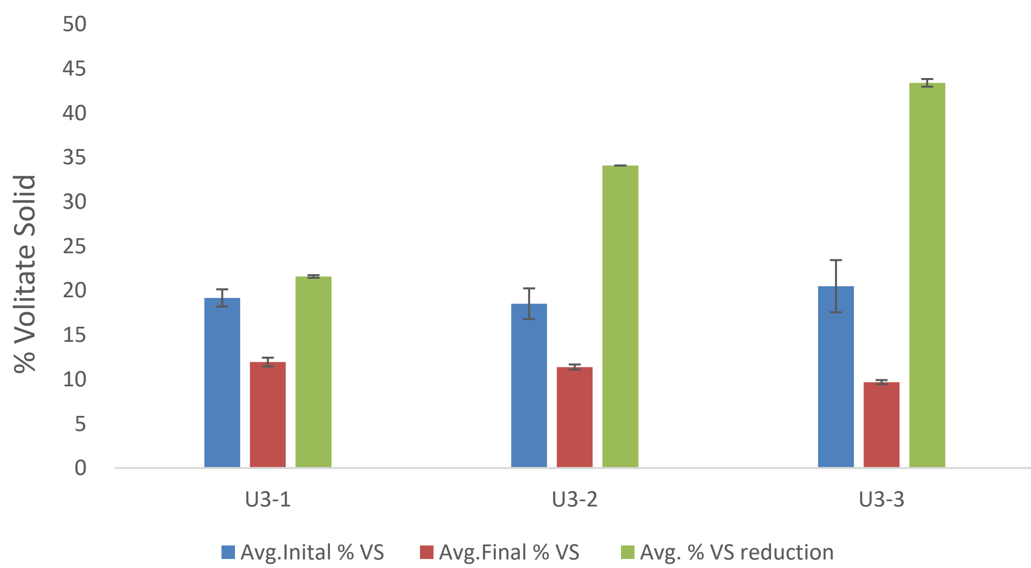

Fig. 24 Total Average Initial, Final Volatile Solid, and Average Percentage Reduction of VS in Upward Flow phase 3 (U3; Improved filter, Compression, No sieve, (See experiment definitions in Table 1)). Error bars indicate +/- one standard deviation.

The leachate COD concentration between U3-1, U3-2, and U3-3 is shown in fig. 25. The COD concentration of U3-1and U3-2 started at the high level and suddenly approached to the LST's COD level in day 5. Due to U3-1 failure at day 8, there was no COD data after day 5. During the first 24 hours, the COD concentration of U3-3 declined, followed by an increase, and reached a peak value at day 12 and finally followed a gradual decline and never approached

LST’s COD level. This incomplete hydrolysis in U3-3 even after day 23 may indicate an inefficient solubilization of HSCM in the LBR due to a poor hydraulics. The COD concentration of U3-2 had a similar tendency as U3-3 between day 5 and day 15. Due to U3-2 failure at day 16, there was no COD data after day 16.

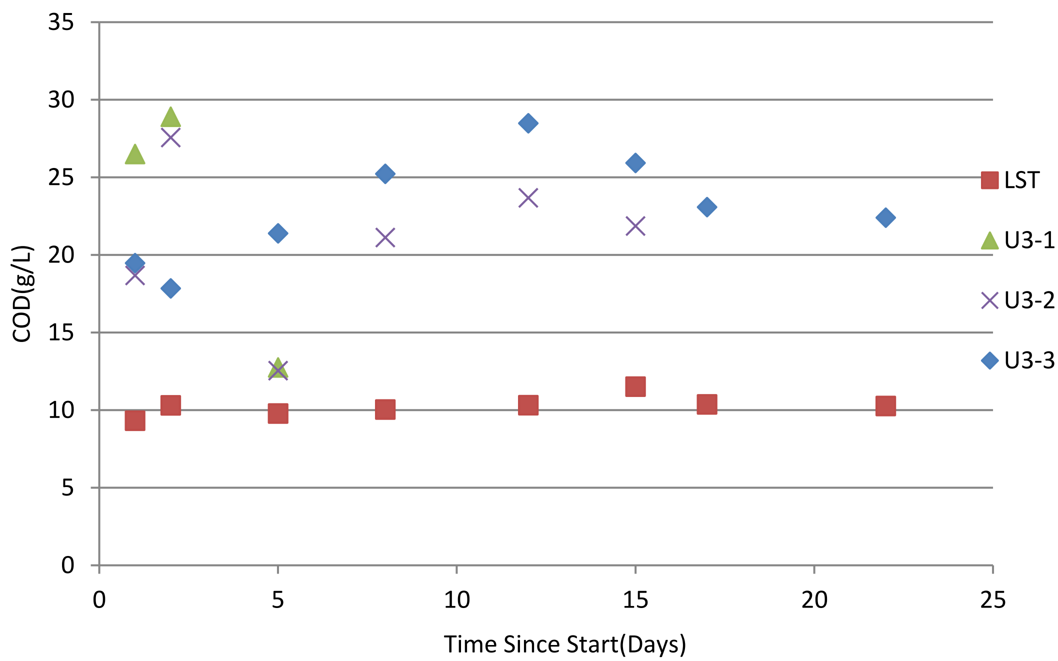

Fig. 25 Leachate COD Concentration Column in Upward Flow phase 3 (U3; Improved filter, Compression, No sieve, (See experiment definitions in Table 1))

The column experiment flow rate in U3 is shown in fig. 26. The LBRs’ flow rates tended to increase and approached 24 ml/min during the first 5 days of operation. Due to U3-1 failure at day 7, there was no leachate flow rate data after day 7. Then, the leachate flow rates in U3-2 and U3-3 decreased and gradually drew down to approximately 10 ml/min until day 15. After day 15, there was no leachate flow rate in U3-2 due to the failure of the column. The flow rate in U3-3 trended to decrease and approached to approximately 5ml/min at the end of experiment. Because no leachate flowed out of the sampling port over the course of one minute, no data measured for U3-2 and U3-3 between day 3 and 5.

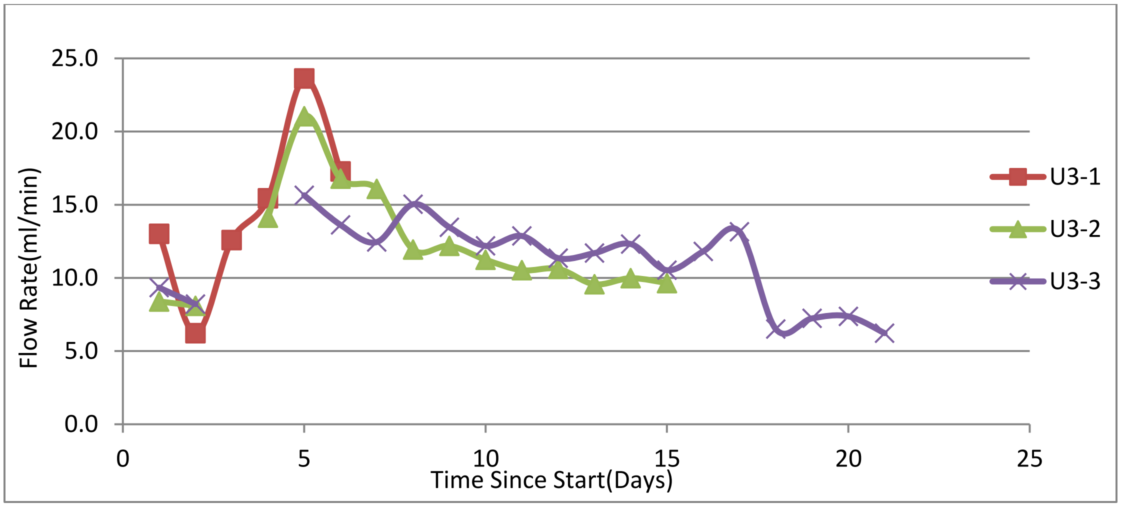

Fig. 26 Column Experiment Flow in Upward Flow phase 3 (U3; Improved filter, Compression, No sieve, (See experiment definitions in Table 1))

The column experiment pressure head difference in U3 is shown in fig. 27. Pressure head of U3-1 gradually increased starting at the 2nd operation day, and it reached 80 inches in day 7. This indicated that the column had failed. U3-2 and U3-3 had constant pressure head around 2 inches during the first 4 operation days, and U3-2 pressure head continually increased after day 4 and approached 80 in at day 16 when failure occurred. This indicated that the pressure started to build up at day4 in U3-2, and the clogging layer formation caused the column failed at the end. U3-3 was the only LBR that operated more than 3 weeks, and its pressure head kept at a constant level around 3 inches until day 16 and suddenly drew up to 76 inches before the experiment terminated. This indicated that although the LBR did not fail at the end, the pressure had already built up. A clogging layer formed inside the LBR. The column may fail in the next few days if this experiment continually operated.

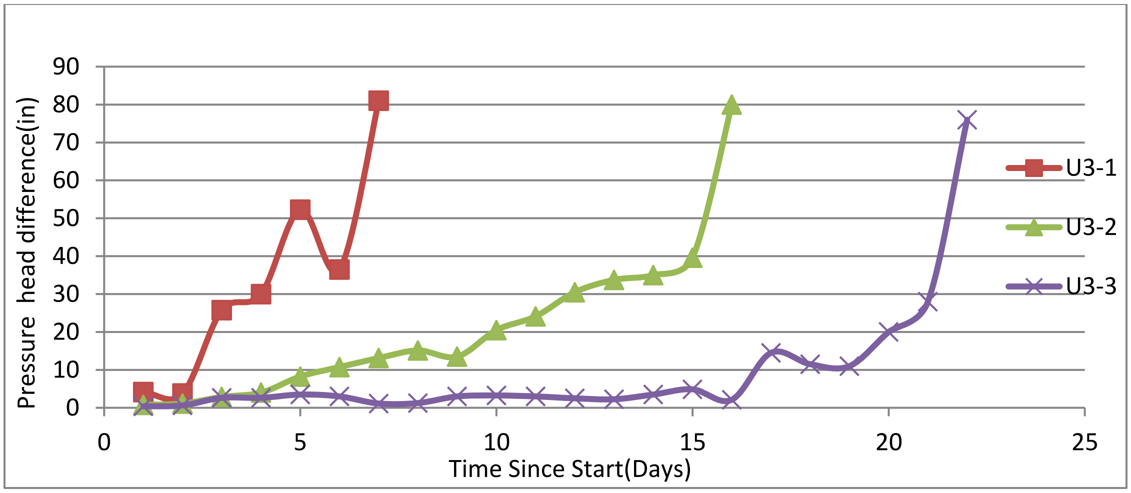

Fig.27 Column experiment pressure head difference in Upward Flow phase 3 (U3; Improved filter, Compression, No sieve, (See experiment definitions in Table 1))

U3 was an unsuccessful upward flow experiment due to two columns' failure. The average percentage reduction of VS in this experiment was below 40% which indicated an ineffective hydrolysis process in the LBR compared to U2. The leachate COD concentration of U3-2 and U3-3 did not approach to LST's COD level during the operation days which illustrated that the hydrolysis process did not complete in the LBR. The high-pressure head rising in three columns demonstrated that pressure head built up inside the LBRs resulting in clogging issues. All data demonstrated that U3 was not successful, indicating that addition of the top material sand layer may have benefited LBR operations in U2.

4.5. Reactor Experiment – Upward Flow Phase 4 (U4)

Causes for the success of U2 and the subsequent failure of U3 were considered. The most important difference in these two experiments was the presence of the sand layer at the top of the columns in U2. Thus, for U4, the columns were loaded with the top sand layer and improved top cap filter.

In U4, U4-1 and U4-3 operated 23 days, while U4-2 only operated for 8 days. One observation was that the top sand layer in the U4-2 was mixed well with the manure in day 3.

The manure inside the column became a slurry texture. The manure in U4-1 and U4-3 was observed to have slurry properties on day 16. The manure which mixed with top sand layer and turned into slurry texture in U4-1 and U4-3 was much later than in U4-2.

The total initial, final volatile solid and average percentage reduction of VS in U4 is shown in fig. 28. Due to U4-2 hydraulic failure at day 8, the average percentage reduction of VS was 1.71% compared with U4-1 and U4-3 which were 67.9% and 35.7% respectively. Degueurce et al. (2016) showed that after 28 days of AD, VS removal ranged from 23% to 44%, which verified cattle manure degradation. These literature values showed that the VS reduction results in U4 except U4-2 were relatively comparable to other reported values. But, the variation of avg % VS reduction between U4-1 and U4-3 was large.

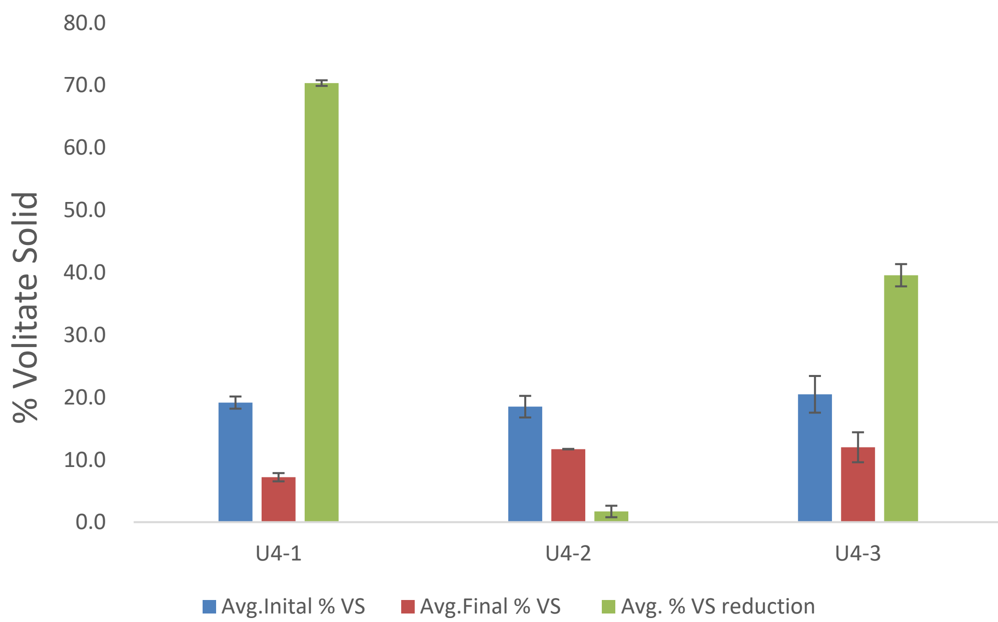

Fig. 28 Total Average Initial, Final Volatile Solid, and Average Percentage Reduction of VS in Upward Flow phase 4 (U4; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)). Error bars indicate +/- one standard deviation.

The average percentage VS in the top, middle, and bottom layers in U4-1, U4-2, and U4-3 are shown in fig. 29, 30, and 31. This percentage distribution of VS profile of U4-2 (fig.31(Middle)) did not have clear tendency between %VS and liquid flow. This percentage distribution of % VS profile of U4-1 and U4-3 (fig.32(Top and Bottom)) indicated a tendency for % VS to decrease with the flow direction. That meant that VS degradation decreased as flow direction in U4-1 and U4-3. U4-1 and U4-3 had the similar trend of VS degradation as the U2. Above all, the profile through upward flow experiments U2, U4-1, and U4-3 indicated that as VS degradation decreased as manure beds level built up in the LBR, more solubilization of HSCM happened in bottom layer than in the top and middle layers. Therefore, the more extensive biological processing of the manure in the bottom layer may have been a result of better hydraulic properties of flow through the material in the bottom.

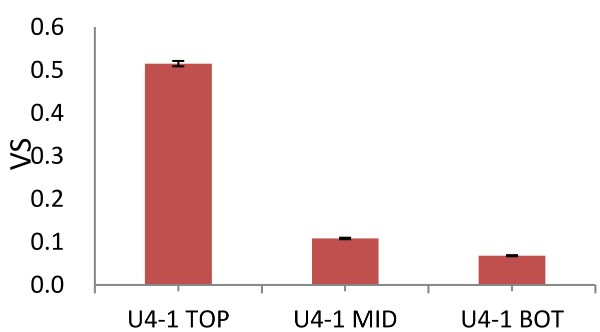

Fig. 29 Average percentage VS in top, mid, and bot layers in Upward Flow phase 4 Column 1 (U4-1; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)). Error bars indicate +/- one standard deviation.

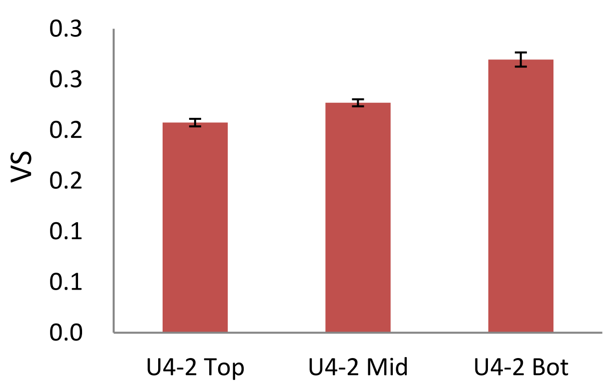

Fig. 30 Average percentage VS in top, mid, and bot layers in Upward Flow phase 4 Column 2 (U4-2; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)).Error bars indicate +/- one standard deviation

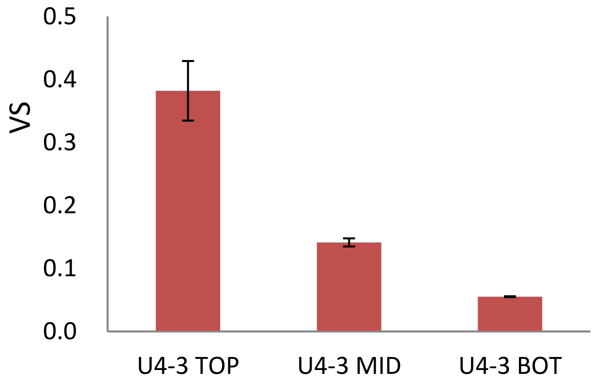

Fig. 31Average percentage VS in top, mid, and bot layers in Upward Flow phase 4 Column 3 (U4-3; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)). Error bars indicate +/- one standard deviation.

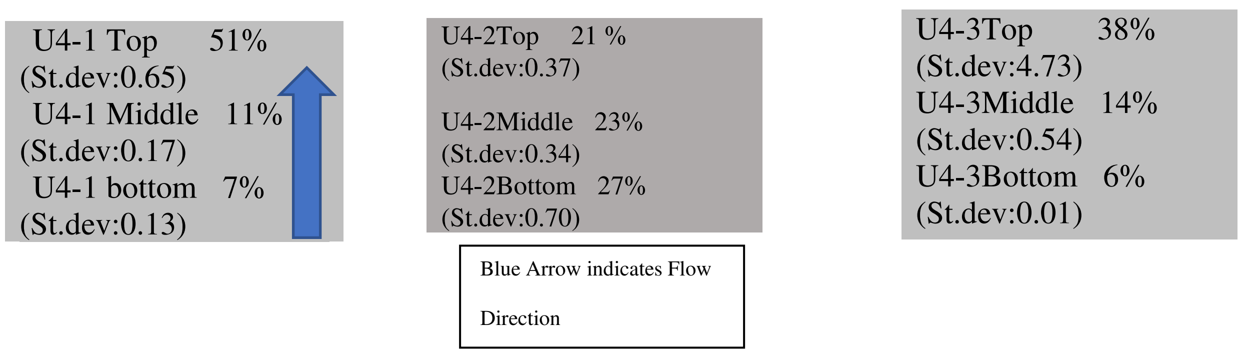

Fig. 32(Left) Percentage distribution of VS in the top, mid, and bot layer in Upward Flow phase 4 Column 1 (U4-1; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)).
Fig. 32(Middle) Percentage distribution of VS in the top, mid, and bot layer in Upward Flow phase 4 Column 2 (U4-2; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)).
Fig. 32(Right) Percentage distribution of VS in the top, mid, and bot layer in Upward Flow phase 4 Column 3 (U4-3; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)).

The leachate COD concentration of U4-1, U4-2, and U4-3 is shown in fig. 33. The COD concentration of U4-1, U4-2, and U4-3 started at the high level and gradually approached the LST's COD level during the first 8 days, and this trend is like U2. Due to U4-2 hydraulic failure at day 8, there was no COD data after day 8. Although U4-2 hydraulic failed at day 8, the COD concentration of U4-2 already approached the baseline of LST COD concentration. This mean that the leaching process of U4-2 was completed. After day 10, the COD concentration in U4-1 and U4-3 kept at a similar level as LST's COD concentration until this experiment terminated.

According to the Karim study (2013), the COD concentration in the leachate deceased after day 4 and approached to 0 g COD/L level after day 9. The trend of COD concentration from the experiment conducted by Karim was similar as U4 experiment. This indicated that most hydrolysis reaction had already finished before day 10; and demonstrated a good leaching potential and hydrolysis in the LBRs in U4-1 and U4-3.

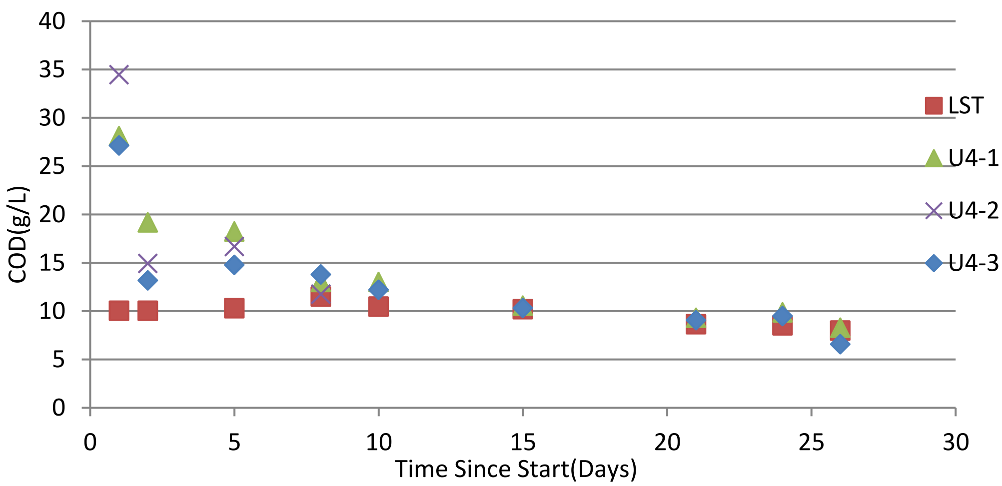

Fig. 33 Leachate COD Concentration Columns in Upward Flow phase 4 (U4; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1))

The column experiment flow rates in U4 is shown in Fig. 34. The variation of U4-1, U4-2, and U4-3 flow rates were approximately between 10 ml/min and 20 ml/min. The two biggest fluctuations of U4-1 and U4-2 flow rates happened between day 9 and day 10 and between day 20 and day 21. Due to U4-2 hydraulic failure at day 8, no flow rate data was showed in the figure after day 9.

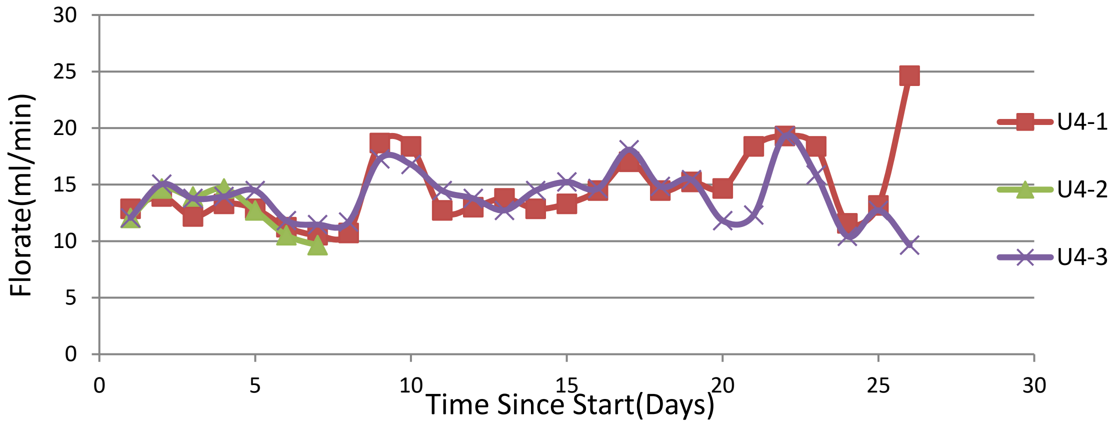

Fig. 34 Column Experiment Flow in Upward Flow phase 4 (U4; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1))

The column experiment pressure head difference in U4 is shown in fig. 35. The variation of U4-1 and U4-3 pressure head was constant between 1 and 3 inches. This indicated no pressure built up inside the columns in U4-1 and U4-3. The pressure head of U4-2 started to increase on day 3 and went above 85 inches at day 8. This indicated the column hydraulic failed due to high pressure built up and clogging issue occurrence inside the LBR.

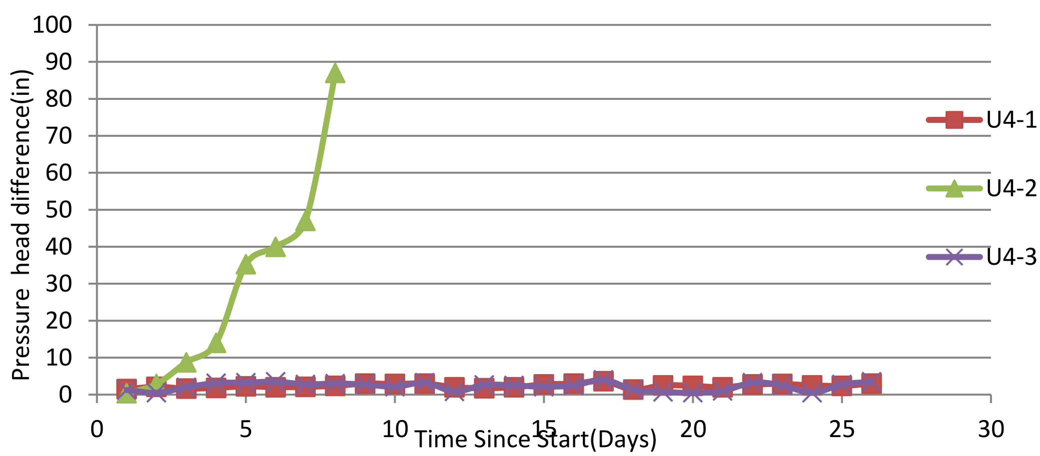

Fig. 35 Column Experiment Pressure Head Difference in Upward Flow phase 4 (U4; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1))

U4 was successful except for U4-2 hydraulic failure. Excluding U4-2, the average percentage reduction of VS in this experiment was above 44% which indicated a satisfactory hydrolysis process in the LBRs. A similar leachate COD trend as the previous study illustrated that most leaching process already finished around day 10 and day 12. The COD concentration of three LBR almost approached to the LST COD concentration. That mean that the leaching of COD was complete in those three columns, and experiments were also complete. The constant pressure head indicated that pressure did not build up inside the LBRs, resulting in no clogging issue (except U4-2 approaching to 85 in high pressure at day 8). Overall, the data illustrated that U4 was a completed upward flow experiment although the U4-2 hydraulic failed. The experiment demonstrated that the combination of a sand layer and improved top cap filter could enhance leachate flow in the LBR. One column did fail in this experiment, indicating the need for further replication.

Based on observations in the failed U4-2 experiment, the possible mechanism for column failure was due to the manure well mixed with the addition of sand as weight on the top of the manure. The layer of sand could keep the manure to stay at the bottom of the column and prevent the manure turning into slurry. The fine particles suspending within the leachate flow could potentially clog the pore space and form clogging layers inside the manure beds

4.6. Reactor Experiment – Upward Flow Phase 5 (U5)

In U5, the columns' setup was the same as the U2 due to the success of that experiment. The experiment included the combination of top material sand on manure beds and the improved top cap filter. In U5, U5-3 operated 21 days after that experiment was initiated. U5-1 and U5-2 hydraulic failed at days 16 and 20.

The total average initial, final volatile solid and average percentage reduction of VS in U5 is shown in fig. 36. The value of avg% VS reduction in U5-2 was 0.5%. The U5-2 operated more than two weeks and hydraulic failed at day 16, and hydrolysis process should almost finish during the first 10 days according to the data from the previous experiments (fig.20). There were no reasons indicating that value of avg % VS reduction in U5-2 should be lower than 1%. The avg % VS reduction of U5-1 and U5-3 were between 16% and 27%. The values of avg% VS reduction were much lower than U2, U3, and U4. The low rate of avg% VS reduction in U5 indicated that the hydrolysis process did not work well and efficiently in the LBRs. The results demonstrated that three columns did not have successful HSCM hydrolysis in the LBRs.

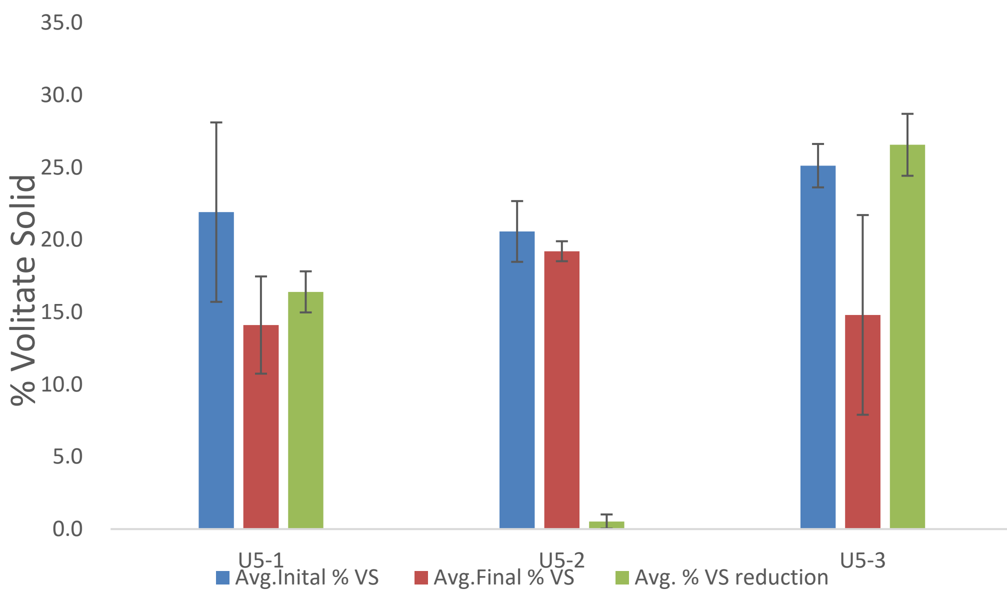

Fig. 36 Total Average Initial, Final Volatile Solid, and Average Percentage Reduction of VS in Upward Flow phase 5 (U5; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)). Error bars indicate +/- one standard deviation.

The average percentage VS in the top, middle, and bottom layers in U5-1, U5-2 and U5-3 are shown in fig. 37, 38, and 39. All three columns formed obvious layers when pouring the manure out of the column body after experiment terminated. According to the distribution of %VS profile (fig.40(Top, Middle, Bottom)), there are not clear tendency between % VS and liquid flow. In general, U5 experiments showed low VS reduction and it is not surprising that trends for successful column operations observed in U2 and U4 were not observed in U5 experiments

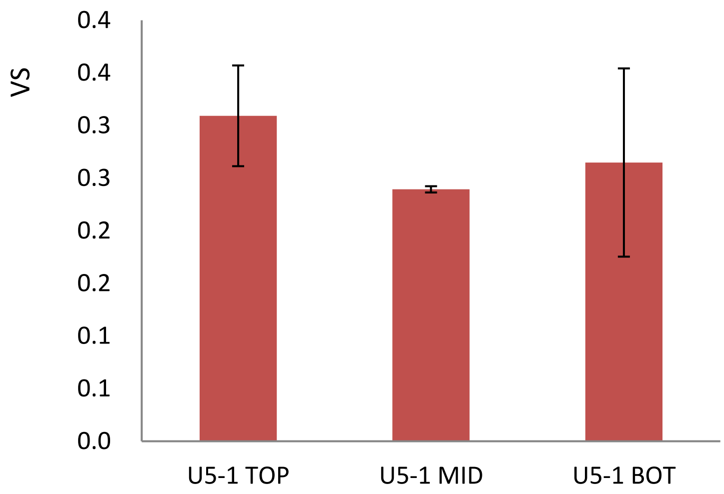

Fig. 37 Average percentage VS in top, mid, and bot layers in Upward Flow phase 5 Column 1 (U5-1; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)). Error bars indicate +/- one standard deviation.

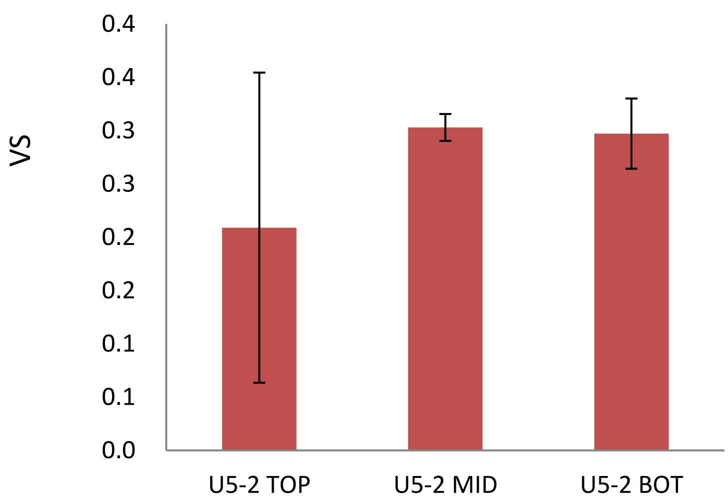

Fig. 38 Average percentage VS in top, mid, and bot layers in Upward Flow phase 5 Column 2 (U5-2; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)). Error bars indicate +/- one standard deviation.

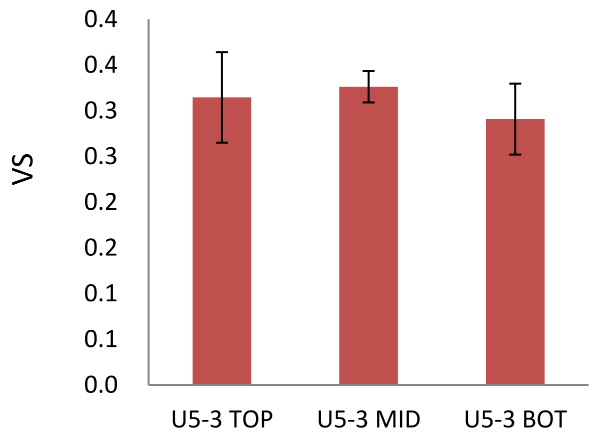

Fig. 39 Average percentage VS in top, mid, and bot layers in Upward Flow phase 5 Column 3 (U5-3; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)). Error bars indicate +/- one standard deviation.

U5-1Top 30.9%
(St.dev:4.79)
U5-1Middle 23.9%
(St.dev:0.03)
U5-1Bottom 26.5%
(St.dev:8.9)

U5-2Top 20.9%
(St.dev:14.5)
U5-2Middle 30.3%
(St.dev:1.26)
U5-2Bottom 29.7%
(St.dev:3.29)

U5-3Top 31.5%
(St.dev:4.95)
U5-3Middle 32.6%
(St.dev:1.71)
U5-3 Bottom 29.1%
(St.dev:3.88)

Blue Arrow indicates Flow Direction

Fig. 40(Left) Percentage distribution of Total VS in the top, mid, and bot layer in Upward Flow phase 5 Column 1 (U5-1; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)).
Fig. 40(Middle) Percentage distribution of Total VS in the top, mid, and bot layer in Upward Flow phase 5 Column 2 (U5-2; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)).
Fig. 40(Right) Percentage distribution of Total VS in the top, mid, and bot layer in Upward Flow phase 5 Column 3(U5-3; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1)).

The leachate COD concentration between U5-1, U5-2, and U5-3 is shown in fig. 41. All three columns started at the high level of COD concentration at initial, and in addition, U5-2 had much higher initial COD concentration than others. The U5-1 and U5-3 decreased shapely after day 2 and approached to LST's COD level at day 6. However, the COD concentration in U5-2 approached to the LST's COD concentration at day 10. After day 10, all three columns' COD concentration was similar as the LST's COD concentration.. Although U5-1 and U5-2 hydraulic failed in day 16 and day 20, COD concentration of those two columns already approached to the baseline of LST COD concentration before their failure day in day 10. This mean that most leaching process of those two columns already completed, and experiments were also completed. The trend of COD concertation in U5 was similar with U2 and U4. This indicated that the hydrolysis process in this phase was almost finished during the first 10 days of operation, and this experiment had successful hydrolysis in the all three LBRs

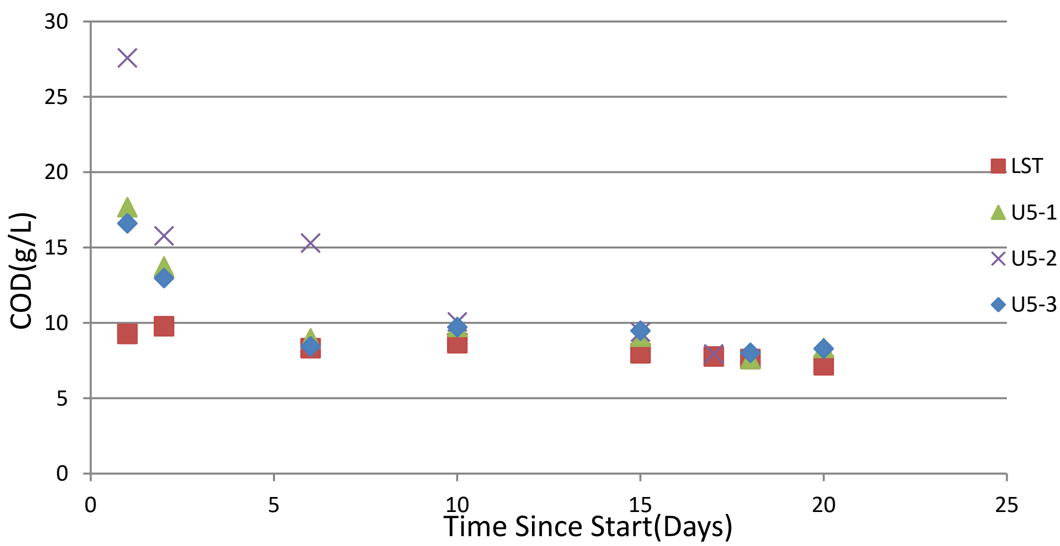

Fig. 41 Leachate COD Concentration in Upward Flow phase 5 (U5; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1))

The column experiment flow rate in U5 is shown in fig. 42. The variation of the flow rate in U5 was approximately at 10 ml/min during the first 10 days. After day 10, the flow rate of three columns approached 15 ml/min. There was no flow rate data after day 16 for U5-2 due to hydraulic failure. One of the biggest fluctuations of flow rates happened between day 13 and day 15 in U5-2. In addition, another large fluctuation of leachate flow rate was happened between day 17 and 19 in U5-1 and U5-3.

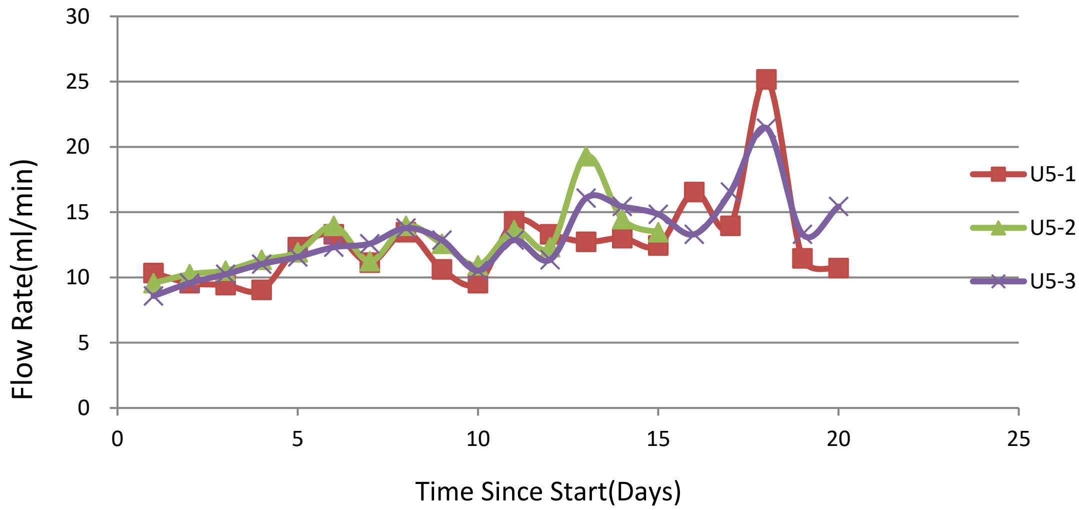

Fig. 42 Column Experiment Flow in Upward Flow phase 5 (U5; S Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1))

The column experiment pressure head difference in U5 is shown in fig. 43. All three columns pressure heads stayed between 1 and 3 inches during the first 8 days. The pressure head of U5-2 started to rise at day 9 and went above 85 inches at day 16. That indicated U5-2 hydraulic failed. After day 15, the pressure head of U5-1 rose immediately and reached 84 inches at day 19 which indicated that the column hydraulic failed and high pressure built up inside the column. The pressure head of U5-3 stayed relatively constant during the first 16 days and approached to 30 inches at the end of the experiment. This indicated that in U5-3 no pressure built up inside the columns until day 16.

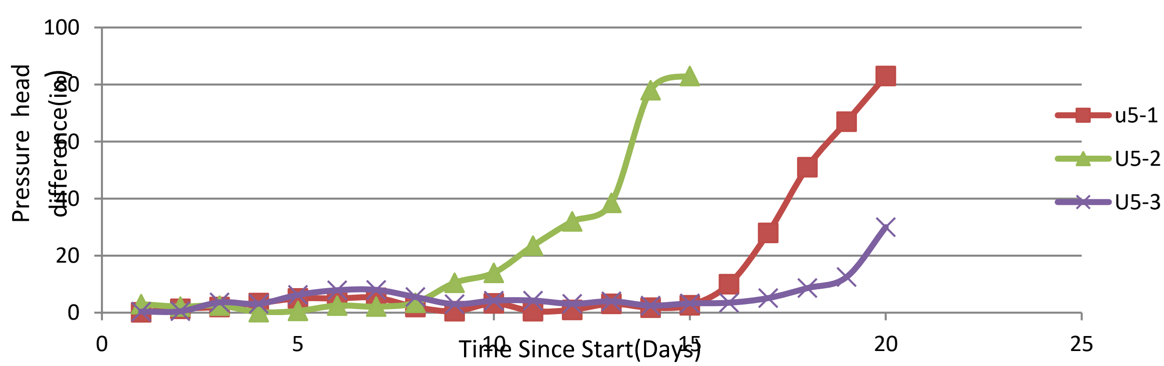

Fig. 43 Column Experiment Pressure Head Difference in Upward Flow phase 5 (U5; Sand & improved filter, No compression, No sieve, (See experiment definitions in Table 1))

While flow was sustained for a reasonable period in U5 experiments (minimum of 16 days), VS reduction was not achieved. The average percentage reduction of VS in this experiment was only around 20% (especially 0.5% VS reduction in U5-2) which indicated a inefficiency hydrolysis process in the LBRs. This was possible caused by the pre-digest manure left outside environment for a long time which resulted into extremely difficult degradable organic compounds remaining in the manure. The percentage distribution of total VS reduction profile in all three columns had the contradictory tendency as U2 and U4. The increasing high-pressure head in all three columns indicated that pressure built up inside the LBRs with progress time resulting in clogging issues. The only data, leachate COD concentration trend which was comparable to U2 experiment, indicated completed hydrolysis in three columns. However, based on observations of VS, organic matter remained in columns that could be solubilized. In addition, according to the observation, the manure did not mix with the top sand layer and turn into slurry texture until the column experiment ended, but U5-2 and U5-3 still hydraulic failed in day 16 and 20. That mean that the manure turned into slurry texture instead of solid in the LBRs may not be the primary reason caused the column failed.

In conclusion, the COD indicated that the leaching of COD in this experiment was completed. But, the hydraulic failure resulted into a low % VS reduction which illustrated U5 had an inefficiency hydrolysis process in the LBRs. The biological process was not successful may cause by some unknown reasons which should be investigated for the future studies.

4.6. Summary

Downward flow LBR experiments were all failed hydraulically in average 4 to 6 operated day. The leachate could not easily permeate through the manure bed and resulted in leachate accumulated on the surface of the manure bed. Finally, the LBR reactors failed. Upward flow

LBR configuration instead of downward flow LBR configuration prolonged the MSAD system operation time by ten days approximately. The results of U2 and U4 indicated that the operation days already passed three weeks except U4-2 failed at day 8. The COD data in U2, U4, and U5 demonstrated that the hydrolysis process almost finished in the first ten operation days. That possibly means that perhaps 10 to 12 days of operation is sufficient. The summary of VS reduction in U2, U3, U4, and U5 is shown in fig.44. The average initial % VS for all upward flow experiments was approximately 20%, and the average final % VS was around 10% except for with U5, which remained 16% of VS in the manure samples. That indicated that in the U5 experiment, while flow was sustained in columns, there was inefficient hydrolysis in the LBRs. The % VS reductions for all experiment phases were more than 30%, with U2 approaching 50% reduction of VS (expect for U5, which had 14% of VS reduction).

In U2, U4-1, and U4-3, the profile of % VS in top, mid, and bottom layers indicated that the trend of increasing % VS with height of the LBRs. The solubilization of HSCM did not evenly happen between bottom, middle, and top layers. The future experiment should investigate the best method to improve the hydrolysis through the whole column more uniformly.

The data indicated that the upward flow setup of the LBRs is a more functional configuration to sustain leachate flow and enhance of solubilizing of organic matter into the liquid phase for high rate of methane generation. The steady pressure head data except U4-2 showed no pressure built up inside the LBRs in U2, U4 and U5 within 16 days of operation. Observation of COD concentrations in leachate indicated that leaching of COD in experiments was complete after 10-12 days of operation so that there is not a need to sustain flow through columns beyond that time. Although some individual column experiments hydraulic failed before three weeks operation time, the hydrolysis in columns could be possible completed if the

COD concentration of those failed columns already approached to the COD concentration of LST. Therefore, the upward flow LBR setup notably improved the operation time and leachate flow through the reactor. Improved liquid flow in the columns was observed. The idea about upward flow LBR configuration will also be applied to the design of multiple leachate modules for future study to develop an implementation strategy for the MSAD system.

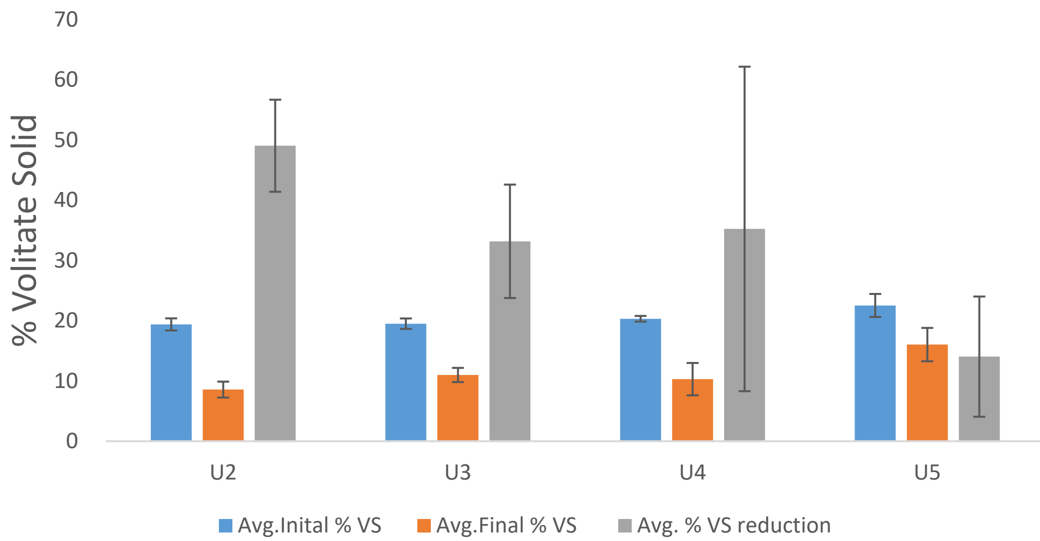

Fig. 44 Total Average Initial, Final Volatile Solid, and Average Percentage Reduction of VS in U2, U3, U4, and U5 (See Table 1 for Experiment Descriptions; in U2 all columns operated for 21 days, in U3 one column operated for 21 days, and other two ended at 8th and 14th day; in U4 two columns operated for 21 days, and only one column ended in 14th day; in U5 one column operated for 21 days, and other two failed at 16th and 20th day). Error bars indicate +/- one standard deviation.

CHAPTER 5: SUMMARY AND RECOMMENDATION FOR FUTURE STUDIES

5.1 Summary

The proposed MSAD technology has the potential to enable implementation of AD systems capable of handling HSCM with up to 90% TS. It is plausible that this technology can be implemented in Colorado where the climate is arid and water limitation existed. The design of the LBR plays a vital role in the MSAD system's ability to handle HSCM. The successful design of LBR required the critical parameter of the hydraulic conductivity in the manure beds (Custer et al., 1990). In this study, an iterative series of column experiments including downward and upward flow experiments were conducted. These experiments were motivated by the need to develop options to prevent clogging issues, preferential flow pathways, mass-transfer limitations in the LBR, and finally sustain leachate flow through the LBR.

Achieving good leachate flow through the LBR manure beds with upward flow LBR configuration is a significant contribution of this study. The upward flow LBR configuration with the combination of a layer of sand and improved top cap filter can generally prevent clogging issues and obtain good leachate flow through the LBR. Although some individual columns' experiments were hydraulic failure before three weeks of operation time such as upward flow experiment 4 and 5, leaching of COD of those experiments was already complete because effluence COD concentration approached to the baseline LST COD concentration before hydraulic failure. That mean the experiments could be considered complete, and perhaps 10 to 12 days of operation is enough to remove COD in LBR reactors. However, clogging issues occurred inside some LBRs causing the experiments to fail prematurely. This indicates that there are unknown reasons that may induce the clogging issues inside the LBR and reduce the hydraulic conductivity of the waste bed. Various physical, chemical, and biological mechanisms of failure should be explored in future studies. Future studies are required to understand the failures of the LBR operation and continue to invesigate the best method to sustain the leachate flow through the waste beds.

The data of % VS redcution in top, middle, and bottom layers in U2, U4-1, and U4-3 indicates that the trend of increasing %VS with height of column. The hydrolysis does not evenly happen through the whole LBR reactor. Hydrolysis should be more completed reactions througout the entire length of the reactor. Future experiments should be investigated to find out the reason and figure out a optimize option to improve hydroloysis become more even throughout LBR reactors.

5.2 Recomndations for Future Research

One option for improved liquid flow through the columns is addition of gypsum, which is a soft sulfate mineral composed of calcium sulfate dihydrate($CaSO4.2H2O$). $CaSO4 \cdot 2H2O$ is a sparingly soluble electrolyte which is present in seawater and industrial water systems (Shukla et al., 2008). When it dissolves into water, it can form divalent cation, Ca^{2+}. Such cations may attract negatively charged organic compounds. This interaction may result in the flocculation of organic molecules (Higgins and Novak, 1997). The aggregation of organic molecules caused by the addition divalent cation may facilitate the prolongment of adequate levels of hydraulic conductivity.

Another option is to investigate new bulking agents, which can degrade and contribute COD to the leachate during the AD process. The previous experiment conducted by Sandefur (2017) used woodchips as bulking agent in the LBR. However, the woodchips could not degrade in the three weeks operation time and thus they did not contribute to COD to the leachate. It may be worthwhile to investigate bulking agents that can enhance the porosity of the waste bed while contributing COD to the leachate.

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