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High rate anaerobic digestion of primary and secondary sludge using the static granular bed reactor (SGBR)

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**High rate anaerobic digestion of primary and secondary sludge using the static
granular bed reactor (SGBR)**

by

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A thesis submitted to the graduate faculty

in partial fulfillment of the requirements for the degree of

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ABSTRACT

The aim of this research was to demonstrate the feasibility of the static granular bed reactor (SGBR) as a replacement for the conventional mixed and heated anaerobic digester typically operating at municipal wastewater treatment plants (WWTP). The static granular bed reactor (SGBR) is a high rate anaerobic granular reactor. The SGBR operates at a short hydraulic retention time (HRT) (i.e 1 to 2 d) while maintaining a long solids retention time (SRT). Conversely to conventional digesters the SGBR separates the SRT from the HRT, reducing energy and volume requirements. The SGBR is downflow reactor with a dense bed of granules that entraps particles which helps to facilitate hydrolysis. Anaerobic treatment of primary and secondary municipal sludge was studied with a laboratory and pilot-scale SGBR at organic loading rates (OLR) from 2.8 ± 0.9 to 5.5 ± 1.7 g COD/L·d and 3.3 ± 2.0 g COD/L·d, respectively. The results of this research demonstrated the SGBR system's potential to be a practical and competitive alternative to conventional anaerobic digestion of wastewater sludges. The laboratory ($76 \pm 4\%$) and pilot-scale ($71 \pm 0.4\%$) SGBR produced a biogas with a higher methane content than the conventional anaerobic digester tested ($59 \pm 2.8\%$). Effluent VFA concentrations remained below 40 mg/L as HAc, indicating the potential to reduce process instability due to the buildup of intermediates. The low VFA concentrations of the SGBR's effluent also reduces odors compared to the liquid streams of conventional anaerobic digesters. The SGBR produced an effluent with low organic matter; average TSS and COD removal efficiencies remained above 90%.

CHAPTER 1. INTRODUCTION

The production of residuals from primary and secondary treatment has been steadily increasing due to the increasing number of municipal wastewater treatment plants (WWTP) and more strict discharge standards (Wang et al., 2008). Stabilizing the residuals from primary and secondary treatment with anaerobic digestion has been proven to be one of the most efficient stabilization technologies (Wang et al., 2008; Riau et al., 2010). It is standard practice to use large well mixed and heated anaerobic reactors to help compensate for the slow growth of methanogenic organisms. To make anaerobic digestion more efficient and economical research is focused on accelerating the digestion process and increasing the production of methane.

Current research trends in the digestion of municipal sludge commonly focus on pretreatment of the feedstock. Frequently researched pretreatment technologies include mechanical (ultrasound, high pressure and lysis), thermal hydrolysis, chemical oxidation (ozonation), biological (thermal phased anaerobic) and alkali treatments (Appels et al., 2008; Carrere et al., 2010). Pretreatments can be used to help the digestibility of the substrate, but they also increase capital and operational costs and have the potential to produce inhibitory compounds (Sakai et al., 2007; Lv et al., 2010).

There has been a lack of research done on modifying the anaerobic microbial communities to enhance the anaerobic digestion of wastewater sludges. The research presented here will propose a different strategy to conventional anaerobic digestion and shows the potential to reduce detention times and decrease the concentration of organic compounds in the effluent (including odor causing compounds). This study takes advantage of the natural granulation of

anaerobic microbes to digest municipal wastewater residuals. Anaerobic granules consist of a dense aggregation of microbes from different trophic groups (Uyanik et al., 2002; Liu and Tay, 2004; Baloch et al., 2008). These aggregates of microbes have desirable settling characteristics, allowing them to be maintained inside of the reactor. The volume of the anaerobic reactor can be minimized along with the hydraulic retention time (HRT) by retaining a larger population of degrading microorganisms inside of the reactor. A shorter HRT will reduce operation and capital costs, making anaerobic digestion a more attractive form of treatment (Ripley et al., 1986).

There are currently several high rate anaerobic reactor configurations that utilize anaerobic granules. The upflow anaerobic sludge blanket (UASB) is the most commonly utilized anaerobic granular reactor. However, the treatment efficiency of the UASB reactor is affected by the influent solids concentration (Bal and Dhagat, 2001). High influent solids concentrations encourage solids to wash out of the reactor and the overall removal efficiency of the USAB reactor suffers. Other granular reactor configurations are faced with similar challenges when treating wastewaters high in particulate matter. This research evaluates the ability of another high rate anaerobic reactor configuration, the static granular bed reactor (SGBR), to treat the high solids waste of primary and secondary sludge.

The SGBR is a simple downflow high rate anaerobic digestion system developed at Iowa State University (Mach, 2000). The SGBR system distributes wastewater over a dense bed of active anaerobic granules. The downflow operation of the SGBR allows influent solids to be trapped within the active granular bed, promoting hydrolysis. Particulate matter can only be biologically hydrolyzed after becoming physically removed either by entrapment in the sludge bed or adsorption (Elmitwalli et al., 2001b). Solids trapped within the granule bed of the SGBR

are within close proximity to degrading biomass, enabling extracellular enzymes to carry out hydrolytic reactions.

Laboratory and pilot-scale SGBR systems have been successfully used to treat a variety of wastewaters including synthetic wastewater consisting of non-fat dry milk, industrial wastewater, pork slaughterhouse wastewater, landfill leachate, and dairy wastewater with excellent results (Evans and Ellis, 2004; Debik et al., 2005; Evans and Ellis, 2010; Park et al., 2012; Turkdogan et al., 2013; Park et al., 2015; Oh et al., 2015). The objective of this research was to evaluate the ability of the SGBR system to treat primary and secondary municipal sludge in a high rate system operating with a short (i.e. one day) HRT.

CHAPTER 2. LITERATURE REVIEW

The main objective of a municipal wastewater treatment plant (WWTP) is to produce an effluent that will maintain or even restore the chemical, physical and biological integrity of the receiving environment. Raw wastewater is subjected to several physical, chemical and biological processes to remove contaminants, making it suitable to discharge into the environment. The residuals (solids and organic material) collected from these different wastewater treatment processes also require treatment. The production of sludge from WWTP in the United States is estimated to be around 6.2 million dry tonnes per year (Kargbo, 2010; Bolzonella et al., 2012). Anaerobic digestion of residuals from WWTP is favorable due to the large diversity of easily degradable organic matter, nutrients and alkalinity from inorganics (Gerardi, 2003).

Anaerobic digestion is an attractive method of treating wastewater residuals due to its ability to reduce the volume of solids, destroy pathogens and produce biogas. The anaerobic conversion of organic matter to biogas reduces its volume, which results in reduced disposal costs. Up to 90% of the degradable organic matter in wastewater can be stabilized by anaerobic digestion, compared to only 50% by aerobic digestion (McCarty, 1964; Demirel et al., 2005; Hassan and Nelson, 2012). When compared to aerobic digestion, anaerobic digestion produces significantly less sludge and requires less energy input (Leitão et al., 2006). Aerobic treatment of municipal wastewater residuals requires substantial operation and maintenance costs (Sing and Viraraghavan, 1999). Anaerobic digestion does not require oxygen, depends on less nutrients than aerobic digestion and produces a renewable source of energy (i.e. methane).

Biogas from anaerobic digestion can be used as a renewable source of energy due to the high methane composition. The anaerobic digestion process produces a gas mixture composed predominantly of methane (65-70%) and carbon dioxide (30-35%) along with trace concentrations of nitrogen, hydrogen sulfide and water vapor (Appels et al., 2008). As the relative concentration of methane increases so does the biogas' energy potential. Anaerobic digestion is a cost effective biological treatment due to its low sludge production, low energy requirements and high energy recovery rate (Chen et al., 2008).

Microbial Process of Anaerobic Digestion

A diverse community of microorganisms is required to carry out the digestion of the complex organic matter found in wastewater residuals. No bacterium is able to produce all of the enzymes required to degrade the large variety of substrates that are found in wastewater sludges (Gerardi, 2003). Several microbial populations are necessary to complete the various reactions required to convert complex organic matter into biogas (i.e. mostly methane and carbon dioxide) and new bacterial cells (Gerardi, 2003). The anaerobic digestion of organic material follows the steps shown in Figure 2.1. The transformation of organic matter to methane can be divided into four distinct stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis.
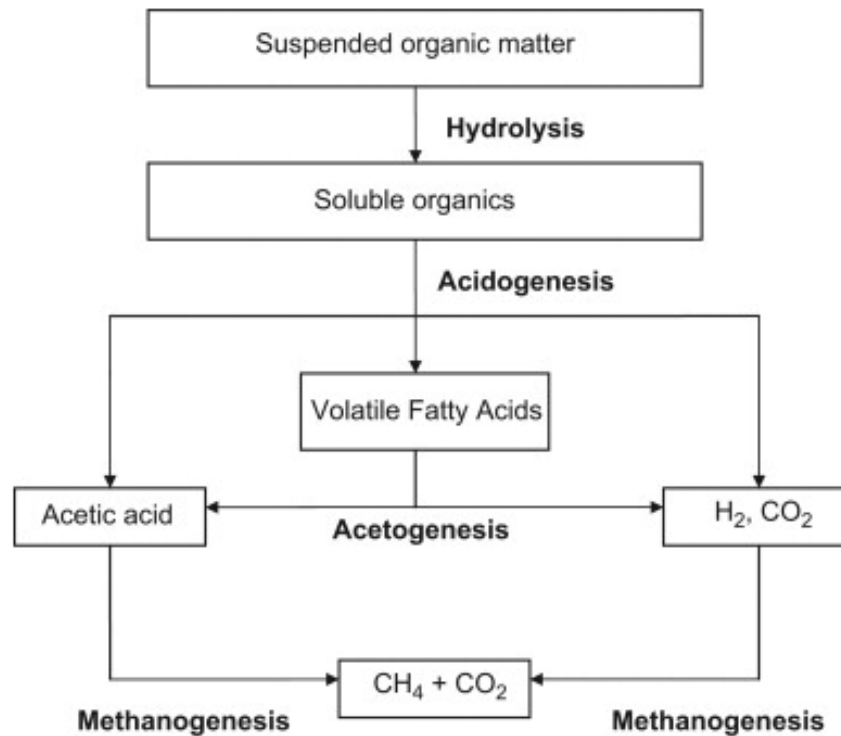
Figure 2.1: Anaerobic degradation pathways of complex organic matter (Appels et al., 2008)

During the first stage of anaerobic digestion, insoluble and/or complex organic matter is

converted to soluble molecules that can pass through bacterial cell walls. Microorganisms release

extracellular enzymes that hydrolyze insoluble organic materials and break down large insoluble

organic molecules into simpler soluble molecules (Verma, 2002). Hydrolyzing bacteria degrade

lipids, polysaccharides, proteins, and nucleic acids into soluble organic matter such as fatty acids

and amino acids (Mondala et al., 2013). These simpler compounds can now be passed along the

food chain and taken up by other bacterial populations. The products of hydrolysis are now the

correct size and form to pass through the cell walls of bacteria where they can be used as energy

or nutrient sources (Parkin and Owen, 1986).

During acidogenesis, the second step of anaerobic digestion, products created during

hydrolysis are further degraded into volatile fatty acids (VFA), ammonia, CO_2, H_2S, and other

products (Appels et al., 2008). During the third stage of anaerobic digestion, acetogenic bacteria convert the products of acidogenesis to simple organic acids (i.e. mostly acetic acid), carbon dioxide and hydrogen (Mondala et al., 2013). Methanogenesis occurs when methanogenic microorganisms convert acetic acid, hydrogen and carbon dioxide to methane.

There are two main pathways for methanogenesis to take place. The main pathway for methane production involves the cleavage of acetic acid (CH_3COOH). Splitting acetic acid into methane and carbon dioxide accounts for approximately seventy percent of the methane produced in an anaerobic digester (Gujer and Zehnder, 1983). Methane in an anaerobic digester is also produced from carbon dioxide and hydrogen gas. Hydrogen is used as an electron donor, while carbon dioxide is reduced and used as an electron acceptor. The two pathways of methane formation are demonstrated below. Equation 1.1 and equation 1.2 show the splitting of acetic acid into methane and the formation of methane from hydrogen and carbon dioxide, respectively.

$$CH_3COOH \rightarrow CH_4 + CO_2 \tag{1.1}$$

$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O \tag{1.2}$$

The initial hydrolysis step is the rate limiting step in the digestion of wastewaters containing substantial amounts of particulate and complex organic matter (Singh and Viraraghavan, 2004). The rate of methane and carbon dioxide production are proportional to decay of particulate material and accumulation of soluble compounds (Gujer and Zehnder, 1983). Methane and carbon dioxide are primarily produced from soluble compounds, therefore methane formation is proportional to the rate of hydrolysis.

Conventional Reactors

A major challenge for using anaerobic digestion to treat sludge is the slower growth rate of anaerobic organisms. Slow growth rates of microorganisms are typically compensated for by process modifications such as increasing the digestion temperature (typically 35°C) and lengthening the detention times (typically between 10 and 30 d) of microorganisms. A schematic of a conventional anaerobic digester is displayed in Figure 2.2.
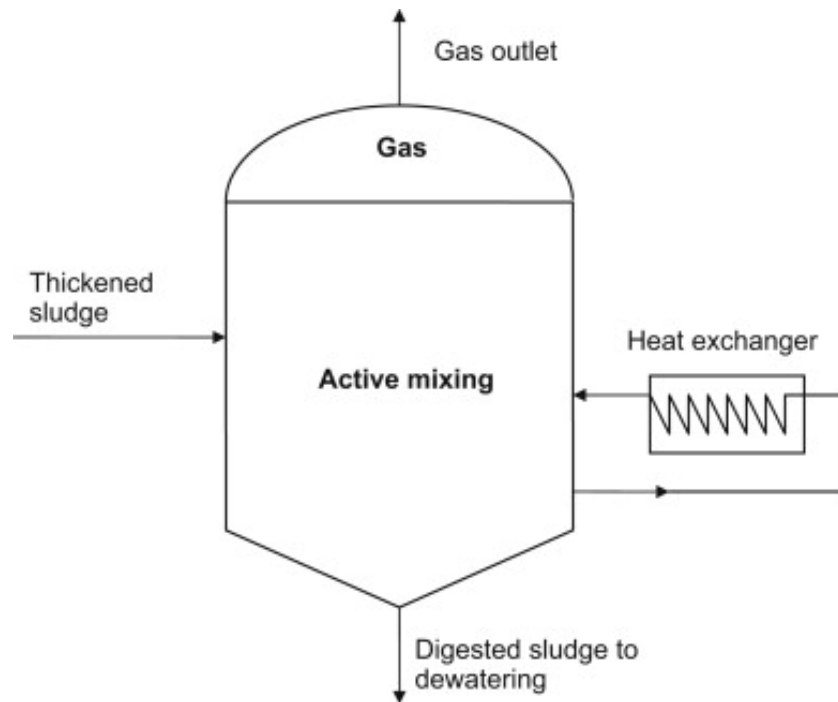
Figure 2.2: Conventional anaerobic digester (Appels et al., 2008)

In a typical municipal anaerobic digester the solids retention time (SRT) and hydraulic retention time (HRT) are both the same. The HRT is defined as the average time liquid spends in the digester while, the SRT refers to the average time microorganisms are retained in the reactor. The SRT must be long enough to allow adequate time for the critical organisms to grow and mature for complete digestion of complex organic matter (Parkin and Owen, 1986). At short detention times the methanogic bacteria will be withdrawn from the digester faster than they can

reproduce. Detention times less than 10 days will result in significant washout of methanogenic bacteria (Gerardi, 2003). In a conventional digester the SRT is controlled by the volume of the digester. To increase the time microorganisms have to digest organic material (SRT) the volume of a conventional digester must be increased resulting in larger capital and operation costs. There are alternatives to the conventional method of anaerobic digestion.

Anaerobic Granular Reactors

Anaerobic granules can be utilized to increase the SRT and reduce the reactor's volume and HRT. By conserving the degrading population of microorganisms (increasing the SRT) in the reactor, the HRT can be reduced. A shorter HRT will reduce reactor volume, operation and capital costs, making anaerobic digestion a more attractive form of treatment (Ripley et al., 1986). Anaerobic granules have desirable settling characteristics, enabling the biomass to be maintained inside of the reactor, thus making the HRT independent of the SRT (Karadag et al., 2015). Anaerobic digesters that utilize granules are the highest efficiency reactors to treat high strength organic wastewater and produce biogas (Soto et al., 2011; Baeta et al., 2012; Wu et al., 2016).

The high biomass concentration in anaerobic granules enables rapid organic matter degradation and requires less reactor volume (Liu et al., 2003). The granules are maintained inside of the system, controlling the growth rate and concentrations of microorganisms and increasing the reactor's SRT. Increasing the SRT reduces the rate at which slow growing microorganisms are washed out of the system (Ittisupornrat et al., 2015). As the SRT increases so does the treatment efficiency because the microbial population has more time to develop and

mature. The active biomass retained within the system is closely related to an anaerobic digester's treatment efficiency (Uyanik et al., 2002). By maintaining the bacterial populations in the system anaerobic granular reactors can treat high strength organic wastewaters without external separation or recirculation of the wastewater (Lim and Kim, 2014).

Increasing the SRT allows slow growing bacteria to become more enriched and increases the diversity of the biological community (Clara et al., 2005). Increasing the diversity of microorganisms also increases the physiological capabilities of the wastewater treatment technology. To ensure effective digestion of complex organics the microbial population must be of adequate quantity and concentration. A long SRT protects against a loss in digestion efficiency caused by fluctuations in temperature, potential inhibitory compounds, and slowly degradable compounds. As the SRT increases the food to microorganism (F/M) ratio is inversely affected. A larger SRT allows more micrograms to compete for the same amount of feed material. As the F/M ratio decreases so does the sludge production, resulting in lower sludge handling costs (Ittisupornrat et al., 2015). The use of dense granules in anaerobic digestion decreases the potential for microorganisms to be washed out, compared to other reactor types (Karadag et al., 2015).

Anaerobic Granule Characteristics

Anaerobic granules consist of a dense and diverse aggregation of microbes from different trophic groups (Uyanik et al., 2002; Liu and Tay, 2004; Baloch et al., 2008). The different groups of microorganisms form a complex food chain, depending on each other for essential nutrients (Diaz et al., 2006). Acetogens and methanogens need to be in close proximity of each

other for efficient hydrogen transfer between species (Lv et al., 2010). It is difficult to provide optimal conditions for the growth and interspecies interactions of microbes in a conventional digester (Lv et al., 2010). Granules can generate methane efficiently and at a high rate because there are different physiological types of microorganism located close to one another, increasing the rate of interspecies electron transfer (Diaz et al., 2006). Several researchers have suggested that the core of the granule consists of mainly methanogens which are sensitive to oxygen, while facultative bacteria dominate the granule's outer layers (Shen and Guiot, 1996; Baloch et al., 2008).

Anaerobic granules contain a well-developed pore structure, allowing fluid flow through the granule and the mass transfer of essential nutrients (Wu et al., 2016). The porous structure of a granule consists of a connected system with many branches similar to a system of arteries. An anaerobic granule typically has a wide main channel with many small sized sub-channels merging into bigger ones eventually connecting to the main channel (Wu et al., 2016). The size and length of a granules' channels depends on its diameter. Wu et al. (2016) concluded that granules with a large diameter (3-3.5 mm) have a larger pore size and a bigger pore volume than smaller granules (0.5-2 mm). A larger granule pore size may allow for increased substrate transport along with an increase in biogas production. After the granule reaches a certain size the well-defined pore structure deteriorates, resulting in vacant areas and channels that penetrate toward the granule's interior (Diaz et al., 2006). The internal geometry is likely to have any effect on the biogas production rates. The biogas production in a granule based reactor could be proportional to the size of the granule (Wu et al., 2016).

Diaz et al. (2006) observed granules of differing sizes and physical properties inside of a single bioreactor. The size and color of a granule may be an indication of its age. Small and

compact granules primarily consisted of younger microbial populations. Younger granules were observed to be black in color and mainly gram negative bacteria (Diaz et al., 2006). As granules aged, they became lighter in color (grey) and dominated by gram positive bacteria and *Archaea*. As granules became older they lost their compact spherical shape and became large and less dense (Diaz et al., 2006). After 4 weeks the granule's center became vacant as a result of biomass decay (Diaz et al., 2006). These older granules were brown in color and contained no gram positive bacteria. A lack of metabolic activity was observed at the interior of the more mature (brown and grey) granules, possibly due to the lack of nutrient transfer to the interior (Diaz et al., 2006). As granules aged microbial activity was observed mainly at the outer edges, possibly due to inefficient nutrient diffusion to the center.

The activity of various microbial populations is determined by measuring their byproducts. These byproducts can be gas production or the accumulation of soluble intermediates such as propionate and butyrate (Gujer, and Zehnder, 1983). Lim (2008) found high concentrations of acetate, propionate and iso-valerate near the top of the SGBR granule bed. The buildup of VFA indicated that most of the insoluble organic matter was hydrolyzed near the top of the digester. Granule reactors that are not mixed encourage different zones of microorganisms at different locations in the reactor. Baloch et al. (2008) found that acidogenesis was the dominant reaction close to the influent point, while methanogenesis dominated downstream of the acidogens.

Baloch et al. (2008) analyzed anaerobic granular sludge samples from different compartments of a granular bed baffled reactor (GRABBR). The morphology of a granule was found to be influenced by the dominant reaction taking place in each compartment of the GRABBR (Baloch et al., 2008). The morphology of anaerobic granules may be influenced by the

dominant species of microorganism along with the growth and decay rates of microorganism (Lim and Kim, 2014). The granules in the methanogenic zone remained densely packed, smooth and relatively stable. Conversely, disintegration and floatation was observed in granules maintained in the acidogenic zone (Baloch et al., 2008). Granules dominated by acidogenic reactions contained a less dense core, an irregular outer surface and reduced structural stability. The high concentration of relatively fast growing acidogenic bacteria could produce fissures and broken surfaces, reducing the structural stability of the granule (Baloch et al., 2008). Granules with fissures and broken surface are less favorable to methanogenic microorganisms due to reduced stability and increased oxygen transfer (Baloch et al., 2008). The morphologies of granules from methanogenic and acidogenic zones are displayed in Figure 2.3.
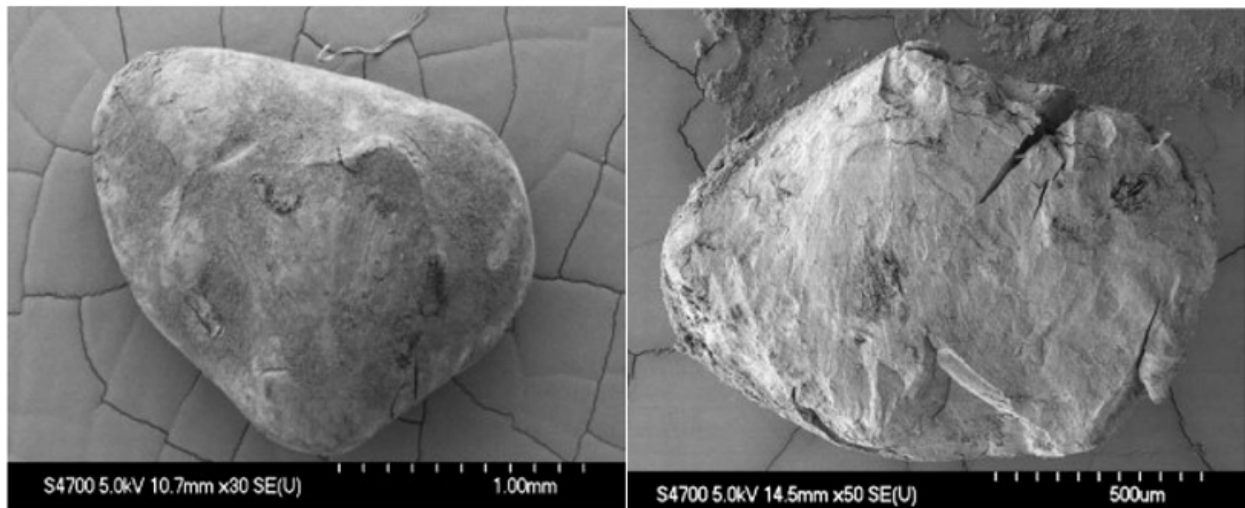
Figure 2.3: Granule from methanogenic dominant zone (left) and one dominated by acidogenic dominant zone (right) (Baloch et al., 2008)

The diversity of the methanogenic subpopulations increases with the complexity of the waste composition (Liu and Tay, 2004). A wider range of enzymes are required to break the various linkages of complex organic matter. Granules grown on complex substrates are larger and more diverse than those grown on simple substrates (Lim and Kim, 2014). The granule

communities have been observed changing and adapting to their food source. Kovacik et al. (2010) demonstrated that a change in feed source from ethanol, propionate and acetate to just acetate resulted in a decrease in microbial diversity. Without ethanol in the feed source, the population of microorganisms that normally converted ethanol and propionate to hydrogen and acetate decreased substantially. When ethanol and propionate were omitted from the influent, hydrogen and formate consuming methanogens gave way to acetate consuming methanogens due to a lack of hydrogen and formate production (Kovacik et al., 2010).

Upflow Anaerobic Sludge Blanket (UASB) System

Currently there are several anaerobic reactor configurations that take advantage of anaerobic granules. The upflow anaerobic sludge blanket (UASB) reactor is often utilized in industrial and municipal wastewater settings. The UASB reactor is one of the most common anaerobic digestion systems currently in use (Nelson et al., 2012). In the UASB reactor contains a blanket of granular sludge which is kept in suspension by the upward flow of wastewater into the system. The density of anaerobic granules in a UASB reactor needs to be large enough to resist the shear stress supplied by the hydraulic upflow of the influent and the biogas. Excessive hydraulic loadings in a UASB can lead to the washout of biomass with the effluent (Bal and Dhagat, 2001).

The UASB reactor requires a gas-liquid-solid separation device, which occupies between 16 and 25% of the reactor volume (Hashemian and James, 1990). This device must be properly operated and maintained to achieve the maximum treatment efficiency. Blockages in the gas separator compartment of the UASB can result in failure to separate solids from the effluent

(Hashemian and James, 1990). Improper alignment of effluent weirs can also result in hydraulic short circuiting and reduced treatment efficiencies in the UASB (Heffernan et al., 2011). The sludge bed height of a UASB reactor needs to be controlled to prevent it from extending above the entrance of the gas liquid separator, thus increasing the potential to discharge solids with the effluent. The UASB reactor also requires an operator to control the sludge bed height in the reactor by appropriately discharging granular sludge (Heffernan et al., 2011).

While treating raw municipal wastewater with the UASB reactor at organic loading rates (OLR) between 1.56 and 1.6 kg chemical oxygen demand (COD)/m^3·d, Singh and Viraraghavan (2004) observed COD removal efficiencies ± standard deviation (SD) of $84 \pm 2.1\%$, $87 \pm 2.5\%$, $81 \pm 3.2\%$, $79 \pm 1.0\%$ and $56 \pm 2.8\%$ at 20, 32, 15, 11 and 6°C, respectively. A significant reduction in COD removal efficiency was observed when the UASB reactor's temperature was reduced from 11 to 6°C (Singh and Viraraghavan, 2004). Singh and Viraraghavan (2004) determined that above 11°C the temperature of the UASB reactor did not significantly affect the total suspended solids (TSS) removal efficiency. The effluent TSS produced by the UASB from raw municipal wastewater ranged from 10 to 30 mg/L and had a volatile suspended solids (VSS)/TSS ratio of 0.8 ± 0.15. However, a decline in TSS removal was noticed once the upflow velocity of the UASB reactor reach a critical point (Singh and Viraraghavan, 2004).

VFA are formed as intermediate products during the anaerobic digestion process. The accumulation of VFA reflects a kinetic imbalance in the microbial populations involved in the anaerobic digestion process. The total VFA concentration as acetic acid was maintained below 35 mg/L for a UASB treating raw municipal wastewater at HRTs from 48 to 6 h and during stable operational periods at 32, 20, 15, and 11°C (Singh and Viraraghavan, 2004). The VFA/alkalinity ratio in the effluent of a UASB reactor treating raw municipal wastewater ranged

from 0.032 to 0.14 at HRTs from 48 to 6 h and during stable operational periods at 32, 20, 15, and 11°C (Singh and Viraraghavan, 2004). While treating raw municipal wastewater with the UASB reactor the average gas composition at 20°C and an HRT of 48 h was 65 to 70% methane, 12 to 15% carbon dioxide and 15 to 20% nitrogen (Singh and Viraraghavan, 2004). A psychrophilic (20 ± 1°C) UASB reactor treating municipal wastewater at an OLR between 0.15 and 1.2 kg COD/m^3·d had a COD removal efficiencies in the range of 80 to 84% (Singh and Viraraghavan et al., 1998). The UASB reactors treated low strength wastewater at temperatures between 6 and 20°C, with removal of COD, BOD, and SS ranging from 38 to 90%, 47 to 91%, and 50 to 92%, respectively (Singh and Viraraghavan, 1999). Singh and Viraraghavan (1999) also found that the average biogas production fluctuated from 0.167 to 0.199 L methane/g COD$_{removed}$ and the methane composition ranged from 65 to 86%.

The average treatment efficiencies of full scale UASB reactors treating municipal wastewater preceding grit removal in semi-tropical regions is displayed in Table 2.1. The average design flowrates for the WWTP varied from 30,000 to 164,000 m^3/d.

Table 2.1: The average treatment performance of UASB reactors located in semi-tropical regions (Heffernan et al., 2011)

UASB Location	Source	Effluent		Removal efficiency %	
		COD (mg/L)	TSS (mg/L)	COD	TSS
India	Sato et al. (2006)	364	357	43	18
India	Heffernan et al. (2011)	285	107	46	49
Brazil	Oliveira & von Sperling (2009)	251	85	65	71
Brazil	Heffernan et al. (2011)	247	112	62	54
Middle East Lit	Nada et al. (2006)	221	63	71	85
Middle East	Heffernan et al. (2011)	337	40	52	84

The Static Granular Bed Reactor (SGBR) System

The static granular bed reactor (SGBR) was developed by researchers at Iowa State University. The SGBR system utilizes anaerobic granules like the UASB reactor to treat wastewater. Unlike other granular reactors the SGBR does not require mixers, gas-liquid-solid separation devices, recirculation pumps, or heat exchangers. The SGBR has a simple downflow configuration, allowing influent to flow through a bed of active anaerobic granules (Ellis and Evans, 2008). The downward flow regime of the SGBR allows for the biogas to be easily separated from the granule bed and liquid at the top of the system. The SGBR uses active granules similar to the UASB reactor, but it operates in a downflow configuration instead of an upflow. Due to the downflow configuration, the SGBR acts like a bioreactor and a filter and is not susceptible to solids washout under high hydraulic loading rates like the UASB.

The SGBR has been used successfully to treat a variety of wastewaters including synthetic wastewater consisting of non-fat dry milk, industrial wastewater, pork slaughterhouse wastewater, landfill leachate, and dairy wastewater (Evans and Ellis, 2004; Debik et al., 2005; Evans and Ellis, 2010; Park et al., 2012; Turkdogan et al., 2013; Park et al., 2015; Oh et al., 2015). The results obtained from Evans and Ellis (2004) using the SGBR system to treat municipal wastewater are displayed in Table 2.2. At steady state operation the SGBR system had COD removal efficiencies between 74 and 84%. Conversely to the UASB reactor, the SGBR's ability to remove TSS increased when the HRT of the system was decreased. As the hydraulic flow into the system increases, the granule bed of the SGBR may become more compact (Evans and Ellis, 2004). This bed compaction and decrease in the bed porosity of the SGBR system improves solid entrapment and retention (Evans and Ellis, 2004).

Table 2.2: Performance of the SGBR system treating municipal wastewater based on TSS and CBOD$_5$

HRT	Municipal Wastewater		SGBR Effluent				
	TSS (mg/L)	CBOD$_5$ (mg/L)	TSS (mg/L)	CBOD$_5$ (mg/L)	pH	Alkalinity (mg CaCO$_3$/L)	Methane (%)
48	106 ± 58	29 ± 6.6	29 ± 12	17 ± 6.5	7.08 ± 0.23	286 ± 18	64 ± 11
36	274 ± 72	170 ± 96	11 ± 2.9	24 ± 6.4	6.75 ± 0.24	465 ± 77	61 ± 24
24	301 ± 99	135 ± 68	12 ± 2.4	26 ± 9.0	6.80 ± 0.19	338 ± 74	64 ± 5.1
18	163 ± 56	84 ± 40	8.2 ± 3.5	31 ± 5.8	6.91 ± 0.20	ND	76 ± 7.6
12	236 ± 109	167 ± 106	7.8 ± 4.1	57 ± 9.4	7.07 ± 0.33	353 ± 32	39 ± 9.8
8	187 ± 100	107 ± 39	5.6 ± 3.3	30 ± 12	7.23 ± 0.17	323 ± 74	23 ± 2.0

Primary and Secondary Municipal Sludge Characteristics

Residuals from primary wastewater treatment are a combination of floating fats, oils and grease along with solids collected at the bottom of the primary clarifier. The residuals from secondary wastewater treatment are composed primarily of microbial cells (proteins and polysaccharides) and suspended solids produced during aerobic biological treatment (Mondala et al., 2009). The mixture of primary and secondary sludge is composed of 60 to 80% carbohydrates, fats and proteins (Gerardi, 2003). Approximately 80% (30% primary sludge and 50% secondary sludge) of the organic waste input into a WWTP ends up in the anaerobic digesters (Gerardi, 2003). While, Foresti et al. (2006) estimates that 40 to 60% of the total organic matter in raw sewage is collected from primary and secondary clarifiers and sent to the anaerobic digesters.

Secondary sludge contains more non-biodegradable solids and is more resistant to degradation than primary sludge (Grady et al., 2011). The biodegradation of secondary sludge is limited by the hydrolysis of compounds produced from death and lysis of activated sludge

bacterial cells (Parkin and Owen, 1986). The biodegradation of primary sludge and secondary sludge are displayed in Figure 2.4. Primary sludge has a higher potential for COD reduction compared to that of secondary sludge. The digestion curve stabilizes at SRT larger than 10 days as all of the sludge compounds are significantly reduced (Appels et al., 2008). A short SRT will result in the washout of methanogenic bacteria and the accumulation of VFA. At a long enough SRT when the substrate is almost completely degraded the system's performance is controlled by the death and lysis of biomass (Grady et al., 2011).
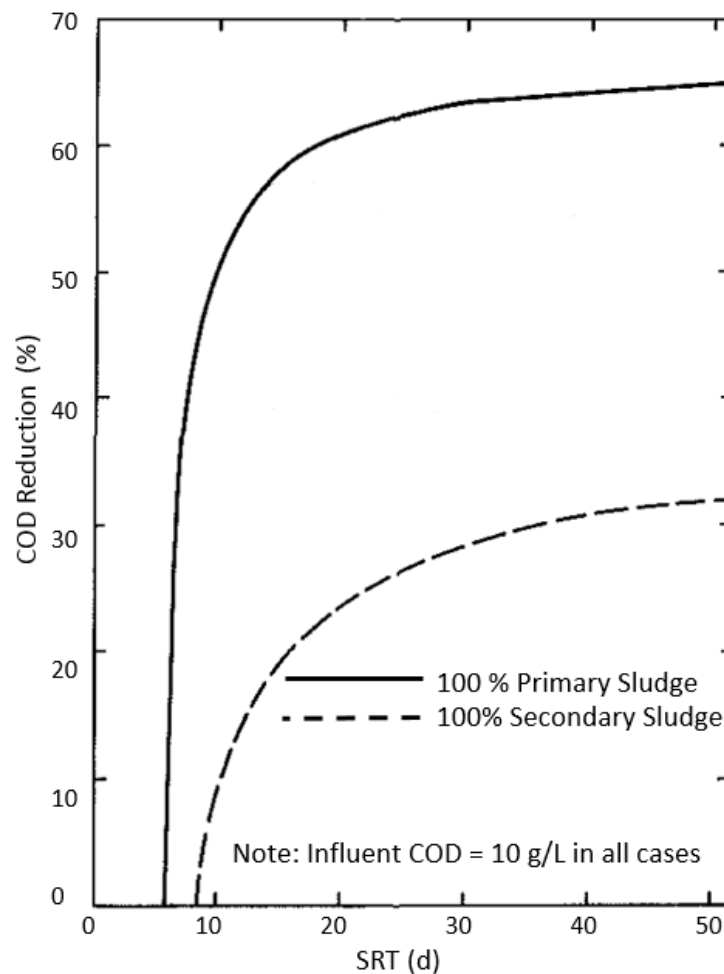
Figure 2.4: Secondary sludge and primary sludge digestion in a conventional anaerobic digester (Parkin and Owen, 1986)

CHAPTER 3. MATERIALS AND METHODS

Influent Characteristics

A laboratory-scale SGBR was used to treat primary and secondary municipal sludge. Sludge samples were obtained from the Ames Water Pollution Control Facility (WPCF) located in Iowa. The Ames WPCF is designed to treat dry-weather flow of 8.6 million gallons per day. Characteristics of influent and effluent of the Ames WPCF are demonstrated in Table 3.1.

Table 3.1: Ames WPCF treatment efficiencies (datum is from calendar years 2010 and 2011)

Parameter	Influent	Effluent
Oxygen Demand (mg/L)	162	4
Suspended Solids (mg/L)	220	7
Ammonia (mg/L)	23	0.2
Dissolved Oxygen (mg/L)	--	9.6

At the Ames WPCF, solids from the secondary clarifiers are returned to the primary clarifiers. Solids from the primary clarifiers are then sent to the anaerobic digesters. The characteristics of the two primary anaerobic digesters operating at the Ames WPCF are demonstrated in Table 3.2.

Table 3.2: Ames WPCF digester characteristics (data is from calendar years 2014 and 2015)

	Temp	pH	Volatile Acids	ALK	Digester		Supernatant	
	°C		(mg/L)	(mg/L)	% T.S.	% V.S.	% T.S.	% V.S.
				Digester #1				
Average	36.3	7.0	579	3621	2.8	65.6	3.2	61.6
Maximum	41.7	7.2	1180	5000	3.7	72.8	14.7	73.0
Minimum	30.6	6.8	234	2780	2.1	56.4	2.2	20.6
				Digester #2				
Average	36.5	7.0	437	3396	2.5	66.4	3.1	62.4
Maximum	40.0	7.6	3710	4520	3.1	72.6	11.8	77.0
Minimum	30.6	6.6	90	600	1.8	58.4	1.9	21.8

The sludge collected from the Ames WPCF was stored in a refrigerator at 4°C before feeding the system. The storage time was between 4 and 5 weeks. The characteristics of the sludge remained nearly constant throughout the study due to the low storage temperature (Han et al., 1997b). The particulate COD (PCOD) consistently made up between 91 and 98% of the total COD (TCOD) in the influent. The low influent soluble COD (SCOD) indicates little activity associated with hydrolysis, the first step of digestion. Primary and secondary sludge was diluted to facilitate hydraulic flow through the reactor. The influent was created by diluting the sludge with tap water at a 1:15 ratio. The characteristics of the influent are demonstrated in Table 3.3.

Table 3.3: Laboratory-scale SGBR influent characteristics

Parameter	Study Mean ± SD
TSS (mg/L)	2267 ± 593
VSS (mg/L)	1694 ± 391
TCOD (mg/L)	5693 ± 1483
SCOD	314 ± 137
VFA (mg/L as HAc)	245 ± 35
pH	6.02 ± 0.69
Alkalinity (mg/L as CaCO$_3$)	457 ± 110

Initially the HRT of the SGBR was set at 48 h. The HRT of the system started at 48 h and was gradually reduced to 24 h over the course of the study. The SGBR's feeding rate was varied by modifying the HRT of the system. The variation in OLR with HRT is displayed in Table 3.4. The HRT was reduced in a stepwise manner while maintaining a consistent substrate concentration in the influent. The HRT was gradually decreased to give the microbial population contained in the SGBR time to acclimate. The reduction of the HRT in a stepwise fashion results in greater reactor stability and performance (Barker and Stuckey, 1999; Saner et al., 2016). The

OLR varied throughout the study to demonstrate the feasibility and resiliency of the SGBR system.

Table 3.4: OLR conditions and corresponding HRT

Operation Time (d)	HRT (h)	OLR (g COD/L·d)	OLR (g TSS/L·day)
62	48	2.8 ± 0.9	1.0 ± 0.4
151	36	4.1 ± 0.7	1.5 ± 0.2
10	30	5.0 ± 1.0	1.5 ± 0.4
89	24	5.5 ± 1.7	1.7 ± 0.7

Laboratory-scale SGBR System Setup

The laboratory-scale SGBR system was operated for a period of approximately 10 months at room temperature (e.g., $22 \pm 2℃$). The laboratory-scale SGBR system consisted of a 2-liter reactor with 1-liter of working volume, a 4-liter tank for influent storage, a Masterflex peristaltic pump, magnetic stir plate, and a gas meter. The peristaltic pump was utilized to feed the influent into the top of the reactor. The hydraulic loading was not large enough to maintain a constant flow into the system. Intermittent feeding can cause surges of acid and hydrogen production and decrease the pH depending on the buffering capacity of the system (Parkin and Owen, 1986). Increasing the system's SRT and/or frequency of feedings can minimize the impacts of intermittent feeding. The laboratory-scale SGBR system was fed once per hour to minimize the impacts of intermittent feeding.

The SGBR reactor was fitted with influent and gas ports at the top, and effluent drain at the bottom and a backwash port on the side. A schematic representing the SGBR system is displayed in Figure 3.1. A stainless steel mesh (2 mm) was installed at the bottom of the reactor,

to prevent granules from being washed out from the reactor. Marbles (1.3 cm) along with gravel were placed over the steel mesh and below the granules, to support the granule bed. Finally approximately 1 L of granules were added to the reactor. The reactor was seeded with anaerobic granules from the City Brew Brewery in La Crosse, Wisconsin. The SGBR system was seeded with approximately 76.5 g of granular sludge with a TSS of 76,500 mg/L.
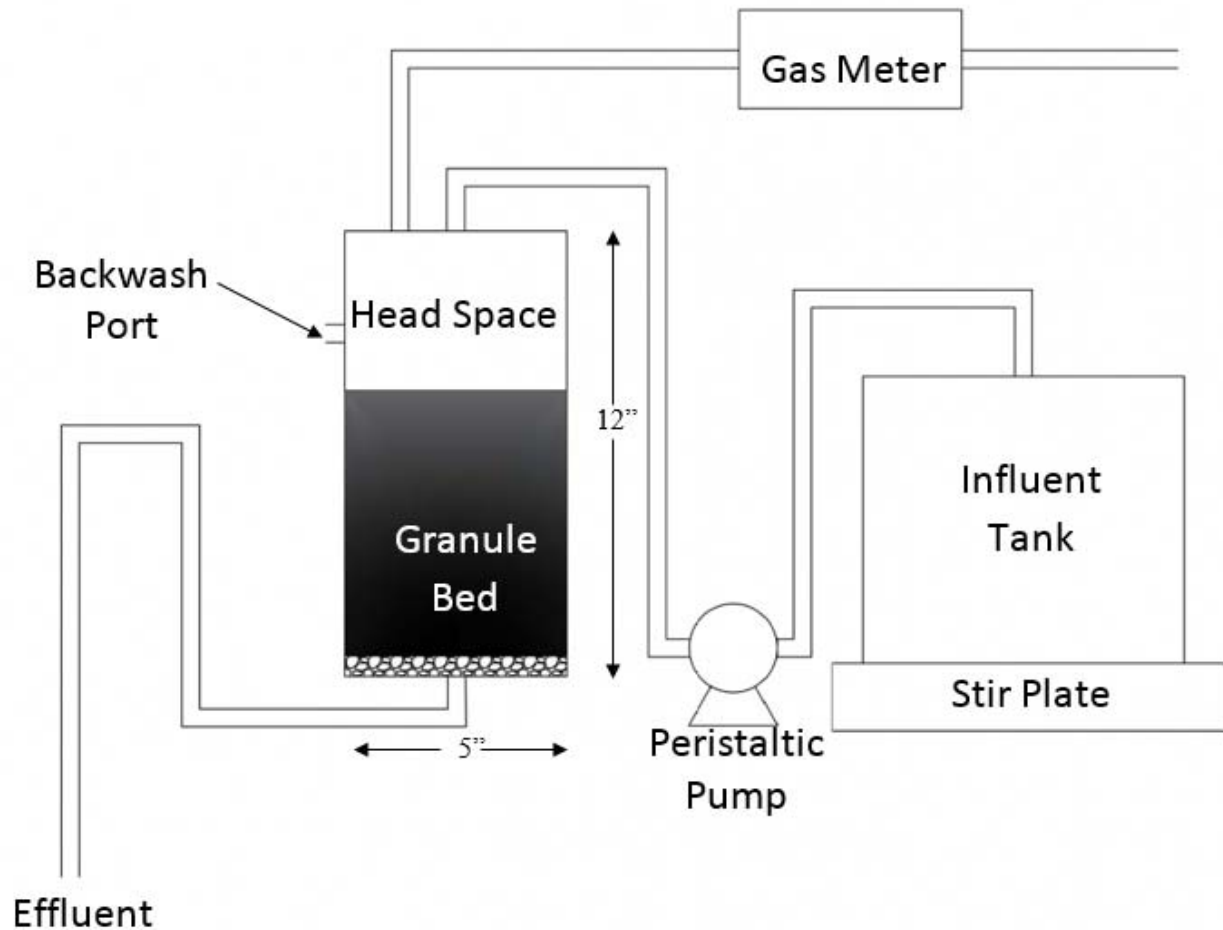
Figure 3.1: Laboratory-scale SGBR system schematic

During the startup of an anaerobic granule digester it is common to use a seed sludge to establish a microbial community in the reactor (Ahring, 2003 and Nelson et al., 2012). Pregranulated sludge is advantageous for reducing the startup phase of granule based reactors.

The seed sludge provides an active microbial community that will kick start the digestion process without the buildup of potentially inhibitory intermediate products such as VFA (Nelson et al., 2012). The buildup of VFA in an anaerobic digester could cause a reduction in pH and ultimately lead to process failure. At a low pH VFA are undissociated and can become toxic when they pass through the cellular membrane, dissociating and reducing the pH (Boe, 2006; Appels et al., 2008). It is important to regularly monitor the performance of an anaerobic digester to detect irregularities in system performance.

The effluent of the SGBR was sampled twice a week to analyze the performance and health of the SGBR system. The SGBR influent and effluent were tested for TSS, COD, alkalinity, pH and VFA according to The Standard Methods for the Examination of Water and Wastewater (APHA, 1998). The test method used for each parameter is displayed in Table 3.5.

Table 3.5: The test method for each parameter

Parameter	Description	Section
TSS	Total Suspended Solids Dried at 103-105°C	2540 D.
VSS	Fixed and Volatile Solids Ignited at 550°C	2540 E.
COD	Closed Reflux, Titrimetric Method	5220 C.
Alkalinity	Titration Method	2320 B.
pH	Electrometric Method	$4500\text{-}H^+$ B.
VFA	Distillation Method	5560 C.
SVI	Settling Characteristics	2710

The influent and effluent samples were filtered with glass fiber filter paper (Whatman GF/C, 1.2µm pore size) to analyze TSS and VSS. The SCOD fraction was determined by measuring the COD of a filtered sample. The influent and effluent pH was analyzed using a Fisher Scientific Accumet excel, model XL15 pH meter. The biogas production was estimated using a wet tip gas meter (Speece, Nashville, TN). The wet tip gas meter was calibrated to

measure a 75 mL volume of biogas per tip. The composition of the biogas was analyzed by a Gow Mac gas chromatograph series 580 (Bethlehem, PA). The gas chromatograph column used detected relative components of nitrogen, methane and carbon dioxide. The gas chromatograph system was calibrated with a gas standard composed of 70% methane, 25% carbon dioxide and 5% nitrogen.

CHAPTER 4. LABORATORY-SCALE RESULTS AND DISCUSSION

COD and Suspended Solids (SS) Removal Efficiencies

The SGBR's ability to remove TCOD and TSS from primary and secondary sludge is demonstrated in Figure 4.1 and Figure 4.2, respectively. The process efficiency of the SGBR system was evaluated under diverse OLRs ranging from 2.8 to 5.5 g COD/L·d. As the OLR increased the TSS and TCOD removal efficiencies remained above 90%. The average suspended solids (SS) and COD removal efficiencies are demonstrated in Table 4.1. The average effluent TCOD and SCOD were maintained between 247-353 mg/L and 122-175 mg/L, respectively.
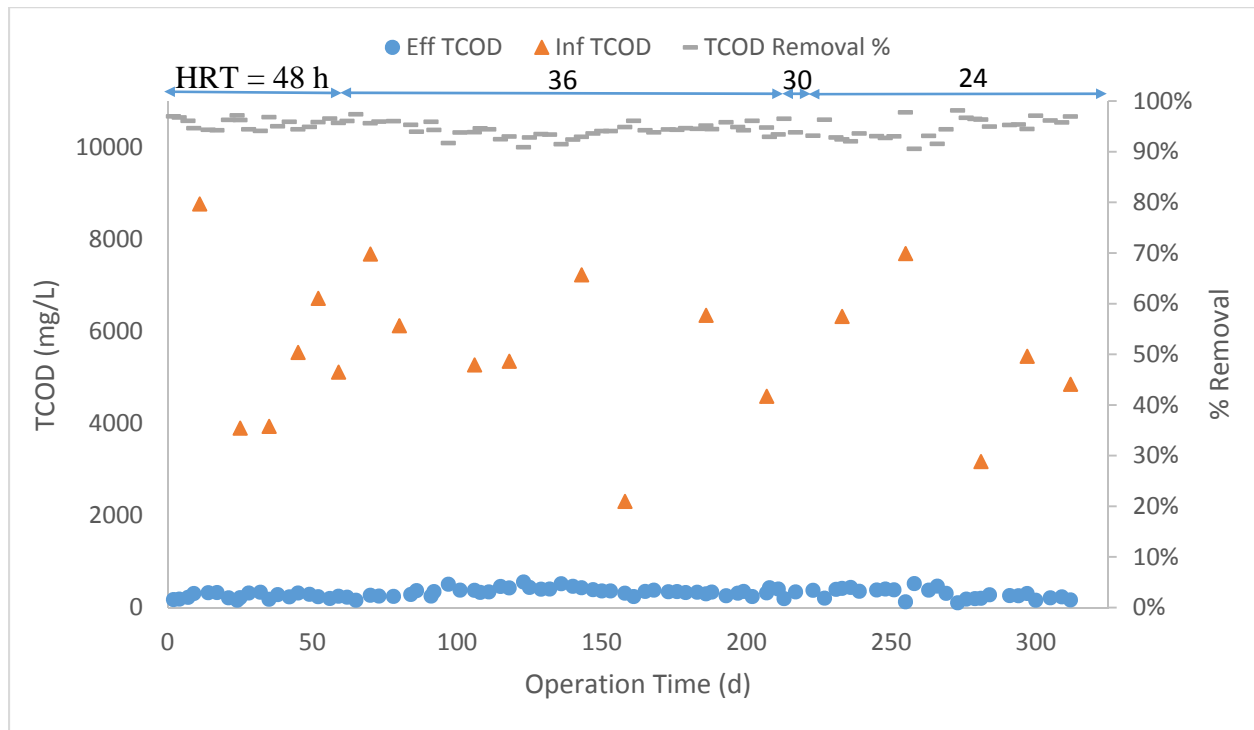
Figure 4.1: Laboratory-scale SGBR TCOD removal
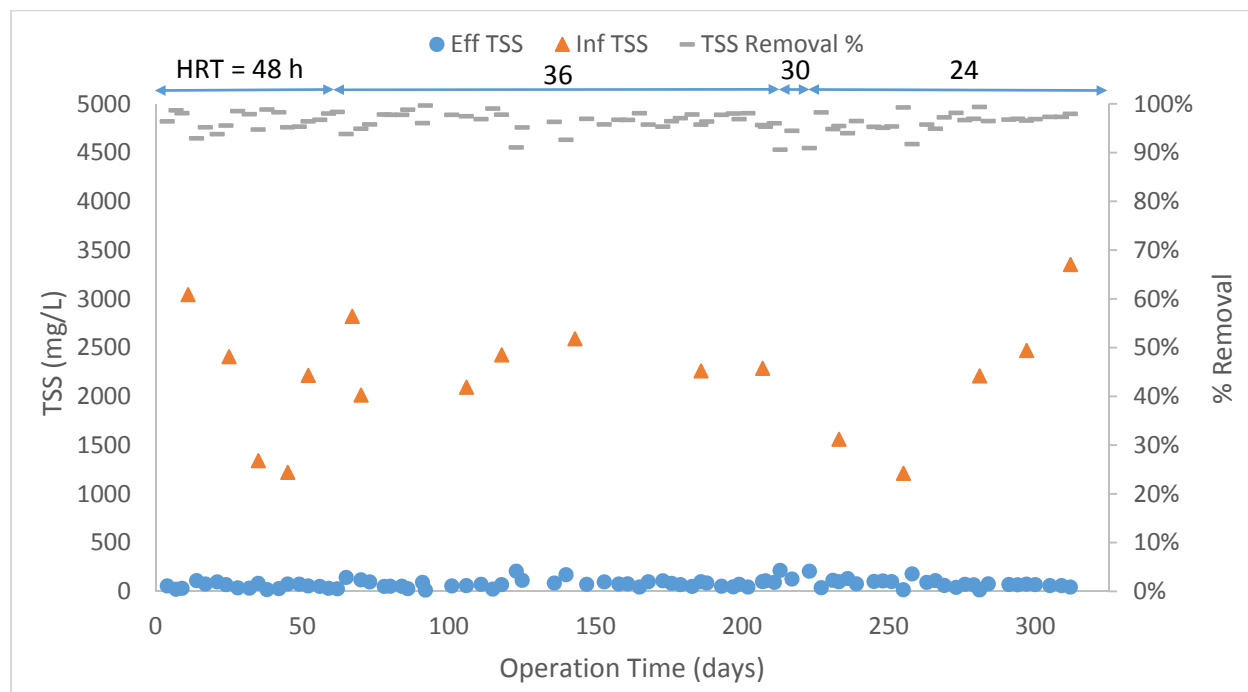
Figure 4.2: Laboratory-scale SGBR TSS removal

Table 4.1: SGBR effluent COD and SS characteristics

HRT (h)	48	36	30	24
OLR (g COD/L·d)	2.8 ± 0.9	4.1 ± 0.7	5.0 ± 1.0	5.5 ± 1.7
TSS (mg/L)	53.0 ± 27.1	79.9 ± 38.8	182 ± 48.7	77.0 ± 36.0
TSS Removal (%)	95.6 ± 1.0	96.6 ± 1.7	92.0 ± 2.1	96.4 ± 1.7
TSS Range (mg/L)	18 - 110	12 - 206	126 - 214	14 - 178
VSS (mg/L)	36.3 ± 19.2	74.5 ± 38.6	143 ± 38 [a]	60 ± 28 [a]
VSS Removal (%)	97.7 ± 1.2	96.0 ± 2.1	92.2 ± 2.1 [a]	96.3 ± 1.7 [a]
VSS Range (mg/L)	6 - 64	8 - 180	99 - 168 [a]	11 - 140 [a]
TCOD (mg/L)	247 ± 57.8	353 ± 82.3	300 ± 96.6	291 ± 115
TCOD Removal (%)	95.6 ± 1.0	94.2 ± 1.4	94.5 ± 1.8	94.7 ± 2.1
TCOD Range (mg/L)	159 - 333	158 - 553	191 - 373	100 - 518
SCOD (mg/L)	122 ± 58.3	175 ± 40.5	126 ± 10.0	129 ± 51.9
SCOD Reduction (%)	61 ± 19	44 ± 13	60 ± 32	59 ± 17
SCOD Range (mg/L)	48 - 173	109 - 275	129 - 134	34.8 - 222

[a] VSS was estimated based on the average TSS/VSS ratio of the 48 and 36 h HRT (i.e. 0.785)

As the OLR increased the average TCOD of the effluent maintained stable. The SGBR system maintained a TCOD removal efficiency above 90.6% for OLR ranging from 2.8 to 5.5 g COD/L·d. The relationship of effluent TCOD and SCOD with HRT is demonstrated in Figure 4.3. After each decrease in HRT an increase in the effluent COD concentration was observed. After the SGBR had time to acclimate to the increased OLR the effluent COD concentrations stabilized. The microbial communities of anaerobic granules are highly structured and capable of responding to quick and major changes in their environment (Kovacik et al., 2010). The diversity of microorganisms contained in anaerobic granules enables them to adapt to changes in their environment.
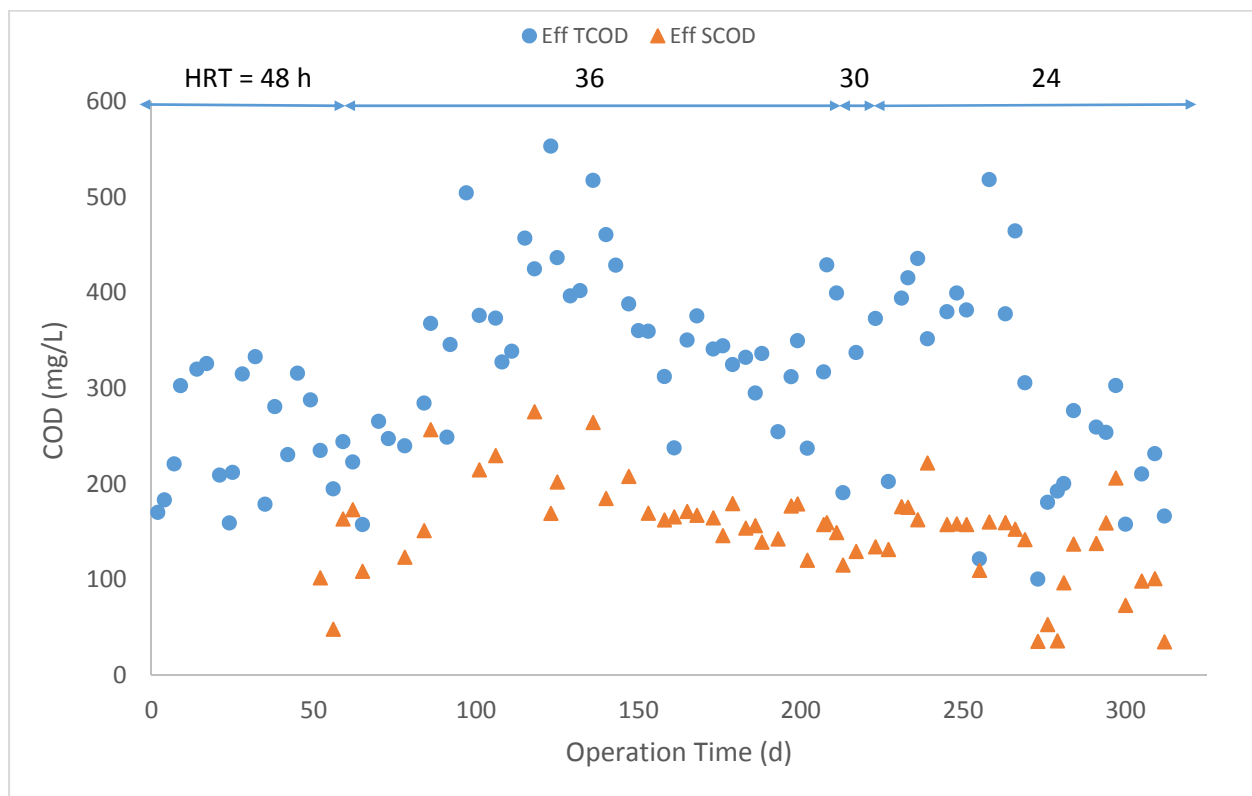
Figure 4.3: Comparison of laboratory-scale SGBR effluent TCOD and SCOD

The SGBR system maintained a TSS removal efficiency above 90.8% under the various OLR applied. The average effluent TSS for the 30 h HRT and 24 h HRT was 182 and 77 mg/L, respectively. The 30 h HRT was applied to the SGBR system for only two weeks. The HRT was gradually decreased to put less stress on the microbial communities in the SGBR system. As the granules became acclimated to the increased OLR the TSS and TCOD removal efficiencies increased during the 24 h HRT. The range of effluent SS concentrations is demonstrated in Figure 4.4.
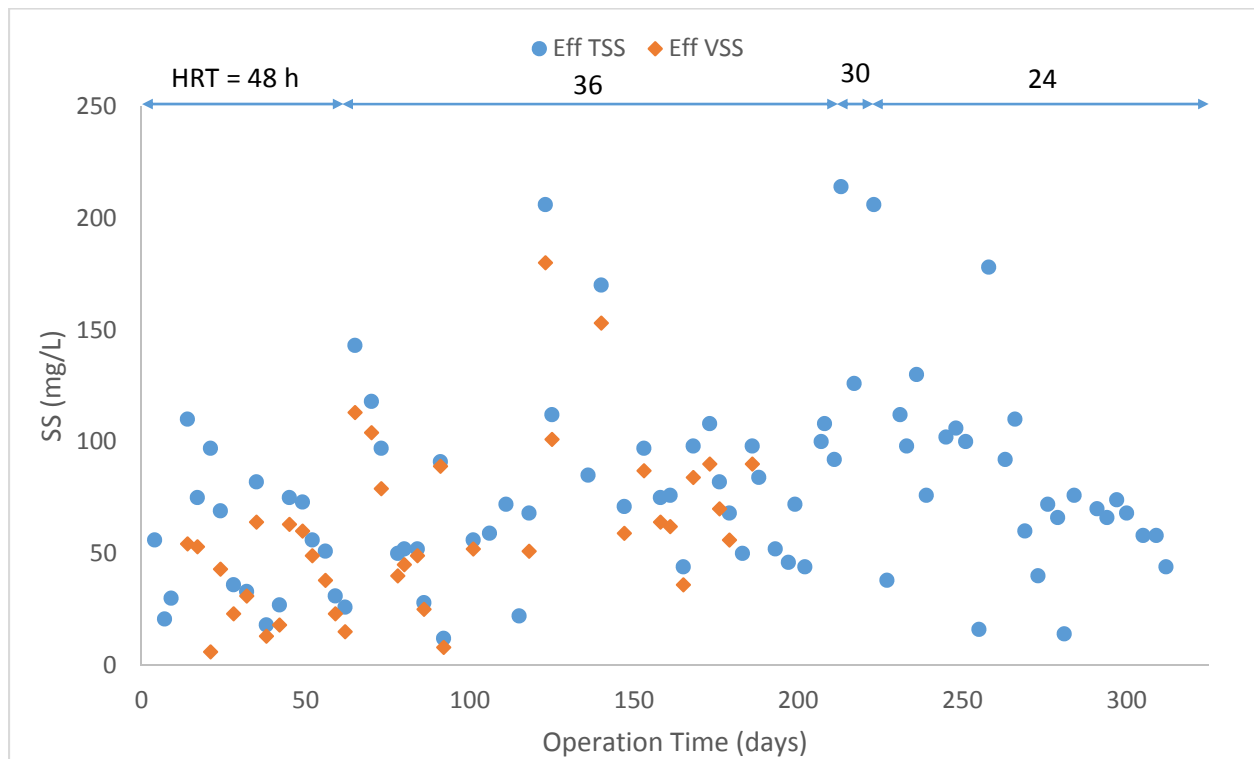
Figure 4.4: Comparison of laboratory-scale SGBR effluent TSS and VSS

The volatile solids (VS) measurement approximates the amount of organic matter present. The VS consist of the solids lost after the sample has been heated to 550°C. The volatile portion of all the solids (suspended and dissolved) in a water sample is referred to as the VS. The destruction of VS is associated with the rate of microbial hydrolysis. The rate of hydrolysis

depends on particle size and composition. While VSS refers to the portion of volatile suspended solids in a water sample. The calculation for percent VS reduction is displayed in Equation 4.1.

$$\% \, VS \, Reduction = \frac{VS_{In} - VS_{out}}{VSS_{In}} \times 100 \qquad (4.1)$$

The reduction in VS of a 14 L conventional mesophilic digester treating primary and secondary sludge from Mashalltown, IA ranged from 32% at a 24 d HRT (1.2 g VS/L·d) to 47% at a 40 d HRT (0.8 g VS/L·d) (Han et al., 1997b). The reduction in volatile solids (VS) of a 15 L conventional mesophilic digester treating sludge from the Ames WPCF ranged from 32.5% at a 10 d HRT (2.9 g VS/L·d) to 46.8% at a 15 d HRT (2.1 g VS/L·d) (Han and Dague, 1997a).

The reduction of volatile solids will vary from plant to plant regardless of the efficiency of the anaerobic digestion process. The non-biodegradable portion of municipal sludge varies widely (35-80%) depending on the wastewater source and previous treatment (Parkin and Owen, 1986). Therefore, the reduction of volatile solids is not the only indicator that should be used to measure the efficiency of an anaerobic digester. As a general guide volatile solids reduction should be above 30 to 40% (Parkin and Owen, 1986). The SGBR is able to remove solids at a higher rate than conventional digesters because it acts like a filter and a bioreactor. The SGBR averaged over 90% solids removal during this study.

Biogas Production and Composition

Biogas composed predominantly of methane and carbon dioxide is produced when organic matter is degraded under anaerobic conditions. The quantity of biogas produced depends on the level of organic loading, amount of volatile solids and the carbon to nitrogen ratio in the substrate (Mata-Alvarez et al., 2000; Senturk et al., 2012; Ketheesan and Stuckey, 2015). The

stability of the SGBR can be estimated by biogas production and composition. Under periods of system imbalance methane composition and biogas production will decrease. For example the carbon dioxide fraction of the biogas can be used as an indicator of digester performance. An anaerobic system that is imbalanced (i.e. organically overloaded) will exhibit an increase in the carbon dioxide fraction of the biogas (Ripley et al., 1986). The carbon dioxide component of biogas can also vary by substrate composition. Waste high in carbohydrates will produce more carbon dioxide than protein rich waste (Parkin and Owen, 1986).

The rate of biogas production will generally increase as the OLR and the microorganism's feed source increases. The growth of microorganisms will continue to increase with an increase in food source until a maximum growth rate is achieved (Clara et al., 2005). The biogas production of the SGBR is displayed in Figure 4.5. The gas production of the laboratory-scale SGBR increased with each increase in OLR.
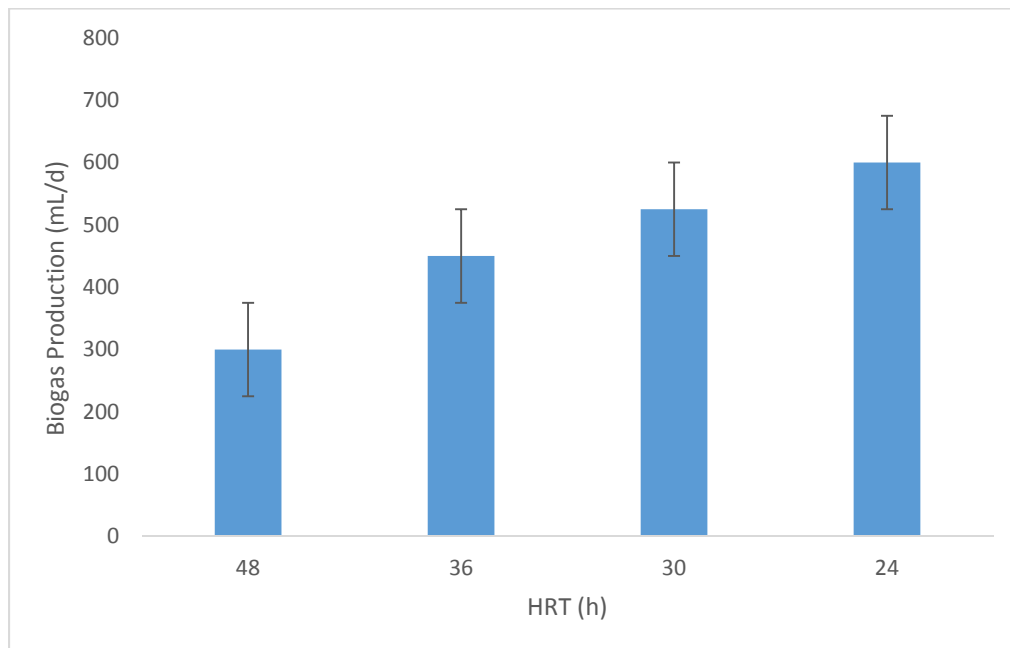
Figure 4.5: Laboratory-scale SGBR system biogas production by HRT

Low methane production could be a result of high influent PCOD and low operational temperature (Singh and Viraraghavan, 1999). The PCOD of this study composed between 91 and 98% of the influent's TCOD. Several research groups have shown the particulate matter can make up to 85% of the TCOD in domestic wastewater (Levine et al., 1985; Zeeman et al., 1997; Elmintealli et al., 2001b). When the influent contains all particulate matter, soluble matter results from the hydrolysis of the particulate matter (Grady et al., 2011). The growth of biomass is controlled by the soluble substrate concentration. The microorganisms must wait for hydrolysis of the particulate substrate to occur before they can degrade organic matter.

The maximum conversion of a substrate to methane is an important parameter in determining the potential of treating wastewater under anaerobic conditions (Elmitwalli et al., 2001a). The ability of a substrate to be converted to methane can determine its biodegradability and the potential treatment efficiency. High amounts of carbon dioxide and hydrogen gas in the biogas is an indication of reactor instability (Leitão et al., 2006). As the methane content in the biogas increases, anaerobic biodegradation increases. The composition and production rate of biogas is an indication of reactor performance.

The gas composition of the SGBR system is displayed in Figure 4.6. During the 24 h HRT of this study the SGBR produced gas with a composition of $76 \pm 4.0\%$ methane, $18 \pm 1.6\%$ carbon dioxide and $4 \pm 2.3\%$ nitrogen gas. The biogas composition for a sample collected from an Ames WPCF digester in December 2011 was $59 \pm 2.8\%$ methane, $37 \pm 1.8\%$ carbon dioxide. The methane content of the laboratory-scale SGBR treating sludge from the Ames WPCF was 22% greater than that of the Ames WPCF digester.
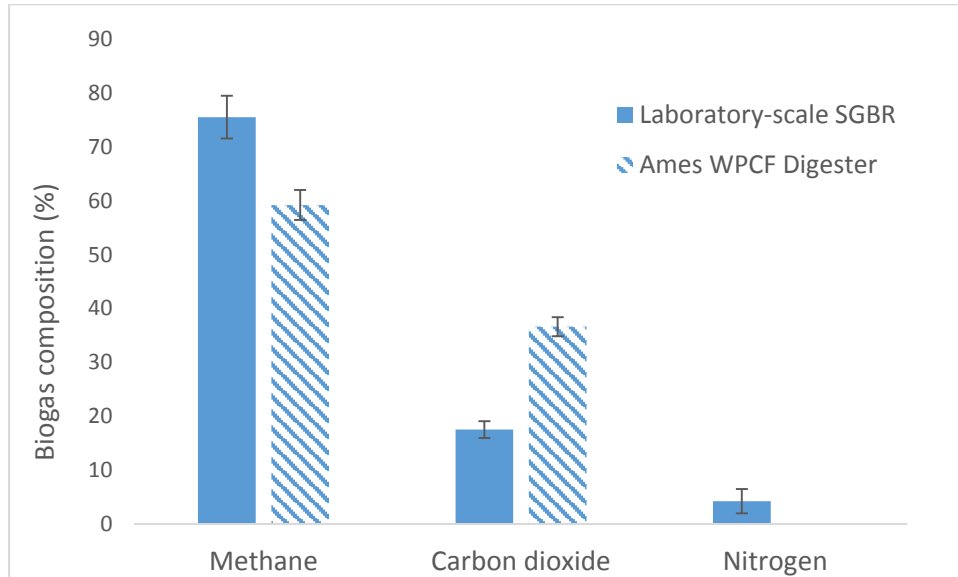
Figure 4.6: Laboratory-scale SGBR system and conventional digester biogas composition treating primary and secondary sludge

The biogas composition of 15 urban WWTP in Canada and the US was $63 \pm 2\%$ methane and $37 \pm 4\%$ carbon dioxide (Lackey et al., 2015). The biogas composition of a 15 L mixed conventional mesophilic digester treating sludge from the Ames WPCF ranged from 67 to 71% methane, 24 to 27% carbon dioxide, and 4 to 7% nitrogen at OLR ranging from 2.1 g VS/L·d (15 d HRT) to 2.9 g VS/L·d (10 d HRT) (Han and Dague, 1997a). The biogas composition of a 14 L conventional mesophilic digester treating primary and secondary sludge from Mashalltown, IA ranged from 65 to 72% methane, 24 to 27% carbon dioxide, and 2 to 5% nitrogen at OLR ranging from 0.8 g VS/L·d (40 d HRT) to 1.2 g VS/L·d (24 d HRT) (Han et al., 1997b).

Theoretical Methane Yield

The theoretical value of methane production is proportional to the organic matter destroyed. The theoretical volume of methane production per gram of COD removed is 0.35 L

CH$_4$/g COD removed. The theoretical volume of methane is generally less than the actual

volume due to a fraction of organic matter being utilized for microbial growth, and not all of the

gas produced is captured and measured accurately (Parkin and Owen, 1986). The theoretical

methane production was based on the assumption that 90% of the COD removed was converted

to methane (Oh et al., 2015). The volume of methane produced per mass of COD removed for

each backwashing period is displayed in Table 4.2. Overall 0.14 L CH$_4$/g COD removed was

observed during this study. The laboratory-scale SGBR methane yield is consistent with the

expected yield values for anaerobic municipal sludge digestion (i.e. 0.08 and 0.18 L CH$_4$/g COD

removed) (Noyola et al., 2006). The theoretical methane yields are displayed graphically in

Figure 4.7.

Table 4.2: Actual methane production based on COD removal

Backwash interval (d)	TCOD loaded by influent (g)	TCOD discharged by effluent (g)	TCOD Removed by backwash (g)	COD removed (g)	CH$_4$ production (L)	L CH$_4$/g COD removed
99	345.6	15.0	62.6	268.0	24	0.10
31	109.7	8.5	73.4	27.9	7.8	0.31
34	143.0	8.7	69.9	64.3	8.5	0.15
43	156.7	9.2	77.6	69.9	11	0.17
56	294.7	18.7	28.7	247.2	25	0.11
18	38.9	4.7	74.2	-40.0	8.8	-0.25
16	88.0	4.2	63.9	19.9	7.8	0.44
15	82.5	3.2	26.0	53.2	7.4	0.15
Total	1259	72.1	476.4	710.5	92	0.14
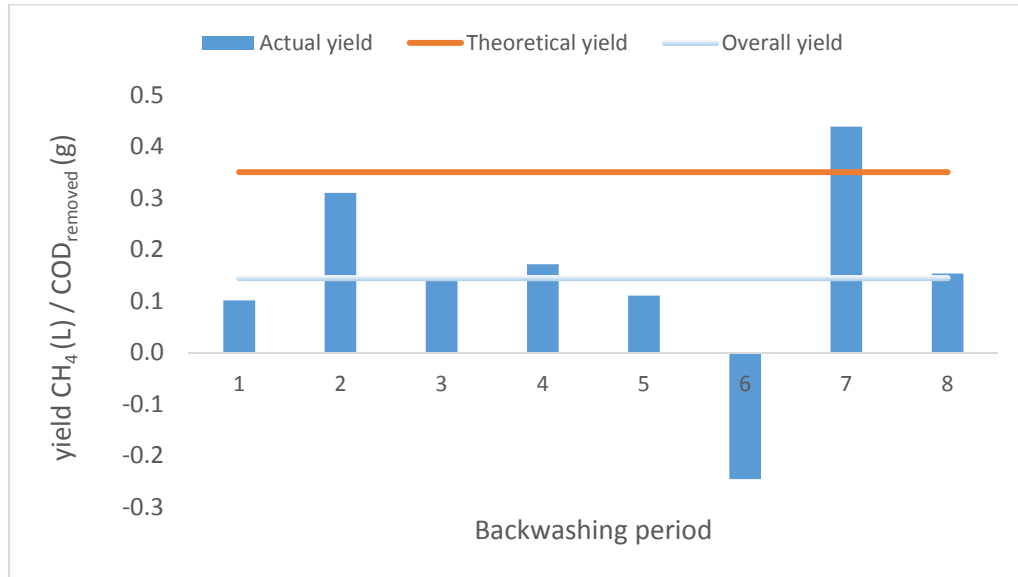
Figure 4.7: Actual and theoretical methane yield (laboratory-scale SGBR)

The formation of biogas pockets inside the sludge bed could result in a discordance in the biogas measurement (Soto et al., 2011). Abrupt release of the biogas from pockets in the SGBR's sludge bed was most notable during backwashing periods. The applied upflow velocity from backwashing was great enough to initiate the separation of gas bubbles from the granule surface (Soto et al., 2011). The volume of biogas released from the SGBR due to backwashing was approximately 1380 ± 114 mL per backwash cycle.

Another possible reason the actual methane yield does not match the theoretical is the supersaturation of methane that can exit the reactor with the effluent. Dissolved methane that leaves with the effluent could result in incorrect estimations of methane production. Contrary to common belief effluent from anaerobic treatment is often supersaturated with methane, resulting in a miscalculation of the methane production (Keller and Hartley, 2003). Approximately 20 to 60% of the theoretical methane production could be dissolved in the effluent (van Haandel and Lettinga, 1994; Agrawal et al., 1997; Singh and Viraraghavan, 1998; Keller and Hartley, 2003; Souza et al., 2011). The amount of dissolved methane in the effluent can be significant,

depending on reactor operational conditions. The solubility of methane is reduced by high ionic strength and increased by the presence of hydrophobic material such as fats and oil micelles (Souza et al., 2011).

The amount of methane dissolved in the effluent composed of between 36 and 41% of the total methane yield for a UASB reactor treating municipal wastewater at 25°C and located 900 m above sea level (Souza et al., 2011). The dissolved methane concentrations for the UASB reactor were 1.37 to 1.67 times higher than those calculated using Henry's Law. The UASB's total methane yield calculated by Souza et al. (2011) increased from 0.14 - 0.15 L CH_4/g COD removed to 0.22 - 0.24 L CH_4/g COD removed when the amount of dissolved methane in the effluent was quantified.

Backwashing the SGBR and Solids Mass Balance

Not all of the influent solids can be converted to methane. Solids can accumulate in and on top of the SGBR's granule bed, resulting in excessive head loss. The SGBR system requires backwashing because it acts as a filter as well as an anaerobic digester. Head loss in the SGBR system occurs periodically due to the entrapment of solids in the granule bed. Slowly biodegradable and non-biodegradable solids will accumulate in the system. The head and treatment efficiency of the system can be restored by periodic backwashing. Evans and Ellis (2004) determined that controlled wastage of solids through backwashing was essential in treating municipal wastewater with the SGBR. Backwashing helps to alleviate head loss, and it can also provide a degree of bed mixing which may be beneficial (Park et al., 2012). Mixing in a

digester disperses metabolic end products and toxic materials contained in the influent (Parkin and Owen, 1986).

The system was backwashed by pumping effluent into the underdrain of the system, pushing accumulated solids out of the system through a drain port above the granule bed. The duration between backwashing was based on the system's head loss and the accumulation of solids on top of the SGBR's granule bed. The time between backwashing decreased as the OLR of the system increased. An upflow velocity of 1 m/h was determined to be sufficient to dislodge accumulated solids while maintaining the anaerobic granules in the SGBR system (Oh et al., 2015). Oh et al. (2015) noticed significant buildup of non-degraded particulate organics in the SGBR system at a HRT less than 18 h and OLR greater than 3.5 kg COD/m^3·day. During this study significant solids build up was first noticed during the 36 h HRT with a corresponding OLR of 4.1 ± 0.7 g COD/L·d.

A solids mass balance was performed on the SGBR system to analyze solids removed by degradation, backwashing, and with the effluent. The following solids balance was adopted from Park et al. (2012). Several assumptions were made in order to calculate the solids balance of the SGBR system. The COD removed was assumed to be converted to methane at a ratio of 0.35 L CH$_4$/g COD adjusted to standard temperature and pressure (STP). Next it was assumed that the solids removed by methane conversion can be approximated by dividing methane production by the ratio of influent PCOD to TSS as demonstrated below in Equation 4.2. TSS$_{conversion}$ is the TSS removed by the TSS conversion to methane.

$$\text{TSS}_{conversion} = \frac{\text{CH}_4 \text{ production(L)from TSS}}{(0.35\ L/g\ COD) * (\text{PCOD}/\ \text{TSS})} \tag{4.2}$$

Equation 4.3 differentiates the methane production from SCOD removal and methane from solids degradation. The total methane production was measured by accounting for measured biogas production, methane solubility and methane that escaped due to backwashing. Dissolved methane can also be discharged from the reactor through the effluent. The volume of dissolved methane that exited the SGBR system with the effluent was estimated using Henry's Law, as displayed in Equation 4.4. The Henry's Law constant at 298.15 (K = 0.0014 mol/L/atm) was used to calculated the solubility of methane at STP (Oh et al., 2015). The process of backwashing the system depressurizes the system. The volume of methane released from the reactor due to backwashing was approximately 1380 ± 114 mL per backwash cycle.

$$CH_4 \ from \ TSS \ (L) = Total \ CH_4 \ production - CH_4 \ from \ SCOD \ removal \qquad (4.3)$$

$$V_{dissolved} = \left(1.4 * 10^{-3} \frac{mol}{L*atm} * e^{\left[1700\left(\frac{1}{273.15+T} - \frac{1}{298.15}\right)\right]} * 1atm * 22.4 \frac{L}{mol}\right) * Q_{out} \qquad (4.4)$$

Equation 4.5 estimates the methane from SCOD removal. This assumes that the SCOD (e.g. VFA) removed was all converted to methane.

$$CH_4 \ from \ SCOD \ removal \ (L) = \left(SCOD_{in} \ (g) - SCOD_{out} \ (g)\right) * 0.35(L/g \ COD) \qquad (4.5)$$

Equation 4.6 combines the soluble and particulate fractions of COD.

$$PCOD = TCOD - SCOD \qquad (4.6)$$

Equation 4.7 estimates the amount of solids unaccounted for.

$$TSS_{unaccounted} = TSS_{in} - TSS_{out} - TSS_{conv} - TSS_{backwash} \qquad (4.7)$$

The solids balance was calculated for each backwashing period as displayed in Table 4.3. The average ratio of influent PCOD to TSS was calculated for each backwashing period. The

average methane composition in the biogas was used for each period as if the methane composition was stable. The accumulation of solids increased substantially during long periods between backwashing. A chart displaying the fate of the solids load into the SGBR system is demonstrated in Figure 4.8. The influent TSS were removed by degradation (22%), backwashing (50%), effluent (3.7%) and 24% were unaccounted for. Solids removal in a conventional heated and mixed anaerobic digester is typically between 40 and 60%.

Table 4.3: Laboratory-scale SGBR solids balance

Backwash Interval (d)	Solids loaded by Influent (g)	Solids Discharged by Effluent (g)	Solids Removed by Degradation (g)	Solids Removed by Backwash (g)	Unaccounted Solids (g)
99	120	3.6	29.9	33.2	54
31	48	1.6	11.5	38.9	-4
34	52	1.8	12.6	37.0	1
43	66	2.3	15.9	41.2	7
56	110	4.9	27.8	15.2	62
18	38.9	1.7	8.4	39.3	-10.5
16	34.6	1.5	7.6	33.9	-8.4
15	32.4	1.4	7.1	13.8	10
Total	503	19	121	253	111
percent of total	100%	3.7%	24.0%	50.3%	22.0%
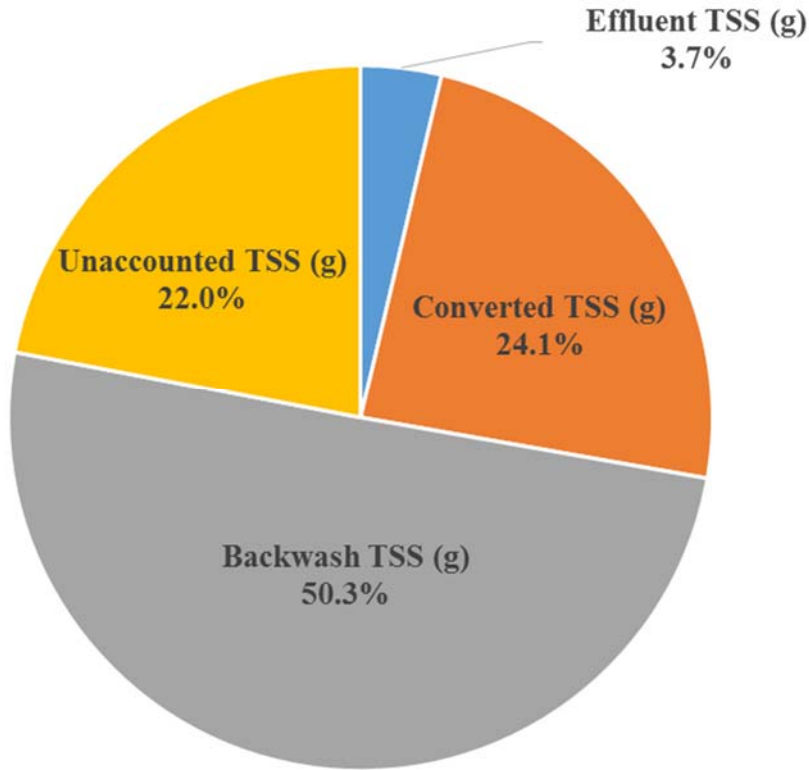
Figure 4.8: Fate of laboratory-scale SGBR solids

Hydrolysis of solids appeared to be rate limiting based on the amount of solids that were backwashed from the SGBR. As the time between backwashing increased so did the solids that were unaccounted for as displayed in Figure 4.9. Carefully monitoring the accumulation of solids on top of the bed and backwashing the system was essential in treating primary and secondary sludge with the SGBR.
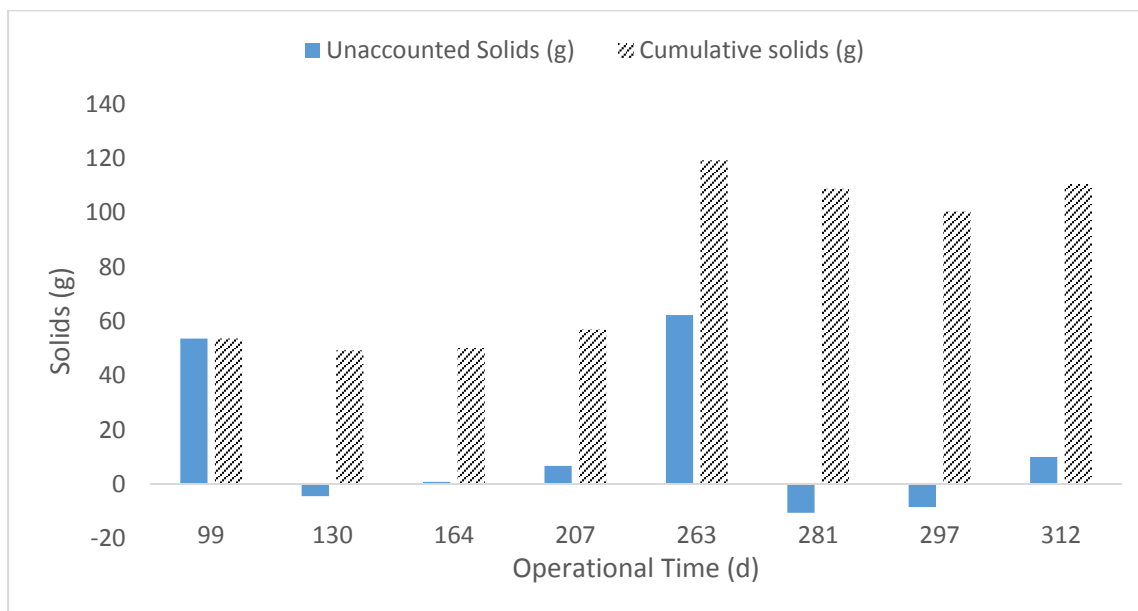
Figure 4.9: Overall solids accumulation in the laboratory-scale SGBR system

The SGBR was capable of separating solids from the influent because it acts like a filter and a bioreactor. Biological and physical processes both play an essential role in anaerobic digestion of particulate matter. Particulate matter can only be biologically hydrolyzed after becoming physically removed either by entrapment in the sludge bed or adsorption (Elmitwalli et al., 2001b). The influent was composed primarily of solids as indicated by the average influent PCOD of 94.5 ± 2.4%. The UASB reactor does not effectively retain particulate matter. When treating wastewaters high in particulate matter the UASB reactor allows solids to pass through the bioreactor with little hydrolysis and stabilization (Grady et al., 2011).

COD Mass Balance

The principal equation for the mass balance is displayed below in Equation 4.8 (Oh et al., 2015). The results of the COD mass balance for each HRT are displayed in Table 4.4. As demonstrated in Figure 4.10, 22% of the COD input into the SGBR system was unaccounted for.

There are several possibilities for the unaccounted COD: accumulated solids in and on top of the granule bed, dissolved methane leaving with the effluent, cell synthesis, methane lost to the atmosphere, and sulfate reduction (Lobato et al., 2012).

$$TCOD_{in} = TCOD_{out} + COD_{CH_4} + COD_{unaccounted} + COD_{backwashed} \qquad (4.8)$$

Table 4.4: COD mass balance of the SGBR system

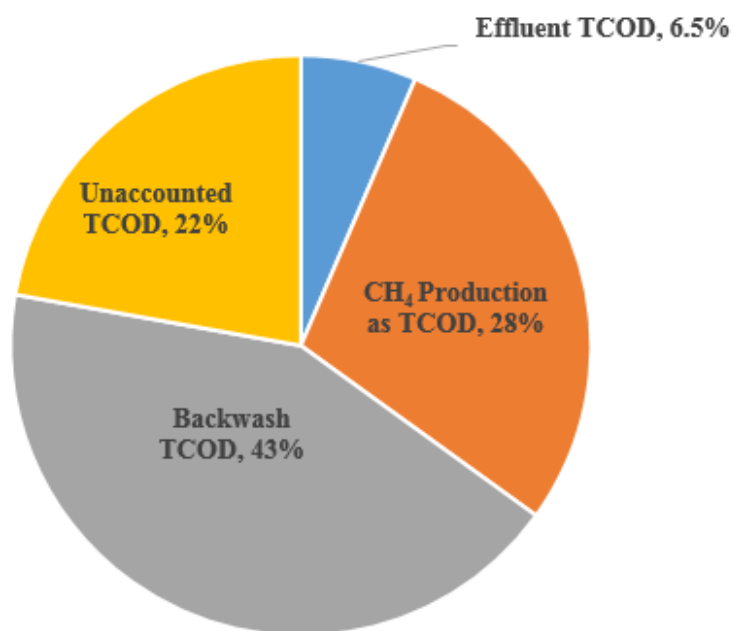| HRT (h)	Influent TCOD (g)	Effluent TCOD (g)	$CH_{4production}$ (g COD)	Backwash TCOD (g)	Unaccounted TCOD (g)
48	88	7.7	40	37	2.5
36	490	36	148	247	59
30	40	2.4	11	5	21
24	489	26	116	185	163
Total	1107	71	315	474	245
% of Total	100%	6.5%	28%	43%	22%

Figure 4.10: COD mass balance of the laboratory-scale SGBR system treating primary and secondary municipal sludge

As discussed above dissolved methane that leaves with the effluent could also result in unaccounted COD. Depending on the reactor configurations and substrate composition, effluent has the potential to be supersaturated with methane. COD removal can also be affected by sulfate reducing bacteria. Sulfate reducing bacteria use sulfate as an electron acceptor to oxidize various organic compounds (Singh and Viraraghavan, 1998). Sulfate reduction reduces the methane yield for each unit of converted COD (Colleran et al., 1995). Problems with competitive inhibition can arise between sulfidogenic bacteria and methanogenic archaea during anaerobic digestion of sulfide containing wastewater (Barber and Stuckey, 2000). While treating low strength municipal wastewater with the UASB reactor Singh and Viraraghavan (2004) determined that sulfate reducing bacteria removed 25 to 35% of the COD, while methanogens removed COD at a rate of 35 to 45%. Conversely, Lobato et al. (2012) concluded that low concentrations of sulfates in domestic wastewater (20 to 100 mg SO_4/L) results in low COD removal due to sulfate reduction.

Unaccounted COD could potentially be used for cell synthesis (Singh and Viraraghavan, 2004). The average biomass yields were between 0.057 and 0.122 g VSS/g $COD_{removed}$ for an SGBR system treating slaughterhouse wastewater at HRT and OLR from 48 to 20 h and 2.97 to 8.23 kg COD/m^3·d, respectively (Park et al., 2012). Typical biomass production rates for high rate anaerobic digesters are between 0.05 and 0.1 g VSS/g $COD_{removed}$ (Grady et al., 2011). The growth yield of biomass is very low in anaerobic systems (Grady et al., 2011).

The COD mass balance performed on pilot and demonstration-scale UASB reactors by Souza (2010) quantified COD removal in the following ways: effluent SCOD (14 to 24%), Sludge in the effluent (10 to 20%), sludge accumulated in the UASB reactor (8 to 10%), methane

in the biogas (24 to 30%), dissolved methane (16 to 18%) and sulfate reduction (4.5 to 5%) (Lobato et al., 2012). Oh et al. (2015) determined an unaccounted TCOD of 19.3% in the SGBR system while treating dairy processing wastewater as displayed in Figure 4.11.
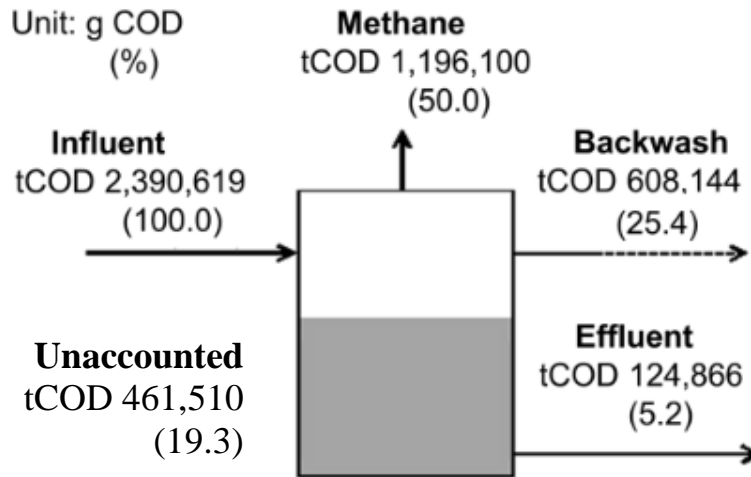
Figure 4.11: COD mass balance on the pilot-scale SGBR system treating dairy processing wastewater (Oh et al., 2015)

Sludge Volume Index (SVI) of the Backwash Material

The SVI measurement quantifies the propensity of sludge to settle and compact. The SVI was calculated to evaluate the ability of the backwash material to be dewatered and settled. The SVI value of the backwash material was obtained by measuring the volume of sludge settled in a 1000 mL beaker. The volume of settled sludge vs time is displayed in Figure 4.12.
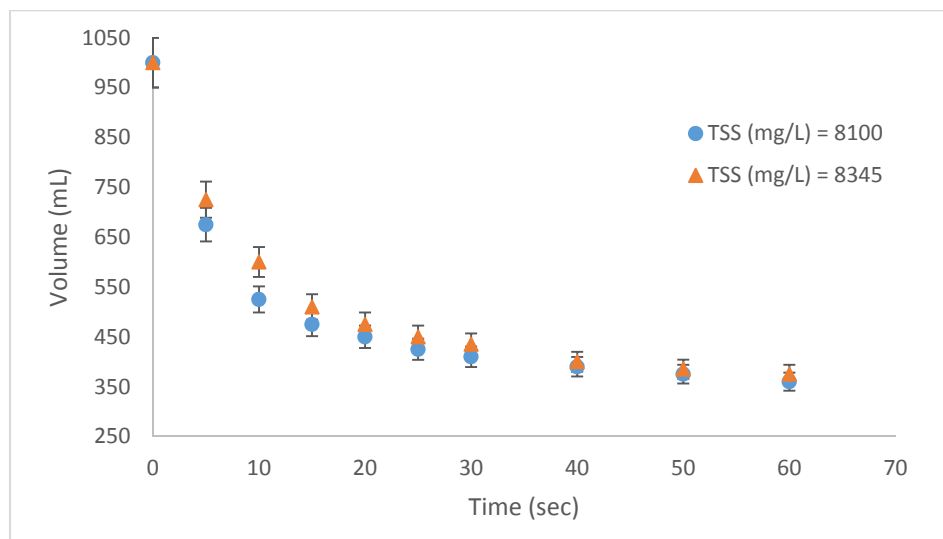
Figure 4.12: Settling volume of laboratory-scale SGBR backwash material

The SVI for backwashed material with a TSS ranging from 8100 to 8345 mg/L was determined to be between 51 ± 2.5 and 52 ± 2.6 mL/g, respectively. A SVI value less than 80 mL/g indicates excellent settling and compaction characteristics (Grady et al., 2011). The solids wasted with backwash water could be dewatered for ultimate disposal or land application.

Hydrolysis, Acidification and Methanogenesis

The percentages of hydrolysis, acidification, and methanogenesis reactions were calculated to determine the SGBR's ability to degrade primary and secondary sludge. Equation 4.9 through Equation 4.12 demonstrate the calculations required to express the volume of methane as COD. Where $V_{dissolved}$ corresponds to the volume of dissolved biogas released with the effluent based on Henry's Law. The Henry's Law constant at 298.15 ($K = 0.0014$ mol/L/atm) was used to calculated the solubility of methane at STP (Oh et al., 2015).

$$V_{CH_4} = V_{measured} + V_{dissolved} \tag{4.9}$$

$$V_{measured} = V_{biogas} * CH_4\%$$ (4.10)

$$V_{dissolved} = \left(1.4 * 10^{-3}\, \frac{mol}{L * atm} * e^{\left[1700\left(\frac{1}{273.15+T}-\frac{1}{298.15}\right)\right]} * 1atm * 22.4\, \frac{L}{mol}\right) * Q_{out}$$ (4.11)

$$COD_{CH_4} = V_{CH_4} * \frac{g\ COD}{0.35\ L\ CH_4}$$ (4.12)

Equation 4.13 through Equation 4.15 calculates the percentages of hydrolysis, acidification, and methanogenesis (Oh et al., 2015). The conversion factor of 1.28 g COD/g VFA was assumed (Danalewich et al., 1998; Rössle and Pretorius, 2001; Oh et al., 2015). The 1.28 g COD/g VFA is based on typical VFA composition distributions for prefermenters in domestic wastewater treatment (Rössle and Pretorius, 2001).

$$H(\%) = 100\left(\frac{COD_{CH_4} + SCOD_{out} - SCOD_{in}}{TCOD_{in} - SCOD_{in}}\right)$$ (4.13)

$$A(\%) = 100\left(\frac{COD_{CH_4} + COD_{VFA_{out}} - COD_{VFA_{in}}}{TCOD_{in} - COD_{VFA_{out}}}\right)$$ (4.13)

$$M(\%) = 100\left(\frac{COD_{CH_4}}{TCOD_{in}}\right)$$ (4.15)

Table 4.5 estimates the percentages of hydrolysis, acidification, and methanogenesis for each HRT. The rate of hydrolysis and methanogensis remained stable as the HRT was decreased during the study. The acidification step was negative because the VFA concentration of the influent (245 ± 35 mg/L as HAc) were greater than the VFA concentrations in the effluent (11 to 30 mg/L as HAc). The results indicate that influent organic matter was not completely converted to methane possibly due to the high fraction of influent PCOD (Oh et al., 2015). Evans and Ellis (2004) determined that hydrolysis or mass transport limitations could be reasons for incomplete degradation of suspended solids in a SGBR system treating municipal wastewater. During high-rate anaerobic treatment at 30°C, Elmitwalli et al. (2001a) estimated the maximum hydrolysis,

acidification and methanogenesis for suspended COD in domestic sewage at $87 \pm 5\%$, $78 \pm 4\%$ and $77 \pm 4\%$, respectively.

Table 4.5: Hydrolysis, acidification and methanogenesis of the SGBR system

Operation Time (d)	HRT (h)	OLR (g COD/L·d)	COD_{CH4} (g)	Hydrolysis (%)	Acidification (%)	Methanogenesis (%)
62	48	2.8	43	22	-5.3	25
151	36	4.1	157	24	-7.1	26
10	30	5.0	12	25	-5.4	28
89	24	5.5	125	23	-9.0	25

VFA, Alkalinity and pH

The VFA, alkalinity and pH of an anaerobic digester are interconnected. The success of a digester depends on maintaining an acceptable buffering capacity along with avoiding excessive VFA concentrations (Ripley et al., 1986). Accumulation of VFA in a digester can result in a decline in system pH, affecting methanogenic populations. As demonstrated in Table 4.6, effluent pH, alkalinity, and VFA values were stable during the duration of the study.

Table 4.6: Effluent pH, VFA and alkalinity examples for the SGBR system

	HRT (h)		
	48	36	24
pH	6.64 ± 0.25	6.63 ± 0.21	6.62 ± 0.15
VFA (mg/L as HAc)	NM	19 ± 5.5	16 ± 4.4
Alkalinity (mg/L as CaCO₃)	463 ± 67	534 ± 89	438 ± 58

A good indicator of the anaerobic process needs to be able to detect metabolic stress before it causes major issues. The VFA concentration is a good indicator of performance because it can detect imbalances in the system relatively quick, within 2 days in most cases (Ahring et al.,

1995). Overloading and sudden variations in HRT and OLR can result in the accumulation of VFA in an anaerobic digester (Leitão et al., 2006).

As discussed above, VFA are formed as intermediate products in anaerobic digestion. The accumulation of VFAs may be a result of unstable kinetics between the microbial groups involved in the digestion process. The acidogenic conversion phase of anaerobic digestion is nearly fivefold faster than the methanogenic conversion phase (Ketheesan and Stuckey, 2015). The accumulation of VFA reflects a kinetic imbalance in the microbial populations involved in the anaerobic digestion process. Stressful operational and environmental conditions can result in an increase in VFA producers and a decrease in VFA consumers, leading to reactor instability (Leitão et al., 2006).

The stability of an anaerobic digester depends on the effective production and consumption of VFA. Operating an anaerobic system at a long SRT results in essentially no accumulation of VFA in the effluent (Parkin and Owen, 1986). The VFA of the SGBR's effluent were between 30 and 11 mg/L as HAc for OLR ranging from 0.85 to 3.4 g VSS/L·d. The average volatile acid (VA) concentrations of the AWPC digesters ranged from 579 mg/L to 437 mg/L. Reducing the VFA concentrations in the effluent also reduces the amount of odorous compounds (Han et al., 1997b).

Maintaining the system's pH in the optimal range prevents the dominance of acidogenic microorganisms which results in the accumulation of VFA (Ketheesan and Stuckey, 2015). The optimal pH range of 6.5 and 8.2 for methane production (Oh et al., 2015). Inhibition of methanogens starts to become noticeable around a pH of 6 (Ketheesan and Stuckey, 2015). The pH of the SGBR's effluent was maintained between 7.17 and 6.19 for the duration of the study.

The pH of an anaerobic system is important in controlling parameters that can significantly affect hydrolysis along with VFA production (Huang et al., 2015).

As the HRT of the system decreased from 48 h to 36 h, there was an increase in the VFA production as demonstrated in Figure 4.13. This increase in VFA production during the decrease in HRT resulted in a reduction in alkalinity as demonstrated in Figure 4.14. The effluent pH remained stable during the increase in HRT (Figure 4.14), indicating that the alkalinity was used to maintain a stable pH in the system (Oh et al., 2015). A decrease in the system's pH would be a result of the buildup of VFA concentration causing the destruction of the bicarbonate buffering capacity (Ripley et al., 1986). Carbonate buffering comes from the degradation of nitrogenous organics (i.e. proteins) to ammonia (Parkin and Owen, 1986). Ammonia will react with carbon dioxide to form ammonium bicarbonate. The SGBR maintained enough buffer capacity to counteract the increase in VFA production due to an increase in the OLR.
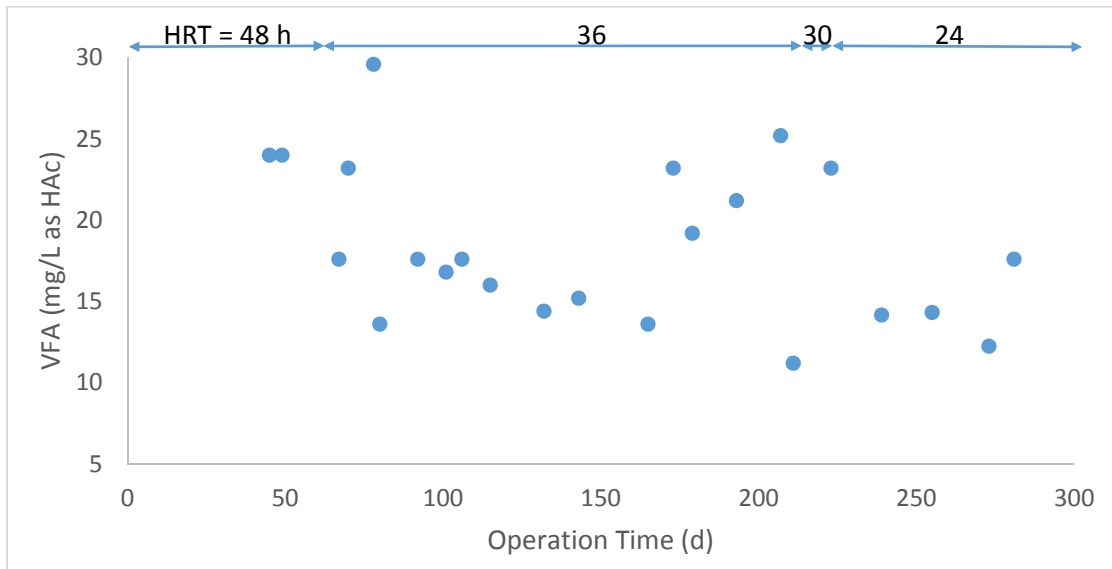
Figure 4.13: Laboratory-scale SGBR effluent VFA concentrations
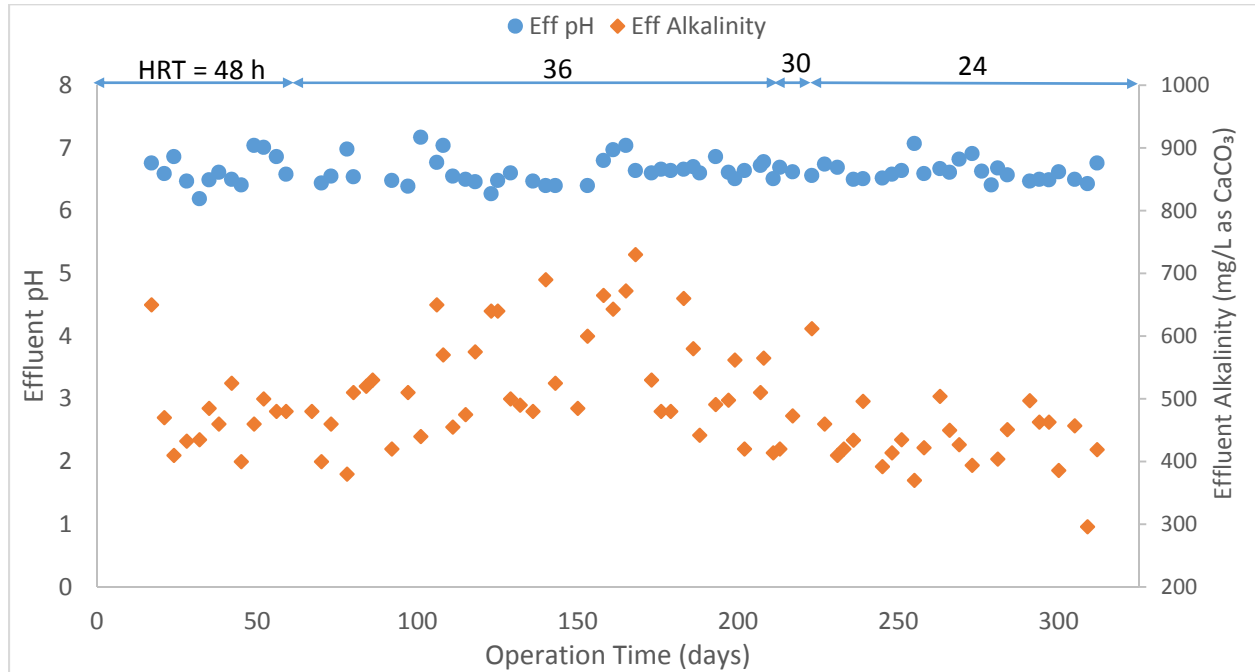
Figure 4.14: Variation of effluent pH and alkalinity

The effluent pH cannot necessarily be used as an indicator of process performance. The accumulation of VFA can be concealed by the digester's buffering capacity, resulting in a stable pH. Due to the possible concealment of VFA, monitoring only the pH is not sufficient to detect process imbalances (Ketheesan and Stuckey, 2015). Monitoring alkalinity and VFA is also important to detect process stability. The VFA/alkalinity ratio can be used to detect the degree of acidification in the anaerobic digestion process (Demirel and Yenigun, 2004; Ketheesan and Stuckey, 2015). A VFA/alkalinity ratio less than 0.3 reflects reactor stability, while a ratio between 0.3 and 0.4 indicates the possibility of digester instability (Oh et al., 2015). As demonstrated in Figure 4.15 the VFA/alkalinity ratio remained between 0.02 and 0.08 for the duration of the study.
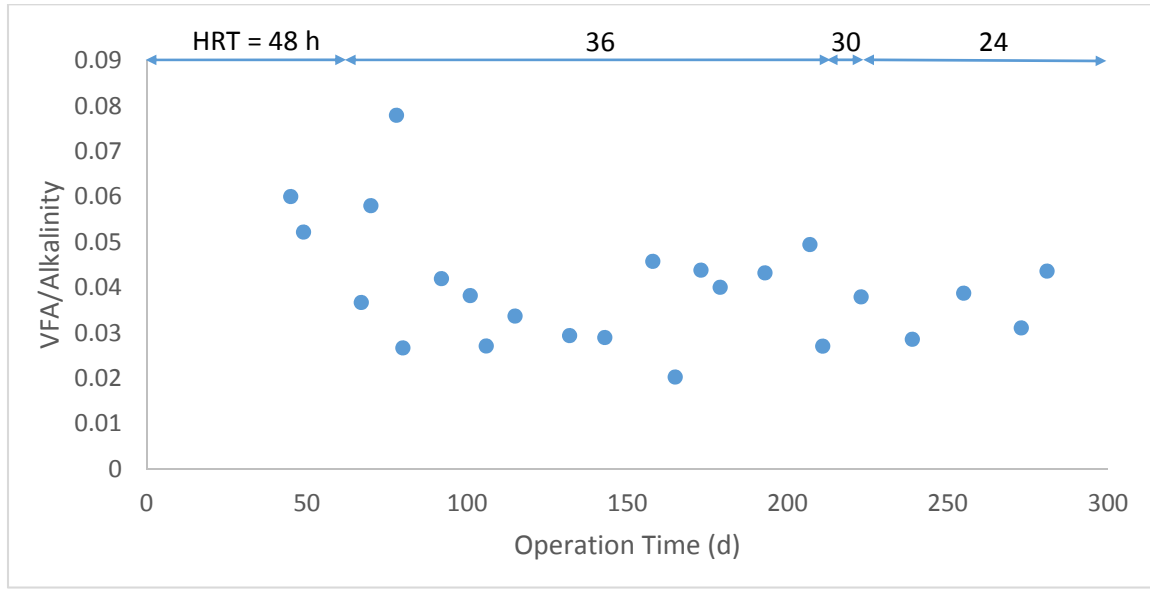
Figure 4.15: Variation in the ratio of VFA to alkalinity of the effluent

The VFA of a laboratory-scale 14 L conventional mesophilic digester treating primary and secondary sludge from Mashalltown, IA at HRT ranging from 24 d SRT (1.2 g VS/L·d) to 40 d SRT (0.8 g VS/L·d) were maintained between 200 mg/L as HAc (Han et al., 1997b). The VFA, Alkalinity and pH of a laboratory-scale 15 L conventional mesophilic digester treating sludge from Ames WPCF at HRT ranging from 10 d SRT (2.9 g VS/L·d) to 15 d SRT (2.1 g VS/L·d) were maintained between 180 and 300 mg/L as HAc, 5000 to 6100 as $CaCO_3$ and 7.0 to 7.4, respectively (Han and Dague 1997a).

CHAPTER 5. SECONDARY AND PRIMARY SLUDGE TREATMENT BY ON-SITE PILOT-SCALE STATIC GRANULAR BED REACTOR (SGBR)

Introduction

A laboratory-scale of the SGBR system was conducted to determine its ability to treat primary and secondary sludge. The laboratory-scale SGBR consistently removed greater than 90% of the TCOD and TSS. The laboratory-scale reactor was successfully operated at HRT and OLR ranges from 48 to 24 h and 2.8 to 5.5 g COD/L·d, respectively. The objective of this research was to evaluate the ability of a pilot-scale SGBR system to treat primary and secondary municipal sludge at a high rate.

Materials and Methods

A 1,000 gallon on-site pilot-scale SGBR system was used to treat primary and secondary sludge at the Ames WPCF. The primary and secondary sludge was diluted with chilled plant water at a ratio of 1:15 to create the influent. The influent was stored in a 650 gallon feed tank. The influent was pumped from the feed tank to the top of the SGBR using a Masterflex peristaltic pumps (Models L/S 77521-40). The influent was distributed across the top cross-section of the anaerobic reactor with a 3/4-inch perforated PVC pipe. A gravel underdrain was installed to support the granule bed. The SGBR system was seeded with approximately 400 gallons of anaerobic granule sludge from an operating UASB reactor at City Brew Brewery in La Crosse, Wisconsin.

A 65-gallon tank was used to collect effluent for backwashing the system. A 4-inch diameter weir was installed above the operating water level in the reactor to allow backwashed water to be discharged from the reactor. The entire SGBR system was retrofitted on site with 3/4-inch PVC piping and fittings. The biogas produced by the SGBR system was collected through a port on the top and vented to the outside of the building. The rate of biogas production during operation was measured using an Actaris Schlumberger oil gas meter. The biogas was routed through a steel wool scrubber to remove hydrogen sulfide. A schematic of the pilot-scale SGBR setup is displayed in Figure 5.1.
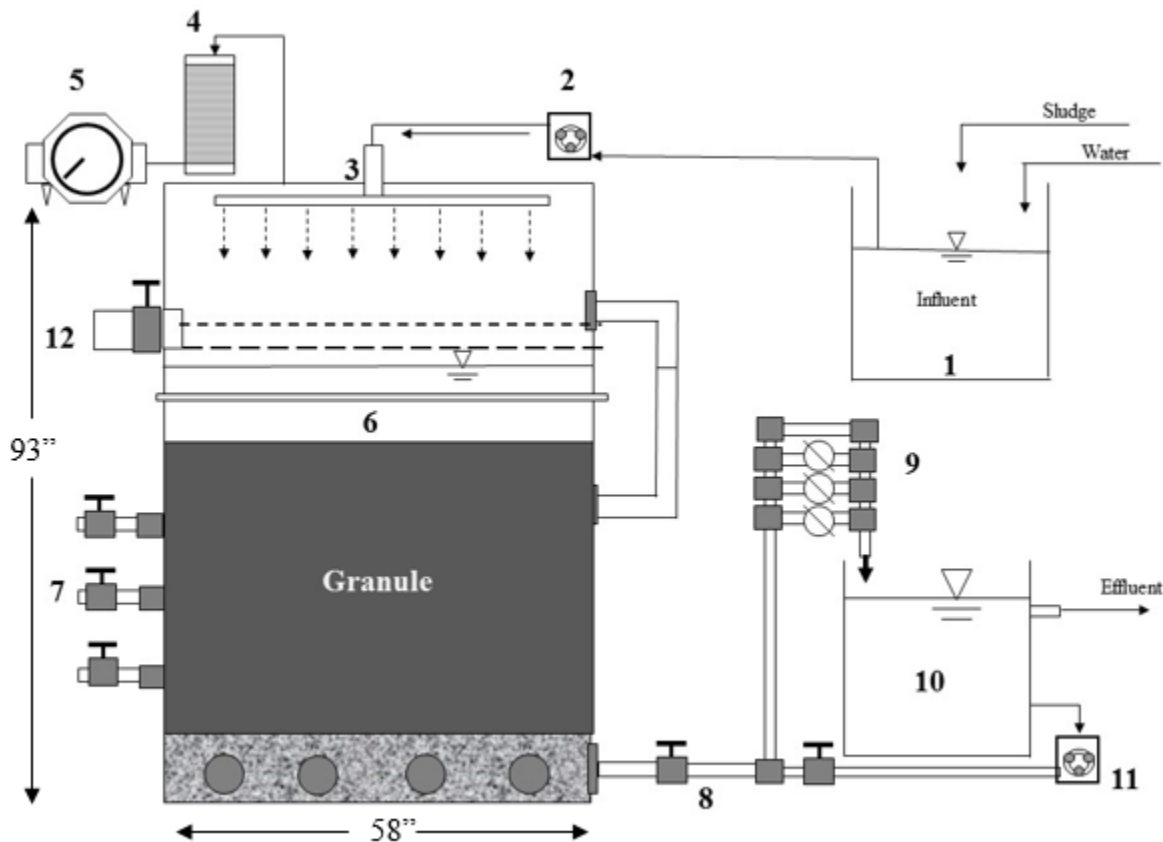

Figure 5.1: Schematic of the pilot-scale SGBR system: (1) Influent storage tank, (2) Influent pump, (3) Influent distributor, (4) H2S scrubber system, (5) Gas meter, (6) SGBR reactor, (7) Sampling port, (8) Drain and backwashing valve system, (9) Effluent overflow pipe, (10) Effluent storage tank, (11) Backwash pump, (12) Backwashing water discharge system

The flowrate of the influent was controlled to set the HRT of the SGBR. The HRT of the reactor was initially started out at 48 h. The reactor's HRT was based on the reactors active volume of granular biomass. Influent, effluent and biogas samples were collected and analyzed frequently to evaluate the health and performance of the reactor. The TCOD, SCOD, TSS, alkalinity, VFA and gas composition were all analyzed. The reactor was backwashed every one to two weeks in order to remove excessive buildup of solids in and on the granule bed.

The SGBR influent and effluent were tested for TSS, COD, alkalinity, pH and VFA according to The Standard Methods for the Examination of Water and Wastewater (APHA, 1998). The test method used for each parameter is displayed in Table 5.1. The influent and effluent samples were filtered with glass fiber filter paper (Whatman GF/C, 1.2μ pore size) to analyze TSS and VSS. The SCOD was also measured using the filtered wastewater sample. The influent and effluent pH was analyzed using a Fisher Scientific Accumet excel, model XL15 pH meter. The biogas production was estimated using a RITTER© wet-test (drum-type) gas meter. The composition of the biogas was analyzed by a Gow Mac gas chromatograph series 580 (Bethlehem, PA).

Table 5.1: The test method for each parameter

Parameter	Description	Section
TSS	Total Suspended Solids Dried at 103-105°C	2540 D.
TCOD	Closed Reflux, Titrimetric Method	5220 C.
Alkalinity	Titration Method	2320 B.
pH	Electrometric Method	$4500\text{-}H^+$ B.
VFA	Distillation Method	5560 C.

Results and Discussion

The characteristics of the influent and effluent are demonstrated in Table 5.2. The influent was sampled at a port on top of the SGBR. There was a significant variation in influent TCOD and TSS due to a lack of mixing in the influent storage tank.

Table 5.2: Pilot-scale SGBR influent and effluent characteristics

	Influent		Effluent	
	Average	SD	Average	SD
pH	5.73	0.16	6.66	0.11
Alkalinity (mg/L as CaCO$_3$)	613	58	828	28.2
VFA (mg/L as HAc)	1069	185	19.7	8.2
TCOD (mg/L)	6586	4073	251.3	110.6
SCOD (mg/L)	1474	530	82.0	33.8
TSS (mg/L)	2604	1904	118.3	63.4

The SGBR system's ability to remove TCOD and TSS from primary and secondary sludge at a 48 h HRT is demonstrated in Figure 5.2 and Figure 5.3, respectively. The process efficiency of the SGBR system was evaluated under an OLR of 3.3 ± 2.0 g COD/L·d. The TSS and TCOD removal efficiencies were $91.9 \pm 7.0\%$ and $95.2 \pm 3.2\%$, respectively. The effluent TCOD and TSS were maintained between 50.6-382 mg/L and 20-213 mg/L, respectively.
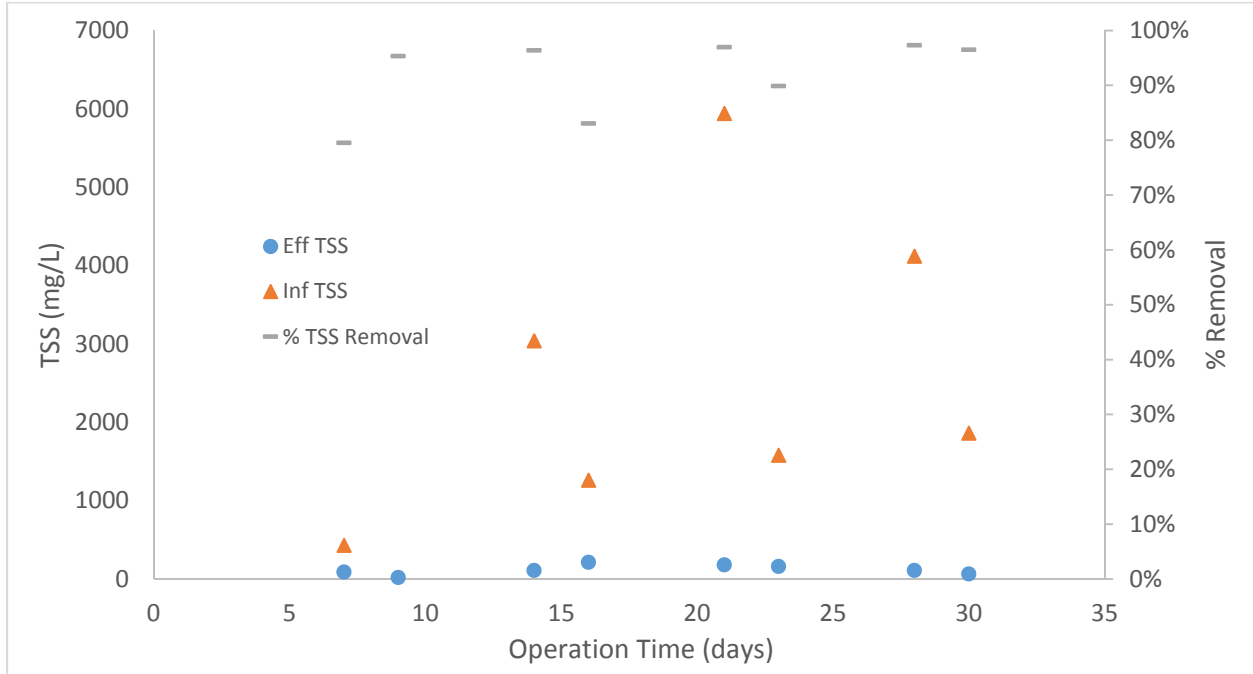
Figure 5.2: Pilot-scale SGBR TSS removal
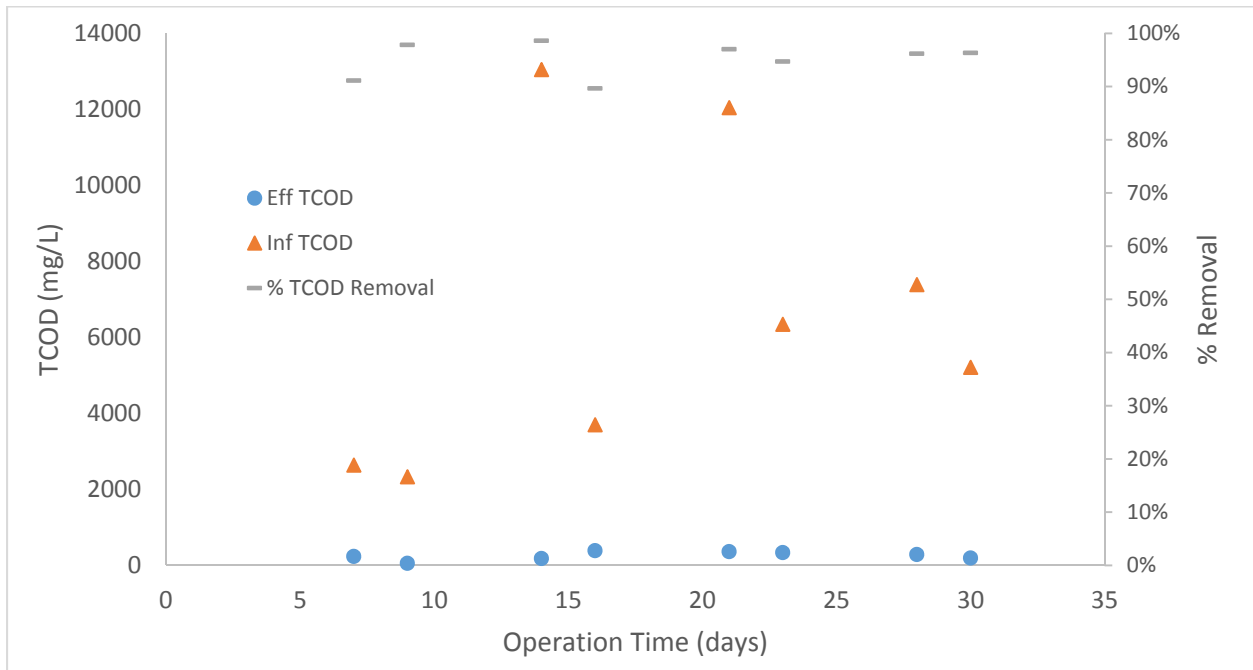
Figure 5.3: Pilot-scale SGBR TCOD removal

As demonstrated in Figure 5.4, the alkalinity, pH and VFA/alkalinity ratio of the effluent were monitored to make sure the system was operating within the appropriate ranges for methanogens. Research has shown that the inhibition of methanogens can start to become noticeable around a pH of 6 (Ketheesan and Stuckey, 2015). The pH increased and the VFA concentrations decreased from effluent to influent. The consumption of influent VFA may have been primarily responsible for the increase in pH. The influent and effluent VFA concentrations were measured between 863 and 1370 mg/L as HAc and 13 and 34 mg/L as HAc, respectively. The VFA/alkalinity ratio of the SGBR effluent remained below 0.04. A VFA/alkalinity ratio less than 0.3 reflects reactor stability.
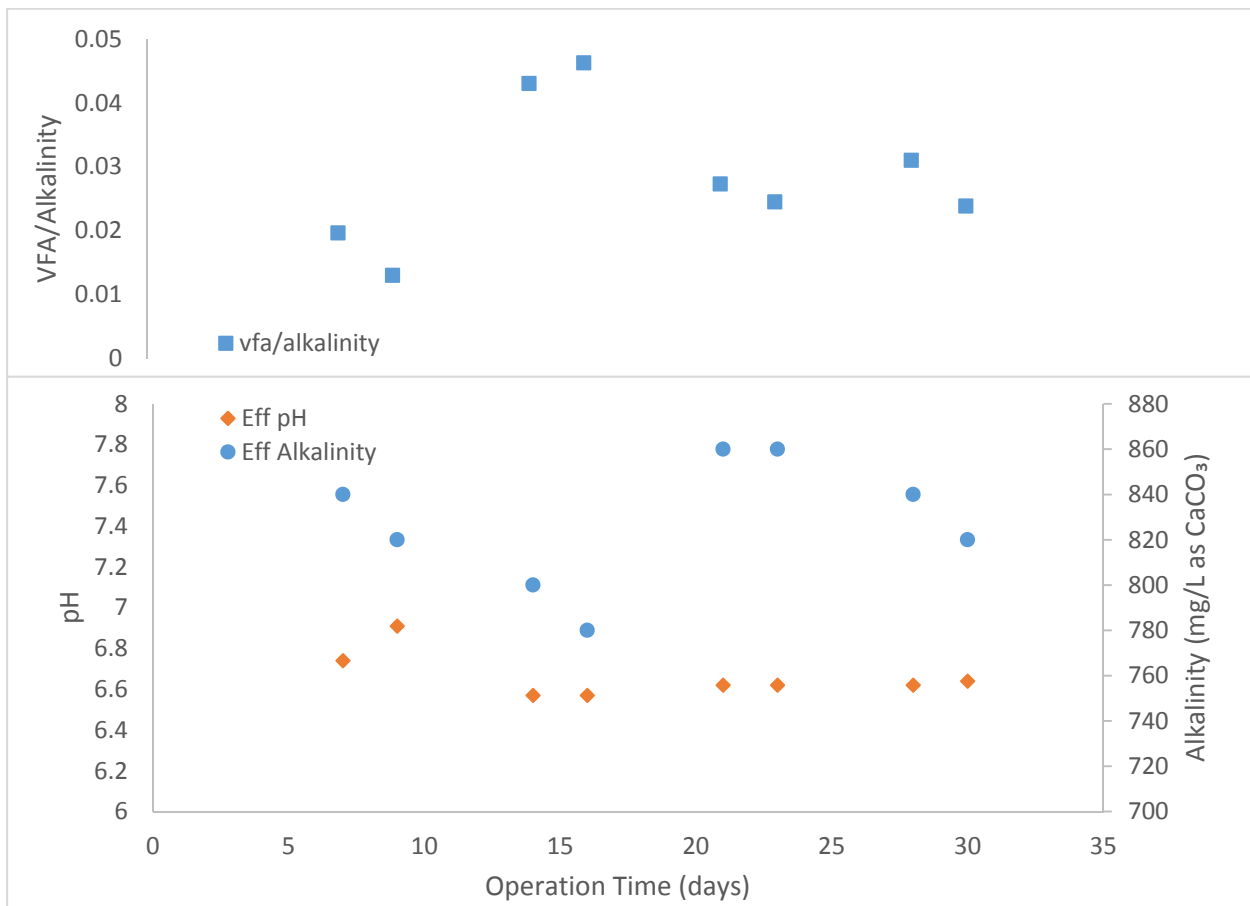
Figure 5.4: Pilot-scale SGBR variation of effluent pH, alkalinity and the VFA to alkalinity ratio

The SGBR system was able to treat and generate methane from a high particulate waste stream (67 ± 24% influent PCOD). At a 48 h HRT and an OLR of 3.3 ± 2.0 g TCOD/L·d the SGBR system produced a biogas with 70.5% methane, 25.8% carbon dioxide and 3.4% nitrogen. As demonstrated in Figure 5.5 the methane content in the biogas of the pilot-scale SGBR was 16% greater than the Ames WPCF digester treating sludge from the same source.
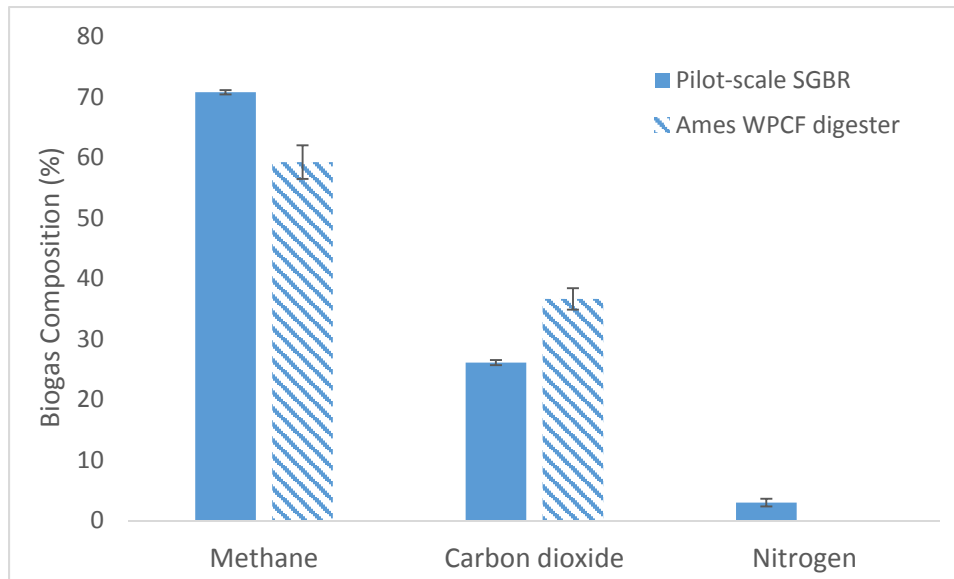
Figure 5.5: Pilot-scale SGBR and Ames WPCF digester biogas composition

The quantity of methane produced is an indication of the SGBR system's ability to degrade solids. The results presented in Figure 5.6 were attained from the solids mass balance as presented in Park et al. (2012). The COD mass balance results are demonstrated in Figure 5.7. The theoretical conversion ratio of 0.35 L CH_4/ g COD was used to estimate the COD consumed to generate methane. The methane production due to particulate degradation was estimated by subtracting the reduction in influent and effluent SCOD from the total methane production. Methane generation was measured daily and the dissolved methane in the effluent was calculated

using Henry's Law. The pilot-scale SGBR was not backwashed during the period analyzed, resulting in higher quantities of COD and solids unaccounted for. The unaccounted TSS could be a result of solids accumulated in the SGBR system, effluent that is supersaturated with methane, methane lost to the atmosphere, sulfate reduction and biomass growth.
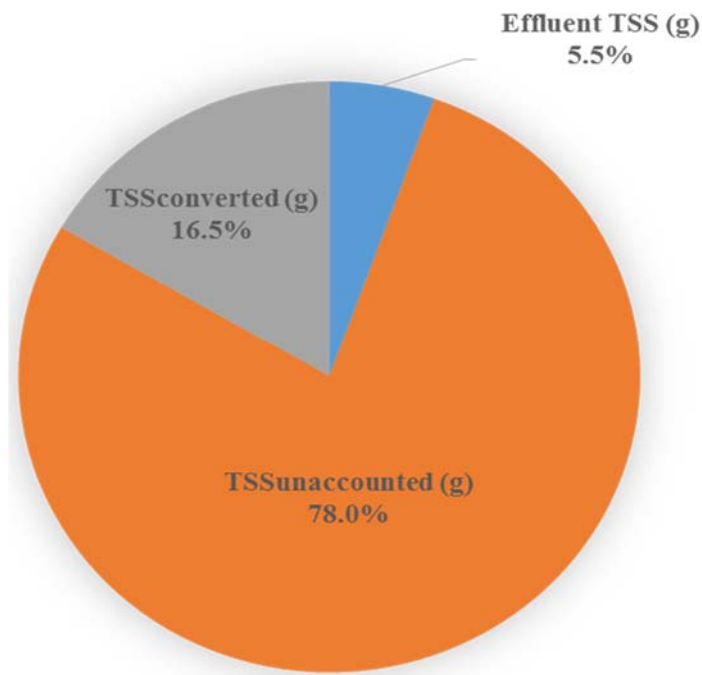
Figure 5.6: Pilot-scale SGBR solids balance
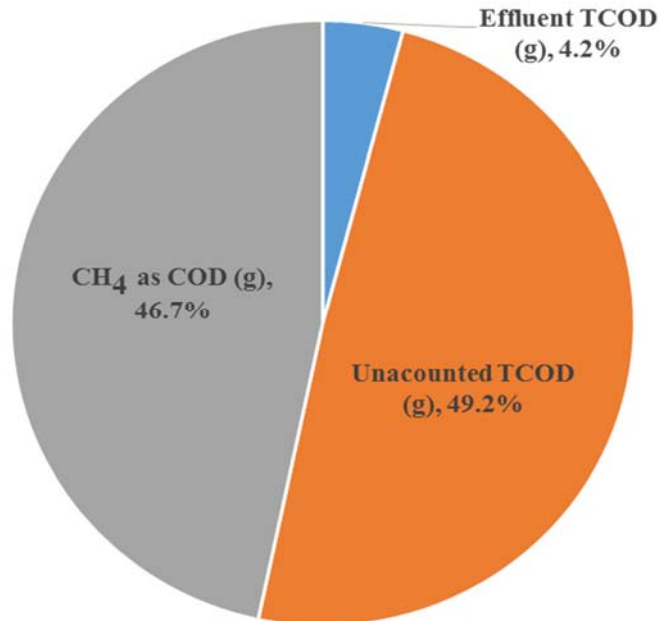
Figure 5.7: Pilot-scale SGBR COD mass balance

The measured cumulative biogas production is displayed in Figure 5.8. A leak was discovered on the 8th operational day, solids degradation was estimated using the average daily methane production after the leak was patched. The average methane production was estimated by separating the weekly operational days (4) and non-operation days (3).
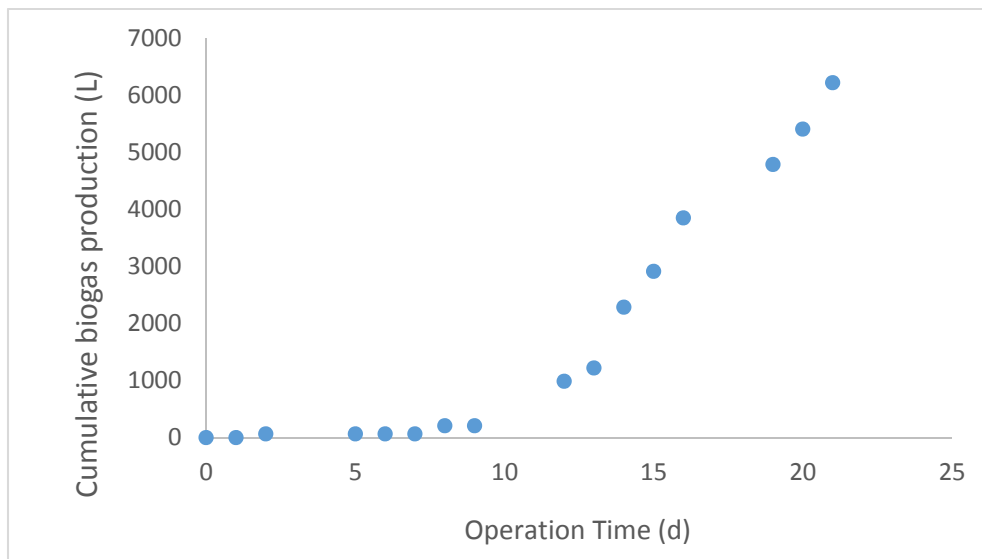
Figure 5.8: Cumulative biogas production

Conclusion

The pilot-scale SGBR showed the ability to degrade and remove solids from primary and secondary sludge. The pilot-scale SGBR has the potential to produce a biogas with a higher methane content than a conventional digester. The pilot-scale SGBR discharged effluent with low organic material, removing $91.9 \pm 7.0\%$ and $95.2 \pm 3.2\%$ of the influent TSS and COD, respectively. The effluent VFA concentrations were maintained between 13 and 34 mg/L as HAc. Low effluent VFA concentrations could potentially reduce process instability from the buildup of intermediates. The low effluent VFA concentrations of the SGBR can reduce odors compared to the liquid streams of conventional anaerobic digesters. 16.5% of the influent solids were converted to methane. The unaccounted COD could be a result of solids accumulated in the SGBR system, effluent that is supersaturated with methane, methane lost to the atmosphere during backwashing, sulfate reduction and biomass growth.

CHAPTER 6. ENGINEERING SIGNIFICANCE

The simple operation and construction of the SGBR system makes it an economical option for anaerobic treatment of wastewater. Ancillary heating and mixing are not required to operate the SGBR system. Due to the slow growth rate of methanogenic organisms it is standard practice to use large mixed and heated reactors in anaerobic digestion. The key to any efficient anaerobic reactor is to develop and maintain a large, stable, viable methanogenic population (Parkin and Owen, 1986). Maintaining a mature population of microorganisms inside of the anaerobic digester allows for a smaller reactor volume and a reduced HRT.

In a conventional anaerobic digester the feedstock is concentrated as much as possible to reduce the volume of the reactor. Dewatering of the sludge before the digester would not be required when using the SGBR, reducing operational costs. The average total solids concentration of the sludge in the Ames WPCF digesters was between 2.5 and 2.8%. The target solids concentration of the influent for this study was 0.2%. When treating sludge with the SGBR the solids could be pulled from the clarifier faster to reduce the solids concentration. Reducing the sludge bed height of the clarifier would also increase its solids removal efficiency. Reducing the height of the sludge bed reduces the possibility for solids to be re-suspended.

Table 6.1 compares conventional mesophilic anaerobic digestion of wastewater sludges with the SGBR. The SGBR has the potential to produce a biogas with higher methane content while operating at lower temperatures and HRT. The methane content of the biogas of a laboratory-scale SGBR operating at a 1 d HRT was up to 14% higher than the conventional digesters operating at 20 d HRT (Table 6.1). The SGBR showed the ability to consistently

produce an effluent with over 90% solids removal compared to the 30 to 50% solids removal

from conventional mesophilic digesters (Table 6.1). The effluent of the SGBR system had lower

VFA concentrations and potentially odor causing compounds than conventional digesters (Table

6.1). Offensive odors are a result of elevated VFA concentrations (Rudolfs and Heukelekian,

1930; Fisher and Greene, 1945; Han et al., 1997b). Conventional reactors are subject to process

upsets from the accumulation of VFA due to organic and hydraulic fluctuations. As the OLR

increased for this laboratory-scale study the effluent VFA concentration remained stable (i.e.

below 30 mg/L as HAc). The effluent VFA concentrations were an order of magnitude lower

than the VFA concentrations present for the mesophilic digesters presented in Table 6.1.

Table 6.1: Comparing anaerobic digestion of municipal sludges with laboratory-scale conventional digesters and the SGBR system

Source	Reactor configuration	HRT d	OLR g VS/L·d	VFA mg/L	Alkalinity mg/L as $CaCO_3$	pH	VS reduction %	Biogas methane %
Han and Dague, 1997a	Single-stage Mesophilic	10-15	2.1-2.9	180-300	5000-6100	7.0-7.4	32.5-46.8	67-71
Han et al., 1997b	Single-stage Mesophilic	24-40	1.2-0.8	200	NR	NR	32-47	65-72
Song et al., 2004	Single-stage Mesophilic	20	1.43	579±97	6412±545	7.67±0.1	43.5±8.4	64.7±2.6
Bolzonella et al., 2012	Single-stage Mesophilic	20	2.2	570±400	8400±790	7.8±0.1	36±3.5	63±5
This study	Ambient SGBR	1-2	0.85-3.4	15-24	429-531	6.62-6.64	NR	76±4

NR = Not reported

The heavy metal concentrations of the sludge treat by the SGBR is important to consider.

Heavy metals are non-degradable and can accumulate to inhibitory and toxic concentrations

(Sterritt and Lester, 1980; Chen et al., 2008). The extent of metal inhibition depends on the

soluble concentration and type of metal along with the distribution of microorganisms in the

digester (Mudhoo and Kumar, 2013). The physical and chemical properties (e.g. redox potential, electronegativity, solubility product of the corresponding metal sulfide complex, pearson softness index, electron density and covalent index) inside of the digester controls the toxicity of heavy metals (Workentine et al., 2008; Chen et al., 2014). Heavy metals can be precipitated in anaerobic treatment by the addition of sulfide (Kieu et al., 2011; Chen et al., 2014). The operating solids level has been shown to impact the toxicity of heavy metals, possibly due by providing protection from metal inhibition (Chen et al., 2008).The ions of heavy metals concentrations can be reduced by precipitation, sorption and chelation by organic and inorganic ligands (Chen et al., 2014).

Municipal sewage sludges can contain significant concentrations of heavy metals. The most common heavy metals include chromium (Cr), iron (Fe), cobalt (Co), copper (Cu), lead (Pb), zinc (Zn), cadmium (Cd) and nickel (Ni) (Mudhoo and Kumar, 2013). Lin et al. (1999) concluded the relative toxicities to anaerobic granular sludge were Cu>Cr>Cd>Zn>Ni>>Pb (1 d HRT) and Cu>Cr=Zn>Cd>Ni>>Pb (2 d HRT). The 50% inhibitory concentrations are displayed in Table 6.2. Copper had the highest toxicity of the metals tested (Table 6.2). The inhibitory heavy metal concentrations were dependent on the HRT. The heavy metal concentrations of the sludge to be treated with the SGBR should be quantified in order to select an appropriate HRT. Modifying the HRT of the system could also result in a change in the inhibitory concentration of certain heavy metals.

Table 6.2: 50% inhibition of methane production in a UASB reactor (mg/L) (Lin et al., 1999)

Metal	HRT 1 d	HRT 2 d
Cd	450	330
Cr	200	250
Cu	90	130
Ni	2000	1600
Pb	8800	8000
Zn	690	270

CHAPTER 7. CONCLUSIONS

A laboratory-scale SGBR was utilized to treat a high particulate waste stream (i.e. 94.5 ± 2.4% influent PCOD). During this study the laboratory-scale reactor operated at HRT and OLR ranges from 48 to 24 h and 2.8 to 5.5 g COD/L·d, respectively. The TCOD and TSS removal efficiencies of the SGBR were maintained above 90%. The backwash material displayed desirable settling characteristics with an approximate SVI of 50 mL/g. The effluent VFA concentrations were maintained between 11 and 30 mg/L as HAc. The biogas of the laboratory-scale SGBR was composed of 76 ± 4% methane (1 d HRT). The influent COD was removed by degradation (28%), backwashing (43%), effluent (6.5%) and 22% were unaccounted for.

The pilot-scale SGBR showed the ability to treat primary and secondary sludge on-site at the Ames WPCF. During this study the pilot-scale reactor operated at a HRT and an OLR of 48 h and 3.6 g COD/L·d, respectively. The average TSS and COD removal efficiencies were 91.9 ± 7.0% and 95.2 ± 3.2%, respectively. The effluent VFA concentrations were maintained between 13 and 34 mg/L as HAc. The biogas was composed of 70.5% methane and 16.5% of the influent solids were converted to methane.

Both the laboratory and pilot-scale SGBR demonstrated the potential to increase the composition of methane compared to the biogas of a conventional anaerobic digester. Low effluent VFA showed the potential to reduce process instability due to the buildup of intermediates and reduce odors compared to the liquid streams of conventional anaerobic digesters. Both the laboratory and pilot-scale SGBR discharged effluent with low organic material, the average TSS and COD removal efficiencies were maintained above 90%. The unaccounted COD could be a result of solids accumulated in the SGBR system, effluent that is

supersaturated with methane, methane lost to the atmosphere, sulfate reduction and biomass growth.

The SGBR performs like a bioreactor as well as a filter. Particulate matter can only be biologically hydrolyzed after becoming physically removed either by entrapment in the sludge bed or adsorption (Elmitwalli et al., 2001b). Particulate matter that is trapped in the granule bed of the SGBR can become hydrolyzed. Conversely the UASB reactor is limited by the influent solids concentration. Treatment of primary and secondary sludge with the SGBR offers the advantages of a long SRT and solids entrapment by the granule bed.

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