Enhancement of the Mesophilic Anaerobic Co-Digestion of Municipal Sewage and Scum

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ABSTRACT

Scum is an integral component of solids management in MWWTP and is composed of fats, oils, grease and other entrained floatable materials that are collected during primary clarification. Lab scale BMP tests showed the addition of 14.5 g VS/L of scum exhibited the greatest increase in biogas production of 1.6 times per g VS added compared to the control, while a higher additional scum loading of 33.7 g VS/L reduced the biogas yield to 32% of the control reactor. Lab scale semi-continuous digestion measured the effects of scum loading and temperature of pretreatment in the scum concentrator. At 15 d and 20 d HRTs the greatest observed improvement in biogas was achieved by adding 3% scum by volume and pretreating the scum at 70°C in a scum concentrator with respective improvements of 24% and 16%.

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Nomenclature

BOD	Biological Oxygen Demand
HRT	Hydraulic Retention Time
HT	Holding Time
TS	Total Solids
SRT	Solids Retention Time
F/M	Food to Microorganism Ratio
DO	Dissolved Oxygen
pH	Concentration of Hydrogen Ions in a Solution
VFA	Volatile Fatty Acids
C/N	Carbon to Nitrogen Ratio
VS	Volatile Solids
COD	Chemical Oxygen Demand
VA	Volatile Acids
LCFA	Long Chain Fatty Acids

List of Abbreviations

MWWTP	Municipal Wastewater Treatment Plant
TWAS	Thickened Waste Activated Sludge
PS	Primary Sludge
BMP	Biochemical Methane Potential
ROPEC	Robert O. Pickard Environmental Centre
SCADA	Supervisory Control and Data Acquisition
AD	Anaerobic Digestion
EPS	Extracellular Polymeric Substances
FOG	Fats, Oils and Greases
RL	Low Scum Containing Assays/Reactors
RH	High Scum Containing Assays/Reactors
CBP	Cumulative Biogas Production
HBSS	Hank’s Balanced Salt Solution
ANOVA	Analysis of Variance

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Chapter 1

Introduction

1.1 Background

Currently global energy consumption is slightly over 13 Terawatts/year with an estimated 80% of which is supplied by the burning of fossil fuels (Rittman, 2008). Concomitantly, society is facing an exponentially increasing global energy demand and declining availability of easily extractable oil. As environmental damages are becoming more evident to society through current climate change research as well as physical impurities exemplified through oil spills, North America is seeking technologies and methods to diminish oil dependence. Although it is unlikely that any one technology will encompass all the energy requirements provided through fossil fuels, the enhancement and development of several location specific renewable technologies has the ability to create a more sustainable society.

Municipal wastewater treatment plants (MWWTPs) in North America were developed in the 1800’s with the primary purpose of disease prevention. As population grew and sewer connections were enhanced new regulations and government grants were provided to protect receiving water quality in the 1940’s. Government investment in infrastructure led to secondary treatment process development to remove biological oxygen demand (BOD) and the development of centrifuges to remove water from sludges to decrease drying time. In 1960, 50% of the US population had access to some form of wastewater treatment creating an increased mass load of sludge to be disposed. Subsequently, the US Clean Water Act was put in place in 1977 to control all pollutant discharges including municipal sludge disposal. In 1999 the Canadian Environmental Protection Act was put in place to prevent pollution and protect the environment with focus on human health in order to contribute to a sustainable development. Through these regulations, one current trend is to recover energy (biofuels, co-generation, fertilizer) through MWWTP sludge stabilization, specifically with anaerobic digestion

The use of anaerobic digestion facilities treating several types of wastes has increased tremendously since the 1980's with over 1,000 vendor-supplied systems in operation worldwide. More than 35 industries have been identified as capable processors of waste including chemicals, fiber, meat, milk, pharmaceuticals, and high solid wastes (Lettinga, 1992). Operation of anaerobic digestion facilities are desirable through their ability to be a net energy producing process with around 75-150 kWh of electricity created per ton of waste stabilized depending on operational parameters. Heat recovery is also viable to lower associated heating costs for the facilities during the winter months.

Current trends in research to improve and enhance biogas production in anaerobic digestion facilities primarily include the pretreatment of thickened waste activated sludge (TWAS) prior to blending primary sludge (PS) and other wastes for digestion. Thermal pretreatment of TWAS at temperatures of 60°C and a hydraulic retention time (HRT) of 8 days leads to an increase of approximately 50% in methane generation (Wang, 1997). Further research has been conducted on microwave, chemical and ultrasonic pretreatment of TWAS to increase biogas production with varied results. Scum as a component utilized in anaerobic co-digestion at MWWTPs has not been subjected to the same detailed studies and requires further investigation to enhance biogas production of anaerobic digestion facilities and improve the net energy balance for MWWTPs.

1.2 Purpose

The purpose of this work is to investigate the effects of scum co-digestion with PS and TWAS on mesophilic WWTP anaerobic digestion and to characterize the effects of thermal pretreatment of scum on biogas production.

1.3 Research Objectives

The primary objective of this research is to investigate the effects of scum loading on mesophilic anaerobic co-digestion of PS and TWAS. Particularly, the work investigates the effects of scum concentration, thermal pretreatment temperatures and holding times (HT) of scum at specific temperatures on methane production and energy recovery by anaerobic digestion facilities.

Objectives specific to biochemical methane potential (BMP) assays were to:

- determine the effects of scum concentration on mesophilic anaerobic co-digestion of scum, PS and TWAS;
- determine the effects of scum pretreatment temperature on mesophilic anaerobic co-digestion of scum, PS and TWAS;
- determine the effects of scum HT at a set temperature on mesophilic anaerobic co-digestion of scum, PS and TWAS;
- investigate the methane yields for each waste;
- investigate operation parameters effected by scum addition.

Objective specific to the semi-continuous mesophilic anaerobic digestion reactors were to:

- determine the methane potential of co-digesting scum, PS and TWAS at 1.6 $kgVS/m^3 \cdot d$ at 15 and 20 d HRTs;
- determine the effects of scum pretreatment temperature on mesophilic anaerobic co-digestion of scum, PS and TWAS;
- investigate the effects of scum co-digestion on effluent quality;
- provide applicable recommendations for scum management at MWWTPs.

1.4 Thesis Layout

The thesis is comprised of 7 chapters, where Chapter 1 presents the introduction to the work. A literature review is given in Chapter 2, which describes the history of scum, collection of scum, anaerobic digestion parameters and reactor configurations, followed by research presented in published literature relevant to this thesis. Chapter 3 summarizes the methods and materials including all set up, operation, analytical, and statistical methods used in this study. Chapter 4 presents the results of the BMP assays in the form of a submitted journal article for publication. Chapter 5 presents the results of the semi-continuous tests in the form of a submitted journal article for publication. Chapter 6 is the projected biogas production at ROPEC with scum addition, which is followed by conclusions and recommendations for future work in Chapter 7.

Chapter 2

Literature Review

This chapter will describe the current knowledge of scum treatment in wastewater treatment plants. This includes a description of the composition of scum at various stages of the treatment process, the collection mechanisms, how it is disposed, and current research on the anaerobic digestion of scum.

2.1 What is Scum?

Scum is defined as a mass of sewage solids, buoyed up by entrained gas, grease, or other substance which floats at the surface of sewage (ASCE, 1928). The following section will define scum as it is produced throughout the wastewater treatment process. This includes the primary settling tanks, secondary treatment, and secondary settling tanks. The generation and constituents of the scum will be specifically addressed as well as the method of removal for each process.

2.1.1 Primary Scum

Primary scum consists of varying quantities of skin, soap, grease, vegetable and mineral oils, bits of wood, paper and cotton. It may also include adhesive bandages, plastic tampons applicators, and condoms (Outwater, 1994). As raw wastewater flows from sewage collectors to the wastewater treatment plant (WWTP), the wastewater flows to the screen and degrit chamber where the above listed constituents float to the surface of the wastewater and adhere to the grease and oils. Many of the solid constituents are not removed by the screens or degrit chambers due to their size and dimensions. The constituents then travel to the primary clarifiers where most plants begin the process of scum removal (Metcalf and Eddy, 2003).

2.1.1.1 Characteristics and Production of Primary Scum

Fats, oils, and grease act as the nucleus for scum formation because of their adhesive character and low specific gravity. Although scum is primarily composed of grease, it only accounts for approximately 10% of the raw wastewater influent grease concentration (Outwater, 1994). Typical WWTPs receive 100 mg/L of grease in the raw influent (Metcalfe and Eddy, 2003). Grease partitioning during primary treatment has been reported by Outwater to be 9% in the scum, 45% in the primary sludge (PS), and 46% in the primary effluent. These values may not appear intuitive, but since the scum production is approximately 4% of the total solids production per day, the values are sensible.

The dry weight of primary scum is variable and ranges from 0.1-19 mg/L with a median value of 5 mg/L. The chemical composition and quantity vary depending on wastewater source, recycled plant side streams, removal efficiency upstream and scum removal equipment (WEF, 2008). The total solids (TS) percentage of scum is also dependent on how long it has been accumulating in the treatment system. Scum that has just entered the queue will have a TS range from 2-10%, but just prior to exiting the system the TS in the same location can range from 5 to 65% (Outwater, 1994). This phenomena occurs due to the evaporation of water and coagulation within the scum as the material collects prior to trough cleansing. This will be described in more detail in section 2.1.1.2.

The volatile solids (VS) portion of scum is typically 94-98%. The VS content of PS and thickened waste activated sludge (TWAS) range from 75-80%. (Metcalfe and Eddy, 2003). The VS content of scum is higher than PS and TWAS as it consists of mostly organic constituents.

Plastics are a major problem in wastewater treatment plants because they are non biodegradable. The majority of plastics in scum enter the system as toothbrush handles and tampon applicators (Outwater, 1994). Plastic tampon applicators have the structural stability to survive throughout the treatment process and hence their presence is particularly undesired. A study performed on the wastewater treatment plant in Boston, Massachusetts reported approximately 50,000 plastic tampon applicators entered the treatment plant daily. Screens and scum collection are able to capture the plastic tampon applicators (Outwater, 1994). The importance of tampon applicator destruction is described in section 2.3.

Scum production and properties is region and plant specific depending on demographic, industry, and sewer system (combined or separated). As a high fat and lipid containing residual, regions with more fried items or high lipid containing industries will generate more scum. Table 2.1 illustrates the reported scum production for 19 plants located throughout the United States of America. As illustrated, regions such as Georgia and Missouri, which are known for deep fried foods and industries generated among the highest quantities of scum (Mg/L). Conversely, Illinois and Wisconsin, which are more agricultural and vegetable oriented societies generated less quantities of scum. The wide variation in dry weight and percent solids shows the non-uniform nature of scum and the necessity of each plant to access the residual prior to devising a management plan. An empirical approach is thus recommended in all cases.

Table 2.1 – Reported scum production and properties (Outwater, 1994)

Treatment Plant	Volume (m^3/ML)	Dry Weight (mg/L)	% Solids
N.W Bergen Co. NJ	0.247	2.3	9
3 NYC Plants	0.003-0.005	0.1-2.0	40-50
Jamaica, NYC	0.224	1.2	-
Wards Island, NY	-	4.8	-
Passaic Valley, NJ	0.187	6	-
Calumet, IL	0.030	1.8	63
Southwest, IL	0.090	5.3	61
West side, IL	0.082	4.8	62
North side, IL	0.022	0.5	53

Detroit, MI	-	3	-
Minneapolis, MN	-	-	-
Milwaukee, WI	-	3.1	-
San Mateo, CA	-	11	-
LA, CA	-	10	-
Sacramento, CA	-	14.4	-
East Bay, CA	0.239	9.8	52
Seattle, WA	0.500	2.9	6
St. Louis, MO	-	10.5	-
Albany, GA	-	17	0.6
Averages	0.135	5.9	40.1
Range	0.003-0.500	0.1-17	0.6-63

2.1.1.2 Collection of Primary Scum

Scum may be laden with pathogens and as such technicians and consultants should be cautious when handling scum during the collection process. As with primary clarifier design, scum collection systems are designed such that the prevailing wind blows away from residential areas as scum is particularly odorous due to the breaking down of the fatty acids it is composed of (Mahlie, 1940).

The removal of scum in primary clarification takes place at the end of rectangular clarifiers or by sweeping arms in circular clarifiers. Vesilind (2003) has shown that attempts made to remove scum at the influent of rectangular clarifiers to reduce the travel time of scum were unsuccessful as build up prior to entry caused more problems than an increased travel time. The two most common units for scum collection are the tilting trough and sloping beach as shown in figures 2.1 and 2.2 respectively.

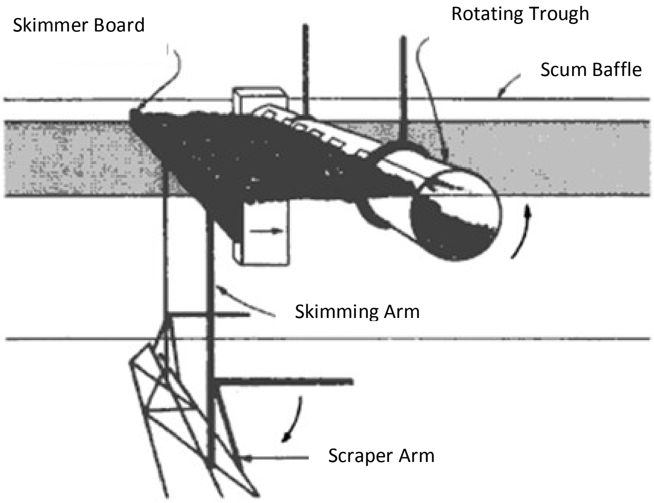

Figure 2.1 – Tilting trough mechanism for scum collection (adapted from Vesilind, 2003)

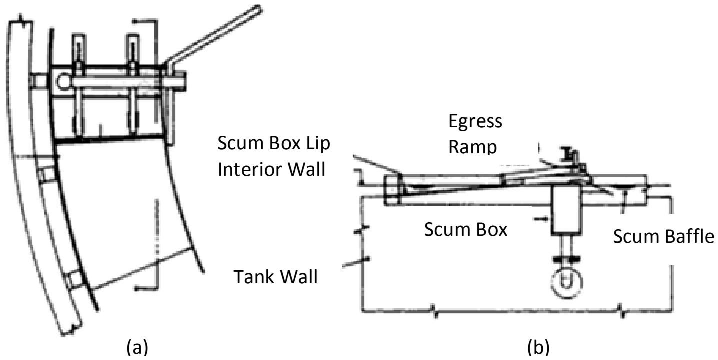

Figure 2.2 – Sloping beach mechanism for scum collection; (a) plan view, (b) profile view (adapted from Vesilind, 2003)

The tilting trough mechanism can be automated through a supervisory control and data acquisition (SCADA) system or manually operated. The mechanism works such that the scum flows on the surface of the primary clarifiers and is stopped at the effluent end by a trough with v-notch weirs. Over time the scum accumulates behind the weirs creating a visible scum layer with measurable length and depth. At a specified interval or by employing a manual override the trough is tipped to allow scum to enter the trough through the v-notch weir and travel to a scum pot. The flow through the trough is achieved by gravity. Once the scum is collected in the scum pot the steps of removal are dependent upon plant design and will be discussed in the next section.

The sloping beach is a stationary device with a collector trough as seen in Figure 2.2. Scum is directed to the trough with water sprays, air sprays, or a blade type scraper that systematically pushes the scum into the trough. The scum is flushed from the trough to a scum pot where steps are taken to remove the scum from the system (Outwater, 1994).

There are several variables that effect the removal of scum in the mechanisms described. Optimal primary tank configuration demonstrates continuous primary clarifier baffling at the outlet and baffles that are deep enough to prevent scum from traveling underneath the baffles. The characteristics of the raw wastewater also effects scum removal, such that wastewaters produced during warmer summer temperatures or at pH values less than 7 results in scum that may stay in suspension and enter and be captured in the settled sludge or not captured and flow to the secondary treatment basins. Performance for scum removal is typically measured by the amount of scum present in the primary effluent channel (WEF, 2008).

2.1.2 Secondary Scum

Secondary scum is defined as the floating grease or foam that occurs during secondary treatment. Scum formed due to grease is less common than in primary clarification and plants rarely have process control problems during secondary treatment (Mahlie, 1940). Activated sludge systems typically have sufficient biomass concentrations that are able to adhere to the remaining grease (~46 mg/L) and settle out during secondary clarification (Vesilind, 2003).

The major antagonists to process operation for secondary treatment in regards to scum are foaming microorganisms (Jolis and Marneri, 2006). In anoxic zones, the scum will be thick and viscous whereas scum in the aerated zones will be a light foam (Handel, 2007). Foams are generally characterized by their color as seen in table 2.2.

Table 2.2 – Description and cause of activated sludge foams (Richard, 2003)

Foam Description	Cause(s)
Thin, white to grey foam	Low cell residence time or “young” sludge (startup foam)
White, frothy, billowing foam	Once common due to non biodegradable detergents (now uncommon)
Pumice-like, grey foam (ash)	Excessive fines recycle from other processes (e.g. anaerobic digesters)
Thick sludge blanket on the final clarifiers	Denitrification
Thick, pasty, or slimy, grayish foam (industrial systems only)	Nutrient-deficient foam: foam consists of polysaccharide material released from the floc
Thick, brown, stable foam enriched in filaments	Filament-induced foaming, caused by *Nocardia, Microthrix* or type 1863

The removal of foaming scum is achieved by adjusting the solids retention time (SRT), food to microorganism (F/M) ratio, dissolved oxygen (DO) concentration, or other activated sludge process operational parameters (Jolis and Marneri, 2006). Chlorination of the sludge may also be used to reduce foaming. Secondary scum is rarely collected on a regular basis and is therefore outside the scope of this project.

2.2 Disposal of Scum

Historically scum has been dewatered, dried, and sent to the landfill for final disposal with other biosolids (Vesilind, 2003). This method has been utilized due to facility restrictions (i.e. no anaerobic digesters) and its ease of operation. Unfortunately, landfilling scum can have a negative impact on the environment due to its high organic composition. The negative environmental impacts have led to several other viable methods of disposal including incineration, chemical fixation, and anaerobic digestion. Each method will be discussed in the following sections.

2.2.1 Incineration

The practice of incinerating scum began in 1939 with the Minneapolis-St Paul Sewage Treatment Plant where two lagoons were alternately filled and set afire. This practice was done for 25 years until air quality emission standards prohibited the practice. Incinerators have since been developed to decrease air pollution emissions and provide a net positive energy value (Outwater, 1994).

In order to meet the regulatory standards scum incineration facilities must utilize the fluidized bed incinerator because it is the only technology able to capture the necessary amount of NOx and decrease the organic smell generated by the exhaust (Sapienza, 1994). The fluidized bed incinerator operates by placing the sludge on top of the silica sand in the incinerator, which is heated to promote uniform combustion. The water in the sludge is then evaporated instantly while the remaining solids are incinerated through contact with the hot air. The sand stabilizes the system as the sludge turns to ash; it is collected as dust and subsequently disposed of as hazardous materials (Sapienza, 1994).

Although incineration does provide a sustainable approach of disposing scum it requires high capital costs and is often subject to public debate. Several municipalities have experienced public demand to close down furnaces making the fluidized bed incinerator a risky investment for scum disposal (Outwater, 1994).

2.2.2 Chemical Fixation

Liquid scum can be chemically stabilized and converted into a pasteurized, odorless, soil-like material through mixing it with cement kiln dust, lime kiln dust, or ground lime. Cement kiln dust is a preferred mixing agent because it is the by-product of cement manufacturing thus increasing the beneficial reuse of residuals (Outwater, 1994).

This process was used at the Deer Island Treatment Plant in Boston, Massachusetts where scum was chemically fixed with cement kiln dust. The process mixed 20 to 75% cement kiln dust by weight and placed the mixture into a curing basin for 1 to 4 weeks. The high pH of the mixture effectively killed all pathogens and eliminated most odors. Once the mixture is stabilized it can be used as landfill cover or embankment fill (Outwater, 1994).

The primary concerns with chemical fixation are the release of ammonia, improper curing, and low concentrations of solids. Although the process does not provide a net energy gain, it is considered a beneficial reuse with low capital investment. With increased product control, chemical fixation can be considered a viable scum processing alternative for treatment facilities that do not have the capability of anaerobic digestion (Outwater, 1994).

2.2.3 Anaerobic Digestion

Anaerobic digestion (AD) of scum is a process where the grease in scum is biologically converted to methane and carbon dioxide under anaerobic conditions. The digestion of scum requires a minimum contact time of 8 to 10 days before it begins to break down as opposed to 4 to 5 days for PS and TWAS. It is likely that for maximum digestion of the co-processed sludge, scum digestion will be the rate-limiting step (Outwater, 1994).

Treatment facilities may harvest the produced methane from the anaerobic digesters using onsite cogeneration facilities. The energy created lowers the energy burden of the facility or may be supplied to the electrical grid. Anaerobic digestion decreases the quantity and stabilizes the sludge, which aids in disposal. These attributes have made anaerobic digestion a popular method of treatment for all types of biodegradable wastes, including scum. The following section will discuss anaerobic digestion in detail.

2.3 Anaerobic Digestion Process

The following section will discuss in detail the anaerobic digestion (AD) process of wastewater sludges and scum. Specifically the process steps, operating parameters, and common types of anaerobic digestion will be presented.

2.3.1 Process

The AD process is the microbial driven biodegradation of organic matter in the absence of oxygen. The process is conducted by several different microbial communities operating in a symbiotic relationship to achieve hydrolysis/liquefaction, acidogenesis, and methanogenesis. The three stages are described in detail below.

2.3.1.1 Hydrolysis/Liquification

Hydrolysis is the first step of AD processes. Hydrolysis converts particulate matter that enters the system to soluble compounds that subsequently may be hydrolyzed further to simple monomers. This process is done by hydrolytic enzymes (lipases, proteases, cellulases, amylases, etc.) secreted by microbial communities. This process is especially important in the digestion of organic waste and may become the rate limiting step of AD (RISE-AT, 1998). Once the complex polymers have been hydrolyzed, the simple monomers can then be fermented by the bacteria (Metcalf and Eddy, 2003).

2.3.1.2 Acidogenesis

Acidogenesis, also referred to as fermentation, is the process where the products of hydrolysis are used as the electron donor and acceptor to convert the substrate to simple organic acids, carbon dioxide and hydrogen. The primary acids produced during acidogenesis are acetic acid (CH_3COOH), propionic acid (CH_3CH_2COOH), and butyric acid ($CH_3CH_2CH_2COOH$). Propionic and butyric acid are not readily available for methanogenesis so they are further decomposed to produce acetate, hydrogen, and carbon dioxide. Acidogenesis is typically not rate limiting, but hydrogen partial pressures must be below 10^{-4} atmospheres or the system can become toxic. Typical acidogenic bacteria in the AD process are *Peptococcus anaerobes, Bifidobacterium* spp., *Desulphovibrio* spp., *Corynebacterium* spp., *Lactobacillus, Actinomyces, Staphylococcus,* and *Eschericia coli* (Metcalfe and Eddy, 2003).

2.3.1.3 Methanogenesis

Methanogenesis is the process in which the acetic acid and hydrogen formed during acidogenesis are utilized by methanogenic archaea to create methane and carbon dioxide. In a typical AD system approximately 72% of the methane is generated from acetic acid and 28% from hydrogenotrophic methanogens (Metcalfe and Eddy, 2003). Typical methanogenic archaea in the AD process are *Methanobacterium, Methanobacillus, Methanococcus,* and *Methanosarcina.* It is important to note that *Methanosarcina* and *Methanothrix* are the only organisms able to use acetate to produce methane and carbon dioxide, the others use hydrogen (Kennedy, 2010). Methane is formed according to the following reactions (Metcalfe and Eddy, 2003):

$$CH_3COOH \rightarrow CH_4 + CO_2 \qquad \text{Equ. 2.1}$$

$$4H_2 + CO_2 \rightarrow CH_4 + 2H_2O \qquad \text{Equ. 2.2}$$

As mentioned before the archaea live in a symbiotic relationship, thus the system operates only if all organisms are present. The hydrogenotrophic methanogens consume hydrogen, which maintains an extremely low hydrogen partial pressure ensuring the system does not become toxic. This effectively acts as a hydrogen sink to speed up the fermentation reaction. If however, there are not enough hydrogenotrophic methanogens to maintain a low hydrogen partial pressure, the fermentation of propionic acid and butyric acid would be slowed and volatile fatty acids (VFA) would accumulate. This would effectively reduce the pH and potentially create an environment unsuitable for microbial growth in the reactor (Metcalfe and Eddy, 2003).

2.3.2 Operational Parameters

The operational parameters of an AD system are the determining factor for the rate of biodegradation. The AD system should be operated to enhance the microbial growth such that the digester can be more efficient in biodegradation of the substrate and reduce the likelihood of inhibitory conditions. The waste composition, pH that controls the AD process, mixing, temperature, and carbon to nitrogen ratio (C/N) are specific operational parameters discussed in the following subsections.

2.3.2.1 Waste Composition

WWTP anaerobic digesters are fed with PS, TWAS, and depending on the facility scum. Each component has a slightly different composition, which inherently affects its digestion properties.

PS is composed of 90% particulate matter, thus it is difficult to initiate hydrolysis/liquefaction and therefore hydrolysis is the rate-limiting step in digestion. The hydrolysis step can be modeled using first order kinetics and exhibits growth through a lag phase followed by exponential growth, stagnation, and eventual degradation. The full digestion of primary sludge is complete in approximately 16 days at 25^{o}C and 8 days at 35^{o}C (Speece, 2008).

TWAS is composed of hydrogenotrophic microbial cells, and extracellular polymeric substances (EPS) that form a floc matrix that offers protections from hydrolysis by the interaction between the EPS, the cells and their cell walls. The interaction of cells within the EPS matrix limits the hydrolysis rate, which decreases the anaerobic biodegradability of TWAS. It is estimated that after 20 days at 35^{o}C there is 47-52% of COD remaining. This leads to a relatively large quantity of biomass remaining after a 20 day digestion period (Speece, 2008).

The last portion of wastewater treatment digestion is from grease. It is important to note that grease is collected as scum, but grease is also entrained in solids collected as PS and TWAS. A significant portion of the methane production is from the anaerobic biodegradation of grease (Speece, 2008). Approximately 30% of the grease entering the AD system is already hydrolyzed or discharged as long chain fatty acids, which suggests hydrolysis is the limiting step. Operating under mesophilic temperatures grease VS destruction is 0, 60, and 92% at retention times of 6, 10, and 15 days respectively.

2.3.2.2 pH Level

pH levels vary in the AD process depending on the process taking place. Acidogenic reactions prefer a slightly acidic environment (5.8-6.2), whereas methanogenic reactions prefer a neutral pH (6.8-7.2). If the pH drops below 6.0, methanogenesis will be inhibited while acidogenesis will be reacting near its maximum specific substrate utilization rate. This will cause a further decrease in pH and eventual fowling of the system. Similarly, if the pH increases beyond 7.6, acidogenesis will become inhibited and the methanogens will not have usable substrate to create methane (Kennedy, 2010). Digesters should be monitored to ensure the pH does not fall outside of these ranges. If the substrate rapidly biodegrades, VFAs will be produced faster than consumed so the pH will decrease. Alkalinity is added to the system when needed in the form of calcium carbonate to reduce pH fluctuations. Calcium carbonate is inert to the system so it does not affect the digestion process (Speece, 2008).

2.3.2.3 Mixing

Anaerobic digesters are continuously mixed for two primary functions. Continuous mixing precludes the development of dead zones of scum and non-mobile sedimentation within the digester. If mixing is not adequate a scum layer will form decreasing the digesters active volume. The second function of mixing is to ensure the feed is mixed with mature cultures of the methanogenic microbial consortia in the reactor. If the methanogenic archaea are not mixed they have been observed to “clump” which does not allow for adequate hydrogen consumption and can result in accumulation of VFAs and digester souring (Speece, 2008).

Adequate mixing is a function of recycle streams and continuously operated stirring mechanisms (mechanical or hydraulic) in the digester. Egg shaped digesters increase the active volume of the reactors by decreasing the amount of surface area at the top of the digester. The smaller surface area at the top of the egg combined with more intense mixing helps control the buildup of scum compared to pancake type digesters. However, since the egg digesters are emptied from the bottom, the scum layer cannot be removed unless a full cleaning is done. This requires a shutdown of the reactor and is costly (Outwater, 1994; Speece, 2008). Figure 3 shows a schematic for the mixing patterns in a conventional digester and egg shaped digester.

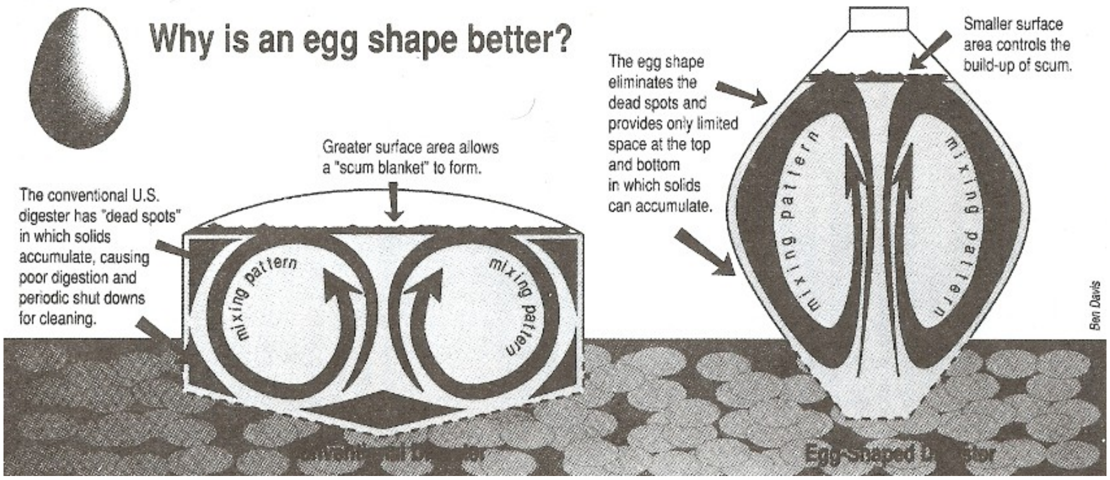

Figure 2.3 – Mixing patterns in conventional and egg shaped digesters (adapted from Outwater, 1994)

2.3.2.4 Temperature

Temperature has a strong impact on the methanogenic consortia present and their kinetic rates. Anaerobic digestion can be operated at a wide variety of temperature ranges, but mesophilic and thermophilic ranges are the most economically viable options.

Mesophilic operation is defined as a digester whose internal temperature is between 25 to 40°C with an optimal value of 35°C. Operating under mesophilic temperatures encourages an environment where the microbial consortia are able to achieve high VS reduction with low VFA concentrations. The heating requirement is lower than thermophilic and because of a more heterogeneous microbial consortia it has been reported to be more operationally stable. Mesophilic digested sludge is typically class B biosolids which can be land applied to grain fields, but are subject to several restrictions. Typical digestion time is 15 days (Speece, 2008).

Thermophilic operation is defined as a digester whose internal temperature is greater than 50°C with a typical value of 55°C. Operating under thermophilic conditions encourages a microbial consortia that have a high specific substrate utilization rate, which allows them to degrade the substrate faster than digesters operating in mesophilic condition. The heating requirement for thermophilic operation is high and the system is less stable to due to rapidly increasing VFA concentrations related to a less heterogeneous microbial consortia. The digested sludge quality is typically class A biosolids, which are not subject to the same restrictions as class B biosolids. Typical digestion time is 3 to 7 days, which offsets some of the heating costs in comparison to mesophilic digestion (Speece, 2008).

Mesophilic and thermophilic digesters have their advantages and disadvantages, but due to ease of operation and lenient biosolids land application regulations in Canada most treatment facilities operate under mesophilic condition. The reactors are much more stable, which decrease the risk of having an unexpected fowling of a digester and stressing the entire system with an increased loading rate to the operating digesters (Kennedy, 2010).

2.3.2.5 C/N Ratio

The carbon to nitrogen ratio is the relationship between the amount of carbon and nitrogen present in the AD system with an optimal C/N ratio of 50/1. A higher

C/N ratio indicates that the methanogens are consuming nitrogen and producing less gas. A lower C/N ratio indicates ammonia accumulation which can increase the pH to inhibitory levels. The C/N ratio is checked periodically and if the system is out of the normal range operators can mix waste to correct the balance (Kennedy, 2010).

2.3.3 Types of Anaerobic Digestion

Anaerobic digestion can be performed as a batch, semi-continuous, or continuous reactor process. This section will describe the three systems along with their advantages and disadvantages.

2.3.3.1 Batch Digesters

Batch digesters are operated by combining an anaerobic inoculum and waste in a sealed container. If possible the vessel should be flushed with carbon dioxide and nitrogen prior to capping to ensure an anaerobic environment. The anaerobic inoculum is typically taken from an active digester to act as a culture for acidogenic and methanogenic microbes.

The biogas generated is drawn from the system and used for cogeneration to provide electricity for the treatment plant or electrical grid. In general 0.395mL CH_4 of biogas is produced per 1 mg of chemical oxygen demand (COD) reduced (Moody et al. 2010). The daily biogas production will vary throughout the process and typically peaks at 15 to 20 days.

Batch digesters are simple to operate because the sludge is pumped to a container until the desired volume is achieved. The system is then allowed to naturally degrade for 30 to 45 days and the remaining solids are pumped out of the system. Fowling is typically not an issue if the system is supplemented with alkalinity to prevent the pH from reaching inhibitory levels (Moody et al.2010)

The biogas production in batch digesters, as mentioned, is not constant so energy output will vary daily. If the treatment facility relies on the energy production, a series of batch digesters should be used at different digestion periods to create semi-constant daily biogas production. Batch digesters also require more cleaning maintenance. After the digestion cycle is completed the remaining solids will adhere to the walls and not be fully pumped out of the system. This will lead to dead space in the digester and require full cleaning. Figure 4 shows a schematic of a batch digester (Delatolla, 2011)

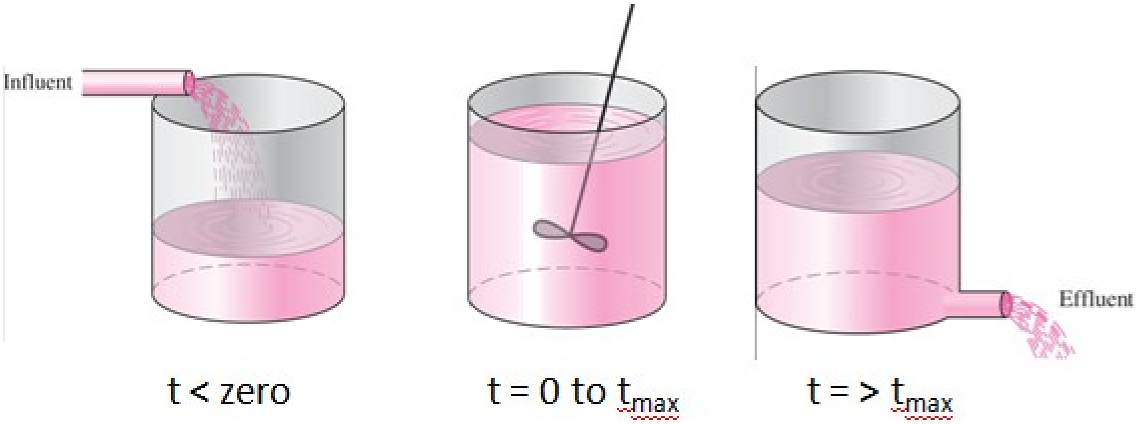

Figure 2.4 – Schematic of a batch anaerobic digester (Delatolla, 2011)

2.3.3.2 Semi-Continuous and Continuous Digesters

Semi-continuous digesters are operated by combining inoculum and waste to a sealed vessel in the same method as the batch system. The semi-continuous digesters will then be allowed to digest the substrate until the desired retention time is reached, typically 12 to 15 days. Digested sludge is then drawn from the system in a known quantity and an equal value of fresh sludge is added to the system. The loading rate of the new sludge should be between 1.6 to 2.2 kg VS / m^3 to avoid excessive VFA production. After the system has been in operation it should achieve steady state biogas production. The removed sludge from the system is tested for VFA, pH, alkalinity, COD, and VS regularly to determine the digester health. These systems are more applicable to lab scale for modeling a fully continuous system as they require more manual control.

Continuous digesters operate and are maintained in the same methodology as the semi-continuous digesters, but are constantly fed and drawn at the same rate. These systems are the most common in treatment facilities due to their low maintenance cost and consistent biogas production. The disadvantages of continuous systems include their difficulty and time requirement to reach steady state. This can pose a problem if a reactor fowls and the backup digester is overloaded for a lengthy period of time. Figures 5 and 6 illustrate schematics of semi-continuous and continuous systems (Delatolla, 2011).

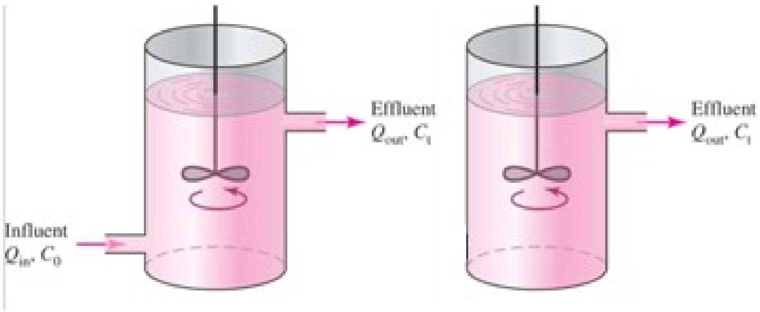

Figure 2.5 – Schematic of semi-continuous anaerobic digesters (Delatolla, 2011)

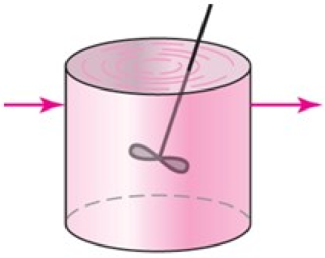

Figure 2.6 – Schematic of continuous anaerobic digester (Delatolla, 2011)

2.4 Anaerobic Digestion of Scum Research

The following chapter will discuss research that has been conducted on the anaerobic digestion of primary scum. There hasn't been a large quantity of research conducted on scum itself so the discussion will be extended to fats, oils, and greases (FOG). This chapter will assess the chemical composition of grease during biodegradation, batch digestion, and continuous digestion of primary scum and FOG.

2.4.1 Chemical Composition of Grease During Biodegradation

According to the Standard Methods (APHA) of water and sewage analysis, grease is defined as "fats, waxes, free fatty acids, calcium and magnesium soaps, mineral oils, and other non-fat substances" (Mahlie, 1940). As previously defined in section 1, scum is composed of a combination of the preceding depending on the influent of the plant. Chemical analysis on the grease skimmed from the primary clarifiers of seven plants across the United States determined the grease can be modeled as a glycerol ester with chemical composition $C_3H_5(C_{17}H_{35}COO)_3$. Using this as the digestible component of scum the theoretical process would be as follows (Mahlie, 1940; Speece, 2008):

$$C_3H_5(C_{17}H_{35}COO)_3 + 3H_2O + lipase = C_3H_5(OH)_3 + 3C_{17}H_{35}COOH \quad \text{Equ. 2.3}$$

$$4C_3H_5(OH)_3 = 5CO_2 + 7CH_4 + 2H_2O \quad \text{Equ. 2.4}$$

$$C_{17}H_{35}COOH + 8H_2O = 5CO_2 + 13CH_4 \quad \text{Equ. 2.5}$$

2.4.2 Batch Digestion

Although the chemical composition of scum will vary slightly, the chemical reactions presented above explain the process scum undergoes during biodegradation in anaerobic systems including anaerobic batch digesters. Batch biological methane potential (BMP) studies of FOG confirm that methane is produced during biodegradation. Kabouris (2008) studied the codigestion of pure FOG obtained from restaurant grease traps with sludge. This study showed that reactors fed with a 10% FOG component based on VS loading produced 25% more methane than reactors fed without FOG over a digestion period of 45 days. The FOG fed reactor achieved 80% of its maximum VS destruction after 11 days. The increased biogas and high VS destruction suggests that pure FOG is readily biodegradable and produces a high quantity of gas per unit of volume or mass.

The ultimate biogas yield can be determined by extending a BMP test to over 100 days. Figure 2.7 shows the various ultimate gas yields according to the EPA's Biodiesel Partnership program (Hansen, 2006).

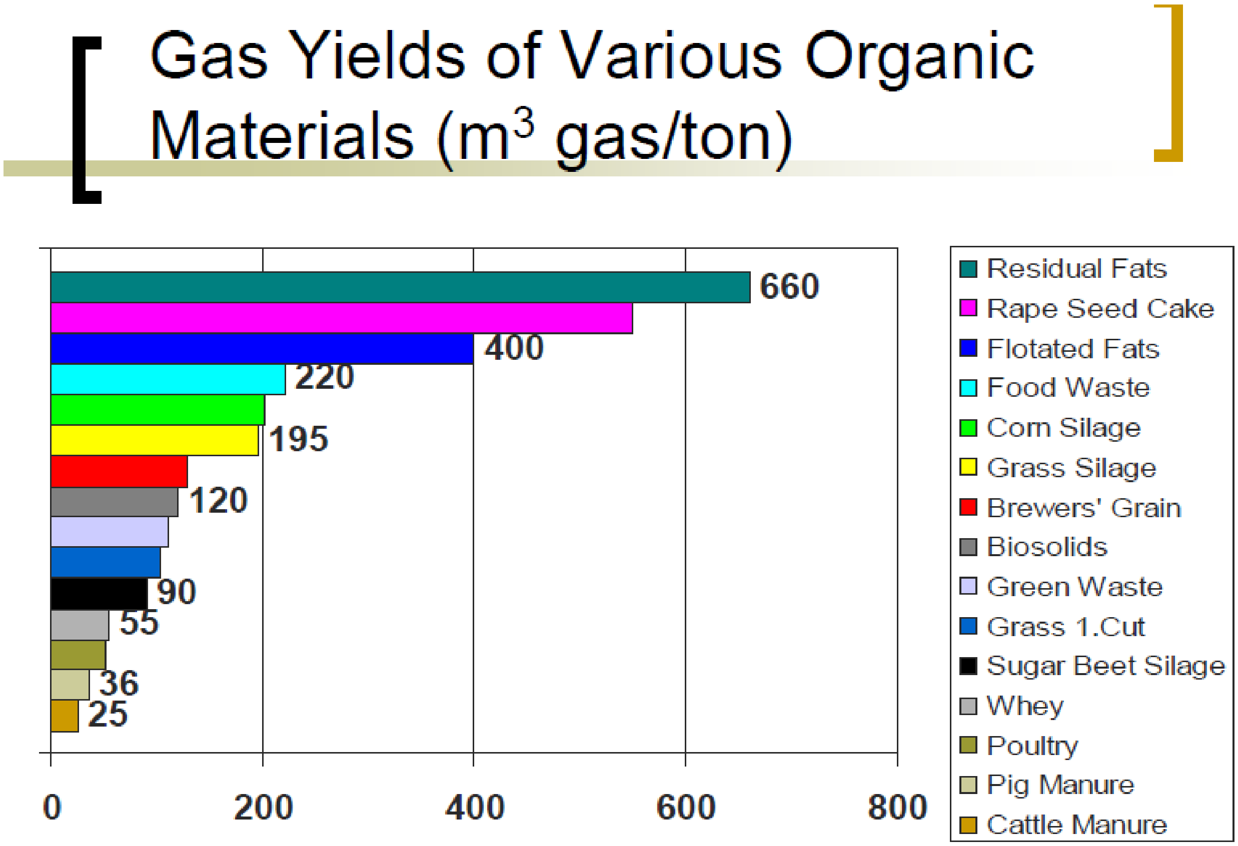

Figure 2.7 – Ultimate BMP of various organic wastes (Hansen, 2006).

The major constituent in scum is a combination of residual and flotated fats, which produces over 600% more biogas per ton of waste in comparison to sludge biosolids. Given that FOG is readily biodegradable the treatment facility should expect an increase in biogas production as scum is added to the digesters.

2.4.3 Continuous Digestion

Since most treatment plants operate continuous anaerobic digesters, the actual behavior of FOG and scum in a lab scale continuous system is vital to the reactors performance. As mentioned before, continuous reactors are more susceptible to fowling and have difficulty reaching steady state if the system is not operating properly.

Due to FOG and scum being readily biodegradable, VFAs and hydrogen production will increase which can cause a decrease of the pH to inhibitory levels. Lab scale continuous reactors have shown similar results that have indicated the addition of 10% scum by volume decreases the pH level to inhibitory levels as acidogenesis occurs at a faster rate than methanogenesis. This problem can be solved by the addition of a base (Gomec, 2006) or adding alkalinity in the form of calcium carbonate (Kabouris, 2009). Both solutions are inert to the system and are able to provide pH control to the system.

If pH is controlled at a level of 6.5 or higher continuous reactors are able to achieve steady state and consume VFA to produce methane on average within 4 days of operation (Gomec, 2006). Complete VFA consumption has been observed as early as 11 days, which lessens the digestion time requirements for the treatment facilities. This also leads to a higher percentage of degradable COD destroyed in the system at normal retention times (Kabouris, 2009).

The digestion of FOG and scum does provide complications to the system if it is not adequately mixed. In conventional digesters the surface area is relatively large creating likely areas that are not strongly mixed. In these areas the scum will form an impermeable layer that can grow to several meters thick and reduce digester efficiency (Outwater, 1994; Downey, 2006). This problem is difficult to solve, and has led to many plants landfilling their primary scum (Downey, 2006). Conventional digesters are not designed properly for adequate mixing of large quantities of scum and FOG, but an egg shaped digester (shown in figure 4) greatly reduces the surface area and likelihood of a problematic scum layer. Plants that are designed with an egg shaped digester are able to utilize the technology and digest primary scum and FOG for the energy benefits (Downey, 2006).

2.5 Conclusion

The process of scum entering the wastewater stream to collection and disposal is well documented through textbooks and process manuals. The research in beneficial reuse of scum is lacking that of other organic solids such as the organic fraction of municipal solids waste, airplane deicing fluids, and animal waste. Although research has shown the biodegradability and potential for scum digestion, specific well organized research has not been conducted. From the literature it is evident that the digestion of scum will require the addition of alkalinity, but biogas production should increase with concentration.

From the literature it is evident that mixing will be an important parameter for ensuring the scum does not form a layer in the lab-scale reactors. The lab scale digesters will need to be monitored for scum formation throughout the digestion process. A solution could be to increase the revolutions per minute of the orbital shaker or create an automated mixing device.

There are several factors that have not been investigated that could have an impact on digestion such as pretreatment. Pretreatment could include conventional heating, microwave heating, physical pretreatment, or chemical pretreatment. Factors such as residence time could also have an impact on scum digestion as well due to the coagulant nature of grease.

Another area of interest would be to operate staged reactors. As mentioned above, pH is a concern in the digestion of scum and FOG because it is readily biodegradable and naturally has a pH less than 6.5. Operating a digestion facility with an acetogenic reactor and methanogenic reactor would allow the scum to lower the pH in the acetogenic reactor without inhibiting the methanogenic activity in the system. Ultimately, the digestion of scum has not been well explored in academia, but it should increase biogas production of a typical treatment facility when codigested with sludge.

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Chapter 3

Materials and Methods

3.1 Materials

The municipal sludge and primary scum used in this study was sampled from the Robert O. Pickard Environmental Centre (ROPEC) municipal wastewater treatment plant (MWWTP), Ottawa, Ontario, Canada. The facility operates as a secondary treatment facility with activated sludge prior to secondary clarification with a solids retention time (SRT) between 5 and 7 days. The solids management plan consists of four circular mesophilic pancake digesters with gas tube mixing and two mesophilic cylindrical pseudo egg shaped anaerobic digesters that are mixed hydraulically. The digesters are loaded with a sludge mixture composed of 58% TWAS and 42% PS by volume operating at 35°C and an SRT of 20 days and organic loading rates between 1.6 and 2.2 kg $VS/m^3 \cdot d$. Samples of active anaerobic digester effluent (inoculum), TWAS, PS and scum were sampled and stored at 4°C until used to prevent unwanted and unquantifiable digestion. Samples of scum were collected in large quantities from the floating beach of the primary clarifier and well mixed to alleviate the variability of scum composition (Metcalfe and Eddy, 2003). All of the mixed scum samples as well as primary sludge and TWAS were characterized for total solids (TS), volatile solids (VS), soluble chemical oxygen demand (SCOD) and total chemical oxygen demand (TCOD) as shown in Table 3.1 with their standard deviations.

3.2 Experimental Set-up and Protocols for BMP Assays

Biochemical methane potential (BMP) batch assays were conducted in order to determine the suitability of scum for mesophilic anaerobic co-digestion and specific biogas production for energy recovery. Prior to the start of each assay, characterization of inoculum, TWAS, PS, and scum was conducted to determine total and soluble chemical oxygen demand (TCOD and SCOD), total and volatile acids (TS and VS) and pH as described in section 3.4 analytical methods.

BMP assays were conducted in duplicate evaluating different factors for the mesophilic anaerobic co-digestion of scum. The factors analyzed were:

- Scum concentration
- Pretreatment temperature
- Pretreatment holding time (HT)

3.2.1 Experimental Set-up

Batch biodegradability tests were performed using the mesophilic biochemical methane potential (BMP) protocol outlined by Angelidaki et al. (2009). BMP assays were conducted using 1 L glass Kimax bottles with an active volume between 600 mL and 642 mL with the control reactor having a 600 mL active volume and the scum added reactors having variable volumes up to a maximum of 642 mL. The different BMP organic loads were determined using a 2^3 factorial design, which included eight reactors plus two controls as described in Table 3.1 on the next page.

Each of the reactors was duplicated and thus the results presented are the averages of the duplicates. R0 consisted solely of the active anaerobic digester effluent (200 mL), which served as the methanogenic inoculum, and Rcont consisted of the anaerobic digester effluent (200 mL at 1.7 % VS) plus TWAS and PS blended at a v/v ratio of 58:42 (400 mL). Rcont had volumetric and specific loads (PS plus TWAS) of 28.0 g VS/L and 5.4 g VS/g VS respectively. RL,50,4 through RH,70,48 consisted of the control components (Rcont), plus an additional amount of substrate added as scum. Scum was added after being subject to various scum HT and temperature (T) in the scum concentrator. The HT within the scum concentrator was investigated at a low exposure time of 4 h and at a high time of exposure of 48 h to low and high temperatures of 50 and 70°C. Scum was loaded at low (L) and high (H) values of 8.2 g VS/assay (13.2 g

VS/L) and 20.3 g VS/assay (31.6 gVS/L) respectively (all scum samples were heated in the lab using a water bath to model the effects of a scum concentrator). The addition of scum at the low and high VS loadings increased the specific loadings of the reactors by 2.4 and 5.9 g scum VS/g VS respectively compared to Rcont. It should be noted that the inoculum volume was kept constant in each BMP assay. The selected range for the scum loading, HT and scum treatment temperature were determined based on ROPEC plant operational capabilities and potential application for current and future WWTPs. Measured biogas productions are normalized for VS loadings.

Table 3.1 – Reactor configurations and initial volumetric and specific VS loadings per assay

Reactor	**Description [1)]**	**T,P and scum VS added (g)**	**Specific Load (kg VS Added / kg VS In)**
Rcont	In + T:P	20.1	5.7
RL,50,4	In + T:P + Scum	28.4	8.1
RL,70,4	In + T:P + Scum	28.8	8.2
RH,50,4	In + T:P + Scum	39.4	11.2
RH,70,4	In + T:P + Scum	40.4	11.5
RL,50,48	In + T:P + Scum	28.4	8.1
RL,70,48	In + T:P + Scum	28.8	8.2
RH,50,48	In + T:P + Scum	39.4	11.2
RH,70,48	In + T:P + Scum	40.4	11.5

Abbreviations: In = methanogenic inoculum, T:P = TWAS:PS v/v 58:42
Scum = (loading rate, temperature in scum concentrator, SRT in scum concentrator)

To ensure sufficient alkalinity and anaerobic conditions in the assay all BMP bottles were spiked with 8 g of calcium bicarbonate/sodium bicarbonate blended at 50:50 m:m ratio and flushed with nitrogen gas for 1 minute prior to being sealed with a rubber septum, and secured with a plastic cap. Immediately following the sealing process, pressure from the nitrogen gas was relieved. All the reactors were maintained at 35°C and continuously mixed using a New

Brunswick Scientific Controlled Environment Incubator Shaker model G-25 set at 115 rpm. Batch assay schematic is shown figure 3.1.

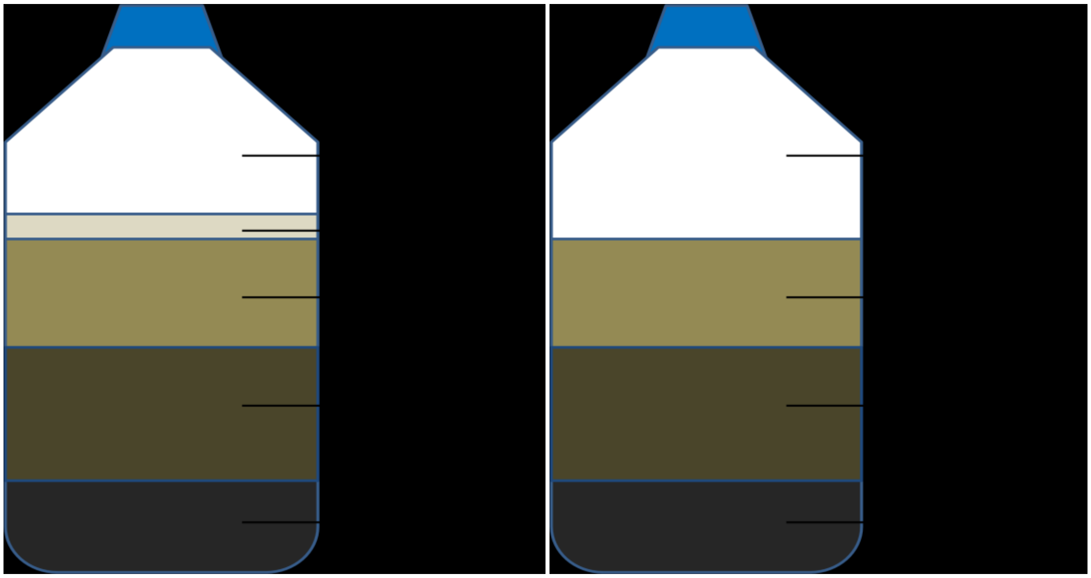


Figure 3.1 – Batch assay schematic with scum (left) without scum (right)

3.3 Experimental Set-up and Protocols for Semi-Continuous Reactors

Semi-continuous reactors were used to determine the effects of scum addition on anaerobic digestion in a lab scale semi-continuous system, with the intention of providing insight into potential effects at full scale operation. Specific biogas production from co-digesting scum with TWAS and PS was determined at desired loading rates and hydraulic retention times (HRTs) applicable to full scale operations.

3.3.1 Semi-continuous reactor design

Semi-continuous digestion was conducted using 1 L glass Kimax bottles with an active volume of 600 mL at a loading rate of 1.6 $kgVS/m^3 \cdot d$ composed of different fractions of TWAS, PS, and scum as shown in table 3.2. TWAS and PS fractions were maintained at a 58:42 v:v for the control and scum added reactors.

The temperature, and holding time (HT) of the scum concentrator prior to scum addition along with the scum loading applied to each of the semi-continuous reactors was based on the BMP assay findings presented in chapter 4. Reactor loads were configured to a full 2^2 factorial design. Loading factors were scum fraction of the total VS and temperature in the scum concentrator. Scum fraction of the TWAS, PS, and scum mixture ranged from 0.24 gVS or 25% by mass and 0.43 g VS or 45% by mass respectively. Scum temperature during a holding time of 48 h ranged from 50°C to 70°C. Hydraulic retention times (HRTs) of 15 and 20 days were investigated for all reactors. All values are typical and feasible for ROPEC plant operation and potential application for current and future WWTPs.

Reactors were started by adding 600 mL of methanogenic inoculum to each reactor and flushing the reactor with nitrogen gas for 1 minute before being sealed with a modified rubber septum and secured with a plastic cap. A 3/8 inch inner diameter glass tube and 1/8 inch inner diameter syringe was inserted through the rubber septum and sealed using silicone grease. Rubber tubing was attached to the syringe and sealed using two zip ties. A 2 L Teflon bag was attached to the rubber tubing to collect biogas during digestion. A 1/2 inch inner diameter rubber tube was attached to the glass tube and sealed using three zip ties. The rubber tube was fastened with a metal clamp to avoid biogas loss as well as air infiltration to the anaerobic system. All reactors were contained in a hot room maintained at 35°C and continuously mixed using an orbital shaker set at 115 rpm throughout the experimental phase.

The reactors were fed daily by extracting 40 mL (1/15th of the active volume) and 30 mL (1/20th of the active volume) through the rubber tube for HRTs of 15 and 20 days respectively. Subsequently, TWAS, PS, and scum were added to the reactors in accordance to table 3.2 to achieve the desired loading rate of 1.6 $\text{kgVS/m}^3 \cdot \text{d}$. Following feeding the rubber tube was resealed with the metal clamp .During the start up phase, all reactors were monitored for biogas production, TS, VS, and pH. Steady state was defined as the period where all parameters were stable within ± 5% daily.

Table 3.2 – Semi-Continuous reactor feed mixtures

Reactor	**Description**	**Loading rate (kgVS/m$^3\cdot$d)**
Rcont	T:P (0.56:0.40 gVS)*	1.6
RL,50	T:P:S (0.42:0.30:0.24 gVS)	1.6
RL,70	T:P:S (0.42:0.30:0.24 gVS)	1.6
RH,50	T:P:S (0.31:0.22:0.43 gVS)	1.6
RH,70	T:P:S (0.31:0.22:0.43 gVS)	1.6

Abbreviations: T = TWAS, P = PS, S = Scum
L = low scum fraction (3% by volume), H = high scum fraction (7% by volume)
50 = Temperature of scum concentrator (50°C), 70 = Temperature of scum concentrator (70°C)
* Mass added/bottle (600 mL active volume)

3.4 Analytical Methods

3.4.1 pH

The pH of all BMP assays was measured using a HORIBA portable pH meter stored in a protective case. Prior to measurements, the pH meter was calibrated using a three point calibration with standard pH solutions of 4, 7 and 10, rinsed with distilled water and dried with Kimwipes task wipers. This procedure was performed between each measurement.

The pH of all semi-continuous samples, substrates, and inoculum was determined using a Fisher Accumet model XL25 dual channel pH/ion meter equipped with a glass electrode. The pH electrode was stored in a pH 7 buffer solution and was removed, rinsed with distilled water and dried with Kimwipes task wipers prior to taking pH measurements. This was procedure was performed between each measurement. The pH meter was calibrated using a three point calibration with standard pH solutions of 4, 7 and 10 prior to the first measurement.

Sample pH was measured immediately after being removed from the reactor to mitigate the effects of escaping carbon dioxide from the solution. It is important to measure the in-situ pH for semi-continuous reactors to adequately analyze the effects within the system. Any measurements taken at room temperature or after storage would be less representative of actual operating conditions.

3.4.2 Alkalinity

Alkalinity was measured using the standard method outlined in Water Environment Federation (1999). Samples were centrifuged in a Thermoscientific Sorvall Legent T+ model centrifuge at 10,000 rpm (relative centrifugal force (RCF) of 11,292) for 30 minutes. Following centrifugation, samples were filtered through a 0.45 µm filter by applying a vacuum using a Fisher Scientific pump.

A measured volume of supernatant (filtered sample) was poured into a Pyrex beaker. A magnetic stirrer was added and the beaker was placed on a Termix stirrer model 120MR, set between speed 3 and 5 depending on sample volume. The sample's pH was measured using a Fisher Accumet XL25 dual channel pH/ion meter and sample titration was performed using 0.1N sulfuric acid dispensed through a 25 mL Kimax burette and the volume of acid required to reach pH of 4.6 was recorded. The same procedure as outlined above for rinsing the pH electrode was performed between each sample. Alkalinity in terms of mg/L as $CaCO_3$ was determined based on the following equation:

$$Alkalinity, mg\frac{CaCO_3}{L} = \frac{AxNx50{,}000}{mL\ sample\ titrated} \qquad \text{Equ. 3.1}$$

where,

A = mL standard acid used

N = normality of standard acid

3.4.3 Volatile Acids

Volatile acids (VA) were measured using standard titration outlined by Speece (2008). The same procedure was followed as outlined in section 3.4.2, but partial alkalinity was measured using Equ 3.1 at pH 5.8. VAs were then determined using the following equation:

$$VA, \frac{mg}{L} = \frac{Total\ Alkalnity - Partial\ Alkalinity}{0.8} \qquad \text{Equ. 3.2}$$

where,

Total Alkalinity = Alkalinity calculated using Equ. 3.1 at pH 4.6

Partial Alkalinity = Alkalinity calculated using Equ 3.1 a pH 5.8

3.4.4 Ammonia as NH_3-N

Ammonia as NH_3-N was determined using HACH TNT 832 (2-47 mg/L). Samples were centrifuged in a Thermoscientific Sorvall Legent T+ model centrifuge at 10,000 rpm (RCF of 11,292) for 30 minutes. Following centrifugation, samples were filtered through a 0.45 µm filter by applying a vacuum using a Fisher Scientific pump.

The supernatant was diluted 30:1 using distilled water and poured into a beaker with a magnetic stirrer. The sample was well mixed using a Termix stirrer model 120MR set on speed 5 and a 0.2 mL sample was pipette from the beaker and added to the HACH TNT 832 vial. After the sample was added, the protective tin foil covering was removed from the HACH TNT 832 vial and screwed on. The vial was turned upside down gently 4 times and let set for 15 minutes. After the reaction was allowed to take place for 15 minutes the vial was wiped using a Kimwipe task wipe and inserted into a calibrated HACH DR 5000 spectrophotometer. The spectrophotometer uses the internal calibration to deliver the NH_3-N concentration in mg/L without accounting for the dilution factor.

It is important to note that all ammonia samples were measured directly after the 15 minute reaction period to avoid degradation and non reliable results. HACH TNT 823 vials were kept in the refrigerator at 4°C to keep the reagent fresh as outlined in the operation manual.

3.4.5 Total and Soluble Chemical Oxygen Demand

Total and soluble chemical oxygen demand (TCOD and SCOD respectively) were measured using HACH TNT 823 (250-15,000 mg/L) and HACH TNT 822 (20-1,500 mg/L) respectively. TCOD measurements were conducted on raw and effluent samples diluted effectively 10 to 1 and 2 to 1 respectively using distilled water. The diluted sample was poured into a beaker with a magnetic stirrer and well mixed using a Termix stirrer model 120MR set on speed 5. The HACH TNT 823 vial cap was removed and 0.3 mL of the well mixed sample was carefully pipette and added to the vial. The cap was replaced and the vial was inverted several times until the vial became very hot as reactions between the sample and reagent were undergoing reactions. The vial was placed in a preheated HACH DRB200 (150°C) reactor for two hours. After the two hour period, the HACH DRB200 reactor was turned off and let cool to 120°C at which point the vial was removed and inverted several times while hot. Once the vial cooled to room temperature, Kimwipes were used to thoroughly clean the outside of the vial and an internally calibrated HACH DR5000 spectrophotometer delivered the COD concentration in mg/L.

To determine SCOD, raw and effluent samples were centrifuged in a Thermoscientific Sorvall Legent T+ model centrifuge at 10,000 rpm (RCF of 11,292) for 30 minutes. Following centrifugation, samples were filtered through a 0.45 μm filter by applying a vacuum using a Fisher Scientific pump. The supernatant was diluted effectively 10 to 1 and 2 to 1 for raw and effluent samples respectively using distilled water. The same procedure outlined above for TCOD was followed for SCOD, expect HACH TNT 822 required 2 mL of the well mixed sample to be added for each vial.

It is important to note that all samples were measured immediately after the vials cooled to prevent unwanted digestion. Also, the HACH DRB200 was covered with a box if the lights were on to prevent light reaction during the digestion process. HACH TNT 822 and 823 vials were kept in the dark at room temperature prior to use as outlined in the operation manual.

3.4.6 Total and Volatile Solids

Total and volatile solids (TS and VS respectively) were analyzed using EPA standard methods (EPA, 2001). Prior to solids determination, aluminum dishes were prepared by scrubbing with soap and water followed by heating in a Thermolyne 62700 muffle furnace model F62730 at 550°C to ensure no organic residues remained. After 60 minutes in the furnace, the dishes were transferred to a Precision mechanical convection oven model 23 maintained at 105°C for 15 minutes. The dishes were then moved to a desiccator and allowed to cool completely.

Following desiccation, each dish was placed on a Mettler Toledo Classic Plus Model AB204-s/fact analytical balance and the weight was recorded. A well mixed sample was then added to the dish and weighed. The dish containing the sample was placed in the Precision mechanical convection oven model 23 at 105°C until all the water in the sample evaporated (approximately 24 hours). After the sampled had completely evaporated, the dish was transferred to the desiccator and allowed to cool for 60 minutes prior to weighing. The dishes were then placed in the Thermolyne 62700 muffle furnace model F62730 at 550°C for 60 minutes. After 60 minutes the dishes were transferred to the 105°C oven for 15 minutes and subsequently allowed to cool in the desiccator for 60 minutes. After complete cooling, the weight was taken and the TS and VS were determined using the following equations:

$$\% TS = \frac{Y - W}{X - W} * 100\% \qquad \text{Equ. 3.3}$$

$$\% VS = \frac{Y - Z}{X - W} * 100\% \qquad \text{Equ. 3.4}$$

where,

W = Weight of dish

X = Weight of dish and wet sample

Y = Weight of dish and sample after drying and desiccation

Z = Weight of dish and sample after ignition and desiccation

3.4.7 Cell Viability Protocol

Live/Dead staining was performed using Molecular Probes Live/Dead Reduced Biohazard Viability/Cytotoxicity Kit #1 (L-7013). The basis of the test is to differentiate insitu cell viability through fluorescence, which can be observed using fluorescent or confocal laser microscopy. The process uses two stains, SYTO 10, a green fluorescent nucleic acid stain that is a highly membrane-permeable dye and stains all cells in a sample along with a second stain, DEAD Red, which is cell-impermeable and only fluoresces membrane compromised cells red, which are accordingly no longer viable.

The protocol used for staining cells in suspension first required the preparation of a working solution of dyes that consists of 2 µL of both dyes diluted into 1 mL of HBSS. The mixed solution was protected from the light with aluminum wrap or placed in a cool dark location. 1 mL of each reactor effluent sample was aliquoted into epenndorf tubes and centrifuged at 250 x g for 10 minutes. The supernatant was discarded and autoclaved water was added to resuspend the cells. This sample was again centrifuged at 250 x g for 1 minute and the supernatant was again discharged. 150 µL of the working dye solution was added to the sample. The sample sat for 15 minutes in complete darkness at room temperature and was centrifuged at 250 x g for 5 seconds to pellet the cells and discharge the supernatant. The pelleted cells were resuspended in 150 μL fresh HBSS washing solution and 10 μL of the washed sample was pipetted onto glass slides and covered (Molecular Probes, 2001). Cells were not fixed as the fixation process can inhibit or disturb the viability analysis; however the samples were immediately investigated.

Immediately after covering the glass slide, the cells were observed using a confocal laser microscope. 1.2 μm thick sections of red (1.4 Au) and green (1.7 Au) planes were examined to quantify the viability of the sample using a 63x oil objective that provided images of 250 μm x 250 μm. Twenty random images were taken and analyzed per sample.

3.4.8 Biogas Measurements

3.4.8.1 Batch Assays

Biogas production was measured using a U shaped manometer equilibrated to atmospheric pressure prior to measuring the liquid volume displacement. Vessels were removed from the incubator, allowed to cool, and vigorously shook prior to biogas measurement. A 10g needle connected to the manometer was inserted through the rubber stopper until pressure in the assay bottle reached atmospheric pressure. The volume of biogas was determined by measuring the difference in starting and finishing point and multiplying the cross sectional area of the water in the tube (2.54 cm inner diameter) connected to the manometer.

3.4.8.2 Semi-Continuous Reactors

Biogas production was measured using 2 L Teflon bags attached to the reactors. Daily, the Teflon bags were attached to a Fisher pump and U shaped manometer equilibrated to atmospheric pressure to measure liquid volume displacement. The Teflon bags were pumped until the liquid volume displacement became stationary in the manometer tube. The volume of biogas was determined by measuring the difference in starting and finishing point and multiplying the cross sectional area of the water in the tube connected to the manometer.

3.4.9 Biogas Composition

Biogas composition was determined using a Hewlett Packard 5712A GC equipped with a metal packed column. Helium, carrier gas was pumped at 25 mL per minute. Samples were collected by inserting a 10g needle attached to a 1 mL syringe through the sample port on the Teflon bags. Gas samples were purged two to three times before the sample for composition was used. National Instruments^{TM} LabVIEW version 6.0 was used to present the percentage of nitrogen, methane and carbon dioxide.

3.5 Sample Preservation

All sample analysis was completed within an acceptable time period. Samples were preserved at most overnight in a refrigerator at 4°C for analysis the following day.

Chapter 4

Batch Biodegradability Assays: Biodegradability and Codigestion of Municipal Sludge and Scum

4.1 Context

The batch biodegradability article presented in Chapter 4 has been submitted for publication in *Bioprocess and Biosystems Engineering* with a title of *Biodegradability and Codigestion of Municipal Sludge and Scum* by B. Young, K. Kennedy, R. Delatolla and R. Sharif. This article describes the results of the BMP assays described in Section 3.2. The results of the BMP test were used to identify the operating conditions of the semi-continuous reactor study presented in Chapter 5.

4.2 Introduction

Scum is the mass of sewage solids, buoyed up by entrained gas, grease, or other substances that float on the surface of primary clarification units in municipal wastewater treatment plants (MWWTPs). Scum accounts for approximately 4% of the total solids collected in the entire MWWTP and is collected in troughs at the end of the primary clarification basin with current disposal methods including landfilling, incineration and anaerobic digestion (Outwater, 1994). In order to benefit from reuse and increases in biogas production MWWTPs may anaerobically co-digest the scum. Prior to pumping scum to the anaerobic digestion facility, the scum is often heated for a set holding time (HT) in a concentrator to facilitate transport to the anaerobic digester (Downy, 2006).

Cortell (2008) studied the effects of mesophilic anaerobic co-digestion of various combinations of fats, oils and greases (FOG) from restaurants with municipal sludge and demonstrated that a 50% increase in biogas production at a volumetric organic loading rate of 0.48 kg VS/m^3·d. Kobouris et al. (2008) reported a 100% increase in biogas production when restaurant FOG were mesophilically digested with primary sludge (PS) and thickened waste activated sludge (TWAS) at a 10% increase in VS loading as FOG. However in both of the above studies no heating or other pretreatment of the FOG was described and the nonpretreated FOG was directly digested as produced. Heating increases the solubility of the various components in FOG and scum resulting in readily available substrate for digestion or in some cases resulting in increased long chain fatty acids (LCFA) concentrations that can be acutely inhibitory to methanogenesis. Furthermore, Speece (20808) has shown that the addition of heated scum to the anaerobic digestion process increases the neutral lipid and LCFAs content of the anaerobic digester as liquid scum from the scum concentrator is added to the anaerobic digester feed stream. LCFAs have low solubility and require proper digester mixing to preclude the development of dead zones, reformation of surface floating scum in the digester and non-mobile sedimentation. Methanogenic bacterial consortia have also been observed to clump if excess scum is trapped within the system, which inhibits hydrogenotrophic methanogenesis and leads to high hydrogen partial pressures and elevated levels of H_2 in the mixed liquor. If this condition persists it can slow VFA oxidation leading to accumulation of butyric and propionic acids which can result in acidic reactor conditions and further exacerbate the inhibition of methane production by acetoclastic methanogens as well as physical reactor fouling.

Previous studies show specific effects of co-digestions of FOG and scum and the direct effects of heating the scum on biogas production, however a current gap in knowledge exists with respect to the effects of varying scum concentrator temperature and HT combined with the concomitant effect on biogas generation when scum is anaerobically co-digested with primary and secondary municipal sludge. The objective of the work presented in this paper is to quantify biogas production and mesophilic anaerobic biodegradation of real municipal scum co-digested with real municipal sludge (PS and TWAS). Specifically, the effects of initial scum loading with municipal sludge, temperature of pretreatment in the scum concentrator and temperature HT on anaerobic digestion are investigated using batch mesophilic biological methane potential (BMP) assays.

4.3. Methods

4.3.1 Raw Waste Sampling and Characterization

The municipal sludge and scum used in this study was sampled from the Robert O. Pickard Environmental Centre (ROPEC) MWWTP, Ontario, Canada. The facility operates as a secondary treatment facility with activated sludge prior to secondary clarification with a solids retention time (SRT) between 5 and 7 days. The solids management plan consists of four circular mesophilic pancake digesters with gas tube mixing and two mesophilic cylindrical pseudo egg shaped anaerobic digesters that are mixed hydraulically. The digesters are loaded with a sludge mixture composed of 58% TWAS and 42% PS by volume operating at 35°C, an SRT of 20 days and organic loading rates between 1.6 and 2.2 kg VS/m^3·d. Samples of active anaerobic digester effluent (inoculum), TWAS, PS and scum were acquired from ROPEC and stored at 4°C until used in the research to prevent unwanted and unquantifiable digestion. Samples of scum were collected in large quantities from the primary clarifier and were well mixed to alleviate the variability of scum composition (Metcalf and Eddy, 2003). The mixed scum samples as well as PS and TWAS were each analyzed a minimum of five times for total solids (TS), volatile solids (VS), soluble chemical oxygen demand (SCOD) and total chemical oxygen demand (TCOD). Table 4.1 shows the raw waste characterization with standard deviations.

Table 4.1 – Characterization of raw waste

Waste	**TCOD (mg/L)**	**SCOD (mg/L)**	**TS (%)**	**VS (%)**
Inoculum	21900 ± 3400	1110 ± 244	3.0 ± 0.0	1.7 ± 0.0
PS	45200 ± 3800	3090 ± 83	5.5 ± 0.0	4.2 ± 0.1
TWAS	56200 ± 5220	2690 ± 116	5.2 ± 0.2	4.1 ± 0.0
50°C Scum	89200 ± 8680	1390 ± 23	50.7 ± 3.5	48.3 ± 3.5
70°C Scum	134000 ± 14200	3420 ± 49	53.9 ± 1.2	50.9 ± 0.9

4.3.2 Batch Biodegradability Test

Batch biodegradability tests were performed using the mesophilic biochemical methane potential (BMP) protocol outlined by Angelidaki et al. (2009). BMP assays were performed using 1 L glass Kimax bottles with an active volume between 600 mL and 642 mL depending on the scum loading to the reactor. An inoculum reactor denoted as R0 and a control reactor denoted as Rcont were operated with active volumes of 200 mL and 600 mL, respectively. R0 consisted solely of the active anaerobic digester effluent (200 mL), which served as the methanogenic inoculum, and Rcont consisted of the anaerobic digester effluent (200 mL at 1.7 % VS) plus TWAS and PS blended at a v/v ratio of 58:42 (400 mL). Rcont had volumetric and specific loads (PS plus TWAS) of 28.0 g VS/L and 5.4 g VS/g VS respectively.

The different BMP organic loadings of the scum loaded reactors were determined using a 2^3 factorial design, which included the eight reactors denoted in Table 4.2. Each of the reactors (inoculum, control and scum added) were duplicated and thus the results presented in this study are the averages of the duplicates. The scum loaded reactors were labeled based on scum loading, the temperature (T) of the scum concentrator and the HT of the scum in the concentrator. Scum loading is denoted as low (L) and high (H) where L refers to a loading of 8.2 g

VS/reactor (13.2 g VS/L) and H refers to a loading of 20.3 g VS/reactor (31.6 gVS/L). The temperature of the scum concentrator was investigated at low and high values of 50 and 70°C, respectively. The HT within the scum concentrator was investigated at a short time value of 4 h and at a long time value of 48 h. The selected range for the scum loading, HT and scum treatment temperature were determined based on ROPEC plant operational capabilities and potential application for current and future MWWTPs.

Table 4.2 lists the configuration of the reactors used in this research, where R0 is not listed as it was not loaded throughout the study. The scum loaded reactors (reactors RL,50,4 through RH,70,48) consisted of the control components (Rcont), plus an additional amount of substrate added as scum. The addition of scum at low and high scum VS loadings increased the specific loadings of the reactors by 2.4 and 5.9 g scum VS/g VS respectively compared to Rcont.

Table 4.2 – Reactor configurations for BMP assays

Reactor	**Description [1)]**	**T,P and scum VS added (g)**	**Specific Load (kg VS Added / kg VS In)**
Rcont	In + T:P	20.1	5.7
RL,50,4	In + T:P + Scum	28.4	8.1
RL,70,4	In + T:P + Scum	28.8	8.2
RH,50,4	In + T:P + Scum	39.4	11.2
RH,70,4	In + T:P + Scum	40.4	11.5
RL,50,48	In + T:P + Scum	28.4	8.1
RL,70,48	In + T:P + Scum	28.8	8.2
RH,50,48	In + T:P + Scum	39.4	11.2
RH,70,48	In + T:P + Scum	40.4	11.5

Abbreviations: In = methanogenic inoculum, T:P = TWAS:PS v/v 58:42, , L = low scum loading of 8.2 g VS/reactor, H = high scum loading of 20.3 g VS/reactor, 50 = temperature concentrator of 50°C, 70 = temperature concentrator of 70°C, 4 = concentrator HT of 4 hours, 48 = concentrator HT of 48 hours.

To ensure sufficient alkalinity and anaerobic conditions in the assay all BMP bottles were spiked with 8 g of calcium bicarbonate/sodium bicarbonate blended at 50:50 m:m ratio and flushed with nitrogen gas for 1 minute prior to being sealed with a rubber septum and secured with a plastic cap. Immediately following the sealing process, pressure from nitrogen gas was released. All the reactors were maintained at 35°C and continuously mixed using a New Brunswick Scientific Controlled Environment Incubator Shaker model G-25 set at 115 rpm.

4.3.3 Analytical Methods

pH was measured using a HORIBA portable pH meter. TS and VS were measured by EPA method 1684 (EPA, 2001). TCOD and SCOD were measured using HACH TNT 823 (250-15,000 mg/L) and HACH TNT 822 (20-1,500 mg/L), respectively. SCOD samples were centrifuged at relative centrifugal force (RCF) of 11,292 for 30 minutes, filtered using a 0.45 µm Millipore G filter and analyzed using a HACH DR 5000 spectrophotometer. Biogas production was measured daily via a 10g syringe attached to a manometer equilibrated to atmospheric pressure prior to measuring liquid volume displacement.

4.3.4 Statistical Methods

All statistical models and results were determined using Minitab 15 statistical software. The design of the experiment was conducted as a complete 2^3 factorial design that was performed in duplicate. Analysis of variance (ANOVA) was used to determine the statistical significance and interactions of each factor. ANOVA determines the p-value for each of the factors and subsequent interactions. A factor or interaction with a p-value less than 0.05 is interpreted in this study to be significant for all biogas production and biodegradability measurements.

4.4 Results and Discussion

Batch anaerobic digestion experiments were performed for a period of 50 days after which the daily biogas production was minimal in all BMP assays. The average cumulative biogas productions (CBP) of the duplicates as well as initial and final pH for each assay are summarized in Table 4.3. All BMP assays had initial pHs between 6.5-6.9. The CBP of the duplicates varied less than 10% for each BMP assay providing additional confidence in the results. While Table 4.3 gives the overall quantity of biogas produced for the BMP assays it does not indicate the rates of biogas production. The rates of biogas production normalized for VS loading are shown in Figure 4.1. The rates of biogas production varied over time, dependant on the conditions of the BMP assays.

Table 4.3 – Summary of BMP results after 50 days of batch digestion

Parameter	**R0**	**Rcont**	**RL 50 4**	**RL 70 4**	**RH 50 4**	**RH 70 4**	**RL 50 48**	**RL 70 48**	**RH 50 48**	**RH 70 48**
Cumulative Biogas (m^3/tonne/VS added)	40	430	538	371	412	419	692	557	140	135
Initial pH	7.8	6.9	6.5	6.5	6.5	6.3	6.6	6.6	6.6	6.6
Final pH	8.1	7.0	7.4	7.3	7.2	7.1	7.2	7.1	7.0	7.0

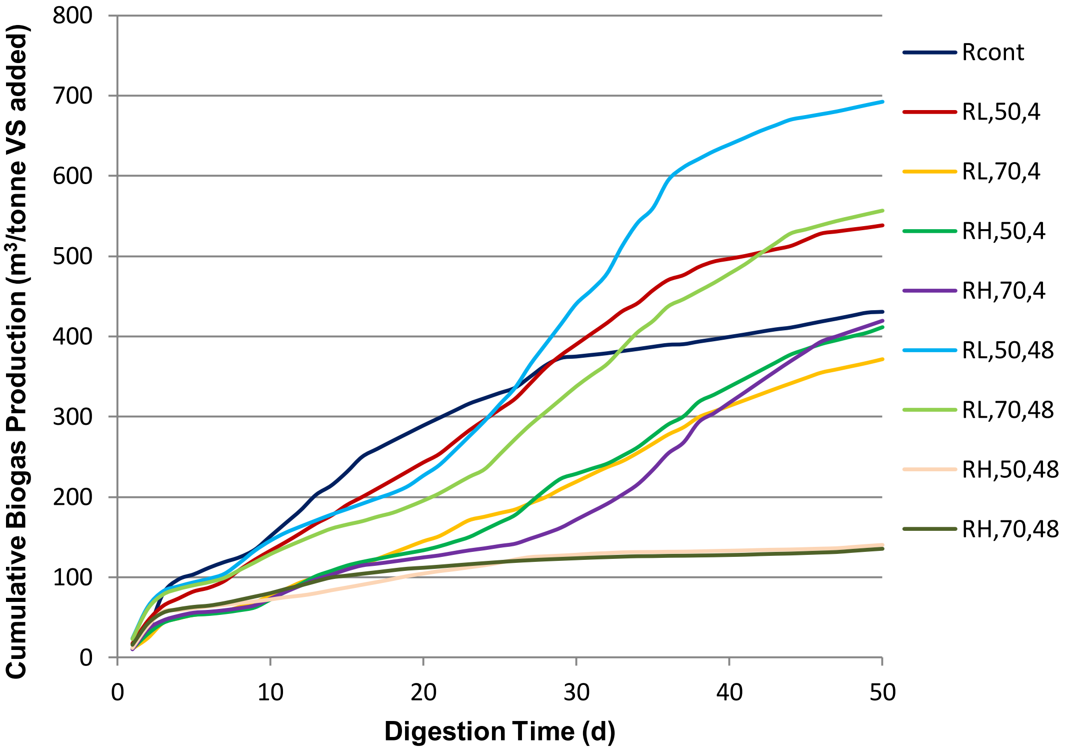


Figure 4.1 - Cumulative biogas production of BMP assays

Comparing CBP yields (per mass VS added) after 50 days of operation we observe that RL,50,48 produced the greatest improvement of biogas production with a 61% improvement over the control reactor, Rcont. RL,70,48 and RL,50,4 also exhibited significant biogas yield improvements in comparison to the control with 25% and 30% improvement respectively. Assays RH,50,48 and RH,70,48 exhibited significantly less biogas production and a decrease in biogas yield in comparison to the control with a 67% and 69% reduction, respectively. The biogas production results demonstrate that co-digestion of lower scum loads (8.2 g VS/reactor) produced a higher biogas yield in three of four reactors operating for 50 days compared to digesting TWAS and PS without scum addition; while co-digestion of higher scum loads (20.3 g VS/reactor) produced a lower 50 day biogas yield at all operational conditions investigated with two reactors producing only slightly less biogas (RH,50,4 and RH,70,4).

All BMP assays exhibited an initial surge in biogas production in the first 3-5 days of the assay indicating that the inoculum used was active and produced biogas from readily degradable components such as VFA present in the waste. However, BMP assays that included scum were followed by a 5-8 day lag phase during which the pH decreased. It should be noted that assays conducted with higher amounts of additional scum (RH assays) had longer lag phases (8 d) and lower scum loaded assays (RL assays) exhibited a shorter lag phase (5 d) while the control reactor (Rcont) did not exhibit any lag phase. Rcont maintained a steady increase in biogas production through days 5-30 of digestion at a rate of approximately 11.2 m^3/tonne VS/day, after which it appears that the usable substrates were depleted from the system and biogas production decreased. It should be noted that the anaerobic inoculum used was sampled from ROPEC and acclimated to TWAS/PS so the lack of a lag phase in Rcont was not unexpected. However the addition of scum as a third component of the sludge mixture either required acclimation by the microbial consortia before rates of biogas production increased or was effected by decreased pH conditions related to increased acidification of the scum component of the sludge mixtures.

Following the extended lag phase, the lower scum loaded reactors (RL,50,48, RL,70,48 and RL,50,4) exhibited the greatest CBP per unit VS of 692, 557 and 538 m^3/tonne-VS as well as increased biogas production rates of 22.6, 16.1 and 15.8 m^3/tonne-VS/day until the 40th day of digestion. Based on the increased specific CBP as well as specific rates of biogas production relative to the Rcont the tri-mixture assays indicate that scum is a readily degradable, high yield energy substrate. Additionally, when the mass of scum added is lower (RL assays) it is readily digested in a tri-mixture of TWAS, PS and scum.

By accounting for the biogas production attributed to the TWAS and PS components in the tri-mixture BMP assays the scum biogas yield for RL assays can be determined on a VS added basis. The TWAS/PS mixture had a biogas yield of 430 m^3/tonne-VS while the calculated biogas yields attributed to scum in RL,50,48, RL,70,48 and RL,50,4 were 1370, 884 and 816 m^3/tonne-VS-of-scum which are 2-3 times higher per mass of TWAS/PS volatile solids. Based on the 58% methane content of the BMP assays the methane yields from MWWTP scum was 794, 513 and 473 m^3/tonne-VS-of-scum added for RL,50,48, RL,70,48 and RL,50,4 respectively. The methane yields from the scum sampled from ROPEC are similar to or slightly higher than what has been reported by Hansen (2006) for residual fats, floatable fats and FOG (400-660 m^3/tonne-VS, respectively). The calculated methane yields expressed by Hansen used residual fats, floatable fats, and FOG as the sole carbon source, were not optimized for a mixture and did not explore any form of pretreatment. Concomitantly the slightly higher scum methane yields found in this study may be attributed to the heat pretreatment or the synergistic effects of the tri-mixture co-digestion or a combination of both.

However, Table 4.3 and Figure 4.1 also show differences in total and temporal biogas production when the scum component was treated and added at a higher loading to the sludge tri-mixture (RH assays). As mentioned above the RH assays tended to have a longer lag phase compared to Rcont and RL assays. In all cases RH assays never produced more total cumulative specific biogas compared to Rcont indicating that there is a limit to the amount of scum that can be successfully co-digested with TWAS/PS without adjusting additional operational parameters. In two assays, RH,50,4 and RH,70,4, the cumulative BMP is 93-95 % of the biogas yield compared to Rcont. However, when HT was increased from 4 to 48 hrs, RH,50,48 and RH,70,48 produced only 140 and 135 m^3/tonne-VS which was only 31-33% of the biogas yield as compared to Rcont. Hence, it appears that a combination of higher scum loading and increased HT tends to have a significant negative impact on the digestion of the sludge/scum tri-mixture. The biogas yield attributed to scum additions of the RH,50,4 and RH,70,4 BMP assays was calculated to be 394 and 408 m^3/tonne-VS assuming a TWAS/PS biogas yield of 430 m^3/tonne-VS. The yields for RH,50,48 and

RH,70,48 are well below the values of the control indicating that digestion of this tri-mixture under the conditions evaluated (scum heated to 50 or 70°C for 48hrs) was extremely inhibitory to its digestion under the conditions applied in this study. In general, digestion of TWAS/PS and scum with a large additional scum load produced negative conditions for successful anaerobic digestion.

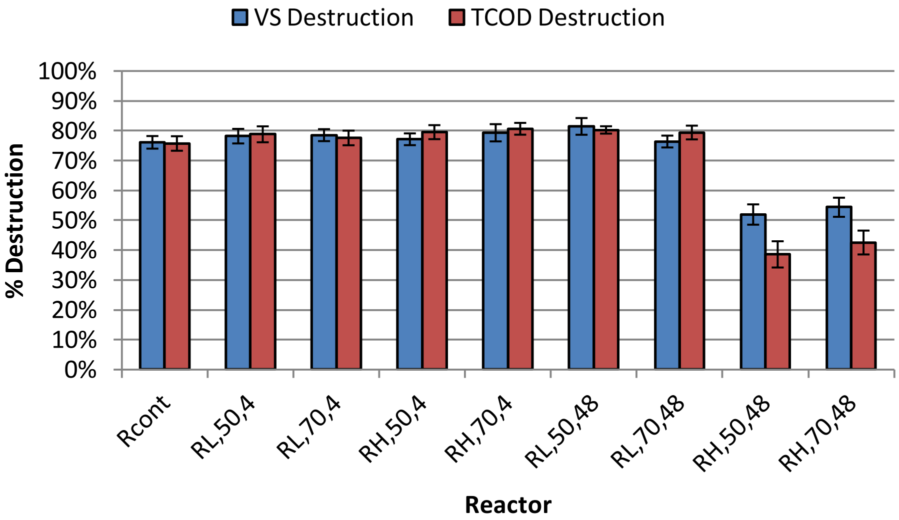


Figure 4.2 – VS and TCOD destruction

All reactors other than the highest scum loaded reactors with the longest HT at elevated temperatures (RH,50,48 and RH,70,48) exhibited VS and TCOD destructions between 75% and 80%. Reactors RH,50,48 and RH,70,48 exhibited lower VS and TCOD destructions of 65% as shown in Figure 4.2. Based on the VS destruction the true methane yields associated with TWAS/PS and scum per mass of VS destroyed was determined. The three low scum loaded assays, RL,50,48, RL,70,48 and RL,50,4, successfully digested the scum/sludge tri-mixture with methane yields per mass of scum VS stabilized determined to be 619, 400 and 378 m^3/tonne-VS-of-scum stabilized. These values are in the range and slightly higher than what has been reported by Hansen (2006), further indicating the potential to increase methane production when digesting scum with TWAS and PS.

The variation in biogas production with and without scum addition and in particular at lower and higher scum loadings at various temperatures and HT have indicated that scum addition to PS and TWAS can have either a positive or negative impact on biogas production by the sludge/scum tri-mixture. All assays exhibited an initial surge in biogas production shortly after start-up, which can be attributed to readily degraded substrate and initial ideal pH conditions for digestion. However as digestion proceeded temporal variations in biogas production were observed for the various BMP assays. While acclimation can be one explanation for the results it is possible that the addition of scum adversely affected the acidogenic/methanogenic balance in the reactors to various degrees. An imbalance of the acidogenic/methanogenic activity can result in the accumulation of VFAs that consume alkalinity and subsequently lower the mixed liquor pH resulting in inhibition or delays in methanogenesis until the VFA concentrations are lowered and pH is increased. Temporal pH changes throughout the experimental assays are shown in Figure 4.3.

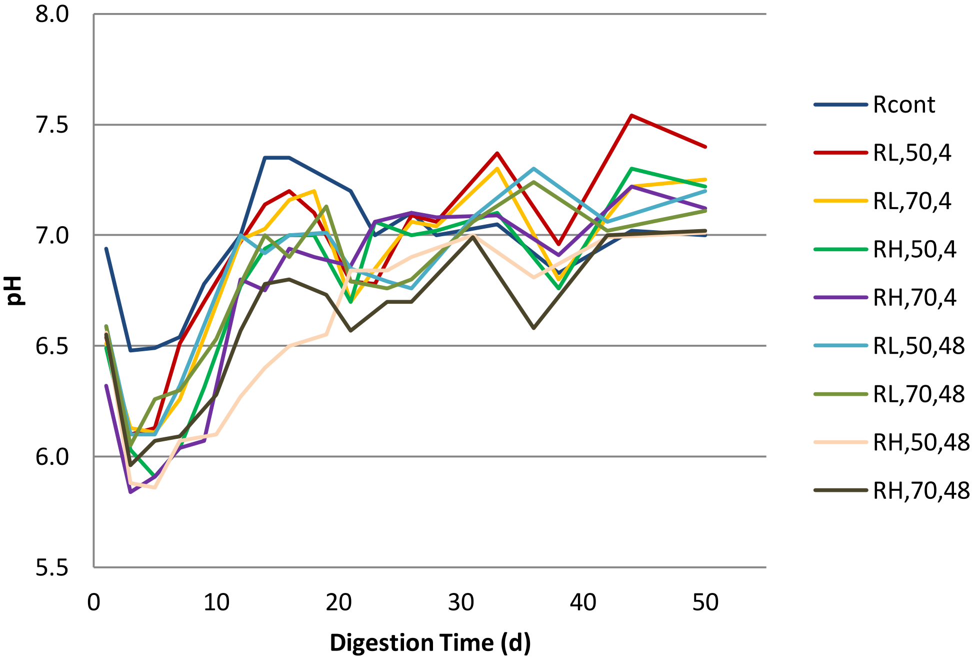


Figure - 4.3 pH of BMP assays over 50 day digestion period

The control assay Rcont which did not exhibit any lag phase and produced biogas at a steady rate until the substrate was exhausted suggests that the acidogenic/methanogenic consortia were balanced in the assay and the pH conditions were appropriate for successful digestion and biogas production. Concomitantly, assays with scum additions and their associated biogas production were compared in relation to the temporal pH conditions that existed in Rcont (Figure 4.3). In the first 20 days, the initial pH values for all the assays loaded with scum were lower than Rcont. The additional readily available substrate components of the added scum which is a heterogeneous mixture of floatable PS from ROPEC resulted in rapid hydrolysis and acidogenesis that subsequently used the available alkalinity and caused the pH to decrease. The effects of scum digestion were observed as early as the first five days of digestion which suggests that a portion of the scum is as readily degradable as the PS and TWAS; this finding is supported by the findings of Outwater (1994) who showed scum digestion after 8 to 10 days.

This decrease in pH was sustained for a prolonged time and was more evident in high scum loaded assays RH,50,48, and RH,70,48 which were subject to long HT at elevated temperatures as well as high volumetric and specific scum loads of 33.8 kg TWAS/PS/scum VS/m^3 and 5.9 kg TWAS/PS/scum VS/kg VS, respectively. The pH of RH,50,48 and RH,70,48 decreased to about 5.8 and required about 20 days to recover to a pH of 6.5 when a gradual increase in biogas production was observed. Conversely, the high scum loaded assays RH,50,4 and RH,70,4 were subjected to a shorter HT and experienced an initial decrease in pH to an approximate value of 6.1; however this drop was not sustained and the pH returned to above 6.5 within 10 days. This indicates that the characteristics of the scum substrate was different in the short HT RH assays such that acidogenesis and accumulation of VFAs was slower. Similarly, all of the low scum loaded assays at both higher and lower temperatures as well as longer and shorter HTs experienced an initial decrease in pH to a value of about 6.1, but returned to above 6.5 within 10 days.

Lay et al. (1997) observed that methanogenic activity becomes inhibitory below a pH of 6.5 and methanogenic archaea operate most efficiently in the pH range of 6.7-7.3. These observations by Lay are based upon the theory that the methanogenic enzymatic functions are disturbed at pH ranges below 6.5 and are susceptible to deactivation if a change is observed. As methanogenic populations become deactivated, a feedback of additional acids ultimately accumulates in the system. It should be noted that the BMP assays that recovered their pH more quickly to 6.5 or above demonstrated better overall biogas production and waste stabilization with respect to VS and TCOD destruction.

In order to better understand the relationship of pH and biogas production Figure 4.4 shows the specific CBP and pH of the best performing lower scum loaded tri-mixture reactors (RL,50,48, RL,70,48 and RL,50,4) relative to Rcont. The trend in relative biogas production is comparable to the observed relative trend in pH in all RL tri-mixture assays. In the first 15 to 20 days the relative pH is decreasing which is indicative of increased acidogenesis and VFA accumulation that ultimately resulted in a decrease in biogas production. Subsequently as the relative pH increased to 1 (days 26-30) the biogas production of the RL assays also increased indicating that VFA accumulation was diminishing and methanogenenesis was improving. After day 30, the relative pH of the RL assays was greater than Rcont and biogas production was also greater as the methanogenic/acidogenic consortia appear to have balanced and the high biogas yield scum was stabilized. These results indicate that hydrolysis and acidogenesis of the scum containing reactors exceeded the buffering capacity of the system during the first 25 to 30 days and hence required additional time to consume the acids in an inhibitory environment before more stable methanogenesis could occur.

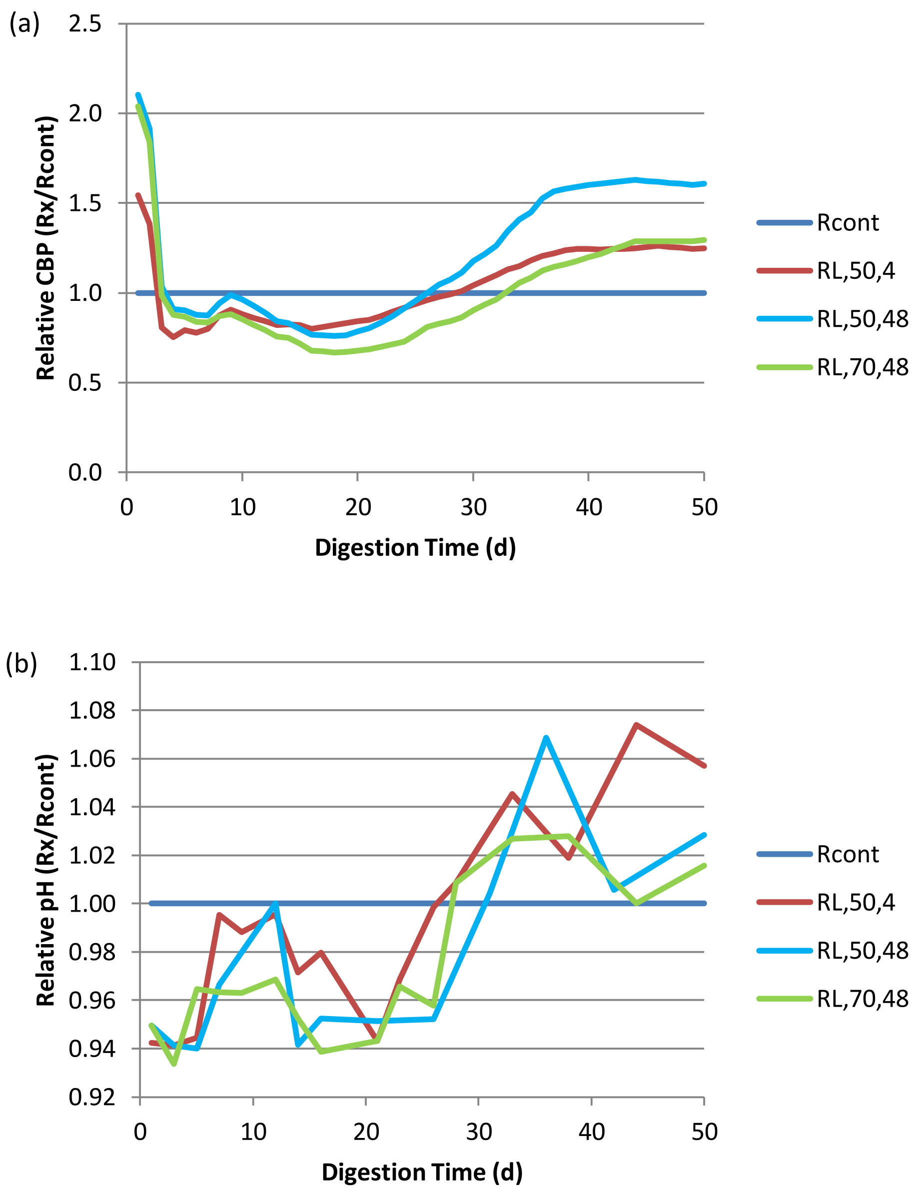
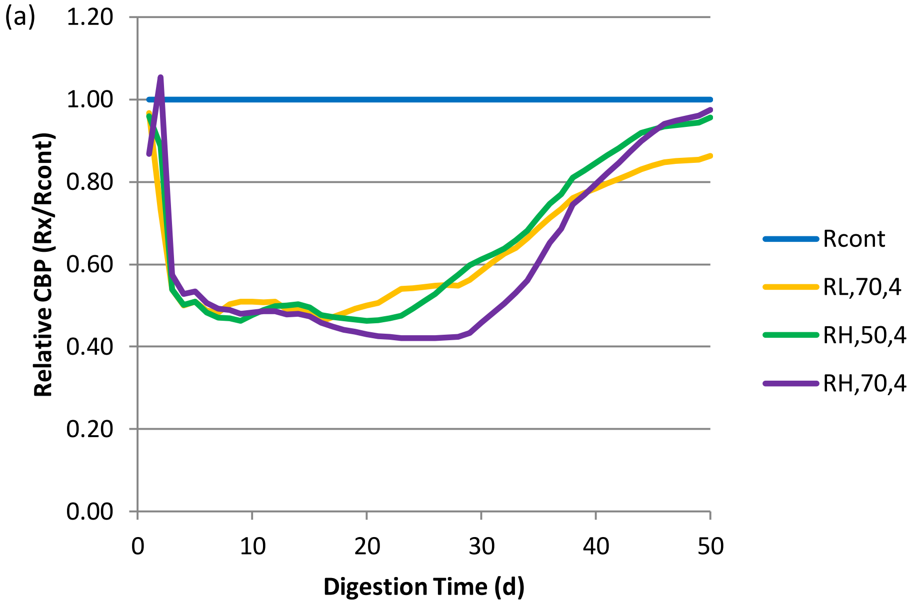


Figure 4.4 – Highest biogas producing assays (a) specific CBP relative to Rcont (b) specific pH relative to Rcont

Figure 4.5 shows the CBP and pH for the moderate biogas producing assays (RL,70,4, RH,50,4 and RH,70,4) relative to Rcont. Similar to the highest biogas producing assays, the moderate biogas producers exhibited an initial drop in relative specific CBP and pH, however unlike the highest biogas producers, as the pH recovered between 25 and 30 days of digestion the relative CBP did not increase substantially nor surpass Rcont. During the initial 10 days of operation, the relative pH dropped to as low as 0.90 in the assays. The methanogenic activity appears to increase after 20 to 30 days as the CBP increases and hence begins to surpass the production of the Rcont reactor accordingly. It is likely that these reactors were slightly overloaded as compared to the highest biogas producing assays due to the higher scum addition and/or higher scum availability through increased heating; the scum load and heat appears to have caused prolonged inhibition, it should be noted however that the inhibition did not completely sour the system.



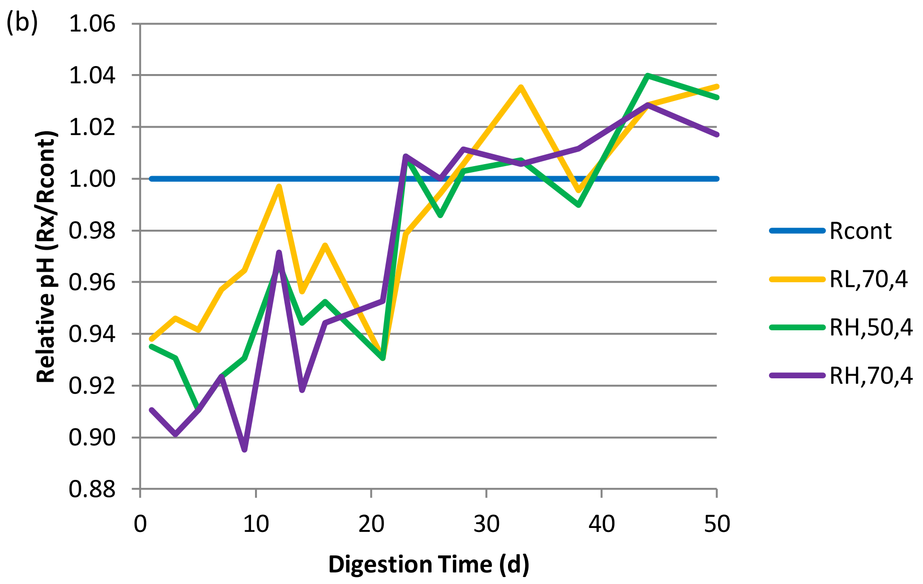
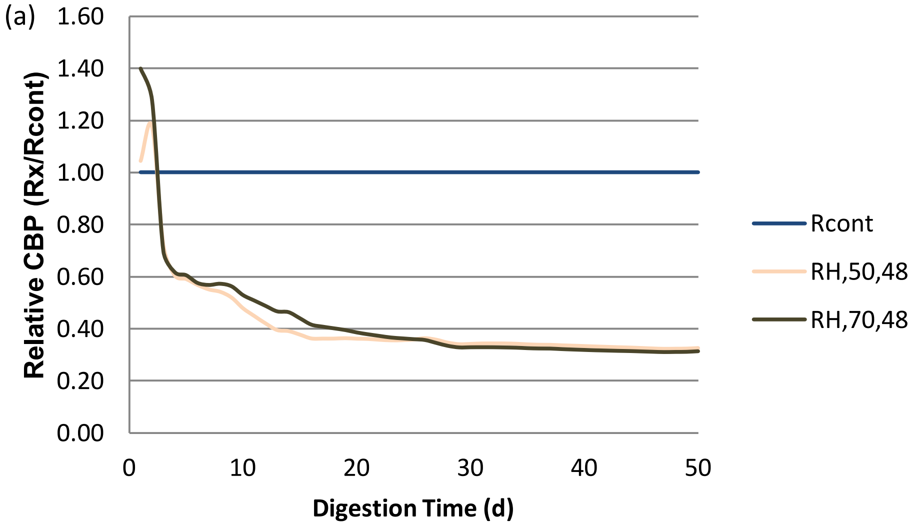


Figure 4.5 – Moderate biogas producing assays (a) specific CBP relative to Rcont, (b) specific pH relative to Rcont

Figure 4.6 shows the CBP and pH relative to Rcont for RH,50,48 and RH,70,48; the highest scum loaded assays that did not successfully digest the tri-mixture. RH,50,4 and RH,70,4 demonstrated only partially successful digestion and subsequent biogas production from the tri-mixture relative to Rcont. Unlike the RL assays, these RH assays did not achieve pH parity with Rcont until day 45 of the 50 day experiment. While the maximum pH difference of the RL assays was 0.4 pH units, RH,50,48 and RH,70,48 demonstrated the largest maximum differences in pH with respect to Rcont of 0.7 and 0.6 pH units, respectively. The minimum pH values in RH,50,48 and RH,70,48 occurred after approximately 15 to 20 days and did not achieve a pH difference of 0.4 pH units until after approximately 25-30 days of digestion. The low pH again is indicative of an accumulation of VFAs and unbalanced digestion as the rate of acidogenesis is assumed to be markedly greater than the rate of methanogenesis. This observation is supported by the relative biogas production curves shown in Figure 4.6a. At no point did the RH,50,48 and RH,70,48 assays achieve parity with Rcont. In fact the difference in CBP relative to Rcont continued to increase, indicating that waste stabilization was unsuccessful. The high scum loaded RH assays with long HT appear to have been unable to recover from the severe decrease in pH that appear to have lead to an inhibitory environment leading to methanogenic deactivation that could not be recovered in the experimental timeframe. Although the scum loading was relatively low on a volume basis, the additional volumetric and specific scum organic loads were high at 33.8 kg-scum-VS/m^3 and 5.95 kg-scum-VS/kg-VS, respectively. Thus, additional pH control within systems operating at high organic scum loading is required. .



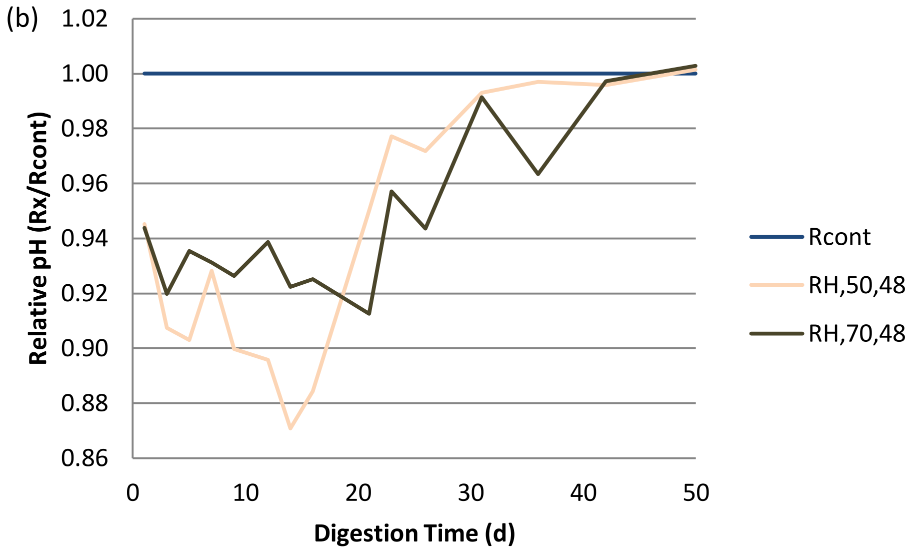
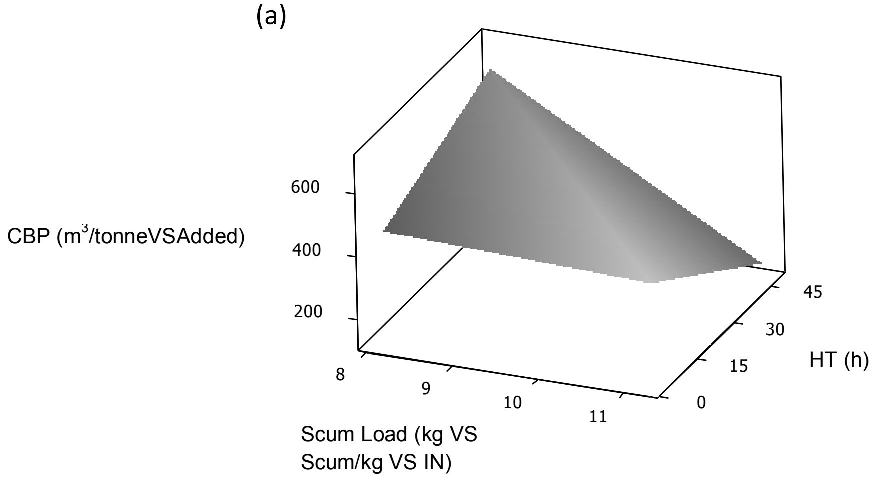


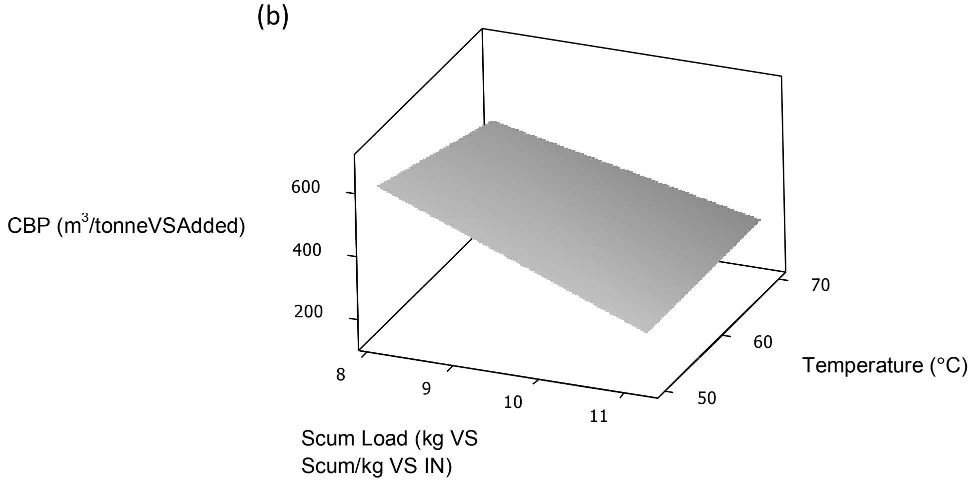
Figure 4.6 – Poor biogas producing assays (a) specific CBP relative to Rcont, (b) specific pH relative to Rcont

An examination of Figures 4.4, 4.5 and 4.6 suggests that interactions between the factors of scum loading, treatment temperature and HT in relation to the response variable of the biogas yield exists. Based on the 2^3 full factorial experiment, three factor surface plots of the factorial analysis for the 50 day specific CBP evaluating the effects of scum load, treatment temperature and HT were plotted (Figure 4.7). Figure 4.7a clearly shows that the highest specific CBP occurs at low scum loads and long HT. In Figure 4.7b the highest specific CBP occurs at low scum loads and low temperature while Figure 4.7c indicates a relatively flat response with little effect of HT or temperature on specific CBP.

(a)


CBP (m^3/tonneVSAdded)
600
400
200
8
9
10
11
Scum Load (kg VS Scum/kg VS IN)
0
15
30
45
HT (h)

(b)


CBP (m^3/tonneVSAdded)
600
400
200
8
9
10
11
Scum Load (kg VS Scum/kg VS IN)
50
60
70
Temperature (°C)

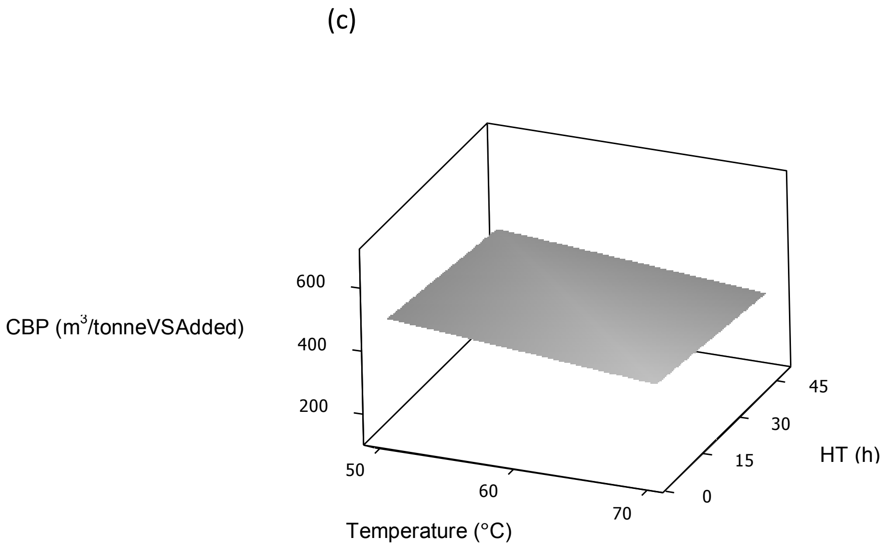


Figure 4.7 - Surface plot of CBP after 50 d (a) scum load and HT, (b) scum load and temperature and (c) temperature and HT

Analysis of the response variable (Y_{biogas}) in relation to all the factors resulted in the following equation that defines the specific CBP surface responses (note interactions were not included if plausibly 0):

$$Y_{biogas} = 408.1 - 131L_{scum} - 37.5T - 27HT + 38L_{scum}T - 112L_{scum}HT \qquad \text{Eqn. 1}$$

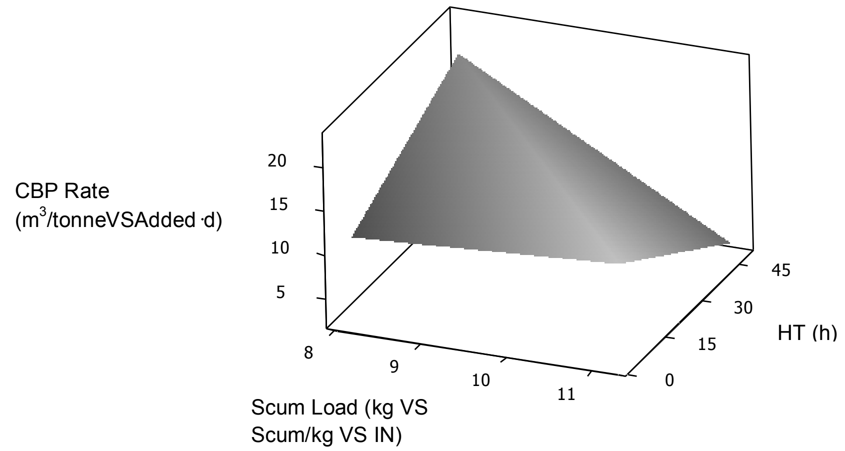
Where,

Y_{biogas} is the expected overall biogas yield (m^3/tonne VS added)
L_{scum} is the coded scum loading (1 high, -1 low)
T is the coded temperature (1 high, -1 low)
HT is the coded HT (1 high, -1 low)

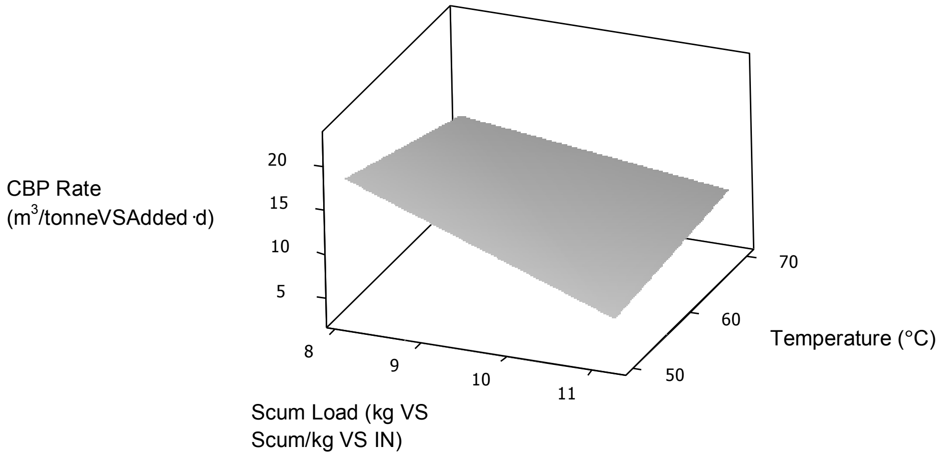
The two-way ANOVA test indicates that scum loading has a significant effect on the 50 day specific CBP with a p-value of 0.002, while the HT in the scum concentrator and temperature of the concentrator did not have as important of an effect on the specific CBP with p-values of 0.094 and 0.287, respectively. The interaction between scum loading and HT in the scum concentrator had a p-value of 0.003 and was also determined to be significant with respect to the 50 day specific CBP, however, it is likely that the poor specific CBP performance of assays RH,50,48 and RH,70,48 via prolonged acidification and decreased pH accentuated the scum loading and HT interaction. The interaction of scum load and temperature had a p-value of 0.251 indicating this two factor interaction was not as significant as the effects of the scum loading and HT interaction. Additionally the two factor interaction of temperature and HT and the 3 factor interaction of scum load, HT and temperature were not significant and as such are not included in equation 1.

In addition to specific factor interaction effects on the CBP after 50 days of operation the effects of the same factors were determined with respect to the rate of the specific CBP (m^3/tonne VS added ·d) in the middle stages of digestion (between days 20 and 40 of the digestion period). In the period between days 20 and 40 the anaerobic inoculum in the assays were allotted time for maturation and adaptation to the waste with pH values in most assays in a range suitable for successful digestion. Figure 4.8 shows that the scum load has the greatest effect on specific biogas production during the middle stages of digestion with the low scum load exhibiting the greatest biogas production. Concomitantly, the interaction between scum load and HT as well as scum load and temperature appear to be more important than the interaction effect of HT and temperature (Figure 4.8).

(a)


CBP Rate
(m³/tonneVSAdded·d)
20
15
10
5
8
9
10
11
Scum Load (kg VS
Scum/kg VS IN)
0
15
30
45
HT (h)

(b)


CBP Rate
(m³/tonneVSAdded·d)
20
15
10
5
8
9
10
11
Scum Load (kg VS
Scum/kg VS IN)
50
60
70
Temperature (°C)

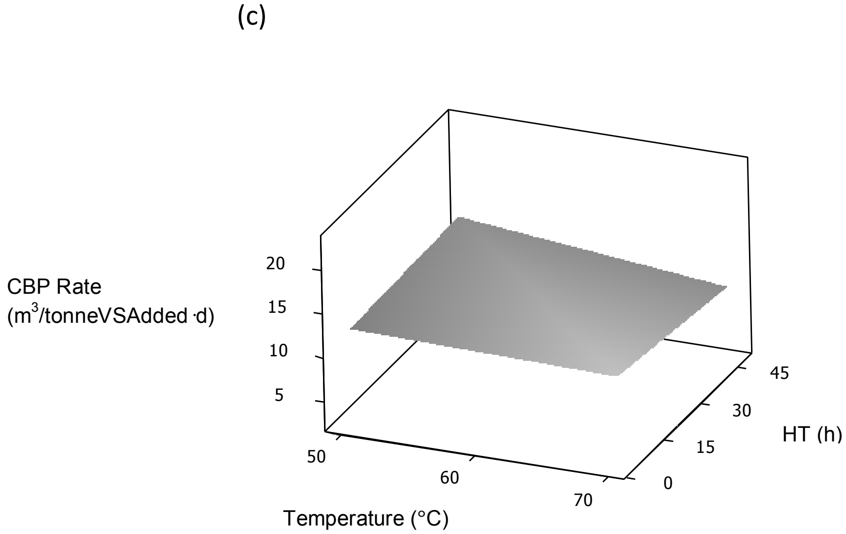


Figure 4.8 - Surface plot of CBP rates between 20 to 40 days of digestion (a) scum load and HT, (b) scum load and temperature and (c) scum temperature and HT

Analysis of the response variable with all interactions resulted in the following equation:

$$Y_{BiogasRate} = 11.5 - 3.7L_{scum} - 0.9T - 0.4HT + 1.9L_{scum}T - 4.6L_{scum}HT - 0.6THT - 0.3L_{scum}THT \qquad \text{Equ. 2}$$

Where,

$Y_{BiogasRate}$ is the expected biogas rate from days 20 to 40 (m^3/tonne VS ·d)
L_{scum} is the coded scum loading (1 high, -1 low)
T is the coded temperature (1 high, -1 low)
HT is the coded HT (1 high, -1 low)

The increase in rate of specific biogas production of the low scum loaded assays during middle stages of digestion in comparison to the high scum loaded assays is significant (p-value = 0.018). Similarly to the 50 day cumulative BMP digestion period, the HT in the scum concentrator and the temperature in the scum concentrator did not show as important of an effect on the middle stage, specific biogas production (p-values = 0.562 and 0.146 respectively). Although neither parameter is significant for the middle stage, specific biogas production, the p-value analysis indicates that HT is more important than temperature relative to the 50 day cumulative biogas production and temperature is more important than HT relative to the specific biogas production. This difference is likely due to the scum concentrator temperature exerting a greater effect on the solubility and mixability of scum within the system during the early stages of the experiment. Increased solubility and mixing promotes increased rates of hydrolysis, acidogenesis and methanogenesis during the early stages of the experiment, while showing little increase in the ultimate biogas production. Similar to the ultimate CBP interactions, the interaction between scum load and holding time was significant with a p value of 0.02. Interactions between scum load and temperature, temperature and holding time as well as scum load, temperature, and holding time were not significant with p values of 0.081, 0.252 and 0.584 respectively. This suggests optimization can be made with little concurrent effects from interactions excluding he scum load should remain low during high HT pretreatment to produce the highest specific rate of biogas production.

4.5 Conclusion

The co-digestion of PS, TWAS, and scum was shown to be beneficial with respect to ultimate biogas production and specific biogas production during early stage digestion. The effect of scum load was significant with respect to the 50 day cumulative biogas production and with respect to the specific biogas production during the middle stage of digestion, while the effects of thermal pretreatment of scum through adjusting the scum HT within the scum concentrator and temperature of the scum concentrator did not demonstrate a significant effect on the ultimate or specific biogas production. The interaction between scum load and HT were significant for both the ultimate CBP and the specific biogas production rate. Concomitantly, the interactions between scum load and temperature as well as temperature and holding time were not significant for either the ultimate CBP or the specific biogas production rate.

The maximum rate of the ultimate biogas production relative to a reactor fed with only TWAS and PS was observed in at low scum loading and with a 48 h HT at 70°C. Specifically, the addition of 14.5 g VS/L of scum exhibited a maximum increase in ultimate biogas production relative per g VS added compared to the TWAS and PS loaded assay, while a higher additional scum loading of 33.7 g VS/L reduced the ultimate biogas yield to 32% of the TWAS and PS assay. Reactors operated with higher scum loading likely exhibited rapid hydrolysis and acidogenesis which consumed alkalinity and lowered the pH in the reactors. The observed prolonged acidic conditions were inhibitory to methanogenesis as demonstrated by a reduction in the biogas production. This study thus shows that care must be taken to monitor the pH within scum loaded digesters to avoid inhibitory effects and potential souring of the system due to overloading and that scum loading and scum loading in combination with HT and concentrator temperature can be used to limit inhibitory effects.

4.6 Contributions

All of the experimental set-up, lab-work, and initial writing and interpretation of the data was performed by Bradley Young. Editing and further investigation of the data was a collaborative effort between Bradley Young, Dr. Kevin Kennedy, and Dr. Robert Delatolla. The process was performed diligently by all over the course of a year.

4.7 References

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Chapter 5

Semi - Continuous Mesophilic Anaerobic Digestion Experiments: Enhancement of Continuous Mesophilic Anaerobic Digestion of Municipal Sludge and Scum

5.1 Context

The lab scale semi-continuous mesophilic anaerobic digestion article presented in Chapter 5 has been submitted for publication in *Bioprocess and Biosystems Engineering* with a title of Enhancement of Continuous Mesophilic Anaerobic Digestion of Municipal Sludge and Scum by B. Young, K. Kennedy, R. Delatolla and R. Sharif. This article describes the results of the semi-continuous reactors described in Section 3.3. The results of the semi-continuous test were used to determine the expected biogas increase from scum presented in Chapter 6.

5.2 Introduction

Scum is the mass of sewage solids, buoyed up by entrained gas, grease, or other substances that float on the surface of primary clarification units in municipal wastewater treatment plants (MWWTPs), with scum accounting for approximately 4% of the total solids collected in the entire WWTP (Outwater, 1994). Scum is collected in troughs at the end of the primary clarification basin and are currently disposed of by landfilling, incineration or anaerobic digestion. In order to benefit from reuse and the production of biogas, WWTPs have been anaerobically co-digesting scum. Prior to pumping scum to the anaerobic digestion facility, it is often heated for a set holding time (HT) in a scum concentrator to facilitate transport to the anaerobic digester (Downey, 2006). The effects of varying scum concentrator temperature and HT operations and the concomitant effect on biogas generation when scum is anaerobically co-digested with primary and secondary municipal sludge is not well documented in the literature. Cortell (2008) studied the effects of mesophilic anaerobic co-digestion of various combinations of fats, oils and greases (FOG) from restaurants with municipal sludge and demonstrated that a 50% increase in biogas production at a volumetric organic loading rate of 0.48 kg VS/m^3·d. Kobouris et al. (2008) reported a 100% increase in biogas production when restaurant FOG were mesophilically digested with primary sludge (PS) and thickened waste activated sludge (TWAS) at a 10% increase in VS loading due to FOG. However in both of the above studies no heating or other pretreatment of the FOG was described and the non-pretreated FOG was directly digested as produced. Heating will increase the solubility of the various components in FOG and scum making them more readily available for digestion or in some cases increase the concentration of components such as long chain fatty acids (LCFA) that can be acutely inhibitory to methanogenesis. Young et al. (2012) performed batch BMP assays on pretreated scum co-digested in a tri-mixture with TWAS and PS. Pretreatment methods used were conventional heating at differentiating holding times. Optimizing the pretreatment parameters and scum load showed a maximum increase in gas yield that was 1.6 times greater than the gas yield of the PS/TWAS system per gram of VS added. The authors concluded that the major factor contributing to an increase in biogas yield was the concentration of the scum loading, with lower relative VS loadings of scum demonstrating the greatest biogas yield. Concomitantly, scum pretreatment temperature was shown to effect the rate of biogas production and the mixability of the scum n the digester. Similarly, Kabouris et al. (2008) showed that a high loading of scum potentially increases the LCFA concentration to inhibitory levels and hence reduces the biogas yield.

The addition of heated scum to an anaerobic digestion process increases the neutral lipid and LCFAs content of the anaerobic digester. LCFAs have low solubility and require proper digester mixing to prevent the reformation of surface floating scum in the digester and non-mobile sedimentation. Methanogenic consortia have also been observed to clump if excess scum is trapped within the system, which inhibits hydrogenotrophic methanogenesis and leads to high hydrogen partial pressures and elevated levels of H_2 in the mixed liquor. If this condition persists it can slow VFA oxidation leading to accumulation of butyric and propionic acids which result in acidic reactor conditions and further exacerbate the inhibition of methane production by acetoclastic methanogens as well as physical reactor fouling (Speece, 2008).

The objective of this study is to quantify biogas production and mesophilic anaerobic biodegradation of real municipal scum co-digested with real municipal sludge (PS and TWAS) in semi-continuous digesters. Specifically, the effects of initial scum loading with municipal sludge, temperature of pretreatment in the scum concentrator and temperature HT on anaerobic digestion are investigated using semi-continuous mesophilic biological reactors operated according to Ontario Ministry of Environment regulated organic loading rates and hydraulic retention times.

5.3 Methods

5.3.1 Samples

The municipal sludge and scum used in this study was sampled from the Robert O. Pickard Environmental Centre (ROPEC) MWWTP, Ottawa, Ontario, Canada. The facility operates as a secondary treatment facility with activated sludge prior to secondary clarification; the activated sludge SRT of the facility is maintained between 5 and 7 days. The solids management plan consists of four circular mesophilic pancake digesters with gas tube mixing and two mesophilic cylindrical pseudo egg shaped anaerobic digesters that are mixed hydraulically. Digesters are loaded with a sludge mixture composed of 58% TWAS and 42% PS by volume operating at 35°C and an SRT of 20 days. Samples of active anaerobic digester effluent (inoculum), TWAS, PS, and scum were sampled on September 13, 2011 and stored at 4°C until used to prevent unwanted and unquantifiable digestion. Samples of scum were collected in large quantities from the beach section of primary clarifier and well mixed to alleviate the variability of scum composition (Metcalfe and Eddy, 2003). All of the samples were characterized for total solids (TS), volatile solids (VS), soluble chemical oxygen demand (SCOD), and total chemical oxygen demand (TCOD) as shown in Table 5.1.

Table 5.1 – Characterization of ROPEC raw waste

Waste	TCOD (mg/L)	SCOD (mg/L)	TS (%)	VS (%)
Inoculum	21900 ± 3400	1110 ± 244	3.0 ± 0.0	1.7 ± 0.0
PS	45200 ± 3800	3090 ± 83	5.5 ± 0.0	4.2 ± 0.1
TWAS	56200 ± 5220	2690 ± 116	5.2 ± 0.2	4.1 ± 0.0
50°C Scum	89200 ± 8680	1390 ± 23	50.7 ± 3.5	48.3 ± 3.5
70°C Scum	134000 ± 14200	3420 ± 49	53.9 ± 1.2	50.9 ± 0.9

Minimum of 5 samples per waste were analyzed

5.3.2 Semi-Continuous Lab Scale Digestion

Semi-continuous digestion was conducted using 1 L glass Kimax bottles with an active volume of 600 mL and loading rate of 1.6 kgVS/m^3·d composed of different fractions of TWAS, PS, and scum as shown in Table 5.2. TWAS and PS fractions was added to the control reactors (PS/TWAS only, no scum added) and the scum loaded reactors in a ratio of 58:42 throughout the entire study. In the case of the scum loaded reactors, a percentage of the PS/TWAS mass measured as VS was simply replaced by scum. The holding times (HT) in the scum concentrator along with the scum loading and temperatures of the scum in the concentrator were chosen in this work based on the batch reactor results presented in Chapter 4 and the work conducted by Young et al., (2012).

The reactor conditions were configured according to a full 2^2 factorial design, with the factors being scum fraction of the total VS and temperature in the scum concentrator. A HT value of 48 hours in the scum concentrator was used for all of the semi-continuous reactors in this study, while a loading rate of 1.6 kgVS/m^3·d was fixed for each reactor. The scum load applied to the reactor was either a low value of 0.24 gVS (25% by mass) or a high value of 0.43 g VS (45% by mass). The scum was either maintained at a temperature of 50°C or 70°C throughout the 48 h HT. Finally, a hydraulic retention times (HRTs) of 15 and 20 days were investigated for all reactors. Table 5.2 summarizes the notation used to identify the reactors and the feed mixtures applied to each reactor. All selected operating values are typical and feasible for the ROPEC plant and are of potential application for current and future WWTPs.

Table 5.2 – Semi-Continuous reactor feed mixtures

Label	**Reactor Configuration**	**Loading rate (kgVS/m^3·d)**
Rcont	T:P (0.56:0.41 gVS)*	1.6
RL,50	T:P:S (0.42:0.30:0.24 gVS)	1.6
RL,70	T:P:S (0.42:0.30:0.24 gVS)	1.6
RH,50	T:P:S (0.31:0.22:0.43 gVS)	1.6
RH,70	T:P:S (0.31:0.22:0.43 gVS)	1.6

Abbreviations: T = TWAS, P = PS, S = Scum
L = low scum fraction (3% by volume), H = high scum fraction (7% by volume)
50 = Temperature of scum concentrator (50°C), 70 = Temperature of scum concentrator (70°C)
* Mass added/bottle (600 mL active volume)

The reactors were started by adding 600 mL of methanogenic inoculum to each reactor and flushing the reactor with nitrogen gas for 1 minute before being sealed with a modified rubber septum and secured with a plastic cap. A 3/8 inch inner diameter glass tube and 1/8 inch inner diameter syringe was inserted through the rubber septum and sealed using silicone sealant. Rubber tubing was attached to the syringe and sealed using two zip ties. A 2 L Teflon bag was attached to the rubber tubing to collect biogas during digestion. A 1/2 inch inner diameter rubber tube was attached the glass tube and sealed using three zip ties. The rubber tube was fastened with metal clamp to avoid biogas loss as well as air infiltration to the anaerobic system. All reactors were contained in a hot room maintained at 35°C and continuously mixed using an orbital shaker set at 115 rpm throughout the experimental phase.

The reactors were fed daily (once/day) by extracting 40 mL (1/15th of the active volume) and 30 mL (1/20th of the active volume) of waste through the rubber tube for HRTs of 15 and 20 days, respectively. Subsequently, the TWAS, PS, and scum mixtures were added to the reactors in accordance to Table 5.1 to achieve the desired loading rate of 1.6 kgVS/m^3·d in all reactors. Following feeding the rubber tube was resealed with the metal clamp .During the start up phase, all reactors were monitored for biogas production, TS, VS, and pH. Steady state conditions were considered to be achieved when all parameters were stable within ± 5% daily.

5.3.3 Live/Dead Protocol

Live/Dead staining was performed using Molecular Probes Live/Dead Reduced Biohazard Viability/Cytotoxicity Kit #1 (L-7013). The basis of the test is to differentiate insitu cell viability using fluorescent stained cells observed by a fluorescent microscope. The process uses two stains with the first being SYTO 10, a green fluorescent nucleic acid stain that is a highly membrane-permeant dye which is designed to stain all the cells of a sample. The second stain, DEAD Red, is a cell-impermeable stain that will only fluoresce membrane compromised cells; these red illuminated cells are accordingly no longer viable.

The protocol used for staining the cells and testing the viability of the cultures included the preparation of a working dye solution by combining 2 μL of both the SYTO 10 and the DEAD Red dyes into a 1 mL volume of Hank's Balanced Salt Solution (HBSS). The working dye solution was protected from light with aluminum wrap or placed in a cool dark location. A 1 mL sample of the effluent of each reactor was pipetted into a 2 mL eppendorf tube and centrifuged at 250 x g for 10 minutes. The supernatant of the eppendorf tube was pipetted out and was added to autoclaved water to re-suspend the cells. The re-suspended samples were centrifuged at 250 x g for 1 minute and the supernatant was again discarded. 150 µL of the working dye solution was added to each sample; this addition resuspened the cells. The dye solution was kept in contact with the sample for 15 minutes in complete darkness at room temperature. Each sample was retrieved and centrifuged at 250 x g for 5 seconds to pellet the cells and allow for the supernatant to be discarded. Finally, the cells were re-suspended in 150 µL fresh HBSS washing solution and 10 µL of each sample was pipetted onto glass slides and covered (Molecular Probes, 2001). The cells were not fixed as the fixation process can inhibit or disturb the viability analysis.

Immediately after covering the glass slide, the cells were observed through a confocal laser fluorescent microscope. Microscopy was performed on 1.2 µm thick sections of red (1.4 Au) and green (1.7 Au) plane. The total thickness of each sample was approximately 6 µm. A 63x oil objective was used to observe the cells and 20 images were acquired for analysis per sample.

5.3.4 Analytical Methods

pH was measured using a Fisher Accumet model XL25 dual channel pH/ion meter equipped with a glass electrode. TS and VS were measured by standard methods (EPA, 2001). TCOD and SCOD were measured using HACH TNT 823 (250-15,000 mg/L) and HACH TNT 822 (20-1,500 mg/L), respectively. Ammonia was measured as NH_3-N using HACH TNT 832 (2-47 mg/L). SCOD and ammonia samples were centrifuged at 10,000 rpm (RCF of 11,292) for 30 minutes, filtered using a 0.45 µm filter and analyzed using a HACH DR 5000 spectrophotometer. Alkalinity was measured using standard method outlined in Water Environment Federation (1999). Biogas production was measured daily by pumping the collected biogas out of the Teflon bags attached to a manometer to measure liquid volume displacement. Live/Dead analytical quantification of the acquired microscopic images was performed using Nikon NI Vision Assistant using color threshold analysis according to Delatolla et al., (2009). Pixels of each color were counted as cells and calibrated through back calculating pixel size to match the relative size of a cell (1 to 10 μm^2)

5.3.5 Statistical Methods

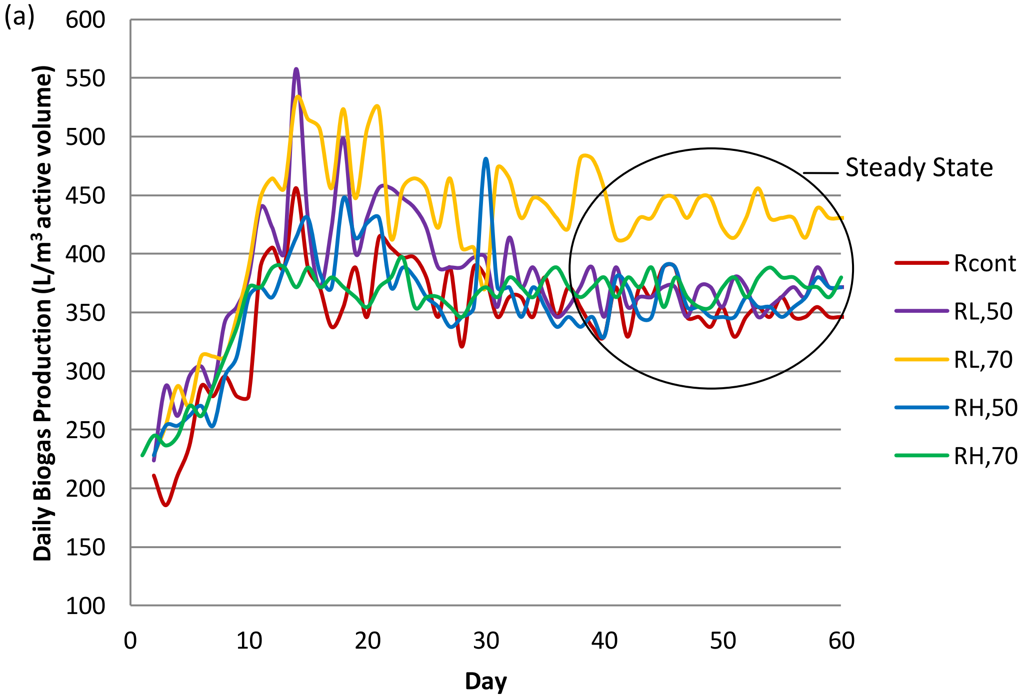
All statistical models and results were determined using Statistica statistical software. The design of the experiment was conducted as a complete 2^2 factorial design. Analysis of variance (ANOVA) was used to determine the statistical significance and interactions of each factor. ANOVA determines the p-value for each of the factors and subsequent interactions. A factor or interaction with a p-value less than 0.05 is interpreted in this study to be significant for all biogas production and biodegradability measurements.

5.4 Results and Discussion

The reactors were run as semi-continuous digesters over a period of 3-4 HRTs until steady state conditions were achieved; once steady state conditions were confirmed the reactors were monitored for an additional full HRT to characterize steady state operation. Prior to steady state the biogas was measured daily and pH, VS, and TS were measured every other day to monitor and ensure reactor health; subsequently all parameters were measured (daily) once steady state conditions were verified in order to best quantify the steady operation of the reactors.

5.4.1 Biogas Production Results

Figure 5.1 shows the daily biogas production at 15 and 20 d HRTs during the start up phase and steady state phase of operation of the reactors. Loading rate was maintained at 1.6 $kgVS/m^3 \cdot d$ for all reactors at both HRTs. Figure 5.1 shows that all reactors demonstrated a linear upward trend in biogas production until approximately one full HRT was achieved. After approximately one HRT the biogas production of the reactors begins to level off and a pseudo steady state phase is observed until approximately 2 to 3 HRTs have expired. After a total of 2 to 3 HRTs the steady state conditions were verified in the reactors as the variance of the biogas production of the reactors above an average value was observed to be less than 5% on a daily basis. The reactors were maintained at steady conditions for an minimum of an additional HRT. All results presented are the averages and corresponding 95% confidence intervals during the steady state operation.



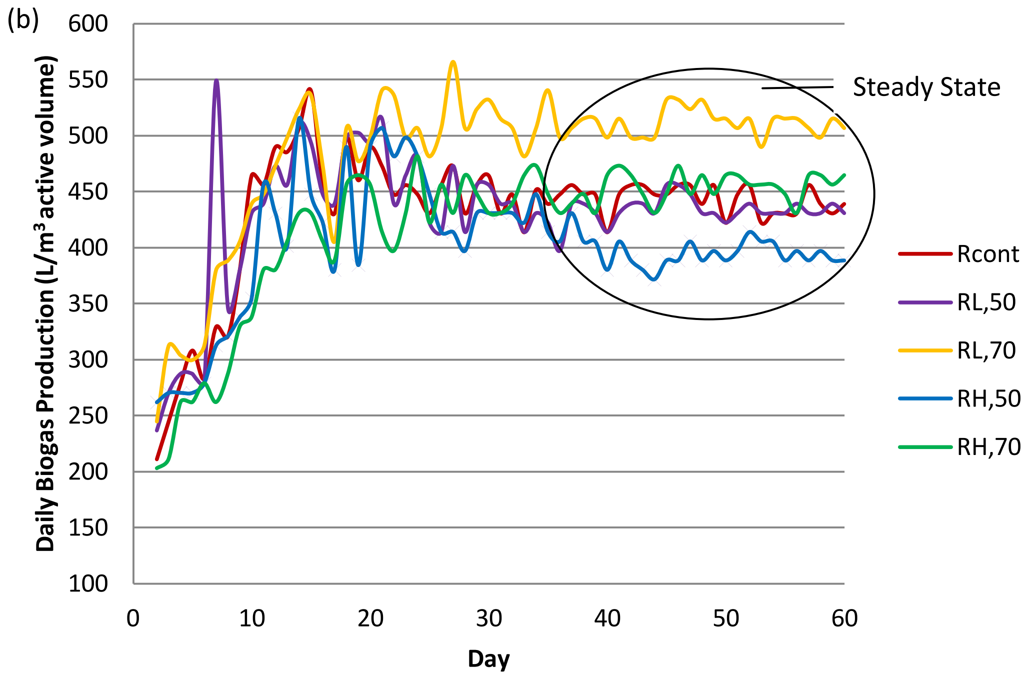


Figure 5.1 Daily biogas production of start-up and steady conditions (a) 15 d HRT, (b) 20 d HRT including start up

The results of the 2^2 factorial design are shown in Figure 5.2, where the the effects of scum concentration, temperature of the scum pretreatment and HRT at steady conditions on the biogas production are displayed as surface plots. The 15 d and 20 d reactors all demonstrated the same response to an increase in the scum treatment temperature; such that an increase in temperature showed a direct increase in daily biogas production. Concomitantly, an increase in scum concentration loading to the reactors at both 15 d and 20 d HRTs did not show any significant direct effects on the biogas production; however, Figure 5.2 displays a significant interaction between pretreatment temperature and scum concentration on daily biogas production. These findings support a the interaction observed between scum loading and pretreatment temperature in the mesophilic BMP assays presented in chapter 4.

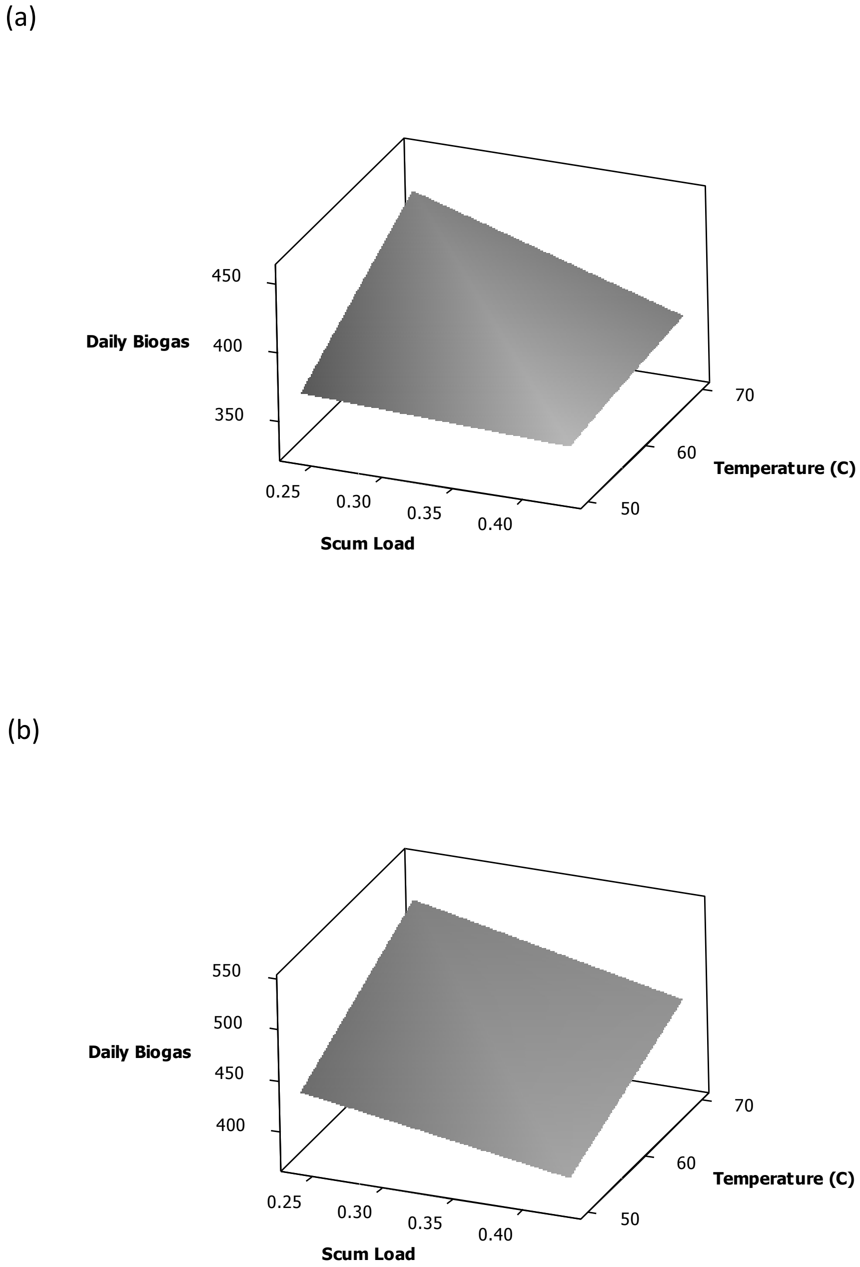


Figure 5.2 – Surface plot of average daily biogas (L/m3 · d) production vs scum load (kg VS Scum/d) and pretreatment temperature at steady state for (a) 15 d HRT and (b) 20 d HRT

Equations 5.1 and 5.2 were produced to define the surface responses shown in Figure 5.2 and to quantify the effects and interactions of the scum concentration and temperature of the scum concentrator on biogas production, (note interactions were not included in Equations 5.1 and 5.2 if plausibly 0):

$$BP_{15} = 383.25 - 17.75L_{scum} + 20.25T - 13.75L_{scum}T \qquad \text{Equ 5.1}$$

$$BP_{20} = 449 - 24L_{scum} + 35T \qquad \text{Equ 5.2}$$

Where,
BP_{15} is the expected daily biogas production at 15 d HRT ($L/m^3 \cdot d$)
BP_{20} is the expected daily biogas production at 20 d HRT ($L/m^3 \cdot d$)
L_{scum} is the coded scum concentration (1 high, -1 low)
T is the coded temperature within the scum concentrator (1 high, -1 low)

A p value of 0.05 or less was used to quantify significance of the ANOVA investigation of the effects of scum concentration and temperature of the scum concentrator on biogas production at an HRT of 15 d and 20 d. having. The effects of both scum concentration and temperature of the scum concentrator, independently, on biogas production were all determined significant at HRTs of 15 and 20 d. The interaction between scum concentration and temperature in the scum concentrator was also determined significant at an HRT of 15d; however this interaction was not significant at an HRT of 20 d. This finding suggests that given a sufficiently long HRT (which is also the sludge retention time (SRT) for the reactor) the advantages of pretreatment is less significant. It also suggests that if the HRT was decreased then the interaction between the scum concentration and the pretreatment temperature would be more significant. However based on the range of operation defined by current regulatory HRT and organic loading rate (OLR) of anaerobic digesters in Canada, the scum concentration and scum treatment temperature produce significant independent and interactive effects.

The R-squared values for Equations 5.1 and 5.2 compared to the biogas yield were 84% and 94% for the 15 and 20 d HRTs. The surface response design equations were generated based on only the reactors digesting the tri-mixture of TWAS, PS and scum. Solving Equation 5.1 for no scum is digested generated average daily biogas production values for Rcont that are within 7% of experimental results. As such the difference between Equation 5.1 and the Rcont biogas yield is attributed to the addition of scum itself which may affect the microbial populations and the kinetics of the microbial consortia. At a 20 d HRT operation, the proposed design equation (Equ 5.2) for expected biogas yield was within 2% of the measured value for Rcont.

These findings shown in Figure 5.2 demonstrate that conventional thermal pretreatment has the greatest positive effect on daily biogas production at low scum concentrations. To more easily visualize the effects of scum concentration and the pretreatment temperature, Figure 5.3 shows the average daily biogas productions during steady state for all of the reactors with error bars representing the 95% confidence intervals of the steady state measurements.

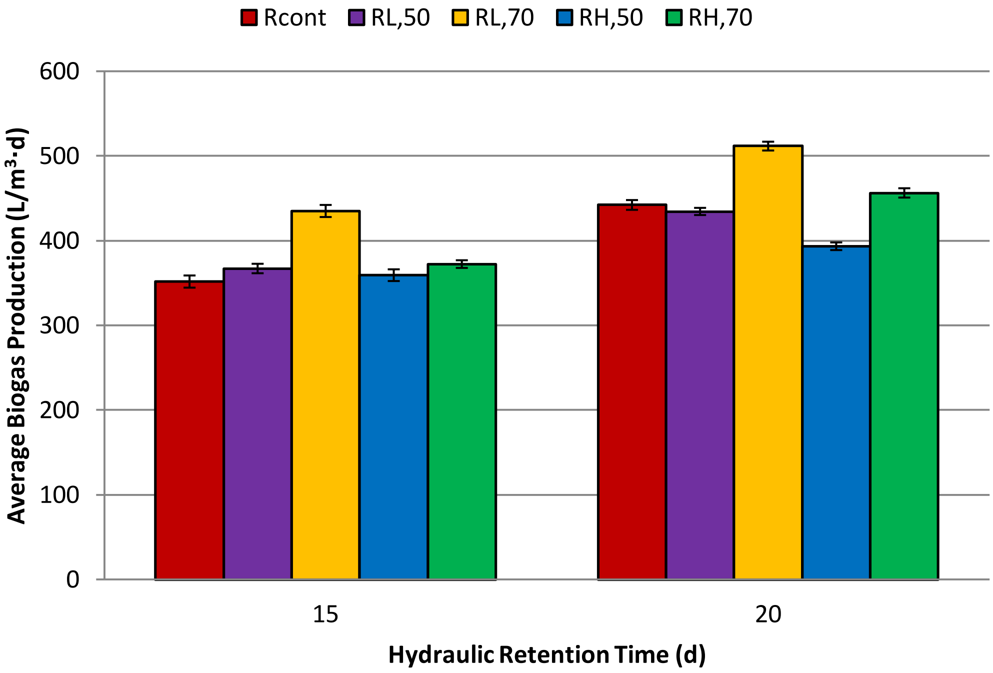


Figure 5.3 – Average daily biogas production during steady state conditions

Operating at a 15 d HRT, RL,70 produced the highest daily biogas production, which represents a 24% improvement over Rcont. RL,50 and RH,70 produced slight improvements that are significant over Rcont while RH,50 was statistically equal to Rcont. Increased biogas yields in RL,70 and RH,70 can be attributed to an increased SCOD load (table 5.1) provided by the heated scum fraction as well as the readily biodegradable nature of scum itself. Although RH,50 did not outperform Rcont, the average daily biogas production would be comparable to current values achieved at WWTPs without changing organic loading rates while also stabilizing the residual.

At a 20 d HRT operation, RL,70 produced the greatest average daily biogas production again, which represents a 16% improvement over Rcont. RH,70 produced a slightly significant increase in biogas production. RL,50 was statistically equal and RH,50 produced less daily biogas than Rcont. Similar to the 15 d HRT operation, reactors with scum concentrator temperatures of 70°C produced the largest increase in biogas production and as such is believed to have better integrated the scum within the reactors and in turn enhanced the SCOD load. Scum concentration produced the best daily biogas production results at the low fraction, or 3% by volume. Observing the reactors over time, the scum appeared to remain well mixed within the system at low loads. Whereas in contrast, high scum fraction, or 7% by volume, appears to separate from mixed liquor and coagulate to the top of the reactors. This phenomenon of separation removed portions of the scum from the microbial environment and hindered the digestion processes.

Figure 5.4 shows the average daily biogas production normalized per the daily biogas produced by Rcont. This figure clearly demonstrates that all the reactors exhibit decreasing biogas production in comparison to Rcont as the HRT is increased from 15 to 20 d HRT. In examining this decrease in biogas production at an HRT of 20 d as compared to an HRT of 15 d, it is important to recall that a a constant loading rate of 1.6 $kgVS/m^3 \cdot d$ was maintained in each reactor. Thus, the microbial consortia of the reactors during the 20 d HRT experiments were provided a longer digestion period to consume the same mass of organic material. Young et al. (2012) proposed that during continuous digestion, scum fractions would have preferential degradation and yield a higher initial biogas production, however, given a longer period of time the ultimate biogas production would produce an improvement, but at a smaller yield. For each of the reactors operated this hypothesis was confirmed as the 15 d HRT produced a greater positive (desired) differential for all reactors when normalized with the control as compared to the 20 d HRT reactors.

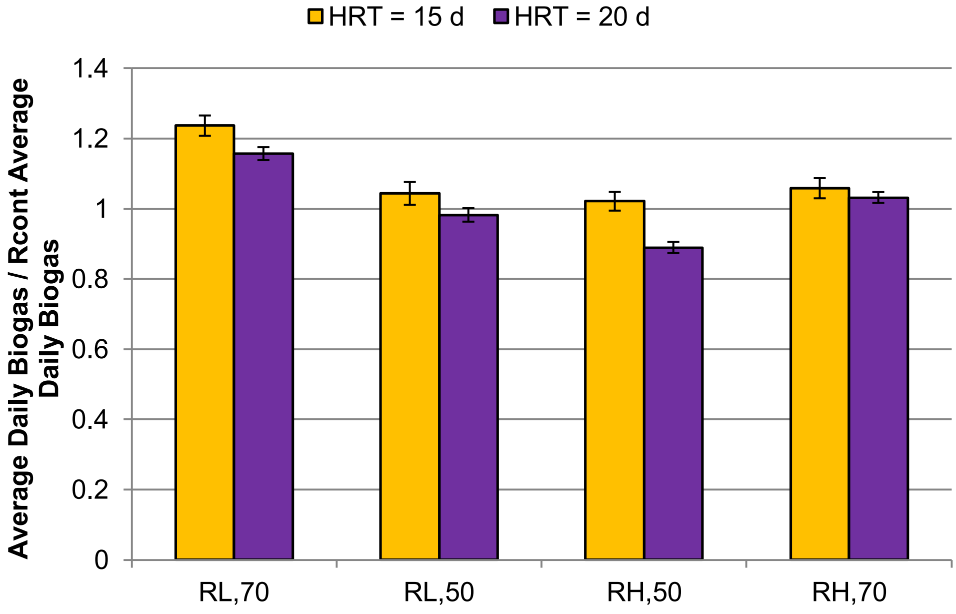


Figure 5.4 – Average daily biogas normalized to Rcont

The biogas contribution from the TWAS and PS mixture is 276 m^3 of biogas/tonne VS added. By accounting for this biogas production attributed to the TWAS and PS components, the tri-mixture reactors can be extrapolated to determine the biogas contribution from the scum component. At a 20 d HRT the RL,70 produced the greatest biogas yield of 452 m^3 of biogas/tone-VS-scum added. RH,70, RL,50 and RH,50 yielded 310, 257, and 221 m^3 of biogas/tonne-VS-scum added respectively. As expected, the biogas yield of scum was higher than the TWAS and PS mixture for the best performing reactors (RL,70, RL,50 and RH,70). Reactors RL,50 and RH,50 experienced mixing trouble which as afore mentioned caused separation of the scum fraction and likely hindered successful digestion of the scum component. Comparing the scum derived biogas yields at 40 day batch assay (Young et al., 2012) the scum did not reach its biogas potential of (794 m^3/tonne-VS-scum added). Operating at 15 and 20 d HRTs, the microbial consortia/sludge contact time was significantly lower, which accounts for the 50% decrease in relation to the batch assays.

5.4.2 Operational Results

Gomec (2006) concluded that pH control is necessary when digesting scum in a continuous system due to rapid hydrolysis and acidogenesis of the scum component while scum lacks natural alkalinity. Lay et al., (1997) showed that the digestion of scum decreased the pH of their system to levels below 5 without the addition of alkalinity, which is significantly below the optimal pH range of 6.7 to 7.3 for acidogenesis and methanogenesis to occur simultaneously. In the herein presented study, R7,50 and R7,70 contained the highest scum fraction and the pH of these reactors was maintained at an average of 7.0 and 7.1 at HRTs of 15 and 20 d, respectively, without additional alkalinity being added. phase. These pH values are within the optimal pH range form anaerobic digestion, however it should be noted that additional scum is likely to decrease the pH further as co-digesting scum with TWAS and PS decreased the rate of hydrolysis and acidogenesis while also adding additional buffering capacity. To maintain anaerobic digestion health, it is recommended that scum addition by volume remain below 7%.

VS stabilization and energy production through methane generation are the primary reasons for the use of anaerobic digestion at MWWTPs. Throughout the experimental phase all reactor effluent was measured for pH, VS, TS, TCOD, SCOD, total alkalinity, volatile acids (VA) and NH_3-N to monitor the health and performance of the reactors. Biogas was analyzed using a gas chromatograph to qualitatively analyze %CH_4. VS stabilization and %CH_4 are shown in Figure 5.5. Percent methane for all of the reactors was statistically equal ranging from an average of 59 to 61%. The VS stabilization data shows that VS stabilization was not representative of biogas production for the scum containing reactors; where the reactors with the highest biogas production did not correspond to the reactors with the greatest VS destruction. The largest discrepancies between biogas production and VS destruction occurs in the high scum containing reactors, RH. These discrepancies between observed VS destruction and expected VS destruction values based on the biogas production may be due to the sampling method used to collect the waste from the reactors and the scum remaining partially separated in the reactors. Samples collected for VS measurements were extracted from the centroid of the reactors after vigorous mixing, while a portion of the scum remained congealed and floating or adhered to the wall of the reactors. Since scum is highly organic its absence in the VS sample may account for the skewed VS destruction measurements presented.

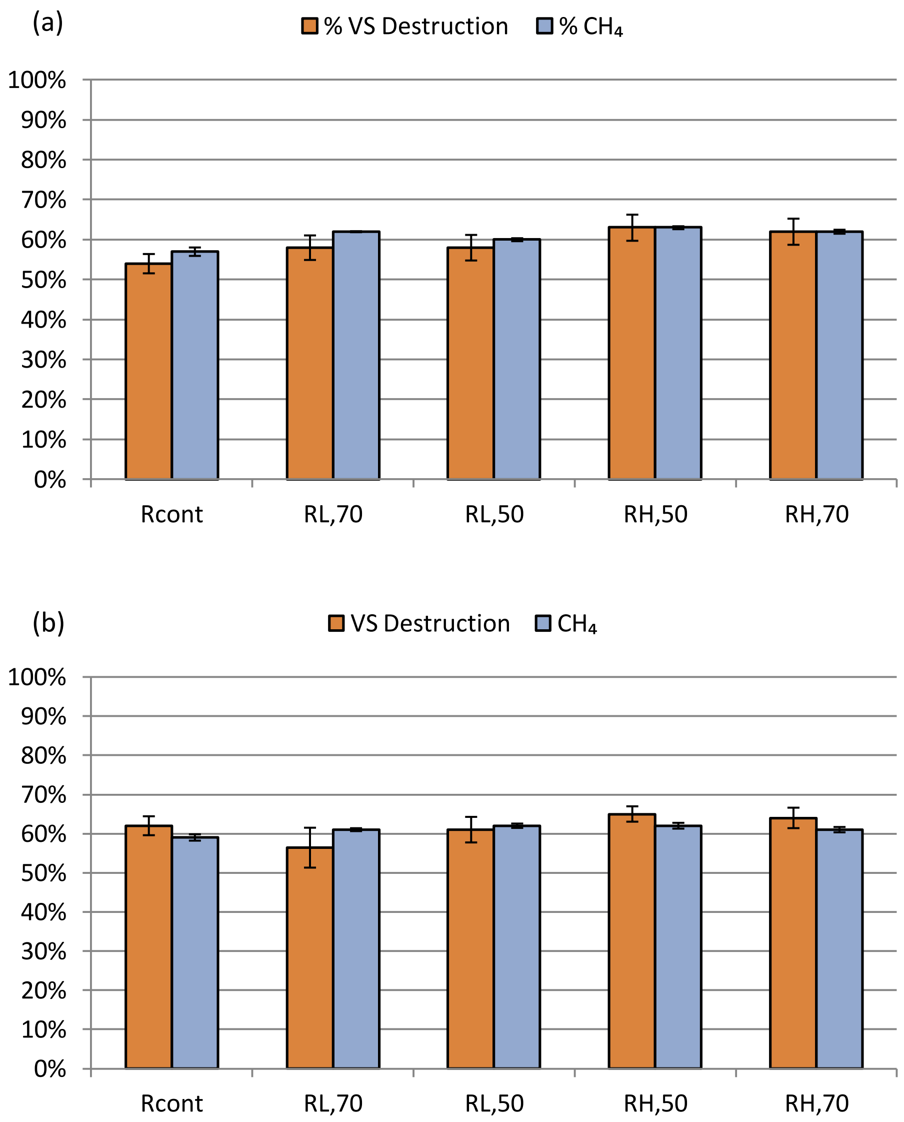
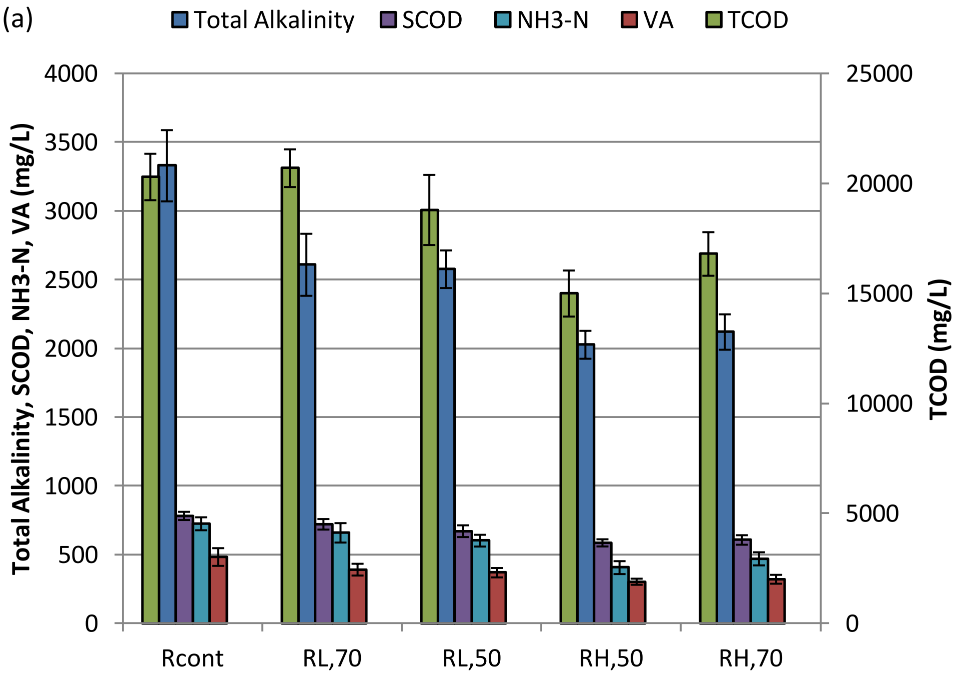


Figure 5.5 VS Destruction and %CH_4 content of biogas (a) 15 d HRT, (b) 20 d HRT at steady state operation

Figure 5.6 shows the total alkalinity, TCOD, SCOD, NH_3-N, and volatile acids (VA) for 15 and 20 d HRTs. In accordance with the pH measurements, the total alkalinity was lowest in the highest scum loaded RH reactors, expressing the significance of not overloading a continuous system with scum. TCOD, SCOD, NH_3-N, and VA were within the expected range of a MWWTP anaerobic digestion facility and are comparable to current ROPEC operation. All values presented confirm lab scale anaerobic digestion was operated comparable to full scale application and were maintained in a healthy state suggesting scum would not adversely affect current solid waste management practiced by MWWTPs.



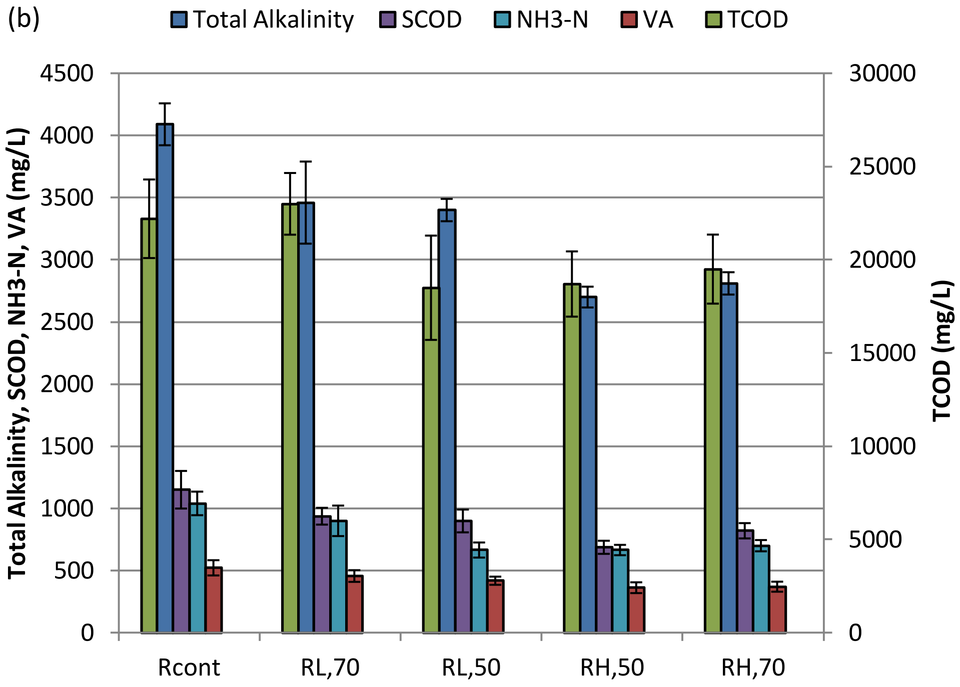


Figure 5.6 – Steady state effluent values (TCOD secondary axis) (a) 15 d HRT (b) 20 d HRT

5.4.3 Microbial Viability Results

Microbial viability analysis was performed on the Rcont, RL, 50, and RL,70 reactors operating at a 20 d HRT. The two lower scum loaded reactors in the study were chosen for viability analysis based on their performance. For comparative purposes, Rcont was used as the standard baseline condition and temperature in the scum concentrator as an additional scum variable. The temperature in the scum concentrator as shown in the previous section was the greatest factor in determining biogas production. Examples of acquired microscopic images are shown in Figure 5.7; where 20 images were acquired and analyzed from each reactor with the images shown in Figure 5.7 being representative of the 20 image data set.. From visual inspection of Figure 5.7, it appears that the control and RL,70 appear to have a greater live fraction (illuminated in green) relative to dead fraction (illuminated in red) as compared to RL,50.

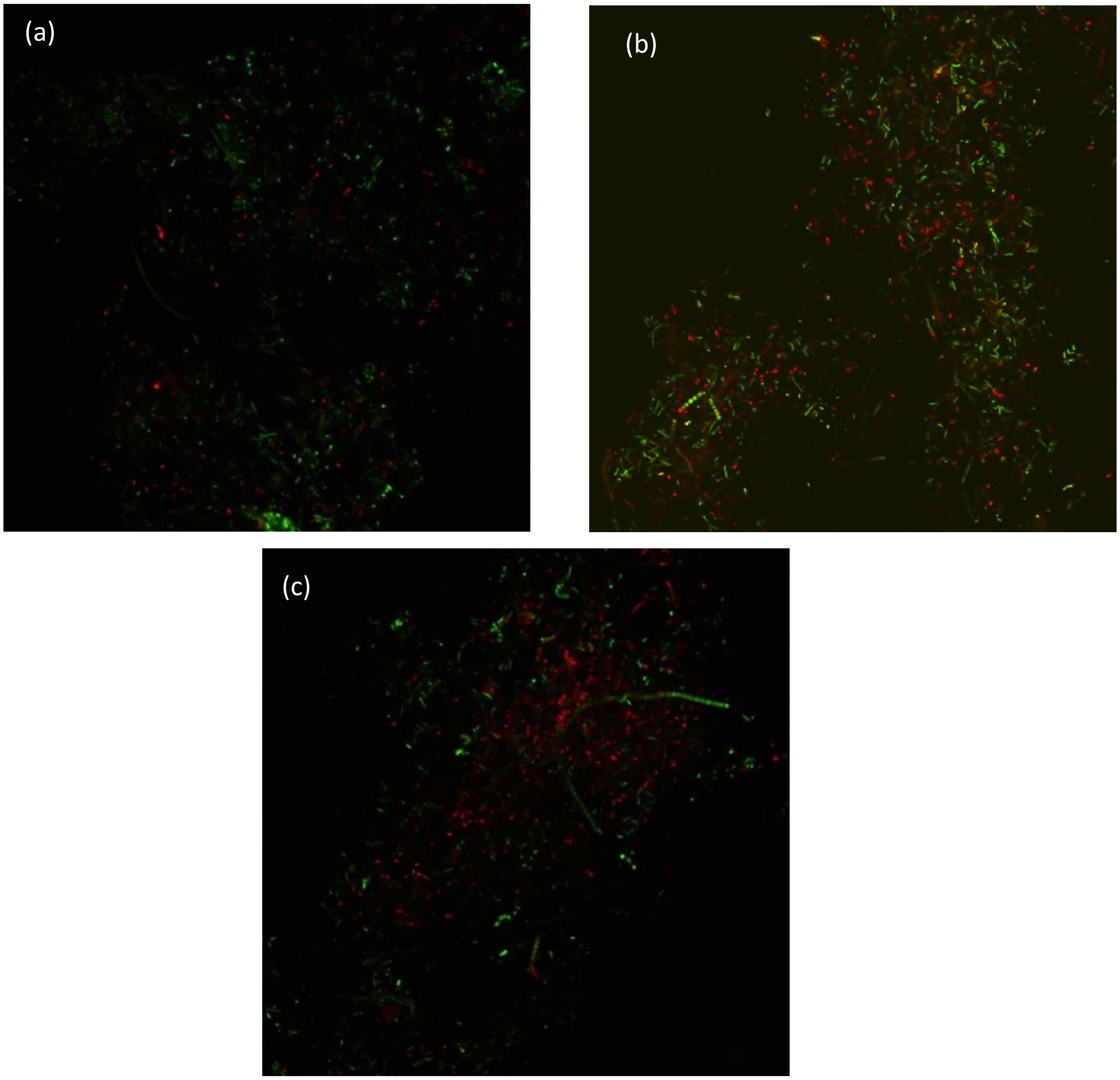


Figure 5.7 – Live/Dead Imagery (a) Rcont, (b) RL,70, (c) RL,50

Figure 5.8 shows the percentage of live cells in the three reactors at 20 d steady state operation. An average value is shown and was calculated by dividing the percent area of live cells by the total percent area of all cells. The error bars show the 95% confidence interval of the 20 images analyzed for each reactor.

The live fraction observed was statistically equal between Rcont and RL,70 as well as between RL,70 and RL,50. Rcont, however, shows a significantly higher live fraction of cells than RL,50.

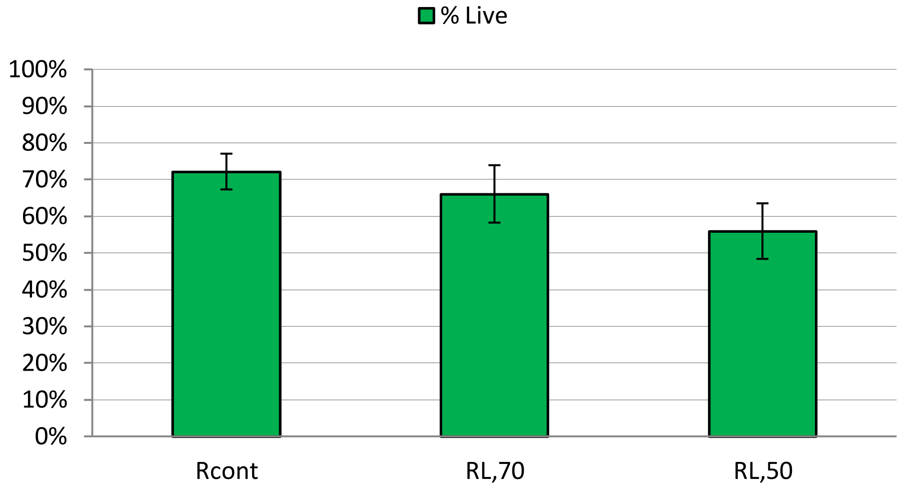


Figure 5.8 – Cell viability at 20 d HRT operation

As was shown in Figure 5.3, Rcont produced statistically the same amount of biogas daily as RL,50, however the cell viability results of Figure 5.8 show that significantly more live cells, 26%, exist in the Rcont reactor. Concomitantly, RL,70 produced significantly more biogas, 16%, then Rcont but the two reactors have statistically the same fraction of live cells. Using NI Vision assistant, the number of live cells per m^3 of waste was calculated to better analyze the reactor results at a microbial level. Figure 5.9 shows the biogas yield per live cells and thus presents the true specific rate of biogas production in each reactor. The error bars represent the 95% confidence interval over a series of 20 images.

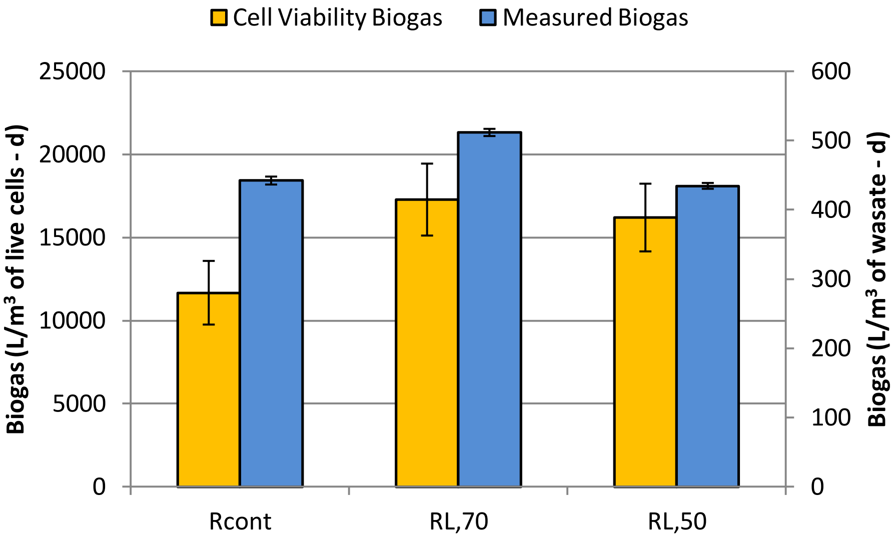


Figure 5.9 – Biogas production normalized for live cell fraction

Rcont produced 11,700 L of biogas/m^3 of live cells · d, whereas RL,70 and RL,50 produced 17,300 and 16,200 L of biogas/m^3 of live cells-d respectively. This suggests either the substrate mixture of TWAS:PS:Scum produces a higher specific substrate utilization rate within the same microbial population or different populations are dominant in Rcont and RL,70/RL,50 reactors. Since both RL,70 and RL,50 produced statistically the same biogas production rate per m^3 of live cells, it is likely different populations are dominant in scum containing reactors vs Rcont. The methanogenic cells present in scum containing reactors produced 48% more biogas per m^3 of live cells than methanogenic cells present in Rcont. In order to determine if the increase is a result of preferential substrates provided by scum degradation or if different microbial populations are present future recommendations entail DNA extraction and analysis. True optimization of the anaerobic digestion process will involve coordinating substrates to the microbial populations and subsequent activities present though the live/dead and DNA extraction techniques.

5.5 Conclusion

This article presents and discusses the operation of continuously co-digesting PS, TWAS, and scum. All tests were performed using municipal sludge and scum collected from a full-scale WWTP, ROPEC, ON Canada. All methodologies and analysis for the tests were performed using standard or proven methods.

Continuously co-digesting PS, TWAS, and scum showed that scum can be beneficial within the anaerobic digestion process. The effects of thermal pretreatment of scum through adjusting the temperature of the scum concentrator showed significant impact on biogas production for both 15 and 20 d HRT operation. Reactors pretreated with thermal temperature of 70°C exhibited more biogas production than reactors pretreated at 50°C. This phenomenon is explained through the higher SCOD fraction as well as a scum fraction more easily integrated to the completely mixed system. Scum treated at 70°C was less likely to congeal and float to the surface.

Scum concentration also had a significant effect on biogas production. Reactors with 3% scum fraction by volume produced more biogas than reactors with 7% scum fraction by volume when heated to 70°C and were statistically equal when heated to 50°C. Reactors fed with higher scum fraction exhibited a decreased pH, although still within the acceptable range. As such the work recommends to not exceed 7% by volume in an anaerobic digester without the addition of an alkaline substrate or chemically adding alkalinity. VS destruction, TCOD, SCOD, NH_3-N, and VA were all in acceptable ranges for digester effluent and percent methane remained at 60% for all reactors.

Cell viability analysis showed a significantly higher biogas yield per live cell in scum containing reactors. Further research should be conducted through DNA sequencing to determine if the yield is due to differential microbial populations or an environment more conducive to methanogens operating at their maximum specific substrate utilization rate. Results could lead to further optimization of the anaerobic digestion process and add additional improvements to biogas production.

5.6 Contributions

All of the experimental set-up, lab-work, and initial writing and interpretation of the data was performed by Bradley Young. Editing and further investigation of the data was a collaborative effort between Bradley Young, Dr. Kevin Kennedy, and Dr. Robert Delatolla. The process was performed diligently by all over the course of a year.

5.7 References

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Chapter 6

Impact of Scum Digestion at ROPEC

6.1 Scum Generation

Empirical scum generation data at the Robert O. Pickard Environmental Centre (ROPEC) and the necessary tools to generate such data are not presently available to plant operators or the University of Ottawa. Thus this investigation uses the average scum generation data shown in Table 2.1; low, average and high mass concentration values of 2.4, 6.0 and 9.6 kg/ML wastewater processed (Outwater, 1994). These scum concentration values were combined with ROPEC's average hydraulic flowrate of 545 ML / d and as such the daily low, average and high mass of scum generated is predicted to be 1300, 3300 and 5200 kg of scum per day respectively.

6.2 Biogas Generation from Scum Addition

ROPEC consists of four gas mixed pancake and two hydraulically mixed pseudo egg shaped mesophilic digesters with approximate individual active volumes of 8,800 m^3 and 12,500 m^3 respectively. Typically one pancake digester is out of service for cleaning, maintenance, or recirculation leaving a total plant active volume of 51,400 m^3. If scum is distributed evenly among the five digesters operating at 1.6 $kgVS/m^3 \cdot d$, the total scum load is 0.01, 0.03 and 0.05 $kgVS\text{-}scum/m^3 \cdot d$ for low, average and high scum generation values respectively. The semi-continuous lab scale reactor results indicated the necessity to provide adequate mixing to maintain a fully mixed system so as to avoid coagulation and flotation of the scum fraction. Scum coagulation and floatation would be more likely to occur in the gas mixed pancake digesters which have been reported by ROPEC staff to be poorly mixed, thus the scum fraction should be diverted solely to the two pseudo egg shaped hydraulically mixed digesters. The resulting low, medium and high scum fractions mass loadings would be 0.03, 0.07 and 0.10 kgVS-scum/m3 · d respectively for each pseudo egg shaped digester.

However, the best tri-mixture digestion lab scale results were achieved with RL,70 which had a scum load of 0.24 kgVS-scum/m^3 · d and overall reactor volumetric load of 1.6 kgVS /m^3 · d. By sending all of the generated scum to one hydraulically mixed pseudo egg shaped digester scum loads could be increased 0.06, 0.14 and 0.20 kgVS-scum/m^3 · d and overall reactor volumetric load of 1.6 kgVS /m^3 · d for low, average, and high scum generation values, respectively. This increased scum loading is in a range closer to what was tested in the lab (RL,70) and it is expected would generate the greatest increase in biogas. Using the biogas/CH_4 yields obtained in the semi-continuous lab scale study, digesting scum as a tri-mixture (scum/PS/TWAS) is expected to yield an additional 340, 790 and 1200 m^3/d of biogas production over digestion of the binary mixture of PS/TWAS for low, average and high scum loads respectively. These values represent a 0.6%, 1.9% and 2.0% biogas increase in respect to current PS/TWAS digestion operations at ROPEC.

Using the specific heat of sludge as 4,200 J/kg · °C, the heating requirement to increase the temperature of scum from an influent value of 20°C to 70°C is 1.1 x 10^9 J/d. Assuming the scum concentrator/heater can be modeled thermodynamically as an anaerobic digester, the heat losses associated with an insulated concrete scum concentrator that will maintain the 70°C holding temperature processing 5200 kg/d of scum (ROPEC high value) with a 2 day HRT and external temperature of 25°C able (with a safety factor of 1.2) is 1.0 x 10^8 J/d. Using the low, average and high scum quantities for ROPEC and the estimated additional biogas/CH_4 production the potential energy benefit of digesting scum can be determined. Assuming a heating value of 22,400 kJ/m^3 biogas (60% CH_4, Metcalfe and Eddy, 2003) mesophilicaly co-digesting heat treated scum with PS/TWAS resulted in a net increase in energy producing 64.0, x 10^8 1.65 x 10^{10} and 2.41 x 10^{10} J/d for low, medium and high scum generation values. Assuming ROPEC produces typical quantities of scum, pretreatment will provide a positive net heating balance.

Chapter 7

Conclusions and Recommendations

7.1 Conclusions

Mesophilic anaerobic co-digestion of scum and municipal sludge has the potential to increase biogas production and improve the energy production at MWWTPs. The addition of scum to the anaerobic digestion system will decrease disposal costs and maintains a healthy continuous digestion process.

From BMP assays, a general conclusion is the readily biodegradable nature of scum. As specific loads in the assays of 11.2 kg VS added/kg VS Inoculum underwent digestion, the VFA accumulation produced an unbalanced digestion process. The pH in these assays dropped to 5.7 and remained below 6.5 for 10 to 15 days, leading to incomplete souring of the system.

The best performing BMP assays were conducted with low scum concentrations and specific loads of 8.1 kg VS added/kg VS Inoculum. These assays also exhibited an accumulation of VFAs, however, the natural and added alkalinity was sufficient to support balanced digestion. The CBP for the best performing tri-mixture assay (RL,50,48) was 692 m^3/tonne VS added representing a 61% improvement over digesting TWAS and PS. All of the RL assays performed comparable or exceeded the CBP of the control suggesting scum is suitable for co-digestion provided specific loads do not exceed the buffering capacity of the system.

Based on the 58% methane content of the BMP assays, the methane yield for scum in a balanced system is 400 – 800 m^3/tone-VS-added placing the residual in par with residual and floatable fats. TWAS and PS co digestion produced 250 m^3/tonne VS added expressing the high energy yield capabilities of scum in the anaerobic digestion process if the system remains balanced. The scum concentration and scum concentrator pretreatment factors affected the CBP and daily biogas production rates. In terms of CBP, the factors affecting BMP assay performance was scum concentration with temperature with HT having minimal effects. The scum concentration and HT interactions was significant with the best performance achieved at low concentrations and high HT. Similarly, the daily biogas production rate was most affected by scum concentration, but the temperature became more significant as it improved the solubility and mix ability of scum within the system. The general conclusion is scum concentration plays the biggest role in the digestion process and scum concentrator temperature should be analyzed further in a continuous system.

Operation of semi-continuous reactors loaded at 1.6 kg $VS/m^3 \cdot d$ for 15 and 20 d HRT indicates an improvement in reactors operated as a tri-mixture of scum, TWAS and PS. Operation at 50 d HRT produced a 24% improvement over the control operating with low scum concentration and 70°C for 48 h in the scum concentrator. The increased biogas production can be attributed to an increased SCOD load as well as the enhanced mix ability of the heated scum. Operating with high scum concentration and lower temperatures were statistically equal to the control, signaling an optimization process to enhance biogas production with scum. Concomitantly, statistically equal biogas production provides confidence that scum addition to anaerobic digesters will not inhibit biogas production and is a beneficial reuse of the residual.

Operation at 20 d HRT produced similar results, except the improvement at low scum concentration and 70°C for 48 h was 16%. The high scum concentration reactors at 20 d HRT appeared to come out of a completely mixed state and coagulate at the top of the reactor hindering the microbial consortia from digesting the scum fraction. This led to slightly lower or statistically equal value daily biogas production to the control. Monitoring effluent quality for the semi-continuous reactors showed all effluent parameters were comparable for scum containing reactors when compared to the control. pH, VS, TS, TCOD, SCOD, total alkalinity, volatile acids, NH_3-N, and $\%CH_4$ were in the acceptable range for all reactors exemplifying the ability for scum to be digested efficiently without hindering process performance or effluent quality.

Cell viability staining was conducted for 20 d HRT, which provided the percentage of live and dead cells in the semi-continuous reactors and the ability to normalize the biogas production in relation to living organisms in the system. It was determined that the control reactor and the low scum containing reactor at 70°C pretreatment were statistically equal with respect to the percentage of live cells, while the low scum containing reactor at 50°C pretreatment contained statistically less live cells than the control reactor. These findings suggests that the scum fraction produces more daily biogas per live organism. However, it should be mentioned that the results may be indicative of a scum containing reactor that was not optimized and as such the scum containing reactor contained less living cells which decreased its biogas production from its theoretical potential.

7.2 Recommendations for future research

The following recommendations are intended to further the research presented in this thesis in order to fully optimize the biogas potential of scum:

- Examine other forms of pretreatment (chemical, microwave, ultrasound etc.)
- Increase loading rate of the continuous system
- Examine a two phase system to mitigate the effects of rapid hydrolysis and acidogenesis

Appendix A

Sample Calculations

A-1 Biogas (L/m^3 of live cells)

Rcont

Daily Biogas (L/m^3 of active volume) = 442

% Cells in Waste = 35 ± 0.15

% Live Cells = 72 ± 7

V1 (mL) = 1

V2 (mL) = 0.15

$$\mathrm{Biogas}\ \left(\frac{\mathrm{L}}{\mathrm{m}^3\mathrm{of\ live\ cells}}\right) = \mathrm{Daily\ Biogas}\ * \frac{1}{\frac{\mathrm{V2}}{\mathrm{V1}} * \%\ \mathrm{Cells\ in\ Waste} * \%\ \mathrm{Live\ Cells}}$$

$$Biogas\ \left(\frac{L}{m^3\ of\ live\ cells}\right) = 442 * \frac{1}{.\frac{15}{1} * 35\% * 72\%} = 11700$$

A-2 Heat Loss

High Scum Production

Scum Produced (kg/d) = 5200

Volume of Scum (m^3) = 5200

Scum Concentrator Dimensions:

Length (m) = 5.5

Width (m) = 1.5

Depth (m) = 1.5

U, Overall Coefficient of heat transfer ($J/m^2 \cdot s \cdot °C$) = 0.7

T scum (°C) = 20

T ambient (°C) = 25

T concentrator (°C) = 70

Specific Heat of Sludge (J/kg · °C) = 4200

$$\mathrm{q_{heat\ scum}} \left(\frac{\mathrm{J}}{\mathrm{d}}\right) = \mathrm{Scum\ Produced} * (\mathrm{Tconcentrator} - \mathrm{Tscum}) * \mathrm{Specific\ Heat}$$

$$q_{heat\ scum} \left(\frac{J}{d}\right) = 5200 * (70 - 20) * 4200 = 1.1 * 10^9$$

$$q_{loss} \left(\frac{J}{d}\right) = U * A_{surface}(Tconcentrator - Tambient) * (86{,}400)$$

$$q_{loss} \left(\frac{J}{d}\right) = 0.7 * 5.5 * 1.5 * (70 - 25) * 86{,}400 = 2.2 * 10^7$$

$$q_{loss\ requirement} \left(\frac{J}{d}\right) = q_{heat\ scum} + \sum q_{loss\ each\ surface}$$

$$q_{loss\ requirement} \left(\frac{J}{d}\right) = 1.1 * 10^9 + 1.0 * 10^8 = 1.2 * 10^9$$