LAB-SCALE RECUPERATIVE THICKENING:

DEWATERING OPTIMIZATION

OPTIMIZING POLYMER ASSISTED DEWATERING IN RECUPERATIVE THICKENING VIA A LAB-SCALE SYSTEM FOR ENHANCED BIOGAS PRODUCTION IN ANAEROBIC DIGESTION PROCESSES

By
JEFFREY PATRICK BELL COBBLEDICK, B. ENG

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AUTHOR:	Jeffrey Patrick Bell Cobbledick, B. Eng. (McMaster University)
SUPERVISOR:	Dr. D. Latulippe
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LAY ABSTRACT

In wastewater treatment (WWT), solid wastes are treated using a technique called anaerobic digestion (AD) which involves the conversion of solids in biogas by anaerobic bacteria. Biogas is a mixture of mostly methane and carbon dioxide and can be used as a fuel source for energy production. There's growing interest in the use of high performance AD processes for the production of biogas at WWT facilities to offset the energy demands associated with WWT. Recuperative thickening (RT) is a promising technique which involves recycling a portion of the digested solids back to the digester. In this work, a detailed and comprehensive study of RT processes was conducted at the lab scale; a demonstration of the optimization of polymer assisted dewatering is given and biogas production and quality monitored. Two 1.5 L custom designed digesters were operated in parallel one as a 'control' and the other operating under a semi-batch RT.

ABSTRACT

There is growing interest in the use of high performance anaerobic digestion (AD) processes for the production of biogas at wastewater treatment facilities to offset the energy demands associated with wastewater treatment. Recuperative thickening (RT) is a promising technique which involves recycling a portion of the digested solids back to the incoming feed. In general there exists a significant number of knowledge gaps in the field of RT because the studies that have been conducted to date have almost exclusively occurred in pilot or full scale trials; this approach greatly limits the amount of process optimization that can be done in a given trial. In this work, a detailed and comprehensive study of RT processes was conducted at the lab scale; a demonstration of the optimization of polymer assisted dewatering is given and biogas production and quality monitored. Two custom designed digesters (capacity = 1.5 L) were operated in parallel with one acting as a 'control' digester and the other operating under a semi-batch RT mode; both digesters were also operated in parallel under RT with alternative polymer flocculants. There were no significant changes in the overall biogas methane composition; however the RT digester had an average biogas productivity over two times higher than the control one. It was found that the recycling of the polymer flocculant back into the RT digester resulted in a significant improvement in dewatering performance. At the highest polymer concentration tested, all polymer flocculants demonstrated equivalent dewatering performance achieving over 6 times lower CST's than the control; at lower polymer concentrations the 4516 polymer flocculant had superior dewatering performance. Thus, there exists an opportunity to decrease the overall consumption of polymer flocculants

through judicious selection of the flocculant and the dose that is used both for the thickening and end-stage dewatering processes in RT digesters.

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LIST OF ABBREVIATIONS

Wastewater Treatment ……WWT
Anaerobic Digestion ……AD
Wastewater Treatment Facility ……WWTF
Combined Heat and Power ……CHP
Environmental Protection Agency ……EPA
Conventional Anaerobic Digestion ……...CAD
Long-chain Fatty Acids ……...LCFA
Fats, Oils, and Greases ..……FOG
Organic Loading Rate ……...OLR
Volatile Fatty Acids ……...VFA
Recuperative Thickening ……..RT
Solids Retention Time ……SRT
Hydraulic Retention Time ……HRT
Specific Resistance to Filtration ……SRF
Capillary Suction Time ……..CST
Primary Sludge ……..PS
Thickened Waste Activated Sludge ……...TWAS
Total Solids ……...TS
Volatile Solids ……..VS
Total Suspended Solids ……..TSS
Volatile Suspended Solids ……VSS
Chemical Oxygen Demand ……...COD
Total Chemical Oxygen Demand ……...TCOD
Soluble Chemical Oxygen Demand ……SCOD
Quaternized-N,N dimethylamino ethylacrylate ……..DMAEA-Q
Total Kjeldahl Nitrogen ……TKN
Carbon : Nitrogen Ratio ……C:N
kg Polymer Flocculant per Total Tonne Solids ……..kgP/TTS

- Chapter 1 -
Introduction

1.1 Anaerobic Digestion

1.1.1 Role in wastewater treatment

The collection and disposal of human waste has been an obstacle since early civilizations and prior to the introduction of wastewater treatment (WWT) most human waste was either released directly into nearby waterways or collected in cesspools. Biological WWT became a necessity when cities became densely populated at the turn of the industrial revolution in order mitigate odours and the contamination of drinking water sources with water-borne illnesses such as cholera. Early WWT used biological tricking filters where sewage was dripped over aggregates covered in a biofilm which removed some contaminants [Henze et al., 2008]. Eventually, this led to the discovery of the 'activated sludge' process in the U.K which involved a sequential batch reactor; this later became known as 'conventional' WWT however there have been several technological advances along with operational changes in order to improve the discharge effluent quality [Henze et al., 2008].

The early WWT processes focused of the treatment of the liquid fraction of domestic wastewater and the solids fraction was often seen as a nuisance product to be used as a fertilizer among the likes of animal manure. While anaerobic digestion (AD) was already an established technology (though not widely used), it was not until the energy crisis in the 1970's along with a demand for industrial WWT that the focus shifted from aerobic treatment of the wastewater to the anaerobic treatment of sewage for energy

production [Henze et al., 2008]. However, in as early as 1922, the energy potential of municipal waste solids has been identified. As can be seen in Figure 1, a newspaper clipping depicts a municipal sewage plant using the sewage waste solids for energy production in March of 1922.

March, 1922 67

Gas from Sewage Waste Runs City Power Plant

How the sewage disposal plant at Birmingham, England, supplies its own power is described in the illustration. Gas from the sewage drives an engine of 20 brake horsepower, which operates a centrifugal sludge pump

SEWAGE that costs large cities tremendous sums each year can be turned into a source of power equivalent to thousands of tons of coal! The waste now dumped into rivers or shipped to sea may be used to run factories or to light buildings!

That conversion of sewage into power is possible has been proved conclusively by the city of Birmingham, England. There a suction gas engine of 20 brake horsepower has been successfully driven by the gases given off by sewage sludge.

On the basis of the Birmingham experiments, an American city that must now pay for the disposal of 400,000 tons of sewage sludge a year might produce 320,000,000 cubic feet of gas suitable for heat and power, or, in terms of energy, 16,000,000 horsepower hours at 20 cubic feet per brake horsepower.

The apparatus for producing gas from sewage consists of two sludge digestion tanks in which the sewage is allowed to ferment. The gases given off are composed of from 25 to 75 per cent of methane, or marsh gas.

A gas engine of the usual type will run on sewage gas without adjustment of the valves. Sewage gas has a higher calorific value than some illuminating gas, averaging about 650 thermal units to the cubic foot, as against 550.

The Birmingham engine runs about six hours a day and is used to operate a centrifugal sludge pump that moves the wet sludge from the gas-generating tank to the drying grounds. In this process a small proportion of the waste material produces enough power to run the pumps of the sewage disposal plant. If all the material were used, there would probably be enough gas available to light the city.

Fig. 1 – Newspaper clipping from 1922 depicting power generation from sewage waste. [Popular Science Monthly, 1922]

AD has several other uses beyond the production of biogas (also known as ‘offgas’ or ‘digester gas’) which can be used as a fuel source. It is also used both for the stabilization of sewage solids and for the destruction of pathogens carried in municipal sewage. It has been estimated that without any modifications to a current AD process, a wastewater treatment facility (WWTF) could produce 39-76% of its electricity demand through AD biogas production and subsequent energy recovery through a combined heat and power plant (CHP) [Silvestre et al., 2015]. A 2007 Environmental Protection Agency (EPA) report to congress outlined the usage of both AD at WWTFs as well as the extent

of biogas utilization in these facilities. The division of waste flows from the EPA 2007 report are summarized below in Figure 2.

Despite the energy and health benefits of anaerobically digesting wastewater solids, it is surprising that as of this 2007 EPA report, only 60% of all wastewater flow in the U.S travels to WWTFs with AD. Furthermore, only one-third of the WWTFs that implement AD undergo both AD and gas utilization; meaning that two-thirds of the biogas produced from AD in the U.S is flared off as a waste product becoming a source of atmospheric carbon dioxide.

WWTFs by Wastewater Flow Rates (MGD)	Total WWTFs	Total Wastewater Flow at WWTFs (MGD)	Wastewater Flow to WWTFs with Anaerobic Digestion (MGD)	Wastewater Flow to WWTFs with Anaerobic Digestion and Gas Utilization (MGD)	Wastewater Flow to WWTFs with Anaerobic Digestion and No Gas Utilization (MGD)
> 200	15	5,147	3,783	1,530	2,253
100 – 200	26	3,885	2,652	1,462	1,190
75 – 100	27	2,321	1,350	604	745
50 – 75	30	1,847	1,125	327	798
20 – 50	178	5,373	2,573	698	1,876
10 – 20	286	3,883	2,036	261	1,775
5 – 10	504	3,489	1,728	257	1,471
Total	**1,066**	**25,945**	**15,247**	**5,140**	**10,107**

Fig. 2 – Wastewater flows in the U.S. from a 2007 EPA report with regards to facilities that treat their sewage waste with AD and use the resulting biogas. [Eastern Research Group, Inc., 2007].

1.1.2 Conventional anaerobic digestion

Conventional AD (CAD) is a well-established process for WWT that reduces the total mass of waste solids by breaking down organic matter in the absence of oxygen at mesophilic temperatures (~37 ^{o}C). The production of methane in CAD follows a specific pathway with several different bacteria and reactions involved at various steps in its production. In 2002 the IWA published a widely cited (over 550 times) model of AD which included an overview of the reactions involved in the degradation of waste solids under conventional anaerobic conditions. CAD begins with the input of substrate (mixture

of primary sludge and wasted biomass), however the degradable substrate available for AD is not typically the same as the total organic input; that is to say a portion of the sludge fed in an AD cannot be broken down into simple macromolecules and remain as either inert particulate (plastics, fibres, etc.) or inert soluble materials (inorganic materials, metal complex's, etc.). The degradable substrate which includes complex particulates as well as inactive biomass is first degraded into complex macromolecules (lipids, carbohydrates, and proteins) by an array of processes such as lysis, physical breakdown by shear forces, and non-enzymatic decay [Batstone et al., 2002]. Carbohydrates and proteins then undergo hydrolysis reactions, degrading them to monosaccharides and amino acids respectively, while lipids are degraded to long-chain fatty acids (LCFA). The monosaccharides and amino acids undergo acidogenesis, a fermentative reaction, by two separate groups of acidogens and are degraded to a mixture of hydrogen, carbon dioxide, and organic acids and alcohols the latter eventually undergo acetogenesis to acetate and hydrogen [Batstone et al., 2002]. LCFAs undergo fermentative reactions to short chain fatty acid intermediates, alcohols, and acetate; the intermediates undergo acetogenesis while the acetate is directly processed. An example of the acetogenesis reaction with propionate, a common organic acid resulting from the fermentation of organics in AD, can be found below in reaction R1:

$$\text{(Propionate) } CH_3CH_2COOH + 4H_2O \rightarrow CH_3COOH + CO_2 + 3H_2 \qquad \text{(R1)}$$

The acetate undergoes acetoclastic methanogenesis to carbon dioxide and methane by acetoclastic methanogens which have a long growth period of ~20 days; these bacteria also tend to be highly sensitive to their environment as they are strict anaerobes and very

sensitive to temperature and pH [Batstone et al., 2002]. The reaction of acetic acid to methane and carbon dioxide by methanogens can be found in reaction R2.

$$CH_3COOH \rightarrow CH_4 + CO_2 \quad \text{(R2)}$$

Hydrogen undergoes hydrogenotrophic methanogenesis to carbon dioxide and methane by methanogens that have a relatively short growth period (~5 days) and are more robust to environmental conditions than acetoclastic methanogens. The methanogenesis of hydrogen reaction can be found seen in reaction R3.

$$4H_2 + CO_2 \rightarrow CH_4 + 2H_2O \quad \text{(R3)}$$

However, hydrogenotrophic methanogenesis accounts for a much smaller portion ($\leq$ 30%) of the total biogas production justifying why CAD has a typical solids retention time (SRT) of $\geq$ 20 days, to allow for the slow growth of acetoclastic methanogens. The biogas produced in CAD is a mixture of approximately 55 to 65% methane and 35 to 45% carbon dioxide, but contains trace amounts of compounds such as hydrogen sulfide and ammonia which need to be removed or 'scrubbed' prior to use in energy generation [Wellinger et al., 2013]. The exact ratio of methane and carbon dioxide components is dependent on several process factors including the methanogenic bacteria population, the food to microorganism ratio, and the digester conditions (e.g. operating temperature) [Kroeker et al., 1979].

The produced biogas can be used for electricity generation via CHP systems such as internal combustion engine-driven generators or so called emerging technologies such as microturbines and fuel cells [Ely et al., 2014]. The produced electricity can be used directly onsite to power the various pieces of equipment (e.g. pumps, blowers) within the

wastewater treatment facility (WWTF) or alternatively it can be sold back into the local electricity grid and thus generate a source of revenue. There is considerable interest in integrating technologies and process improvements that would enable WWTFs to achieve net energy neutrality (i.e. total energy needs are balanced by the amount of energy produced) or even net energy production. However, CAD may not be able to provide enough energy production by itself and might require additional organics to increase the biogas production.

1.1.3 Co-digestion and alternative feed sources

Since CAD may not always produce enough energy to achieve net energy neutrality, other methods to improve the biogas production may be incorporated. One method that has been widely considered is the co-digestion of municipal wastewater with high energy, easily biodegradable materials such as household food waste, industrial/agricultural wastes, and fats, oils and greases (FOGs) [Schwarzenbeck et al., 2008]. These highly biodegradable materials are attractive for increased energy production as they are typically seen as waste products otherwise headed for landfill, but are low in content of inert material and micropollutants (important not to introduce into a WWTF which can struggle to remove them) and characterized by their high-energy yield [Cecchi et al., 2011]. A 2014 report summarized the performance of six different WWTFs that incorporated co-digestion processes [Ely et al., 2014]. The breakdown of the facilities and their average percent of electricity demand generated on-site can be seen below in Figure 3 where two facilities generated less than 30% of their electricity demand and were also the two smallest producers of biogas in total. Three facilities generated between

60% and 90% of their electricity demand (close to net energy neutral) and employed codigestion with several different materials (at each site) including FOGs, industrial food wastes, and biodiesel waste. One facility (EBMUD) generated nearly 130% of their electricity demand with a variety of codigestion materials and was the only plant to operate under thermophilic conditions; they also processed the largest amount of FOGs per day which in combination with the other alternative feed sources resulted in the highest total biogas production of any of the plants surveyed. It is interesting to note that all plants (with the exception of one which began with codigestion ie. no baseline) saw an increase in electricity production when AD was coupled with co-digestion with some plants seeing as high as a 250 and 300% increase. However, none of the plants operating under mesophilic temperatures (conventional operating conditions) were able to achieve energy neutrality. Thus, the use of co-digestion processes on their own does not guarantee that energy neutrality will be achieved. Since the available co-digestion material is so varied in composition its effects on the digester performance cannot always be predicted ahead of time, and can lead to operational challenges such as low pH levels and ammonia inhibition [Mata-Alvarez and Llabres, 2000]. Also, there may be capacity issues in some WWTFs where the digester volume is not large enough to accommodate the increased organic loading rate (OLR) associated with the use of co-digestion processes. This is a significant concern as the capital costs of new digesters are quite high [Seiger and Parry, 2004]. Some of these capacity concerns can be addressed by high performance AD techniques which can be implemented by retrofitting current digesters or altering the current operating conditions.

Facility	CMSA	EBMUD	Hill Canyon	Sheboygan	West Lafayette	Janesville
% increase w/ co-digestion	60%	Over 100%	250%	150-300%	N/A**	40%
Biogas production (cubic feet/ day, averaged)	252,000	2,400,000	450,000	560,000	92,160	120,000
Biogas Use	CHP ICE Boiler*	CHP ICE Boiler Turbine	CHP ICE Boiler*	CHP Microturbines Boilers	CHP Microturbines	CHP Microturbines CNG
% of electricity demand generated on-site (annual average)	60%	128%	80-85% (soon to be 100%)	90%	16-18%	27%

Fig. 3 – Percent of electricity demand generated by on-site AD undergoing co-digestion with various materials at each site. [Ely et al., 2014].

1.1.4 High performance anaerobic digestion

The term 'high performance' in an industrial setting typically refers to the energy or power of a process; CAD is an energy conversion process from fed organics to biogas which can be used to produce power. High performance AD (also known as high rate) is simply modified techniques that allow anaerobic digesters to produce more energy or decrease the energy consumption used for the overall solids handling in WWT [Sieger and Parry, 2004]. There are several reasons to upgrade to high performance AD including reducing the pathogen concentration to achieve high quality biosolids; improved biogas production for reduced energy costs; reducing the final volume of wasted biosolids which decreases the amount of dewatering required and the cost/energy of hauling to their final destination; treatment of wastewater with a high chemical oxygen demand; and reducing the reactor size/footprint or increasing the current loading capacity [Sieger and Parry, 2004; Chan et al., 2009]. There are several different high performance processes which can be implemented each with their own advantages and shortcomings. Some of these

processes will be discussed in this section including operating the digester at higher temperatures (often referred to as thermophilic digestion); physical or chemical breakdown of the feed sludge making it easier for the digester to process; and increasing the retention time of solids in the digester.

1.1.4.1 Thermophilic digestion

Thermophilic digestion refers to AD operated at an increased temperature above the mesophilic range, typically between 50-57 ^{o}C. Thermophilic digestion has several benefits which can lead to a shorter SRT in the digester without increasing the size and increased volatile destruction along with improved biogas production [Kelly, 2005]. In thermophilic AD the breakdown of volatile solids occurs quickly during acidogenesis; however the methanogenesis is still a fairly slow process which often leads to the use of two-stage thermophilic digestion [Roberts et al., 1999]. Thermophilic digesters are able to achieve increased volatile destruction rate due to improved reaction rates of the anaerobic bacteria at higher temperatures [Nges and Liu, 2010]. The two-stage thermophilic digestion is a design where the incoming sludge is first digested at thermophilic temperatures accelerating the acidogenesis of the substrate to produce volatile fatty acids (VFAs), hydrogen, and acetic acid with a relatively short SRT. The partially digested sludge is then pumped into a second digester operating at mesophilic temperatures to allow for methanogenesis to occur with a longer SRT.

Despite the secondary mesophilic digestion, this initial thermophilic digestion (or stand-alone) of the feed sludge still allows for effective pathogen reduction as pasteurization is described as 6 hours or greater at 55 ^{o}C [Roberts et al., 1999]. The

pathogen reduction in thermophilic digestion is especially important if the end goal of the dewatered biosolids is for land application or other agricultural use [Sahlström, 2003; Roberts et al., 1999]. There are pitfalls to thermophilic digestion one of which being the cost to operate the digester at such a high temperature. If the biogas produced from the AD is used on site then some or all of the heating can be off-set by efficient heat capture from the combustion process. Additionally thermophilic digesters are often subject to more unstable behaviour with increased VFA content and often require pH control during the acidogenesis phase which in turn increases alkalinity and salinity [Fernandez et al., 2015].

1.1.4.2 Feed sludge pre-treatment

As a substitute to changing the operating conditions or upgrading the current digesters, the original digester's performance can be enhanced by reducing the number of reaction steps necessary to be performed on the incoming feed sludge for biogas formation. This can be achieved using a pre-treatment method on the incoming feed sludge inducing hydrolysis of large macromolecules by thermal, mechanical, or chemical processes which make the incoming feed easier for the anaerobic bacteria to digest. Thermal pre-treatment can aid in the lysis of wasted biomass in the feed sludge and also increase the hydrolysis rate of material entering the digester resulting in an increase in volatile solids destruction as high as 10% [Ge et al., 2010]. Thermal pre-treatment is a good alternative to thermophilic digestion as only the feed stream needs to temporarily be heated and this heated feed can help reduce the energy load on heating the digester. The feed sludge can also be mechanically broken down physical techniques such as ultrasonic

conditioning [El-Hadj et al., 2007] or high pressure in combination with chemical addition [Wahidunnabi and Eskicioglu, 2014]. Chemical treatments can vary but most of them involve breaking down wasted biomass by disrupting cell membranes and attacking macromolecules causing hydrolysis with processes such as ozonation [Komatsu et al., 2011] or the addition of caustic soda. The limitations for sludge pre-treatment include limited practical application outside of thermal pre-treatment; the high cost of chemical processes and the energy intensity of some mechanical and thermal treatments; and a minimal increase in available feed rate (or SRT) despite the increase in overall solids destruction observed.

1.1.4.3 Recuperative thickening

In order to address the capacity issues which can occur during codigestion, a portion of the partially digested solids in the digester outlet stream are flocculated and then thickened before being blended with the incoming feed and re-introduced into the digester. This process is known as recuperative thickening (RT), however it is also described in the literature as 'Torpey's process', to acknowledge the original work in this area [Torpey and Melbinger, 1967], and also as 'extended solids retention' process [Sieger and Parry, 2004]. Compared to other 'high performance' AD processes, RT is more effective than the feed pre-treatment method at increasing the treatment capacity but does not require the additional energy input to operate at thermophilic conditions [Kelly, 2005]. Operation at mesophilic conditions can also lead to more stable and robust operating conditions when compared to thermophilic treatment [Labatut et al., 2014]. As mentioned in RT processes, the thickened partially digested solids in the digester outlet

stream are blended with the incoming feed and re-introduced into the digester. This operation results in a higher solids retention time (SRT) than the hydraulic residence time (HRT); just like a conventional activated sludge system by decoupling these two process parameters the digester is now bound by the solids loading rate and not the volumetric flow into the digester which leads to an increase in capacity. In addition to the increase in capacity, RT also leads to a decreased volume of waste solids produced; this outcome has the added effect of reducing the amount of polymer flocculant required in an end-stage dewatering process used to create final solids cake. Previous studies have shown that the short-term exposure of the anaerobic bacteria to oxygen in the digested solids dewatering step has no significant effect on the activity of the acetoclastic methanogens [Conklin et al., 2007; Batstone et al., 2015].

1.1.4.3.1 Plant scale recuperative thickening

Various dewatering (aka thickening) technologies have been used to recycle the partially digested solids in the outlet stream. The original RT processes used gravity thickening, centrifugation, anoxic gas flotation, or belt filter press thickening [Kelly, 2005]. It has been shown that the overall process can be improved by incorporating a polymer flocculation pre-step.

In 2011, Ostapczuk et al. [Ostapczuk et al., 2011] showed that using RT in combination with gravity belt thickeners with a polymer flocculation pre-step increased the WWTF capacity to allow for co-digestion of dairy whey with municipal sludge; the amount of biogas produced generated 95% of the facility's electrical demand via a CHP process. They concluded that RT led to an increased SRT, improved biogas production

and quality, but additional research was necessary to establish operational guidelines for the start-up of the RT process [Ostapczuk et al., 2011].

Between 2009 and 2011, two WWTFs in Sydney, Australia were upgraded to incorporate RT by adding rotary drum thickeners with pre-injection of polymer flocculants; a significant number of process improvements were realized including improved raw sludge capacity, increased biogas production, and improved end-stage dewatering performance [Tang, 2009; Ireland, 2011]. The increased biogas production as a result of the RT digestion now provides enough biogas to run their current co-generation plant at full load; at full load the co-generation plant can produce up to 1.4 MW of energy, sufficient enough to power the entire plant plus export a surplus to the grid [Tang, 2009]. This increased energy production comes from digesting municipal solids only, and does not even take into account the potential energy production achievable from implementing co-digestion of high-strength organic material.

Since 2014, Anaergia Inc. has operated a pilot plant with RT in Victor Valley, CA. The RT digester has the ability to operate at 3 times the OLR of the CADs on-site and saw similar results to other full plant trials with increased biogas production and improved dewaterability. However, the flocculation pre-step used to improve the RT separation efficiency was an area of particular concern. Optimizing the process involved switching between multiple polymer flocculants and evaluating the change in dewaterability achieved. Unfortunately this is both labour and energy intensive to perform on the full-scale and involves months of operation with each polymer flocculant to achieve separate steady state operation with each. Polymer flocculant builds up in the

digester during recuperative thickening, after switching to a new polymer flocculant the built up residual polymer flocculant must first be flushed out through several SRTs, at least 3 to achieve greater than 95% removal [Marlin, 1995], before the digester can approach a new steady concentration of the new polymer flocculant. I had the opportunity to travel to Victor Valley with Anaergia Inc. to view the RT plant, perform some baseline tests, and become familiar with the operation which would later be invaluable to the design, implementation, and completion of my work. Below in Figure 4 and 5 pictures are shown from the Victor Valley RT pilot plant. In panel A of Figure 4 the polymer flocculant injection ring and mixing valve are depicted; the made-down polymer is injected at a constant flow rate controlled by a supervisory control and data acquisition (SCADA) system which would increase or decrease polymer flowrates based off of corresponding changes in incoming digestate flowrate. The flowrate of polymer is determined by the polymer make-down concentration, the total solids content of the digester and the required polymer dose. Panel B displays the sampling unit which allows real-time sampling of the filtrate quality and incoming digestate total solids for the mechanical dewatering process. An example of well flocculated solids cake after being mechanically dewatered to approximately 10 wt% can be found in panel C. The mechanical dewatering took place in Anaergia's Sludge Screw Dewaterer (SSD) a screw press type thickener depicted in Figure 5 panel A with the flocculation step occurring upstream by approximately 45 seconds. The corresponding polymer make-down unit is shown in Figure 5 panel B has a small 5 gallon chamber with a high shear overhead mixer to ensure proper dilution of the stock polymer emulsion before use.

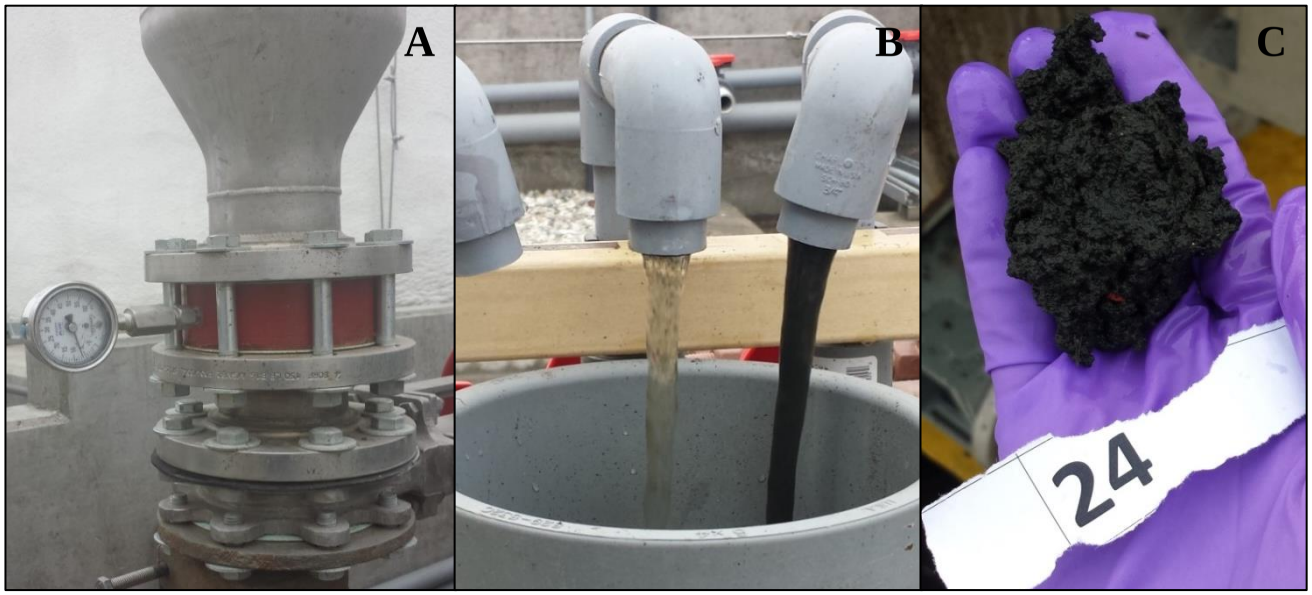


Fig. 4 – Trial pictures from Anaergia's Omnivore™ (RT) process in Victor Valley, CA, USA. In panel A the polymer injection ring and mixing valve are shown. Panel B depicts the filtrate quality from the mechanical dewatering unit (left) and the RT digestate as it exits the digester. Panel C depicts well a flocculated solids cake as it exits the mechanical dewatering unit.

Fig. 5 – Biosolids dewatering equipment used in the Anaergia's Omnivore™ (RT) process in Victor Valley, CA, USA. In panel A, Anaergia's Sludge Screw Thickener 225 (SST) mechanical dewatering unit is shown. Panel B depicts the polymer make-down unit equipped with Anaergia's SST 225

1.1.4.3.2 Lab-scale recuperative thickening

To date, most studies involving RT have occurred on the full or pilot scale. This is surprising given the minimal understanding of the economics and nuances of operation of RT at the plant scale. Very few studies have been published on RT at the lab scale leaving ample opportunity for future research in the area. One of the first lab studies involving RT was performed in 2011 [Vanyushina *et al*., 2012] where solids from a thermophilic digester were removed and thickened via centrifugation with no flocculation and then mixed with feed sludge, first at a recycle to feed ratio equal to 75% of the incoming feed volume then 100% [Vanyushina *et al*., 2012]. They observed an increase of 10-30% in volatile solids (VS) destruction vs the control thermophilic digester and a marginal gain in biogas yield from 0.31 to 0.35 m^3/kg VS [Vanyushina *et al*., 2012]. They also measured the dewaterability of the digestate via specific resistance to filtration (SRF) on the control and the RT digesters; however they only performed this test 5 times throughout each test cycle and did not flocculate the digestate before testing. There was a distinguishable increase in dewaterability observed during the first study and a minimal improvement in the second. This same group produced a follow-up paper in 2012 which explored the effect of different methods of separation during the RT step in lab-scale thermophilic digesters. They explored centrifugation, gravity settling, and polymer flocculant assisted dissolved air flotation; determining that centrifugation was the most effective in terms of increased volatile solids destruction (27%) and approximately equal biogas yield [Vanyushina *et al*., 2012]. They again performed SRF test for dewaterability of the unflocculated digestate however they only performed this test on the gravity

settling and centrifugation digesters, and not on the polymer flocculant assisted flotation technique [Vanyushina *et al*., 2012].

A more recent study in Australia in early 2015 looked at RT the lab scale using stainless steel reactors seeded with 20 L of digested sludge at mesophilic temperatures [Yang et al., 2015]. Solids were removed from the RT and flocculated with a Zetag brand polymer at a dose of 4 g polymer/kg dry sludge then gravity settled to return a thickened cake of approximately 5 wt% [Yang et al., 2015]. They investigated the effect of SRT on digester performance in terms of biogas production and volatile solids destruction however despite the use of a polymer flocculant made no conclusions on the flocculation efficiency achieved or the change in dewaterability observed.

1.2 Flocculation

1.2.1 Mechanisms

In several separation techniques, increased particle size is beneficial to the efficiency of the separation process. Flocculation is an effective way to increase the particle size as it causes small particles in suspension to aggregate by forming larger particles or flocs; these flocs can be more easily separated from the suspending liquid [Gregory, 2013]. A flocculant is the term for the material added which causes small particles to aggregate. Flocculants can be charged particles, cationic or anionic, or not charged at all, non-ionic. In the water/wastewater treatment industry a ‘flocculant’ is the term for a charged polymer or large organic compound used to aggregate small particles to form flocs. In aqueous suspensions such as water/wastewater the small particles are stable due to electrostatic repulsion between particles preventing them from aggregating

and settling out of suspension [Gregory, 2013]. The charges on the flocculant interact with the surface charges on the small particles in a process called charge neutralization. This causes the small particles to become unstable as the overall charge of the particle is neutralized and the electrostatic repulsion diminished [Shammas, 2005]; if too much polymer flocculant is added the additional charge after neutralization can result in a restabilization of the particles in suspension, this is known as charge reversal and can hinder separation performance. In addition to inducing charge neutralization, the addition of a polymer flocculant also allows for a second mechanism of flocculation known as particle bridging. Particle bridging is a mechanism by which several of the small aggregated flocs can be linked by the long polymer chain forming even larger flocs which allow for faster settling and can increase the mechanical strength of the floc [Gregory, 2013].

1.2.2 Polymer flocculants in biosolids dewatering

Biosolids dewatering is an important process in WWT and is especially important when determining the economic feasibility of operating an AD in RT. Sørensen (1996) estimated that as much as 50% of the annual plant operating costs is directly related to the dewatering process. These costs include those associated with the processes that are necessary to dry, transport, and dispose of the residual biosolids. The most common mechanical devices used to separate the water from the biosolids are centrifuges, belt filter presses, and drying beds.

To enhance the dewatering process performance, chemical coagulants and/or polymer flocculants are added to promote the aggregation of the colloidal material into

large dense particles that settle in solution. The amount of water in the interstitial spaces is reduced via a combination of both charge neutralization and particle bridging effects [Vesilind, 1994]. Various water soluble cationic polymers have been used to promote the flocculation of these anionic biosolids materials. Synthetic polymers including poly(ethyleneimine), poly(diallyldimethyl ammonium chloride), poly(acrylamide), and co-polymers such as acrylamide and dimethylaminoethyl acrylate (DMAEA) are most widely used [Bolto and Gregory, 2007]. There are multiple commercial suppliers that offer polymers with different structures (e.g. linear, branched) and a range of molecular weights and charge densities.

Dewatering is a very complex process since it involves many different 62 parameters such as polymer dosage rates [Mikkelsen and Keiding, 2001], mixing intensity [Novak and Bandak, 1989], and the digestate composition itself, which can vary widely over time at the same treatment plant [Eriksson and Alm, 1993]. The challenge is to precisely optimize the amount of polymer that is required – too little (i.e. under dosing) will lead to unsatisfactory dewatering performance, but too much (i.e. overdosing) is cost prohibitive and can also lead to unsatisfactory performance due to charge reversal effects [Christensen et al., 1993; Lee and Liu, 2000]. Unfortunately, the precise control of polymer dosing is a technical challenge because there has been little innovation in the field of dewatering. Traditional polymer screening techniques involve the flocculation of a particular sludge with a few polymer flocculants (typically from one manufacturer) done by a polymer vendor and the dewaterability assessed via a number of different tests. Typical practices for measuring dewaterability include jar test settling rates, specific

resistance to filtration (SRF) tests, and capillary suction time (CST) measurements. Previous work has shown good correlations between these various methods [Karr and Keinath, 1978; Lee and Liu, 2000; Sawalha and Scholz, 2010; Peng et al., 2011]. However, the 'optimizing' of the polymer dose and selection is usually left to the polymer vendor and/or manufacturer and the WWTF does not typically do any further testing resulting in a constriction of the knowledge involved with solids dewatering.

1.3 Problem Statement

The incorporation of RT at a WWTF requires a capital investment to upgrade the mixers (to account for the increased solids content) and to integrate the recycled stream into a dewatering process. Given the importance of the dewatering process to the overall techno-economic performance of RT, it was surprising to find that the studies published to date have almost exclusively occurred in pilot plant or full scale trials at WWTFs. That approach has severely restricted the amount of RT process development work that can be undertaken and thus led to so-called knowledge gaps in the field. For example, a proper techno-economic analysis of the performance and operating costs is required when selecting a polymer flocculant [Kelly, 2005]. Also, the potential toxicity associated with excess polymer in the effluent [Bolto and Gregory, 2007; Biesinger et al., 1976] and increased polymer concentrations in the biosolids are a significant concern [Campos et al., 2002]. This is a valid concern because the use of polymer flocculants in biosolids dewatering is still one of the most poorly understood and practiced areas in WWT. In previous work, we reported significant variations in dewatering performance for multiple types of polymer flocculants [Cobbledick et al., 2014]. In addition, the minimal studies

that have explored RT on the lab-scale have not or poorly considered the change in dewaterability experienced by the digestate when undergoing RT and have not investigated the effects of various recycled polymers on dewaterability as well as digester performance.

1.4 Objectives

In this work, a complete system was developed for the operation of two bench-scale digesters in parallel where one is operated in a semi-batch RT mode (i.e. SRT greater than HRT) and the other was operated as a conventional single pass 'control' (i.e. SRT same as HRT). Both digesters were operated in an automated mixing mode with continuous temperature and biogas production monitoring. This was accomplished with each digester having a volume of 1.5 L and thus the entire footprint was less than 0.5 m^2. I am not aware of any previous 'bench-scale' studies of dewatering and biogas production performance during RT with polymer enhanced separation. Given the number of outstanding issues cited above in regards to polymer-induced flocculation it was chosen to focus our initial work in that area. The main focus of this study was to investigate the influence that various polymer flocculants used during RT would have on the dewaterability of the digester as well as the overall digester performance. However, the system described in this work is ideally suited for studying other process variables for RT operations such as the co-digestion of high strength organics.

- Chapter 2 -
Materials and Methods

2.1 Digester Feed

2.1.1 Sample collection and storage

The feed sludge was obtained periodically as needed over the 9 month testing span from the Dundas Valley WWTF in Dundas, Ontario. The samples were taken from the combined primary sludge (PS) and thickened waste activated sludge (TWAS) line from plant 2. Samples were stored in 4 L Nalgene containers and then transported back to McMaster where they were subsequently stored at 4^{o}C when not in use. The combined PS and TWAS digester feed was a mixture of approximately 60% PS and 40% TWAS. This ratio likely varied during the duration of the test's due to variable operation of both the primary settler and aerobic bioreactor.

There was a slight variation in the total solids (TS) content of the as-received feed (between 2 and 3 wt% (w/v)) and thus simple gravity settling or low-speed centrifugation processes were employed to increase the TS content to the desired range of 3.5 to 4 wt% (w/v). Sludge was centrifuged in 200 mL bottles at 4000 RPM for 5 minutes. A portion of the liquid was then decanted off and the remaining (lightly packed) sludge pellet was gently re-suspended and added back to the original 4 L container. As mentioned previously all digester feed samples were stored at 4^{o}C and were not kept for more than 4 weeks to avoid sample breakdown and a change in the feed characteristics.

2.1.2 Feed total/volatile solids test

The TS and VS content was measured with each new sample of digester feed sludge obtained from Dundas Valley WWTF. An aluminum weigh tin (VWR was used for the TS test; the initial mass of the tin was measured and then a known volume of sample added to the tin, 10 mL for PS. The TS test was performed in a 105 ^{o}C oven for a minimum of 24 hours, cooled, and then weighed again. To measure the VS the same, but now dried, sample was placed in the 550 ^{o}C furnace for two hours then cooled and weighed for a final time. The TS and VS were calculated using Equation 1 and 2 respectively:

$$TS\ (w/v\ \%) = \frac{(24\ hour\ dry\ mass\ (g)) - (Pan\ mass\ (g))}{Volume\ of\ Sample\ (mL)/1000} / 10 \quad (1)$$

$$VS\ (w/v\ \%) = \frac{(24\ hour\ dry\ mass\ (g)) - (After\ furnace\ mass\ (g))}{Volume\ of\ Sample\ (mL)/1000} / 10 \quad (2)$$

The TS content was used to determine if the digester feed sludge required any additional thickening via the gravity settling or centrifugation mentioned above and the VS content was used to calculate the OLR and determine the food to microorganism ratio (F/M) which is a key parameter for the design and operation of an anaerobic digester (Wang et al., 2008). For a detailed breakdown of the OLR for each digester during the two 40-day cycle tests see Table 1 and 1 at the end of Section 2.6.

2.1.3 Feed total chemical oxygen demand test

The chemical oxygen demand (COD) is an indirect way to measure the organic content of a sample. It is determined by oxidizing organic compounds to carbon dioxide using a strong oxidizing agent under acidic conditions. This test can be carried out by manually mixing a strong acid with other corrosive and hazardous chemicals or by using a commercially available kit such as the one provided by Hach. For the total COD (TCOD), Hach kits were used for both safety and practicality purposes. They also reduced the number of corrosive and hazardous chemicals that would be required to be stored in the lab.

When each new sample of digester feed sludge was collected the TCOD was measured. The TCOD refers to all the organic compounds in a given sample and in the case of the digester feed sludge this would refer to compounds such as fatty acids, polysaccharides, and other complex organics. The TCOD of the feed was measured to confirm that the volatile compounds determined from the VS test were organics.

The Hach COD test kits used were high range vials featuring a measuring range of 20-1500 mg/L. Despite the high range test kits samples still required dilution before testing. Before the TCOD test the digester feed sludge was diluted 1:100 in a volumetric flask using deionized (DI) water. 2 mL from the diluted sample was pipetted into a Hach COD high range vial and shaken vigorously. The vial including one ‘blank’ containing 2 mL of DI water were placed in a Hach DRB200 digital reactor block and digested at 150 ^{o}C for two hours. After digestion, the COD vials were cooled to room temperature and the outside of the vials cleaned with a lint-free wipe (VWR) to remove any debris or smudges

from the vials. The vials were then analyzed using a Hach DR3900 spectrophotometer with each vial being read twice to ensure a correct reading.

2.2 Polymer Flocculants

2.2.1 Clarifloc C-6267

The polymer flocculants used in this work were all cationic in charge with various charge densities, chemical composition, and molecular weights. The Clarifloc C-6267, manufactured by Polydyne (same polymer as FLOPAM 640 CT, manufactured by SNF), is a linear polyacrylamide. According to the manufacturer, the Clarifloc C-6267 is a high molecular weight (3 to 20 million Da), very high charge density polymer with a specific gravity of 1.04 and an active solids content of 43%.

The as-received stock emulsion of polymer was stored out of direct sunlight and at room temperature. The stock emulsion was diluted with 50 mM sodium phosphate monobasic buffer (pH of 4.8) in a two-series dilution to obtain a final polymer concentration of 0.25 wt% (w/w). First, half of the required buffer was added to a glass beaker with a magnetic stir bar (VWR, size dependant on volume of polymer solution made) and mixed at 1000 RPM. The stock polymer emulsion was then removed with a pipette with care to ensure the entire volume of emulsion was in the pipette tip; this can prove to be difficult as the stock emulsion has a high viscosity and it takes care and practice to master. The stock emulsion was then pipetted into the mixing buffer solution and allowed to mix for 10 minutes. After mixing, the second half of the required buffer was added to reach the final working concentration of 0.25 wt% and mixed for an

additional 10 minutes. The dilutions were allowed to age at room temperature for at least 90 minutes, but no longer than 12 hours, before use.

2.2.2 FLOPAM 440 LH

The FLOPAM 440 LH is produced by SNF and is linear polyacrylamide. According to the manufacturer the FLOPAM 440 LH is a low molecular weight, high charge density polymer with a specific gravity of 1.04 and an active solids content of 44%. It was prepared in the same manner as mentioned previously to a final working concentration of 0.25 wt%.

2.2.3 SuperFloc 4516

The SuperFloc 4516 is produced by Kemira and is a copolymer of polyacrylamide and quaternized-N,N dimethylamino ethylacrylate (DMAEA-Q). According to the manufacturer the SuperFloc 4516 is a very high molecular weight, high charge density polymer with a specific gravity of 1 and an active solids content of 33%. It was prepared in the same manner as mentioned previously to a final working concentration of 0.25 wt%.

2.3 Lab-scale Setup

2.3.1 Digester design

Two custom cylindrical 2 L digesters (125 mm height, 150 mm diameter) were designed and fabricated ‘in house’. Pictures of the digester can be found in Figures 6 and 7 below with numbered labels corresponding to each of the connection ports. The flanged lid had a rubber o-ring seal and four connection ports for the following: an inlet port to establish a nitrogen (99.9% pure, Air Liquide) blanket in the headspace above the digester

contents during feeding and wasting [1]; a biogas outlet port [2]; an access port to load the digester with fresh feed and remove digestate samples [3]; a thermocouple connected to a data logger (Omega) to monitor the temperature inside the digester [4]. A magnetic stir bar (76 mm × 13 mm) was placed inside each digester and the contents were continuously mixed at approximately 250 rpm via a QuadMag Genie (Scientific Industries Inc.) magnetic stirrer.

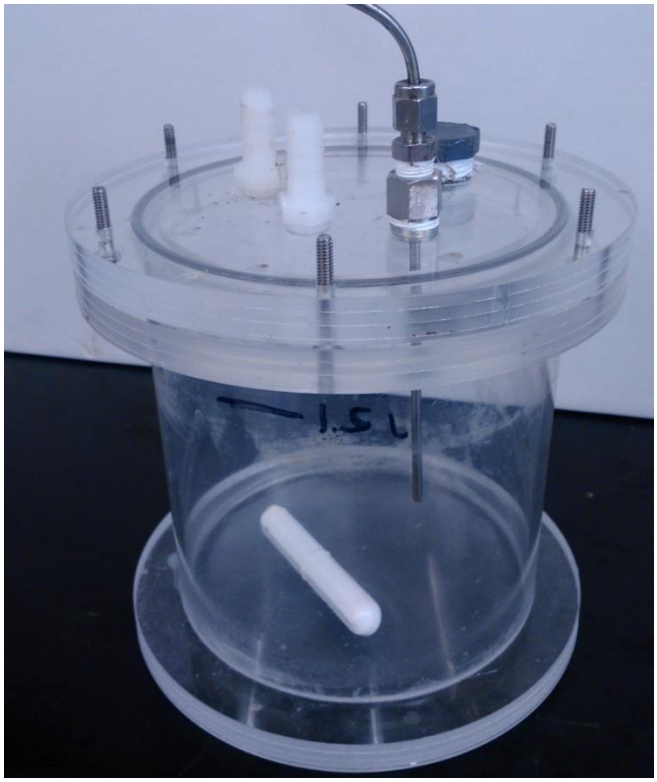


Fig. 6 – Side-view of Lab-scale digester with magnetic stir bar inside.

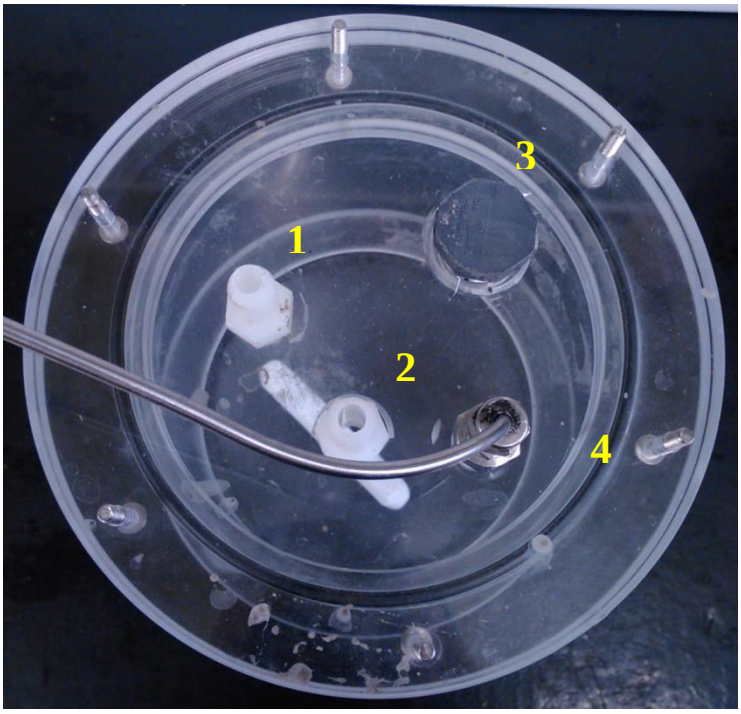


Fig. 7 – Top-view of Lab-scale digester with magnetic stir bar inside. Connection ports are labelled as follows: 1 – Nitrogen blanket inlet, 2 – Biogas outlet, 3 – Feed inlet/digestate extraction, 4 – Thermocouple and fitting.

2.3.2 Temperature control

Both digesters were placed inside a water-filled rectangular tank that sat directly on the magnetic stirrer. The temperature of the water in the tank, and thus the contents of the digester, was controlled to 37-37.5 ^{o}C via an immersion overhead heater from VWR. Overall the control was excellent with the actual temperature inside the digester varying by less than 0.5 ^{o}C over a 24 hour period; see Figure 8 below for a typical one week temperature profile from each of the two 40-day cycles. Water evaporation from the tank was minimized by blanketing the surface with 20 mm diameter hollow plastic spheres.

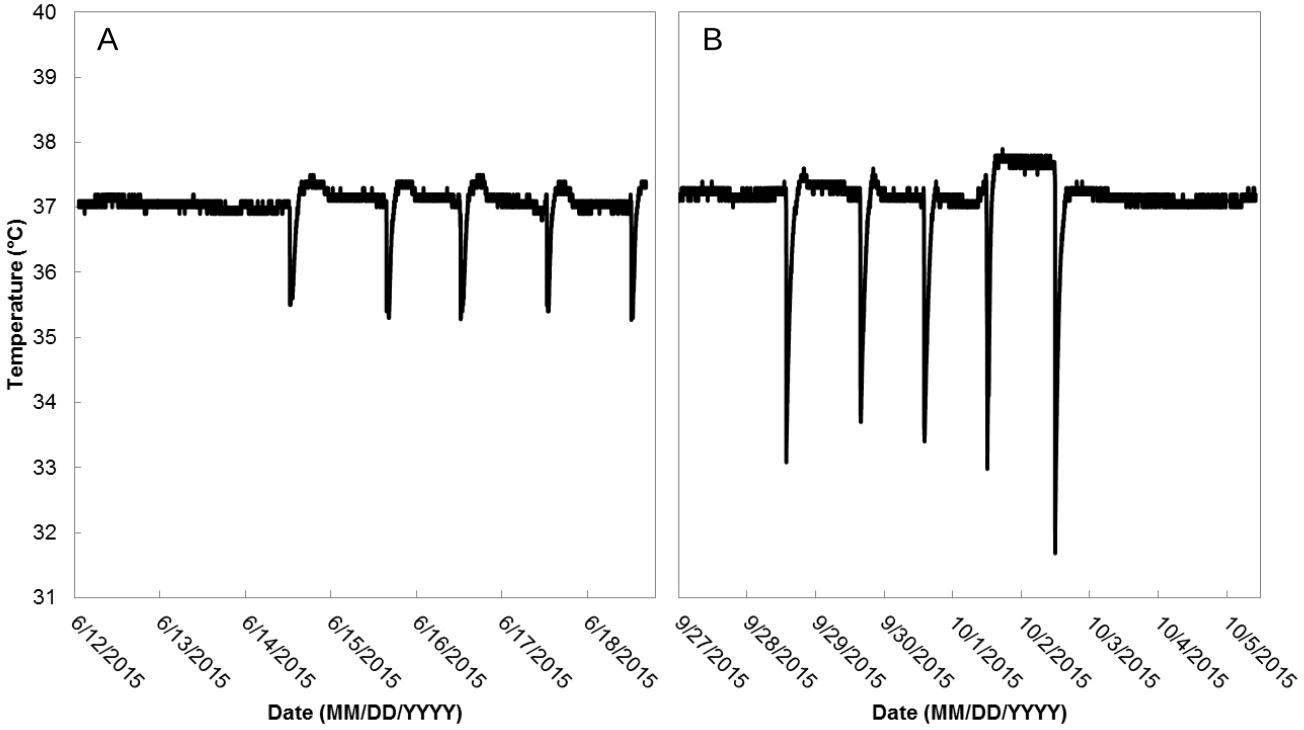


Fig. 8 – A typical one-week temperature profile as recorded by the thermocouple in each digester in the water tank with the immersion overhead heater. Panel A represents one week in digester 1 from the first 40-day cycle and panel B represents one week in digester 2 from the second 40-day cycle. The data were logged at 1 minute intervals. The five downward 'spikes' indicate when the digester was loaded once each weekday with fresh feed with digester 2 undergoing larger spike due to the increased feed rate. There are no such 'spikes' during the first two days of the temperature profile in panel A or the first and last two days in panel B because those days corresponded to the weekend.

2.3.3 Biogas measurement and sampling

The overall digester setup can be seen in Figure 9 below including the water bath and temperature control as well as all tubing which was all silicon based but of various diameters. Each reactor was isolated by the flowmeter and a shutoff valve located in the main nitrogen blanket line; an individual isolation valve in each nitrogen blanket line to the digester; and on each biogas outlet line which included a 3-way valve for gas sampling of the head space. These isolation valves were important for both gas sampling and ensuring that the digester gas from each unit did not cross over into the other through the nitrogen blanket line. Prior to the isolation valves a 5" long and 1 ½" diameter sealed cylinder with inlet and outlet tube attachments was placed in each gas line. The objective of these tubes was to prevent digestate from potentially rising up the gas outlet line and clogging the 3-way valve which would cause an unsafe buildup of biogas and could lead to a rupture of the gas outlet tube and eliminate the anaerobic environment.

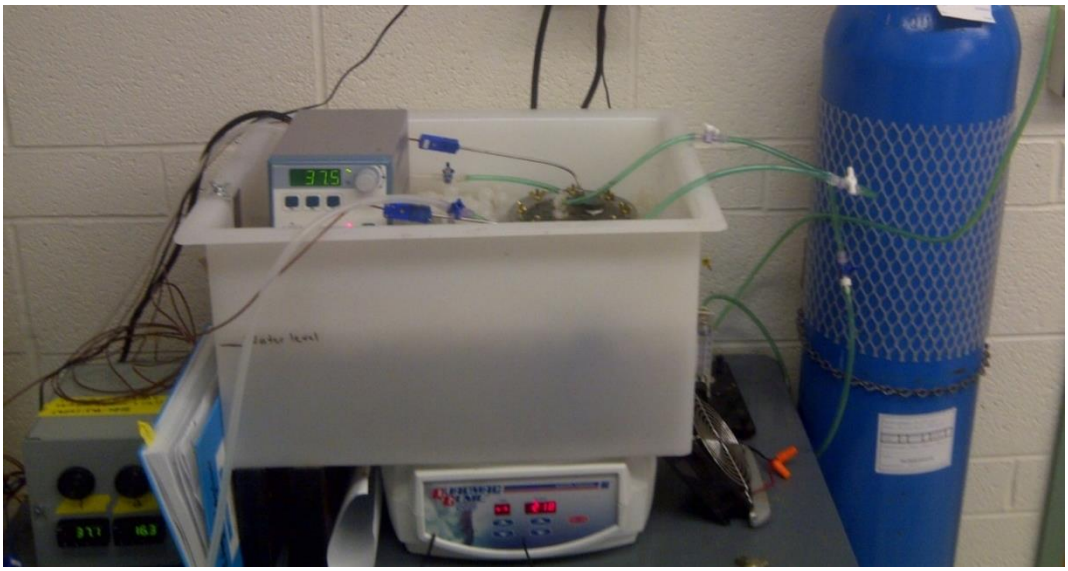


Fig. 9 – Picture of complete lab-scale set up including temperature monitoring and alarms, nitrogen blanket, water bath with heater, and magnetic mixer.

The actual biogas production was measured via a Ritter type MGC-10 Mili-Gascounter (Litre Meter Ltd.) which can be seen below in Figure 10. They were located after the 3-way valve in the biogas outlet line and located in a fume hood for safe venting of the flammable biogas.

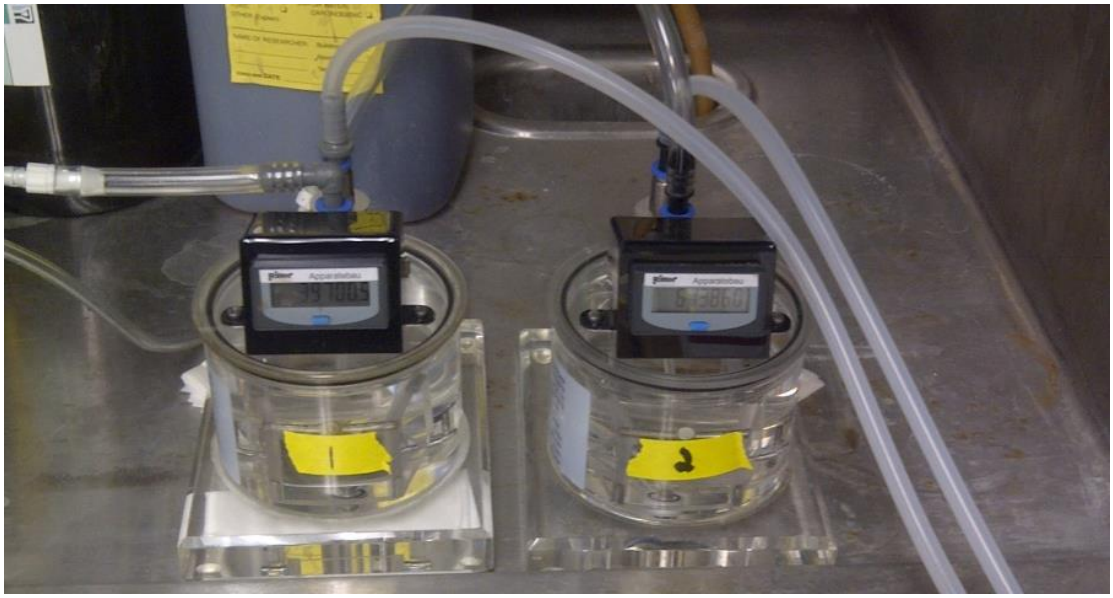


Fig. 10 – Picture of Mili-Gascounters for real-time measurement of the actual biogas production.

The biogas from each digester headspace was sampled one to two times per week during the 40-day cycle. Biogas sampling occurred before the nitrogen blanket had been applied for feeding on the day of sampling and at least 24 hours after the blanket had been applied on the previous day. A 60 mL syringe with a short (5 cm) piece of silicon tubing and a locking luer-lock valve was used to draw the biogas from the headspace via the three-way valve in the gas outlet line seen in Figure 11. The silicon tubing was slotted over the 'to-atmosphere' side of the valve and the valve turned as to open a direct line from the headspace to the atmosphere and close off the path to the gas counter. 30 mL of biogas was then drawn out of the headspace by the syringe; after the luer-lock valve on

the syringe was closed and the silicon tubing pulled off the three-way valve. Since there was now a vacuum caused by the 30 mL of gas removed and some air would inevitably be pulled into the headspace the nitrogen blanket was introduced in order to maintain the anaerobic conditions after biogas removal. The gas samples were then transferred from the syringes into CaliBond gas storage bags (Calibrated Instruments) which are depicted in Figure 11 below for storage until testing later in the week at Anaergia's lab facility. The bags contained one luer-lock valve for adding the sample and one rubber stopcock for sample removal and testing and were stored at ambient temperatures until the time of testing.

Fig. 11 – Picture of Calibrated Instruments Inc. CaliBond gas sample storage bag and three-way valve for gas sampling, nitrogen purge, and biogas volume measurement.

2.4 Digester Operation

2.4.1 Control Digester

The feed schedule for both digesters was semi-batch with 5 days of feeding (corresponding to a 5 day work week) followed by a 2 day starvation period (corresponding to the weekend). Initially, both digesters were operated at the exact same condition of removing 75 mL of the 1.5 L digester contents as waste then adding 75 mL of fresh feed every weekday; thus the SRT and HRT were identical at 20 days. This 'pre-test' step was done for 12 weeks (i.e. 60 weekdays or the equivalent of three SRTs) to ensure that there was no difference between the contents of each digester at the start of the subsequent eight week test cycle. The control digester was operated at the exact same conditions as the 'pre-test' step during the test cycle; refer to Figure 12 for a detailed schematic of the operation and testing procedures. The OLR for the control digester was determined on a mass of VS added per day per working volume of digester and was targeted at 1.75 gVS/(L*d); refer to Table 1 at the end of Section 2.6 for a detailed comparison of the OLR values.

In order to maintain the anaerobic nature of the digesters some important steps had to take place before the feeding could occur. Before opening the nitrogen blanket isolation valves to the digester, the nitrogen tank shutoff valve was opened followed by the pressure control valve which was adjusted until the pressure read 40 PSI. The three-way valve located after each digester was then opened to atmosphere in order to avoid blowing the nitrogen blanket into the gas counters and causing a false reading. After the three-way valves were opened the isolation valves for each tank were opened and the

inline flowmeter was opened and adjusted to 3 LPM. Once these steps were complete the feed port could be opened and both digesters could be worked on in an oxygen free environment.

Once the nitrogen blanket has been applied to the digester the feed port could be unscrewed. Using a 60 mL syringe with a 4 inch piece of silicon tubing on the end, the 'wasted' portion of the digest, 75 mL each day, could be removed. As seen in Figure 12 below this 75 mL wasted portion was used for a variety of experiments. The 75 mL of digester feed sludge was then measured with a plastic graduated cylinder and added to the digester feed port with a funnel to assist with pouring. Since the viscosity of the control digester was not high no additional mixing of the digester was required after feed addition. The feed port screw cap could then be replaced and the isolation valve closed to shutoff the nitrogen blanket to digester 1 only followed by opening the three-way valve in the gas line to the gas counter and shutting it off from the atmosphere.

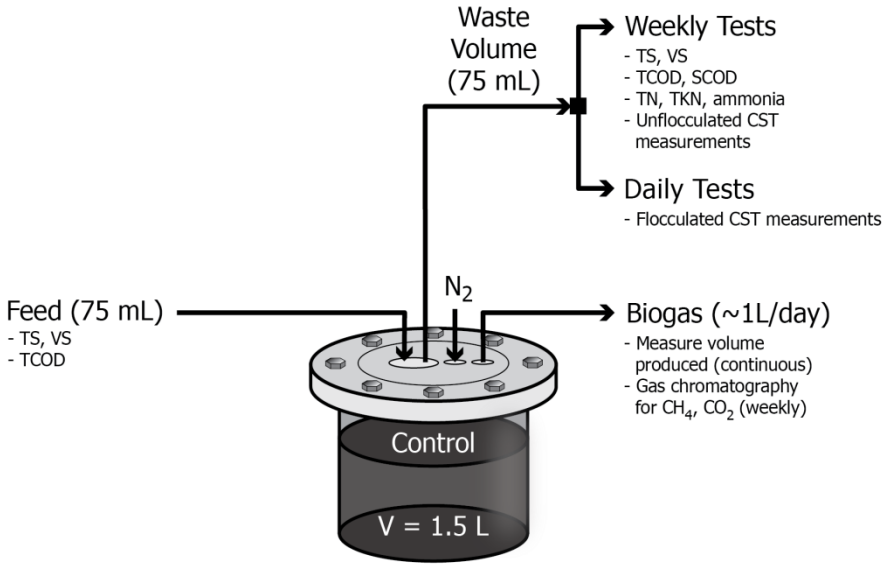


Fig. 12 – Schematic of the operation of the control digester including the various analytical tests that were performed and the source of the samples for those tests. For comparison, the schematic for the RT digester is shown in Figure 13.

2.4.2 Recuperative thickened digesters

The feed schedule for the RT digesters remained the same 5-day feed 2-day starvation as the control however the amount of feed added each day was doubled leading to an increased OLR. The target OLR for the RT digester was 3.5-4.5 gVS/(L*d) for the two 40-day test cycles; refer to Table 1 and 2 at the end of Section 2.6 for a detailed comparison of the OLR values. The HRT and SRT for the RT digester are 10 and between 22 and 30 days respectively. The SRT in the RT digester varies due to the minor amount of solids that are lost during the separation step and the daily calculated SRT can be found in Section 2.6.2 in Figure 15.

Before the feeding and recuperative thickening steps could occur the nitrogen blanket was applied in the same manner as the control mentioned previously. Once the nitrogen blanket was in place the feed port could be opened and the initial 50 mL of

digestate corresponding to the 'wasted' portion could be removed with the same 60 mL syringe and tube. This wasted portion was then used for evaluating the current dewaterability of the digestate and other digestate properties. Refer to Figure 13 for a detailed schematic of the operation and testing of the RT digesters.

After removing the waste portion the 225 mL of the RT portion could be removed and set aside. While the digester was still open the 150 mL of daily feed was added using a plastic graduated cylinder to measure the volume and a funnel to assist with pouring the feed into the digester. The feed port cap was reinserted back into place and the nitrogen gas turned off; the isolation valve was then closed and the three-way valve closed to the atmosphere.

In order to enhance the efficiency of the solids-liquids separation a flocculation pre-step was performed with one of the cationic polymer flocculants. The RT portion of digestate was poured into a 600 mL plastic beaker with a large magnetic stir bar (76 mm x 13 mm, VWR) and placed on a stir plate (VWR). While mixing at 700 RPM the appropriate volume of diluted polymer flocculant was added to the digestate quickly and mixed for 10 seconds. This initial high shear mixing mimics the typical conditions found in the flocculation pre-step before mechanical dewatering occurs during biosolids management. The mixer was then reduced to 300 RPM to mimic the laminar flow in a pipe allowing for floc formation, mixing for 1 minute at this condition. After the flocculation pre-step the digestate underwent gravity driven mechanical dewatering. To carry out the separation, a funnel was lined with a fine mesh (Tyler series 150 grade) and placed over a graduated cylinder. The flocculated digestate was then poured into the

funnel and gravity separated with the mesh retaining most of the flocculated solids; improved flocculation led to increased solids retention in the mesh. Liquid was removed until the remaining solids reached a volume of 125 mL and a sample of the liquid stored in a 50 mL Falcon tube for later testing. The nitrogen blanket was then reapplied to the digester being RT only and the feed port opened. The RT solids were then returned to the digester with the help of a funnel; when the viscosity of the RT digesters increased due to the increasing polymer concentration in the digester additional mixing was performed with the syringe/tube in order to help disperse the feed and RT solids.

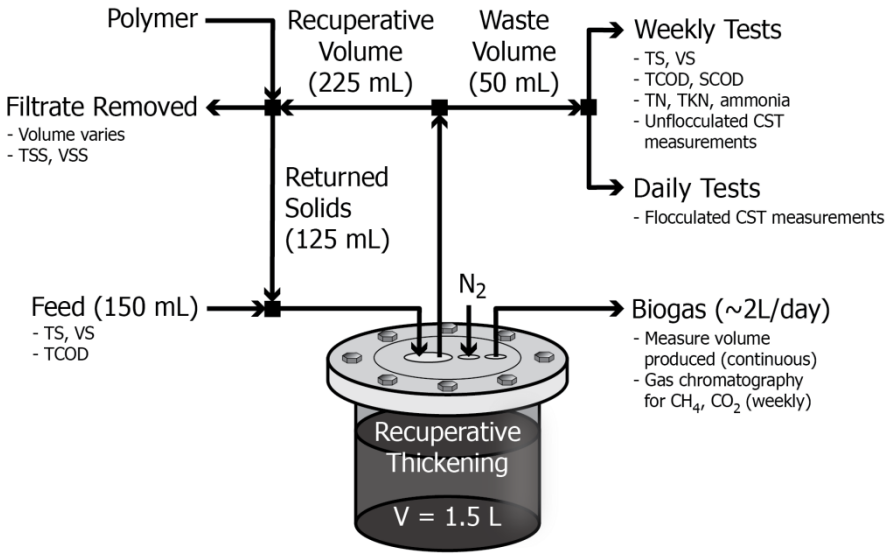


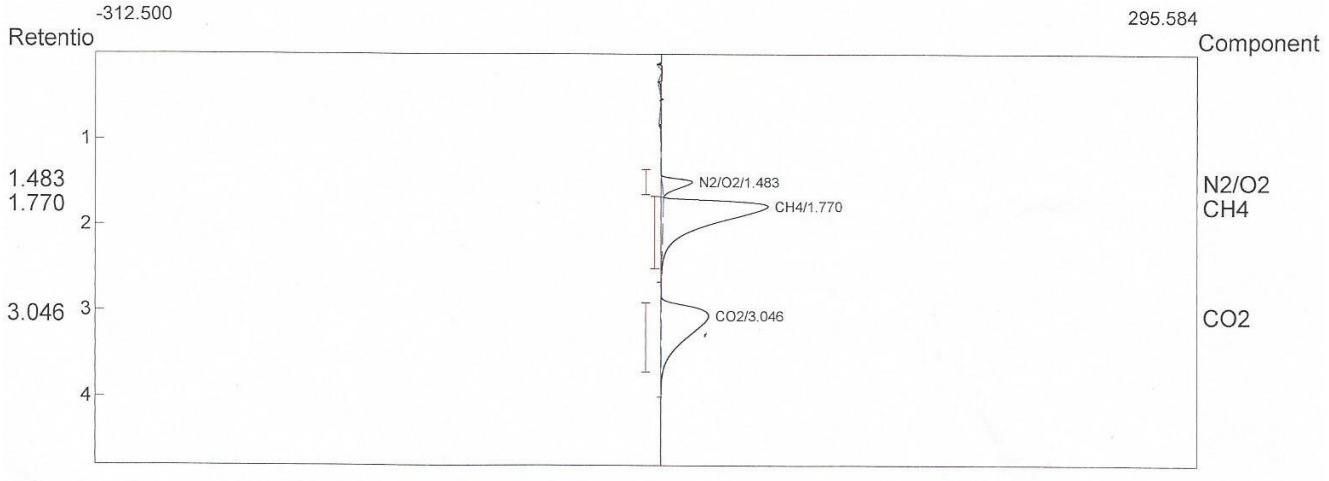
Fig. 13 – Schematic of the operation of the RT digester including the various analytical tests that were performed and the source of the samples for those tests. For comparison, the schematic for the RT digester is shown in Figure 12.

2.5 Biogas Characterization

The biogas samples collected during the week were transported to Anaergia's lab facility once each week to undergo composition analysis. The composition of the produced biogas from each digester was assessed using gas chromatography; which is a

method in which a gaseous mixture is passed through a chromatography column filled with a packing material with the help of an inert gas (also known as a pusher gas). The various compounds in a gaseous mixture have a different binding affinity to the columns packing material which causes the individual compounds to have different residence times in the column. As the gases exit the column various types detectors can be used to analyze exiting compound and determine the quantity at that retention time. This can be correlated to a pure sample of a known gas to determine the typical residence time for this compound in the column; allowing for the quantity of specific compounds in a gaseous mixture to be determined. An example of the raw chromatogram and the resulting area under the corresponding peak for each typical compounds residence time can be found in Figure 14. Which compounds can be detected is dependent on several factors such as the column packing material, the inert pusher gas, and the detector. In this case the inert gas used was helium and the gas chromatography unit used was an 8610C gas chromatograph (SRI) with a Restek MXT-WAX column and thermal conductivity detector. For these experiments the compounds which could be detected include methane, carbon dioxide, and nitrogen/oxygen however the last two compounds could not be detected separately which can be seen in the raw chromatogram in Figure 14 below; it was assumed that the nitrogen/oxygen peak was virtually all nitrogen. It should also be noted that the ratio of nitrogen in these samples is not indicative of results that would be expected on the plant scale where there should only be trace amounts; rather the increased fraction of nitrogen is due to the nitrogen blanket applied when removing waste digestate and feeding. The typical volume fractions of nitrogen were less than 25 percent of the total volume in a

typical GC measurement. Because full-scale AD processes would not have a nitrogen purge step, the composition results are presented on a nitrogen-free basis using the peak areas for carbon dioxide and methane to calculate the percent volume of carbon dioxide and methane. This is done by calculating the mass of each compound using the area under each corresponding curve on the chromatogram (seen at the bottom of Figure 14) and a calibration curve with the system software provided by the chromatograph supplier SRI. The mass of each compound was converted to the volume using the density of each gaseous compound at 1 atm; from these values the total volume, or the sum of the volume of methane and carbon dioxide could be calculated and the % volume of each compound determined.



Component	Retention	Area	Height	External	Units
N2/O2	1.483	122.4262	16.850	0.0000	
CH4	1.770	1002.2785	57.774	0.0000	
CO2	3.046	657.9839	26.185	0.0000	
		1782.6886		0.0000	

Fig. 14 – Example of one gas chromatography raw chromatogram. Area under each curve calculated by SRI provided software. Retention time (left axis) for the area under the curve for each component determined by calibration curve provided by SRI for biogas analysis. Methane to carbon dioxide ratio then calculated using the area under the curve spanning the typical residence time for each component in a spreadsheet also provided by SRI.

2.6 Digestate Quality Testing

2.6.1 Total/Volatile solids

The TS and VS content was measured weekly for each digester and additionally with each new sample of primary feed sludge obtained from Dundas Valley WWTF. The test was performed identically to the one described in section 2.1.2 with the only change is the volume tested was 20 mL. The TS and VS were also calculated using the equations 1 and 2 found above. The results for each week of the two 40-day cycles for all digesters can be found in Table 1 and 2.

2.6.2 Total/Volatile suspended solids

The total (TSS) and volatile (VSS) suspended solids content of the filtrate recovered during RT was measured daily for each RT digester. These tests were performed in order to determine the daily SRT for the RT digesters. The filter paper used had a pore size of 0.35 μm and was dried in the 105 ^{o}C oven before use. The initial mass of the dried filter paper was measured and then a known volume of filtrate was passed through the filter paper under vacuum. The filter paper was then placed in an aluminum weigh tin and dried in a 105 ^{o}C oven for a minimum of 24 hours, cooled in a dry environment, and then the filter paper weighed again. To measure the VSS the same, but now dried, sample was placed in the 550 ^{o}C furnace for two hours then cooled and weighed for a final time. The TSS and VSS were calculated using equations 3 and 4 respectively:

$$TSS\ (w/v\ \%) = \frac{(Filtered\ mass - 24\ hour\ dry\ (g)) - (Fresh\ filter\ mass\ (g))}{Volume\ of\ Filtrate\ (mL)/1000}/10 \quad (3)$$

$$VSS\ (w/v\ \%) = \frac{(Filtered\ mass - 24\ hours\ dry\ (g)) - (Furnaced\ filter\ mass\ (g))}{Volume\ of\ Filtrate\ (mL)/1000}/10 \quad (4)$$

The TSS values determined from the RT filtrate along with the total volume of filtrate and the TS of the digester were used to calculate the SRT on a day-by-day basis for each of the RT digesters. Equation 5 below was used to calculate the SRT where the 'Daily Wasted Volume' refers to the daily 50 mL of waste from the digester and the 'Digester Working Volume' is a constant 1.5 L:

$$SRT = \frac{(Digester\ Working\ Volume\ (L) * TS\ of\ Digester\ (g/L))}{(TSS\ (g/L) * Volume\ of\ Filtrate\ (L)) + (TS\ of\ Digester (g/L) * Daily\ Wasted\ Volume\ (L))} \quad (5)$$

The daily SRT for each digester had various ranges depending on the polymer flocculant and dosage used for RT; the day-by-day SRT values can be found in Figure 15. SRT during RT with the C-6267 polymer flocculant had a range of 22.8-29.8 days, a range of 23.9-29.8 for the 440 LH flocculant, and 26.7-29.9 for the 4516 flocculant. As expected, all RT digesters consistently had an SRT greater than 20 days which was the SRT for the control digester.

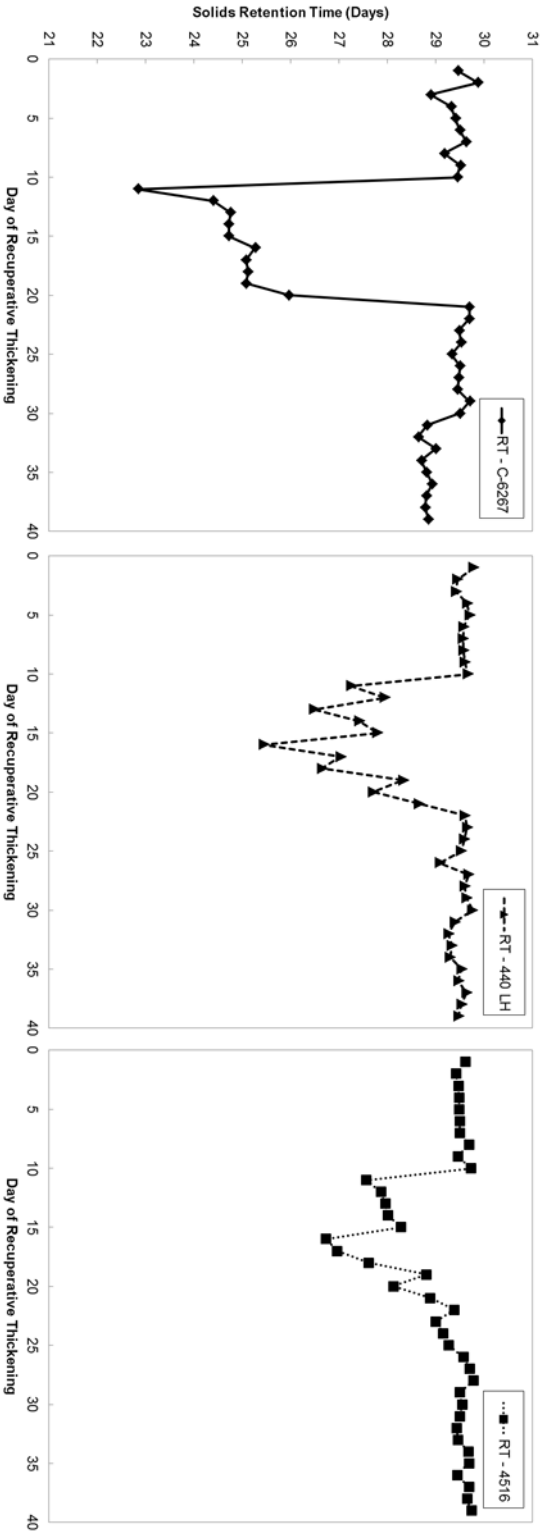


Fig. 15 – Daily SRT for the three recuperative thickened digesters over the 40-day cycle. SRT was calculated using a mass balance involving the TSS of the filtrate during the RT step to measure the solids lost during RT. The control digester had a constant SRT = 20 days.

2.6.3 Total and soluble COD

Each week both the total and soluble COD was measured for each digester. In the case of the digestate the TCOD refers to all the organic compounds in a given sample which would include the anaerobic bacteria as well as any extracellular material, undigested feed, and additional dissolved particles. For this paper the soluble COD (SCOD) refers to organic matter that is smaller than 0.35 µm, which can include undigested feed and other dissolved particles. While the TCOD gives us a good reference to the total organic mass currently in the digester, the SCOD can be used to measure the stability of the digester. A rising SCOD while the TCOD remains constant can be an indication (among other factors) of over-loading; a case where the anaerobic bacteria cannot breakdown and consume the digester feed sludge as fast as it is introduced.

The Hach COD test kits used were also the high range vials featuring a measuring range of 20-1500 mg/L. Despite the high range test kits samples still required dilution before testing. Before the TCOD test the anaerobic digestate was diluted 1:50. For the SCOD test the anaerobic digestate was first placed in a disposable centrifuge tube (Falcon) and centrifuged at 7500 RPM using a (insert model) Beckman-Coulter centrifuge for 5 minutes to separate some of the large particles and aid in the ease of filtration. The resulting centrate was then passed through a syringe filter with an average pore diameter of 0.35 µm into a glass beaker. This filtered centrate was then diluted 1:25 with DI water before the COD test was performed. The COD test was then performed identically as described in Section 2.1.3 for the diluted TCOD and SCOD samples. The

results were then corrected for each given dilution and recorded in Table 1 and 2 at the end of Section 2.6.

2.6.4 Total Kjeldahl nitrogen

The Total Kjeldahl nitrogen (TKN) was measured weekly for each digester direct from the digestate samples using the Hach Simplified TKN kit. The Simplified TKN kit allowed for the measurement of both the total nitrogen and the TKN in a given sample. The TN/TKN can be used to calculate the carbon-to-nitrogen ratio (C:N); when the C:N begins to rise it can affect the biogas composition by changing the metabolic pathways of bacteria resulting in a decreased methane concertation in the biogas [Lin and Lay, 2004; Hills, 1979].

In order to obtain a reading within the range of the test the raw digestate sample was first diluted 1:500 in a volumetric flask using DI water. In digesting vial, 1.3 mL of diluted sample was mixed with 1.3 mL of liquid reagent A and 1 tablet of solid reagent B. The vial was then placed in a Hach DRB200 digital reactor block at 100 ^{o}C for one hour then subsequently cooled to room temperature. After cooling, solid reagent C was then added to the digesting vial and mixed into solution. From the resulting solution 0.5 mL was pipetted into Vial 1 of the test kit along with 0.2 mL of reagent D, this mixture was then allowed to reactor for 15 minutes. Simultaneously, 1 mL of the same raw diluted sample was pipetted into Vial 2 from the test kit along with 0.2 mL of reagent D and allowed to react for 15 minutes. After 15 minutes the vials were read in the order of Vial 1 then Vial 2 in a Hach DR3900 spectrophotometer to determine the TN and the TKN.

The results from these experiments for each 40-day cycle can be found in Tables 1 and 2 with the values corrected for the initial dilution of the raw digestate sample.

2.6.5 Ammonia

The ammonia concentration was measured once per week for each digester using a Hach Ammonia vial kit. High concentrations of ammonia in an AD can lead to inhibition of anaerobic bacteria through a decrease in the specific growth rate causing lowered biogas yields [Hansen et al., 1997]. The sample for each digester was prepared in the same way as the SCOD sample described in section 2.6.3; however the filtered centrate was instead diluted 1:50 with DI water before it was tested with the ammonia vial kit. After dilution, 0.2 mL of sample was added to the ammonia test kit vial and the cap flipped upside-down, exposing a reagent to the sample. The vial was shaken well, until all the reagent contents in the lid became mixed into the vial; it was then left to react for 15 minutes. After 15 minutes the vials were analyzed on a Hach DR3900 spectrophotometer. The results from these experiments for each 40-day cycle can be found in Tables 1 and 2 with the values corrected for the initial dilution of the filtered centrate sample.

2.6.6 pH measurement

The pH of each digester was measured once per week except if there was a pH measurement that was outside of the operating window of 7.0-8.0 in which case the pH would be measured daily in that digester. The pH of the digester plays an important role in a digester; a pH outside of the operating range can lead to the inhibition of the methanogenic bacteria population [Appels et al., 2008]; it can also be an indication that

there is an inhibition of the acetogenesis step involving the breakdown of fatty acids (FA) into hydrogen and acetic acid leading to an increase in the FA. The pH was measured from the daily waste using a VWR sympHony B30PCI Benchtop Multi-meter with pH probe. The results from the weekly pH readings for each 40-day cycle can be found in Tables 1 and 2; during the duration of both 40-day cycle tests there was no pH measurement that fell outside of the operating window stated above.

Table 1 – Summary of testing done during the first 40-day cycle on both Control and RT digesters throughout the experiment. Weeks are labelled to match the previous figures for additional clarity.

	10 kgP/TTS Build Up				2 kgP/TTS Constant				10 kgP/TTS Build Up				4 kgP/TTS Constant			
	Week 1		Week 2		Week 3		Week 4		Week 5		Week 6		Week 7		Week 8	
	Control	RT	Control	RT	Control	RT	Control	RT	Control	RT	Control	RT	Control	RT	Control	RT
pH	7.24	7.43	7.19	7.46	7.45	7.59	7.46	7.68	7.22	7.30	7.29	7.71	7.24	7.29	7.41	7.23
TCOD($\frac{g}{L}$)	29.10	29.50	22.15	36.50	22.45	32.45	22.10	33.10	24.85	30.60	28.35	36.95	24.25	48.10	23.55	42.05
SCOD($\frac{g}{L}$)	0.86	1.73	1.22	0.98	0.77	1.44	0.74	0.94	0.82	1.27	0.81	1.64	1.00	1.08	0.95	1.12
TN ($\frac{g}{L}$)	1.52	1.83	2.03	1.81	1.62	2.18	1.78	1.91	2.36	1.95	1.78	2.14	1.77	2.59	1.62	2.28
NH_3 ($\frac{g}{L}$)	0.72	0.67	0.77	0.72	0.73	0.57	0.83	0.71	0.94	0.78	0.87	0.80	0.84	0.80	0.81	0.76
OLR ($\frac{gVS}{L*d}$)	1.78	3.55	1.78	3.55	1.78	3.55	1.41	2.82	1.41	2.82	1.41	2.82	1.67	3.34	1.67	3.34
TS (wt %)	2.34	2.49	2.35	2.97	2.22	3.20	2.31	3.14	2.51	3.23	2.40	3.51	2.53	4.07	2.47	4.71
VS (% of TS)	47.0	62.7	59.1	59.9	53.5	56.9	58.4	55.7	58.6	55.1	49.1	55.0	55.3	50.0	57.9	49.1

Table 2 – Summary of testing done during the second 40-day cycle on both digester 1 (440 LH) and digester 2 (4516) throughout the experiment. Weeks are labelled to match the previous figures for additional clarity.

	10 kgP/TTS Build Up				2 kgP/TTS Constant				10 kgP/TTS Build Up				4 kgP/TTS Constant			
	Week 1		Week 2		Week 3		Week 4		Week 5		Week 6		Week 7		Week 8	
	440 LH	4516	440 LH	4516	440 LH	4516	440 LH	4516	440 LH	4516	440 LH	4516	440 LH	4516	440 LH	4516
pH	7.34	7.30	7.17	7.05	7.28	7.25	7.31	7.28	7.24	7.21	7.23	7.20	7.31	7.28	7.33	7.26
TCOD($\frac{g}{L}$)	30.10	29.6	34.10	28.05	38.70	41.30	32.30	35.05	35.95	37.95	43.70	47.20	49.20	51.25	48.85	55.25
SCOD($\frac{g}{L}$)	1.51	1.45	1.03	0.89	1.15	0.91	0.87	0.78	0.73	1.05	0.77	0.87	0.77	1.04	0.86	0.91
TN ($\frac{g}{L}$)	1.82	1.65	1.90	1.81	1.97	2.29	2.29	1.96	1.90	2.19	2.59	2.50	2.70	2.31	2.64	2.46
NH_3 ($\frac{g}{L}$)	0.67	0.66	0.74	0.72	0.75	0.62	0.75	0.71	0.74	0.61	0.75	0.78	0.83	0.85	0.81	0.80
OLR ($\frac{gVS}{L*d}$)	4.65	4.65	4.65	4.65	4.65	4.65	4.20	4.20	4.20	4.20	4.11	4.11	4.11	4.11	4.11	4.11
TS (wt %)	2.46	2.41	3.43	3.31	3.44	3.56	3.84	3.68	4.08	3.85	4.64	4.36	4.61	4.82	4.61	4.72
VS (% of TS)	62.8	60.5	63.8	62.9	61.8	61.7	58.9	59.4	61.0	62.0	61.4	58.7	62.0	64.0	62.9	63.0

2.7 CST Measurements

The dewaterability of both flocculated and un-flocculated samples from both digesters was analyzed using a capillary suction time (CST) instrument (Triton Electronics). The CST was measured using Whatman^{TM} 17 chromatography paper and the cylindrical 'slow-filter' reservoir insert. A minimum of 3 mL of sample was used for each test and each sample was tested in triplicate.

For the daily dewaterability testing, 25 mL of the daily wasted digestate from each digester was placed in a 50 mL beaker on a magnetic stir plate. A magnetic stir bar (VWR, 15 mm) was added and the beaker and the magnetic stirrer turned to 700 RPM. Using a pipette, the required dosage of polymer flocculant was added to the beaker and mixed at 700 RPM for 10 seconds. The stirring was then lowered to 300 RPM and mixed under these conditions for 1 minute. Approximately 10 mL of the flocculated digestate was then transferred into the CST 'slow' filter and a CST measurement completed. As mentioned, this was done in triplicate for both digesters flocculated at the same flocculant dosage; the control digester was flocculated using the C-6267 polymer flocculant and each RT digester was flocculated using their corresponding polymer flocculant. Similarly, weekly CST tests were done in triplicate on the digestate from both the control and recuperative thickened digesters without the addition of polymer flocculant to establish baseline dewaterability in each digester.

For the end-stage dewaterability test a smaller scale high-throughput method of testing was performed in order to maximize the number of experiments with a limited amount of sample. The end-stage dewatering tests were performed on the digestate from

the daily wasted volume from each digester which remained after the daily dewatering tests were performed and any other weekly digestate quality testing such as a TS/VS test. The system was identical to the one used in a recent study on polymer flocculant screening for digestate dewaterability [LaRue et al., 2016]. A disposable polystyrene 12-well cell culture plate (VWR) was filled with approximately 3 mL of digestate sample in each well with a small magnetic stir bar (VWR, 7.5 mm). The wells were mixed simultaneously at an initial speed of 650 RPM using a VP-710C Tumble stirrer from V & P Scientific. Various polymer dosages were added in triplicate and mixed at the 650 RPM speed for approximately 10 seconds; the speed was subsequently dropped to 350 RPM and mixed for one additional minute. The stir bars were removed and custom plugs placed over the top of each well, with one well left open to pour out of. Using a custom built pour spout the digestate was poured into the CST 'slow' reservoir and the dewaterability measured. A photo of the tumble stirrer set up with digestate can be found below in Figure 15 [LaRue et al., 2016].

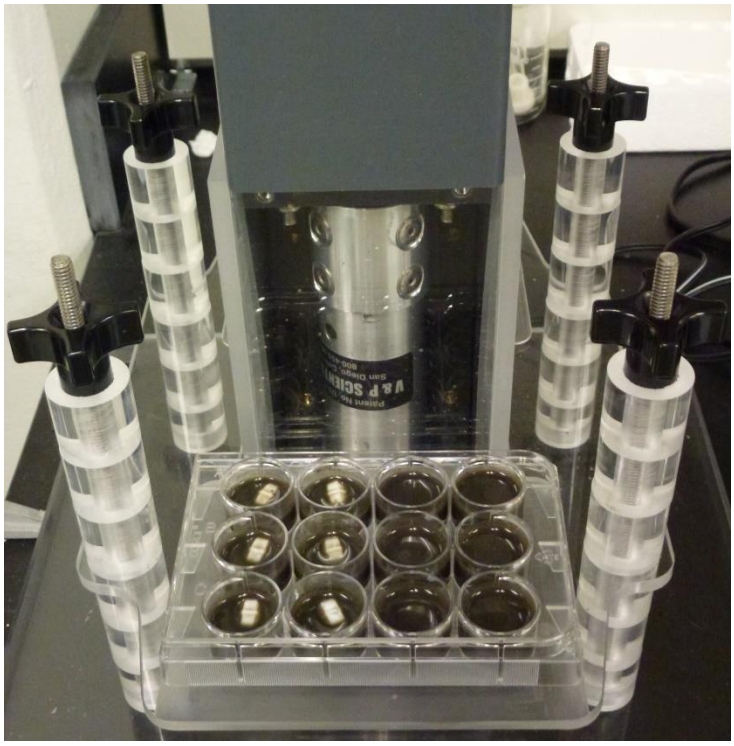


Fig. 16 – Tumble stirrer and 12-well plate depicted with digestate and stir bars in a custom built stand. [LaRue et al., 2016]

- Chapter 3 -
Modelling and demonstration of lab-scale recuperative thickening

The majority of the work presented in this chapter will be published in a forthcoming issue of *Water Research* [Cobbledick et al., 2016]. Since this lab-scale RT setup was designed to emulate the full-scale RT plant Anaergia is currently operating, it was important to complete the initial testing with the same polymer flocculant that they were currently using; this is why the C-6267 polymer was chosen to be the first flocculant tested and compared to the control digester. Throughout the following chapter the C-6267 polymer flocculant is the only referred to.

3.1 Recycled Polymer Modelling

3.1.1 Long term single polymer dose model

Due to the recycling of flocculated digested solids during the RT operation, there is an initial buildup of polymer flocculant. In order to understand both the steady state concentration achieved at different doses as well as the time it would take to reach these steady state conditions, a simple mass balance model was created to track the accumulated polymer concentration in the lab-scale digester. The model results are shown in Figure 17 for doses ranging from 1 kg of active polymer per total tonne of solids (kgP/TTS) to 10 kgP/TTS. A key assumption in the model is that all the polymer chains added during the flocculation step become bound to solids particles and therefore are returned to the digester with the solids fraction. According to previous full-scale plant studies performed by Anaergia, the target polymer doses for the RT digester were 2 and 4 kgP/TTS dose. It was expected that these conditions would allow for a good contrast

between the control and RT digester operated with the same polymer flocculant and also a comparison of different polymers used for the RT digester. As shown in Figure 17, it takes approximately 80 days for the concentration to reach a value within 5% of the final constant polymer concentration. This was found to be quite unacceptable and thus an alternative mode of operation was sought.

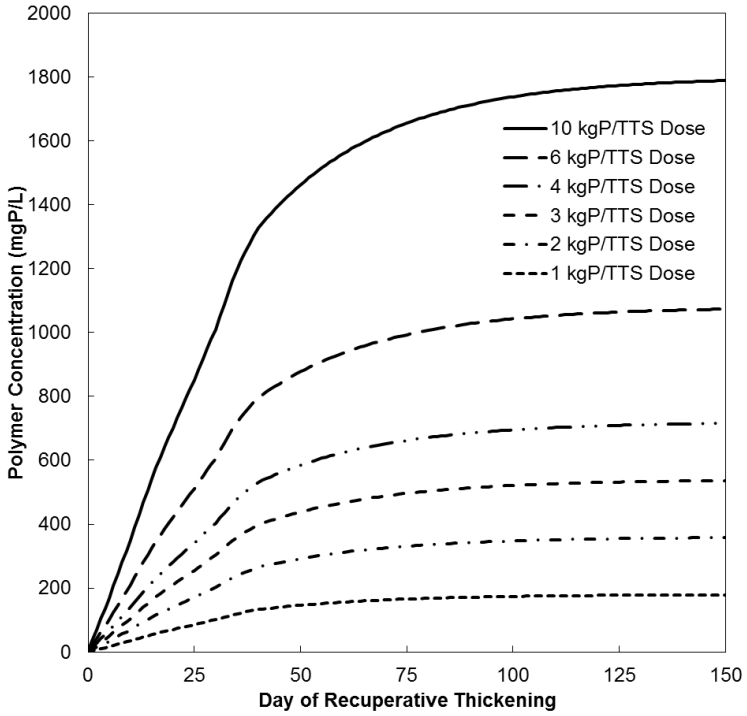


Fig. 17 – A comparison of the kinetics of polymer 'build-up' in the RT digester for various doses of polymer flocculant added to the recuperative volume. The model was developed for a total solids (TS) content of 4 wt% (w/v) in the digester.

3.1.2 'Accelerated' polymer dosing model

An alternative approach is to initially start at a very high polymer dose to build-up the concentration of polymer in the digester and then decrease the dose to the desired level; this strategy has been successfully used in full-scale plant trials [Wang et al., 2008; Ostapczuk et al., 2011]. The actual predicted polymer concentration result of running

under this ‘accelerated’ mode of operation is shown in panel A of Figure 18 using the real TS data from the RT digester in the first 40-day cycle. By initially operating the RT digester at a 10 kgP/TTS dose, the desired constant polymer concentration corresponding to a 2 and 4 kgP/TTS dose can be reached in approximately 10 and 20 RT days respectively. In our process 5 RT days correspond exactly to one full week of testing because the digesters were not fed over the weekend. Panel B of Figure 18 depicts the predicted polymer concentration in a RT digester operating under ‘ideal’ conditions to reach the exact constant polymer concentration of 360 and 720 mgP/L corresponding to the 2 and 4 kgP/TTS long term dose.

Then the polymer concentration is maintained at the desired level for 2 weeks to allow for monitoring of the dewaterability and digester performance. During this time, the RT digester TS content increased from approximately 2.3 to 4.7 wt % (w/v); however the final TS content is well below the value where mass transfer limitations become apparent, which is 10 wt% in AD [Abbassi-Guendouz et al., 2012]. The weekly TS values for both the control and RT digester can be found in Table 1 in the Section 2.6. Overall, it took just eight weeks (i.e. 40 RT days) to complete the two ‘build-up’ stages (at the 10 kgP/TTS dose) and run the two weeks of testing at the two polymer concentrations of 2 and 4 kgP/TTS. This is a considerable advantage over the strategy of operating at a single dose until the desired polymer concentration was reached as it would require more than double that amount of time (see Figure 17).

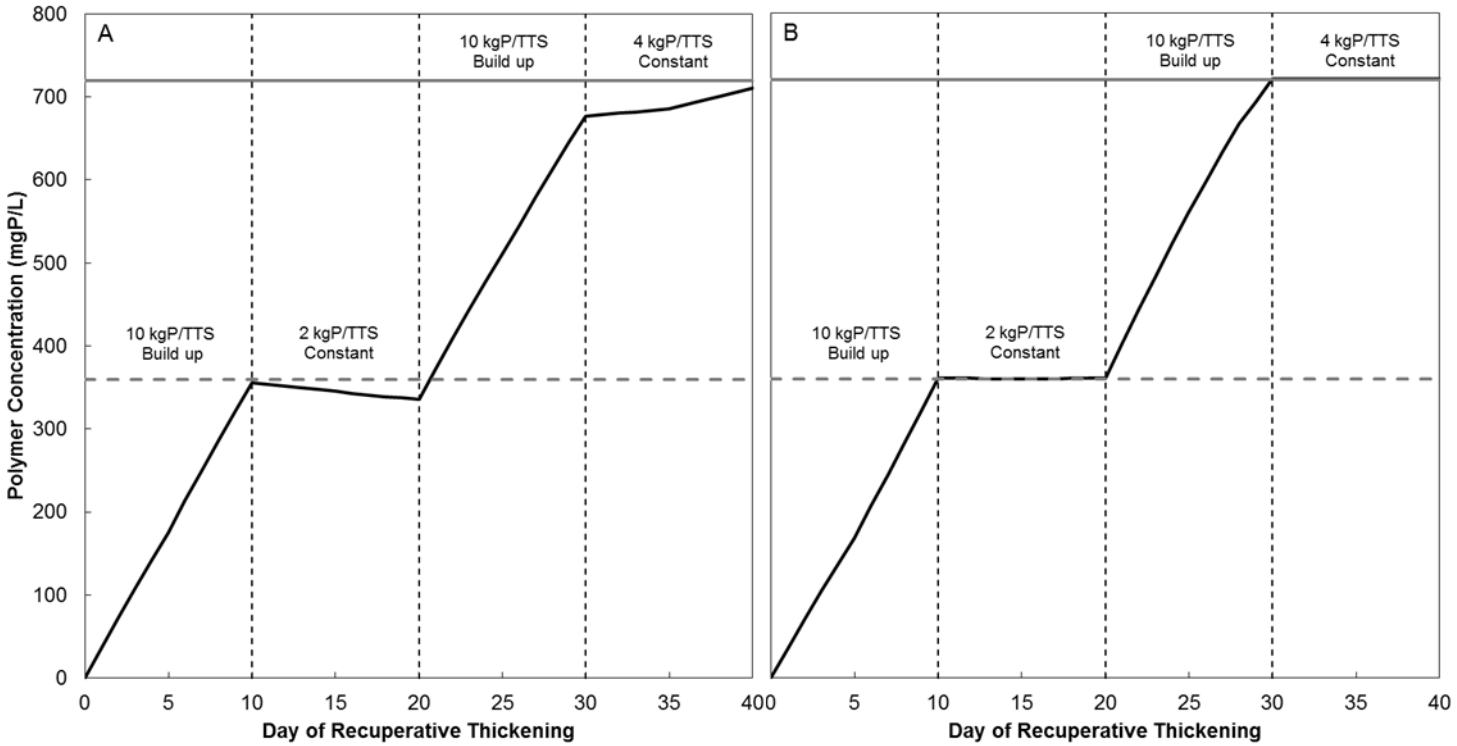


Fig. 18 – Panel A depicts the polymer concentration profile during recuperative thickening as predicted by a digester mass balance and using actual total solids content measurements. Panel B depicts the ideal operation to reach the exact desired constant polymer concentrations. The horizontal dashed and solid lines indicate the steady state polymer concentrations (as shown in Fig. 17) for a 2 and 4 kgP/TTS dose respectively. The vertical dashed lines are shown to identify the two different stages of the polymer flocculant dosing cycle – a high dose (10 kgP/TTS) is used to rapidly build up the polymer concentration in the digester and then a constant dose is used to evaluate the digester performance.

3.2 Digestate Dewatering

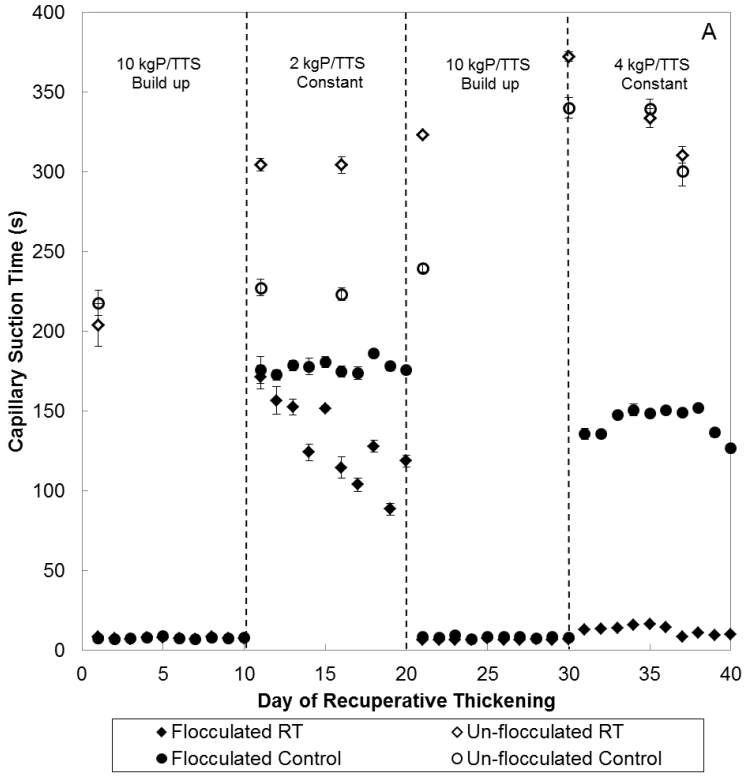
3.2.1 Control vs. Recuperative thickening performance

The CST of the flocculated sample from the waste volume from both digesters was measured on a daily basis. Each sample was analyzed in triplicate and the results are shown in panel A of Figure 19 for the entire test cycle. Also shown in panel A of Figure 19 are the CST values for un-flocculated digestate. As an additional measure of dewatering performance, the quality of the filtrate (as indicated by the TSS values) obtained with the RT digester is displayed in panel B of Figure 19. During the first ten RT days, a 10 kgP/TTS dose of C-6267 polymer flocculant was used. It is important to note that this value actually specifies different information for the two digesters – for the RT digester it specifies both the dose used to thicken the solids that are blended with the feed and returned to the digester and the dose used to flocculate the waste volume from the digester, however for the control digester it specifies only the latter as there is no thickening/blending step. The CST values for the flocculated samples from the control and RT digesters are indistinguishable; for all ten days of testing the average CST was less than 10 seconds and the TSS measured was less than 0.35 g/L indicating excellent solids capture and dewatering performance. This result was expected because the typical doses used for end-stage dewatering processes range from 6 to 12 kgP/TTS [Boráň et al., 2010]. Also, there is no significant difference in the CST values for the un-flocculated digestate on day one as the initial TS values are nearly identical (see Table 1) because both digesters were operated according to the control feed schedule during the twelve week period that immediately preceded the 40-day test cycle.

During the next ten RT days (i.e. days 10 through 20), both of the digesters were evaluated at a constant polymer concentration of 2 kgP/TTS. The un-flocculated CSTs for the RT digester are approximately 40% higher than those for the control digester because of the higher TS values in the RT digester (3.1 to 3.2 wt%) compared to the control digester (2.2 to 2.3 wt%) – refer to Table 1 in the Section 2.6. It is interesting to note that during this period, the CST values for the control digester did not change while those for the RT digester gradually decreased from an initial value of approximately 170 seconds to a final value of approximately 100 seconds. A similar trend was found for the filtrate TSS values; the TSS decreased from approximately 4.5 g/L to 2.2 g/L over the 10 RT days. As shown in Figure A.3 in the Appendix, it was found that the CST results are well correlated to the filtrate TSS values. This phenomenon is likely due to the polymer flocculants in the RT digester being partially active and enhancing the solids capture and floc formation during the consequent flocculation leading to improved dewatering performance during RT. Previous work has shown that the charged groups on the polymer flocculants can be partially degraded in AD processes but not the polymer backbone [Chang et al., 2001]. It is generally well-accepted that the desired CST to achieve effective dewatering performance in mechanical processes is less than 20 seconds. While the recorded CST values at the constant polymer concentration of 2 kgP/TTS are over 5 times higher than this target, the significant difference in CST values for the RT and control digesters indicates that there is a significant improvement in dewaterability. It can also be seen that the percent improvement in CST for the flocculated versus un-flocculated samples is significantly higher for the RT digester.

During the next ten RT days (i.e. days 20 through 30), a 10 kgP/TTS dose of polymer flocculant was used again to 'build up' the amount of polymer in the RT digester to the long-term equivalent of a 4 kgP/TTS dose. The recorded CST results were essentially identical to the first 'build up' stage indicating the robustness of the process. The final ten RT days were conducted at a constant polymer concentration of 4 kgP/TTS. At this condition, it was found the recycled polymer flocculant in the RT digester had a dramatic effect on the dewatering performance. For example, the CST values for the flocculated samples from the RT digester were over 6 times lower than the corresponding values from the control digester. The TSS of the filtrate was also significantly lower at an average of 0.6 g/L compared to the lowest TSS value (2.2 g/L) that was found for the 2 kgP/TTS constant polymer concentration. Also, the CST values for the RT digester were consistently less than 20 seconds indicating acceptable dewatering performance; in comparison, the CST values for un-flocculated samples from the RT digester were greater than 300 seconds. The dewatering performance for the RT digester at 4 kgP/TTS constant concentration was nearly identical to that which was achieved at the 10 kgP/TTS dose while the TSS values were slightly higher. However at the same polymer flocculant dose of 4 kgP/TTS it could be strongly hypothesized that the control digester would achieve significantly higher filtrate TSS values if filtration was performed. Overall, the dewatering performance is much improved in the RT digester compared to the control one for the same polymer flocculant dosage; our hypothesis is that there exists an opportunity to significantly reduce the amount of polymer flocculants required for RT processes by judicious selection of the polymer type and dosage conditions. This is

important not just from a techno-economic perspective but also an environmental perspective as polymer flocculants have been reported to be toxic to aquatic lifeforms even at low concentrations [Costa et al., 2014; Harford et al., 2011; Liber, 2005].



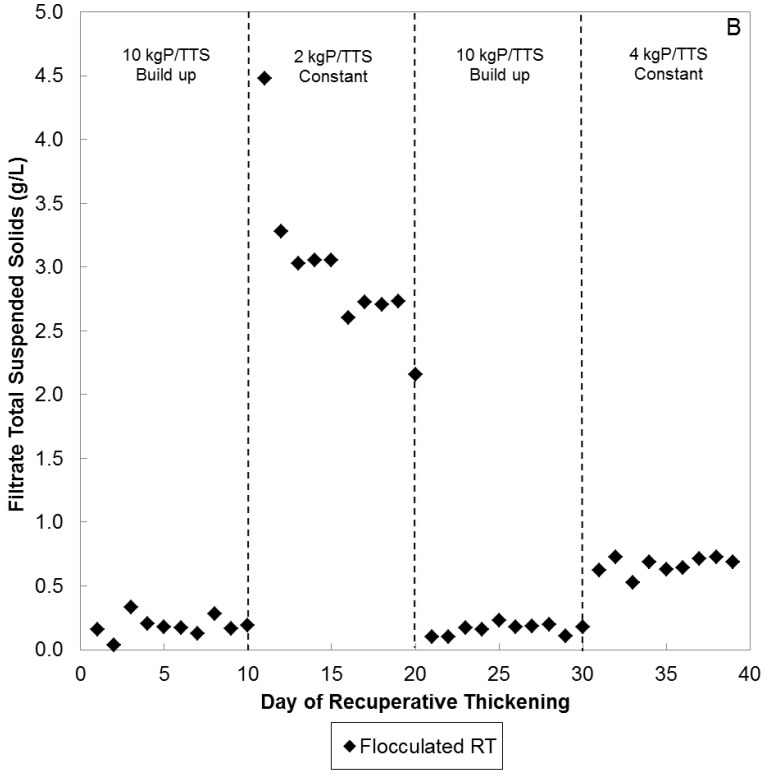


Fig. 19 – A comparison of the dewaterability performance during the first 40-day test cycle for both the RT (diamonds) and control (circles) digesters. Panel A depicts the dewatering performance as measured by the capillary suction time; the un-flocculated and flocculated samples are represented by open and filled symbols respectively. Each sample was analyzed in triplicate and thus each data point is the average and the error bars are the standard deviation. Panel B depicts the dewatering performance based off of the TSS of the filtrate collected during the daily RT`. For panel B the symbol shape was used to intentionally match that for the 'Flocculated RT' results in panel because the flocculated CST dewatering performance is well correlated to the concentration of suspended solids in the filtrate as shown in Figure A.3 in the Appendix. The vertical dashed lines in both panels are shown to identify the different stages of the polymer flocculant dosing cycle as shown in Fig. 18.

3.2.2 End-stage dewatering performance

In order to better understand the effect of the polymer flocculant dose used for an end-stage dewatering process on RT digester performance, a series of tests were conducted for both the 2 and the 4 kgP/TTS constant concentration doses. Six different doses were evaluated in triplicate analysis with the results shown in Figure 20. Note that the CST results for the combination of 2 kgP/TTS constant concentration and 2 kgP/TTS

end-stage dose match the results for the flocculated RT during the second 10 RT days in Figure 19; similarly the CST results for the combination of 4 kgP/TTS constant concentration and 4 kgP/TTS end-stage dose match the results for the flocculated RT during the fourth 10 RT days in Figure 19. All the CST values for end-stage doses greater than or equal to 4 kgP/TTS are less than 25 seconds, and thus it was found that there exists an opportunity to decrease the amount of polymer without affecting the dewatering performance. However, at doses lower than 4 kgP/TTS, the CST values increase significantly. For example, the average CST for the 4 kgP/TTS constant concentration increases from 13 to 70 seconds as the end-stage polymer dose decreases from 4 to 2 kgP/TTS. It is interesting to note that operating at 2 kgP/TTS constant concentration in the RT digester will result in acceptable CST values at end-stage doses greater than or equal to 4 kgP/TTS. While it is not feasible to operate at this condition continuously, it does indicate that there is a sufficient amount of active polymer flocculant such that when enough additional polymer flocculant is added at the end-stage process an acceptable dewatering performance can be obtained. Furthermore, the results show that the 4 kgP/TTS constant concentration requires a minimum amount of additional flocculant to achieve the desired dewatering performance. Our results support the claim that recycling of the polymer flocculant in the digester can lead to a decrease in polymer consumption in the end-stage dewatering processes. We hypothesize that by optimizing the polymer flocculant and the process conditions there exists an opportunity to achieve an overall decrease in polymer consumption in AD processes as was suggested by Sieger and Parry [Sieger and Parry, 2004]. In our previous work, we demonstrated that there is a

significant variation in dewatering performance across a variety of commercially available polymer flocculants [Cobbledick et al., 2014]. We also hypothesize that additional improvements can be realized by optimizing the type of polymer flocculant used in RT. The bench-scale system developed in this work is ideally suited for conducting a systematic evaluation of the performance of different polymer flocculants in RT processes.

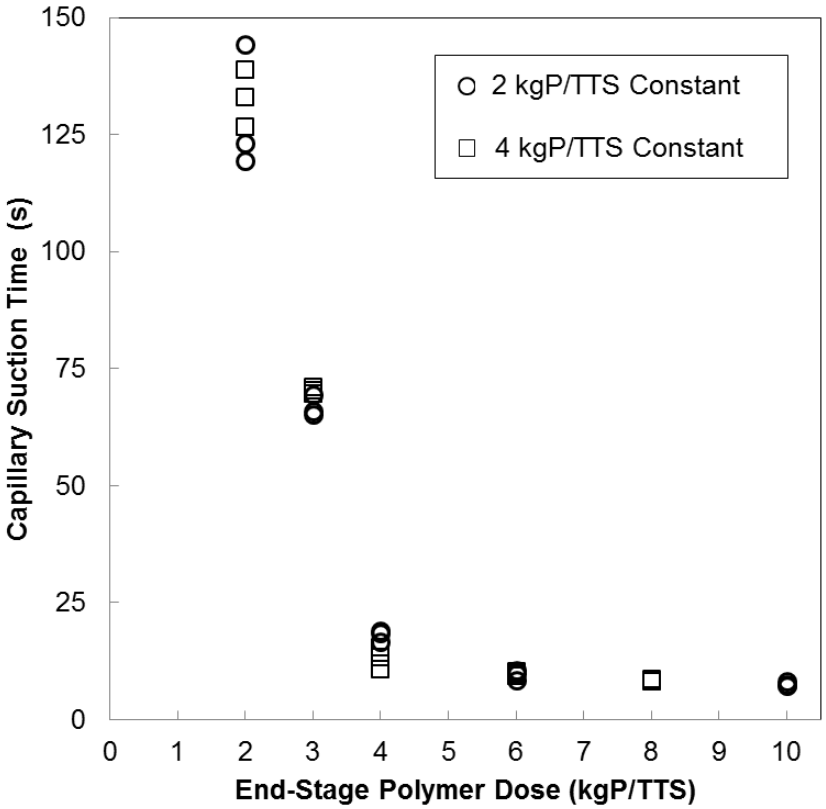


Fig. 20 – A comparison of capillary suction time results for RT digestate from the two different constant polymer concentration levels of 2 kgP/TTS (○) and 4 kgP/TTS (□) after they were again dosed with the same polymer flocculant (C-6267) to evaluate the 'end-stage' dewatering performance.

3.3 Biogas Production and Quality

3.3.1 Recuperative thickening effect on biogas yield

In order to monitor the quantity of the biogas produced in both the RT and control digesters, the produced volume was continuously monitored using Ritter type Mili-Gascounters. The daily biogas yield was calculated using the gas production values and the mass of VS fed with the results shown in Figure 21. For both digesters there is a regular pattern to the biogas yield over each five day RT cycle due to the semi-batch feed schedule (i.e. 5 days of feeding, corresponding to a 5 day work week, followed by a 2 day starvation period, corresponding to the weekend). The first day of each five day RT cycle always has the lowest yield because the microbial communities in the digesters are recovering from the 2 day starvation period. A fairly even yield is obtained during the three days in the middle of the five day RT cycle and the final day has the highest yield. For example, the biogas yields from the RT digester on days 21 through 25 were 0.30, 0.48, 0.48, 0.53, and 0.72 L/gVS respectively. Also, there is no significant difference between the biogas yields of the two digesters. Over the entire test cycle, the ratio of the RT digester biogas yield to control digester biogas yield ranged from 0.806 to 1.37 with an average value of 1.10. Our results indicate that the RT digester has an improved 'degradation efficiency' in VS destruction indicated by its equivalent biogas yield while at almost twice the OLR; this observation is in good agreement with the expectations for high performance AD processes [De la Rubia et al., 2006]. Additionally, the biogas productivity, defined in equation 1, of the RT digester is over two times greater on

average than the control digester as shown in Figure A.1 in the Appendix.

$$Productivity\left(\frac{L_{biogas\,produced}}{day*L_{digester}}\right) = OLR\,(\frac{gVS_{fed}}{day*L_{digester}}) * Yield\,(\frac{L_{biogas\,produced}}{gVS_{fed}}) \quad (1)$$

As shown in Table 1 in Section 2.6, the SCOD values for the control and RT digesters are similar which indicates that both processes are stable at their respective OLR. There was no buildup of SCOD in the RT digester, an indication of overloading, and the biogas productivity was 2.2 times larger than the control output. This would be expected if the feed rate was doubled and the VS destruction rate was at least maintained or improved.

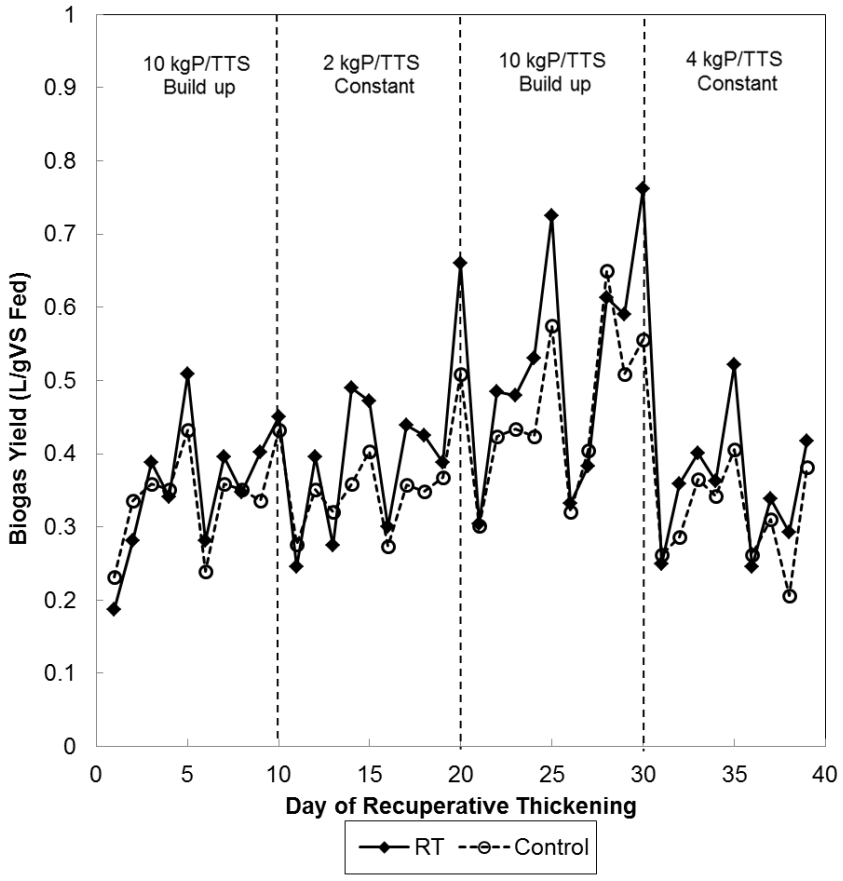


Fig. 21 – Biogas yield during the 40-day test cycle for both the RT (♦) and control (○) digesters. The vertical dashed lines are shown to identify the different stages of the polymer flocculant dosing cycle as shown in Fig. 18.

3.3.2 Recycled polymer effect on biogas quality

In order to monitor the quality of the biogas produced in both digesters, samples were collected and analyzed by gas chromatography once or twice per week. As shown in Figure 22, the biogas produced in the RT digester was very similar in composition to that from the control digester. During the first 30 RT days, the average amount of methane in the RT and control digesters was 65.8% and 67.0% respectively. There was a slight variation in biogas composition during the last 10 RT days where the methane content increased to over 70%. While the typical methane content for anaerobic biogas production is in the range of 60 to 70 % [Appels et al., 2008], our result is likely due to a variation in the incoming feed but additional research is required to confirm this hypothesis. Our results support the claim that the recycling of the polymer flocculant and short term exposure to oxygen during the thickening process did not adversely affect the methanogen activity and are in good agreement with Chu et al. (2003) in regards to the ratio of methane to carbon dioxide and the overall biogas production. Overall, the results from this study indicate that the incorporation of RT in AD processes leads to an improvement in VS destruction and thus increased capacity to accommodate more feed solids. The combination of RT with co-digestion of high strength organic wastes is expected to lead to increased gas production of equivalent quality and thus is ideally suited for WWTFs to achieve net energy neutrality or perhaps even net energy production.

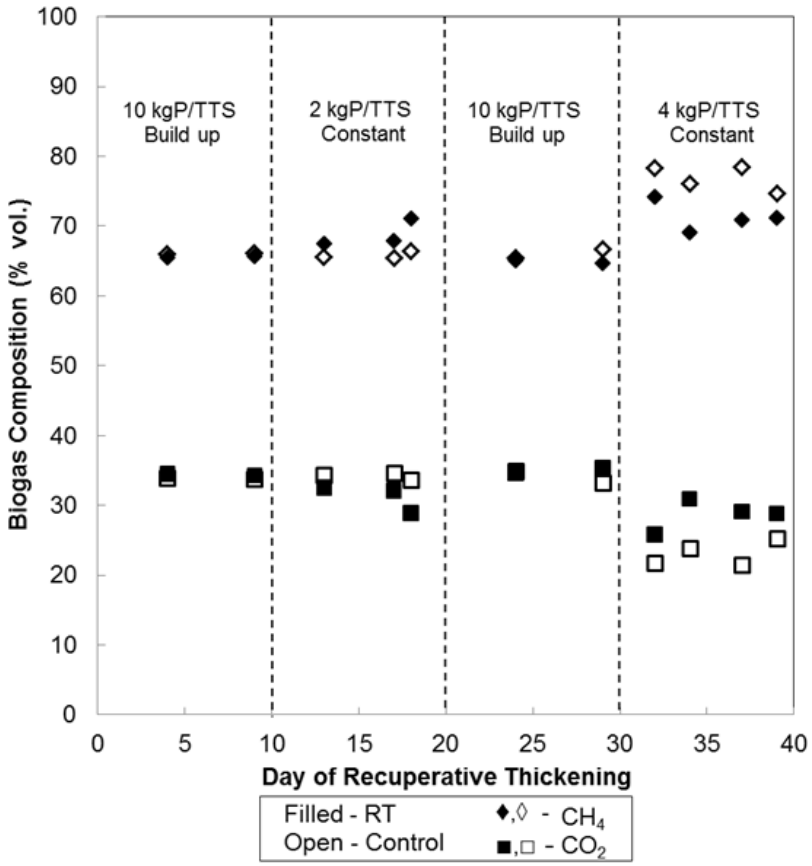


Fig. 22 – Biogas composition during the 40-day test cycle for both the RT (filled symbols) and control (open symbols) digesters. The methane and carbon dioxide values are represented by diamonds (♦,◊) and squares (■,□) respectively. The vertical dashed lines are shown to identify the different stages of the polymer flocculant dosing cycle as shown in Fig. 18.

- Chapter 4 -
Polymer flocculant optimization in lab-scale recuperative thickening

4.1 Digestate Dewatering

4.1.1 Comparison of various polymers' performance

One of the primary objectives of the lab-scale RT system was to assess the effect of various polymer flocculants via the dewaterability of the digestate; the manufacturer describes the C-6267 polymer flocculant as a very high charge density, high molecular weight polymer thus a polymer flocculant from another manufacturer with the same properties was chosen. The 4516 polymer flocculant from Kemira was selected as the second polymer flocculant to be tested; it is described as a high charge density, very high molecular weight polymer and had worked well previously in other dewatering applications. Since the particle bridging process is important in flocculation for dewatering applications and likely plays a role in maintaining some floc structure when the RT digestate returns to the digester decreasing the molecular weight which controls the size of the polymer may affect the observed dewaterability during RT. The 440 LH polymer flocculant from SNF is described as having a low molecular weight and high charge density and previously performed well in dewatering applications, hence it was chosen as the third polymer.

As performed in the previous 40-day cycle the CST of the flocculated sample from the waste volume from each digester was measured on a daily basis. Each sample was analyzed in triplicate and the results for the entire test cycle are shown in Figure 23. The CST values for the un-flocculated digestate also appear in Figure 23. Since both of

these digesters were operating under RT the specified dose refers to the amount of polymer flocculant used to both thicken the solids that are blended with the feed and then returned to the digester as well as the amount used to flocculate the waste volume from the digester. During the first ten RT days both the 440 LH and 4516 polymer performed similarly at a 10 kgP/TTS dose of polymer flocculant. For all ten days of RT at this dosage the CST was less than 10 seconds which indicates exceptional dewatering performance; this was a predicted result based off the prior 40-day cycle. There is no significant difference in the CST values for the un-flocculated digestate for each digester; in fact over the course of the 40-day cycle the CST values for the un-flocculated digestate appear to change similarly and never deviate more than 50 seconds apart while.

During the next ten RT days (days 10 through 20) both of the RT digesters were evaluated at a constant polymer concentration of 2 kgP/TTS. There was no significant change in the CST values for the un-flocculated digestate despite the increase in TS from an initial 2.4 wt% in both digesters to 3.8 and 3.7 wt% from digesters 1 and 2 respectively by the 20^{th} day of RT. It was observed that both digester 1 and 2 had decreasing CST values over this time period with digester 1 decreasing from approximately 77 seconds to a final CST value of approximately 57 seconds and digester 2 decreasing from approximately 70 seconds to 22 seconds. This phenomenon was also observed in the RT digester during the previous 40-day cycle and can likely be contributed to polymer flocculants remaining partially active during RT. While the 440 LH polymer flocculant did not perform poorly its final CST of approximately 57 seconds was still more than two times the CST of the 4516 flocculant. It should be noted that by the end of this low

polymer flocculant dose the 4516 flocculant was nearly able to attain what would be considered a CST low enough (≤ 20 seconds) to achieve effective mechanical dewatering performance.

The polymer flocculant concentration was increased during the next ten RT days (20 through 30) at a 10 kgP/TTS dose to again 'build up' the polymer concentration to a value of 4 kgP/TTS. The CST values recorded were basically identical to the first 'build up' stage (days 0 through 10). The final ten days of RT were conducted at a constant polymer concentration of 4 kgP/TTS and digestate from both digesters were flocculated at this 4 kgP/TTS dose. At this condition, the 440 LH polymer flocculant displayed a sizeable improvement in dewaterability as seen by the decrease in CST values from greater than 50 to less than 13 seconds. Meanwhile the 4516 polymer underwent only marginal improvements in dewaterability with CST values decreasing from just over 20 seconds to less than 10 seconds. This indicates that the long term minimum flocculant dose for the 4516 polymer is likely somewhere between the 2 and 4 kgP/TTS constant polymer concentrations. In both cases, the polymer flocculants were able to achieve a CST value of much lower than 20 seconds at this constant polymer concentration indicating excellent dewatering performance. As was observed in the previous 40-day cycle the dewatering performance of both RT digesters at the 4 kgP/TTS constant concentration was extremely close to that which was achieved at the 10 kgP/TTS dose further indicating the opportunity to significantly reduce the use of polymer flocculants in the RT process.

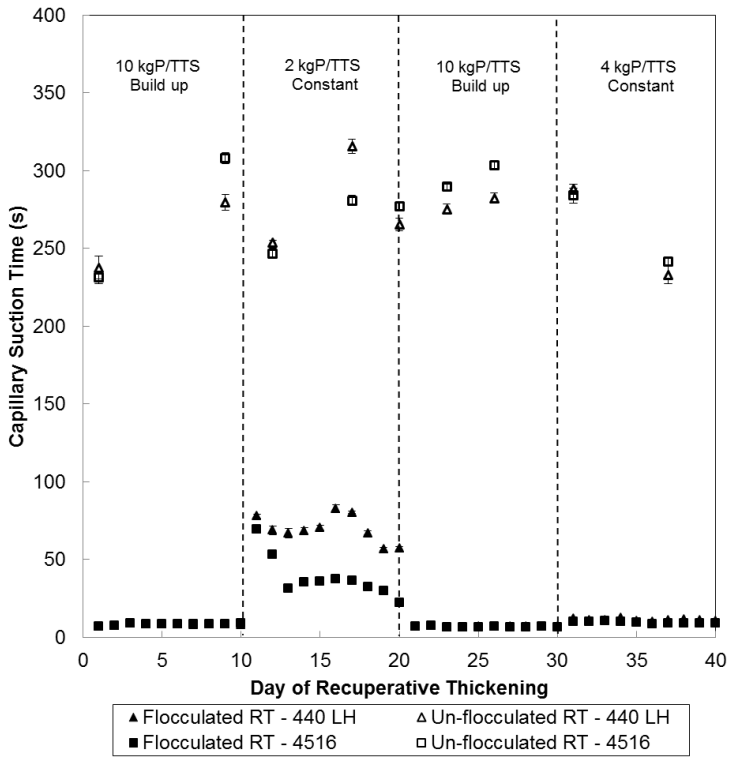


Fig. 23 – A comparison of the dewaterability performance during the second 40-day test cycle for two different polymer flocculants used for RT. The RT samples flocculated with the 440 LH polymer flocculant are represented by (▲) and 4516 polymer flocculant by (■) as measured by the capillary suction time; the un-flocculated and flocculated samples are represented by open and filled symbols respectively. Each sample was analyzed in triplicate and thus each data point is the average and the error bars are the standard deviation. The vertical dashed lines are shown to identify the different stages of the polymer flocculant dosing cycle as shown in Fig. 18.

We can further compare the dewatering performance during RT with three different polymer flocculants against a CAD which can be seen in the top panels of Figure 24 for both 40-day test cycles. It is abundantly clear by the low CST values that all three digesters operating under RT achieve superior dewatering performance compared to the control at the 4 kgP/TTS constant polymer dosage. It is interesting to note that based of the CST values, at the 4 kgP/TTS and both 10 kgP/TTS constant polymer concentrations all three polymer flocculants achieve what would be termed as 'acceptable' dewaterability. However, as can be seen in the bottom panels of Figure 24

when considering the TSS of the filtrate the 440 LH and 4516 polymer flocculant achieve approximately 0.2 g/L lower TSS than the C-6267 polymer flocculant. At the 2 kgP/TTS constant polymer concentration there is clearer separation between the dewatering performances of the three polymer flocculants in terms of both the CST values and the filtrate TSS. At this dosage it is clear that the 4516 polymer flocculant outperforms the others. Interestingly the C-6267 polymer flocculant undergoes the largest and steepest improvement in dewaterability over the ten days of RT; a decrease in CST values of over 70 seconds was observed. Whereas the 440 LH and 4516 polymer flocculants improve over the ten day, but much more slowly and do not decrease as much overall. Similar trends in improved in flocculation performance are observed for each of the polymers in the TSS results in the bottom panels of Figure 24. As was mentioned before there was a good correlation between the TSS measured in the filtrate and the CST value measured from the flocculated waste digestate; this correlation can be found in Figure A.3 in the Appendix and helps to strengthen the flocculation performance analysis as the tests were performed on two different samples but under the same conditions with mirrored performance. Due to the minimal resolution between the CST values at the 4 kgP/TTS constant polymer concentration it would likely be more beneficial to operate at a 3 kgP/TTS dosage as the second constant polymer concentration for future experiments. Hopefully, this would allow for confirmation that polymer flocculants capable of nearly achieving ‘acceptable’ dewaterability at the 2 kgP/TTS constant polymer concentration could operate successfully at only a slight increase in polymer concentration. Additionally, since the overall polymer concentration is reduced, five days of RT could

be removed from the test cycle and still attain a 3 kgP/TTS constant polymer concentration in the digester reducing the total test cycle to 35 days from 40.

Beyond achieving the best dewatering performance the 4516 polymer flocculant also had other operational improvements such as the smallest range of SRTs at 26.7-29.9 days; see Figure 14 for a day-by-day analysis of all RT digesters. This is a result of another improvement in flocculation performance as this indicates the separation efficiency was improved via the decreased TSS of the filtrate even at the lowest flocculant dosages.

It was predicted that the molecular weight of the polymer may have an effect of the dewatering performance during recuperative thickening. However, this was not observed as the 440 LH polymer flocculant performed indistinguishably at the 4 kgP/TTS constant polymer concentration and even outperformed the C-6267 flocculant at the 2 kgP/TTS dose. This observation is consistent with previous work we performed where the manufacturers specified properties could not be used to predict future dewatering performance [Cobbledick et al., 2014; LaRue et al., 2016]. It becomes beneficial to be able to operate multiple parallel RT digesters to compare the dewatering performance of various polymer flocculants when the dewaterability cannot be predicted prior to RT.

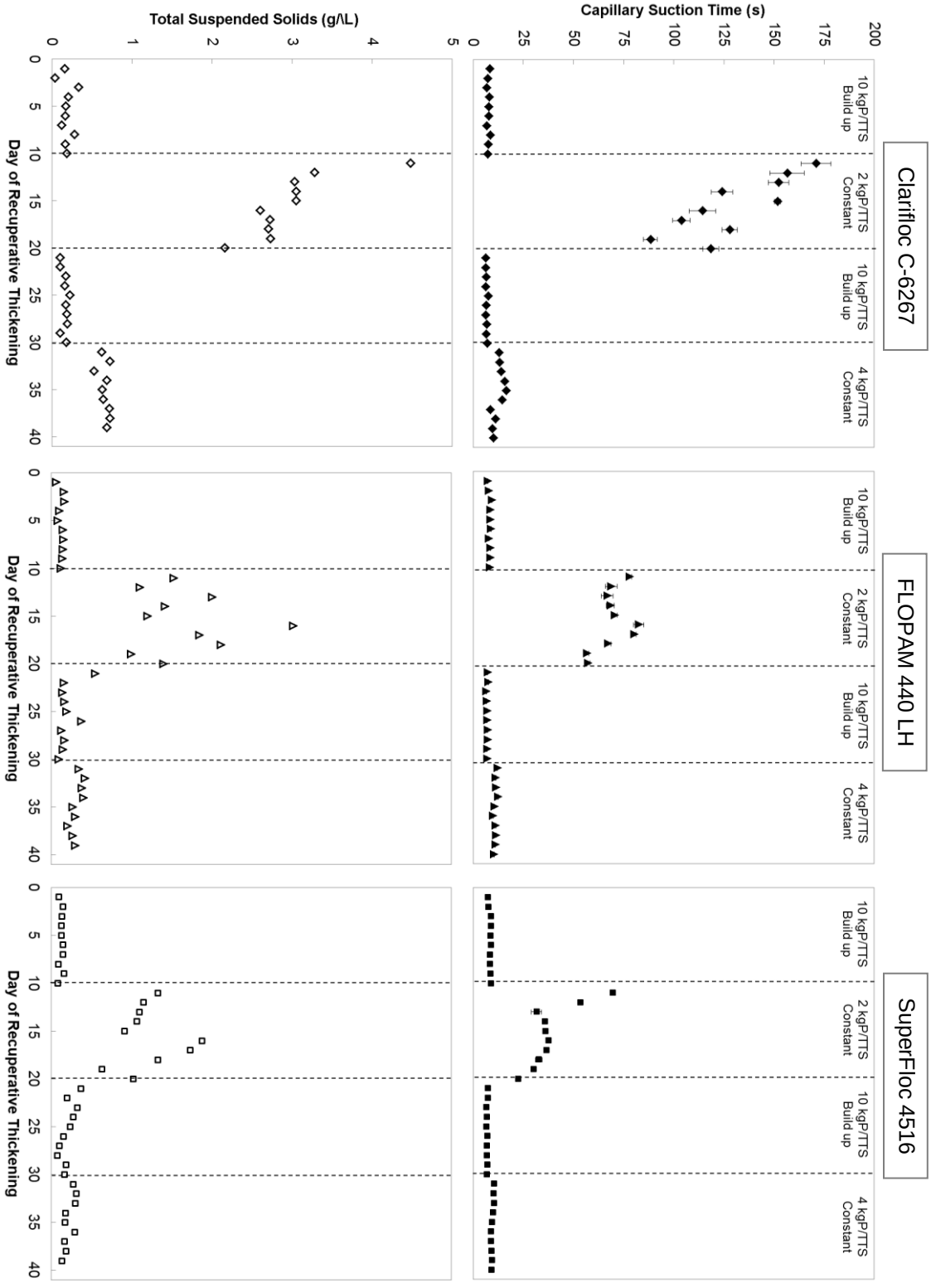

Clarifloc C-6267
FLOPAM 440 LH
SuperFloc 4516
Capillary Suction Time (s)
Total Suspended Solids (g/L)
Day of Recuperative Thickening
10 kgP/TTS Build up
2 kgP/TTS Constant
10 kgP/TTS Build up
4 kgP/TTS Constant

Fig. 24 – A comparison of the dewatering performance after flocculation during two different 40-day test cycles for all polymer flocculants used for RT (▲,Δ – 440 LH, ■,□ – 4516, ♦,◊ - C-6267) digesters. The top three panels depict the dewatering performance as measured by the capillary suction time. Each sample was analyzed in triplicate and thus each data point is the average and the error bars are the standard deviation. The bottom three panels depict the dewatering performance based off of the TSS of the filtrate collected during the daily RT`. For the bottom panels the symbol shape was used to intentionally match that for the 'Flocculated RT' results in top panels because the flocculated CST dewatering performance is well correlated to the concentration of suspended solids in the filtrate as shown in Fig. A.3 in the Appendix. The vertical dashed lines in both panels are shown to identify the different stages of the polymer flocculant dosing cycle as shown in Fig. 18 and the top lines are directly overlapped with the corresponding bottom lines for continuity between figures.

4.1.2 End-stage dewatering performance

In order to further evalute the effect of the the two new polymer flocculants used for RT on the end-stage dewatering process, another series of experiments were conducted for both the 2 and 4 kgP/TTS constant polymer concentrations. For the 4516 and 440 LH polymer flocculants, five different dosages were evaluated in trplicate anaylsis with the average values and standard deviation error shown in Figure 25; the figure also shows the six different dosages for the C-6267 polymer flocculant found previously in Figure 20 as the average values and standard deviation error. At the end-stage dewatering dose of 4 kgP/TTS the CST values for the 4516 and 440 LH RT digestates are all less than 25 seconds as was observed previously with the C-6267 polymer flocculant. CST values began to increase rapidly at dosages lower than 4 kgP/TTS with the C-6267 polymer flocculant; however this is not observed with the 4516 polymer flocculant which even at an end-stage dewatering dose of 2 kgP/TTS did not top CST values of 50 seconds. The end-stage dewatering curve for the 4516 polymer flocculant could be described as 'robust' as experiences no sudden decrease in performance despits significant reductions in polymer flocculant treatment; 1 kg of

polymer per tonne of solids is a fairly significant mass when considering the number of tonnes of solids processed in end-stage dewatering. The actual working dose for an end-stage dewatering unit is typically higher than the lowest possible dose required to achieve the dewatering performance criteria. This is because the digestate is a variable product subject to periodic changes in TS content and other factors which may hinder dewatering performance if sufficient polymer is not applied. A robust end-stage dewatering curve is beneficial as it allows for the actual working flocculant dose to be reduced. For example, the 4516 polymer flocculant can operate within the dewatering performance criteria at a dose as low as 4 kgP/TTS, but at a dose of only 2 kgP/TTS it achieves CST values as low as 34 seconds. Therefore the actual working dose required for end-stage dewatering of RT digestate with the 4516 polymer flocculant is likely very close to the 4 kgP/TTS dose; whereas RT digestate treated with the 440 LH and C-6267 polymer flocculants would likely require actual working dosages closer to 5 or 6 kgP/TTS as the CST values increase more steeply at doages lower than 4 kgP/TTS.

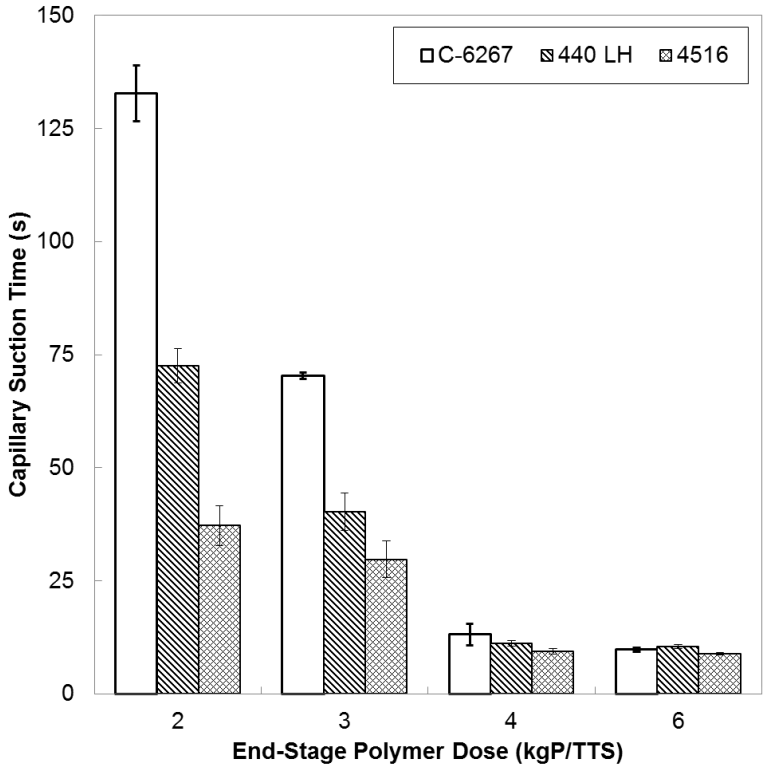


Fig. 25 – A comparison of capillary suction time results for various polymer flocculants used for RT digestate from the constant polymer concentration level of 4 kgP/TTS after they were again dosed with the same respective polymer flocculant used for RT (empty - C-6267, diagonal – 440 LH, hatched - 4516) to evaluate the 'end-stage' dewatering performance. Error bars represent one standard deviation of each sample set.

4.2 Biogas Production and Quality

4.2.1 Various RT polymers' effect on biogas yield

Biogas production in both RT digesters was monitored during the second 40-day cycle also using Ritter type Mili-Gascounters. The daily biogas yield was calculated using the gas production values and the mass of VS fed with the results shown in Figure 26. The same, regular pattern to the biogas yield was also observed in these RT digesters due to the feed schedule involving five days of feed followed by two days of starvation. Since the feed composition is variable it is not possible to directly compare the daily biogas yields from the second 40-day cycle to the first. However the low value of 0.2 and the high values less than 0.8 L/gVS are consistent from the first 40-day cycle to the second. As shown in Figure A.2 in the Appendix, both RT digesters in the second 40-day cycle achieve far greater biogas productivity values than the initial control with low values of approximately 0.7 and high values of 3.5 litres of biogas produced per day per volume of digester (L/L*day) in the second cycle RT digesters and high values of only 0.8 L/L*day for the control. There did not appear to be any adverse effects on biogas yield due to the other two polymers tested. As shown in Table 2 in Section 2.6, the SCOD values for the both RT digesters are low and comparable to those found in the control digester which indicates that these processes were also stable at the higher OLR.

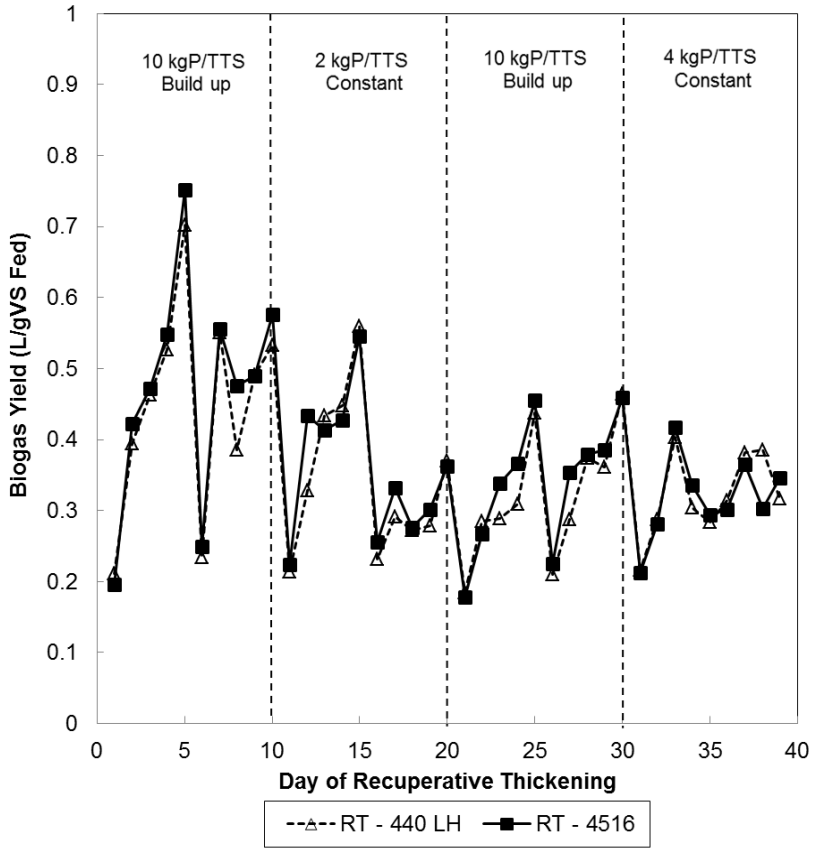


Fig. 26 – Biogas yield during the second 40-day test cycle for both the RT – 440 LH (Δ) and RT – 4516 (■) digesters. The vertical dashed lines are shown to identify the different stages of the polymer flocculant dosing cycle as shown in Fig. 18.

4.2.2 Recycled polymers effect on biogas quality

Biogas quality was monitored through the collection of biogas samples on a regular basis throughout the second 40-day cycle. Samples were analyzed once per week for the volume fractions of methane and carbon dioxide. As shown in Figure 27, the biogas composition for both RT digesters in the second cycle was very similar in composition throughout the experiment. The biogas composition of the RT digesters cannot be directly compared on a day-by-day basis to the first cycle digesters as the feed was variable across the two cycles. However, the average methane composition over the entire second cycle was comparable to the first 30 RT days of the first cycle with 67.9%

and 68.9% methane found in the 440 LH and 4516 RT digesters respectively. While there were four instances of high methane composition in the last 10 RT days in the second cycle, overall the methane composition did not spike as consistently as the last 10 RT days of the first cycle. There did not appear to be any decrease in biogas quality due to the use of these alternative polymers as all of the samples tested had over 60% methane which is consistent with typical mesophilic AD [Appels et al., 2008] and the same improved VS destruction was observed.

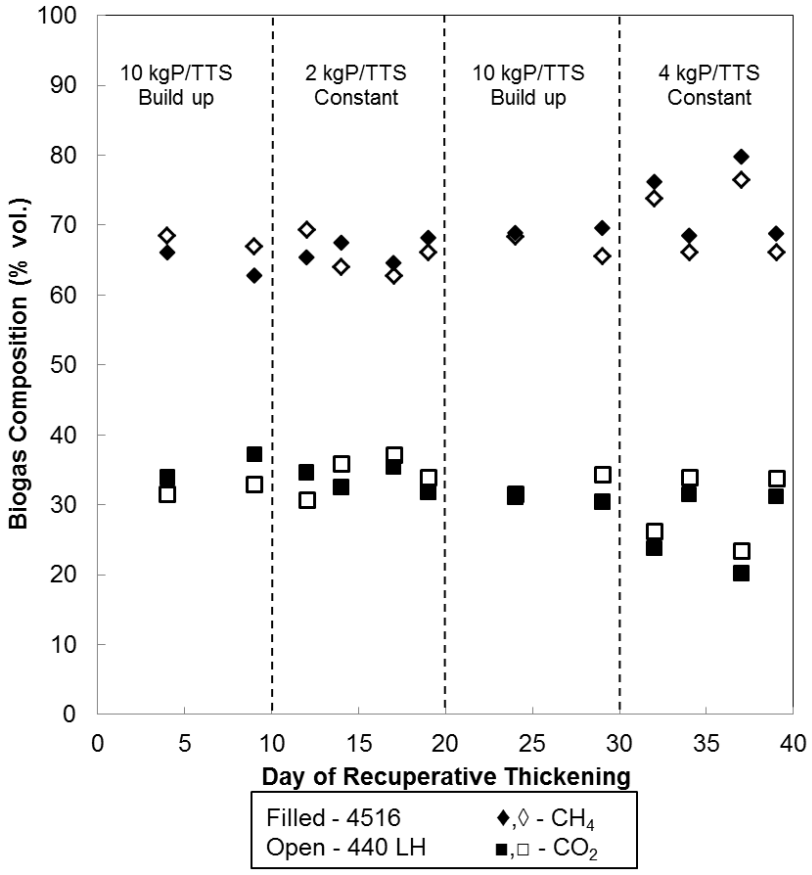


Fig. 27 – Biogas composition during the second 40-day test cycle for two RT digesters with different polymer flocculants, the 440 LH (open symbols) 4516 (filled symbols). The methane and carbon dioxide values are represented by diamonds (♦,◊) and squares (■,□) respectively. The vertical dashed lines are shown to identify the different stages of the polymer flocculant dosing cycle as shown in Fig. 18.

- Chapter 5 -
Conclusions and Recommendations

5.1 Conclusions

Energy production from waste products is just one of several ways our current society can reduce its carbon footprint. The ability for a municipally owned asset such as a WWTF to become a net producer of energy as opposed to a large consumer of energy is both economically and environmentally beneficial. While current CAD may not provide the necessary biogas to produce energy at a high enough rate to achieve net neutrality or greater, the addition of high strength organics can help to boost biogas production and reduce solid waste disposal in the process. The additional OLR of the digesters can cause stress from over-feeding; high performance AD can be used to overcome this issue and boost biogas production while maintaining or increasing the digester capacity for increased OLRs. RT is a promising high performance AD technique which utilizes a recycle stream thickened by polymer flocculant assisted, mechanical dewatering. This dewatering process increases operating costs through the consumption of polymer flocculant; optimization of the polymer flocculant during RT will help to improve the economic feasibility of the process. To date, mainly plant scale studies have been performed on RT and the few lab-scale studies have not considered the influence of recycled polymer flocculants on the overall dewaterability for the digestate. We have developed a lab-scale RT setup and 'accelerated' mode of operation that is ideal for studying the dewatering performance across a variety of dosing conditions. This

technique also lends itself to being able to accommodate several digesters in parallel to screen various polymers for the same sludge source or for various sludge sources.

The recycling of the polymer flocculant back into the RT digester resulted in a significant improvement in dewatering performance compared to the control digester. At the highest steady polymer concentration tested, the CST values for flocculated samples for the RT digester were over 6 times lower than the corresponding values for the control digester. The filtrate quality results, as measured by TSS, were in good agreement with the measured CST values. The 4516 polymer produced by Kemira had the best overall performance with the lowest CST values by far at the 2 kgP/TTS constant polymer concentration and slightly lower values at the 4 kgP/TTS constant polymer concentration. The 2 kgP/TTS constant polymer concentration appears to be an ideal condition to evaluate the dewatering performance of various polymer flocculants as it gives a wide range of CST values from as high as 170 seconds to as low as 22 seconds. When considering the end-stage dewatering of the RT digestate the 4516 flocculant had the most 'robust' end-stage dewatering performance of all three polymers tested. It can be concluded that there exists an opportunity to decrease the overall consumption of polymer flocculants through judicious selection of the dose of polymer flocculant that is used both for the thickening and end-stage dewatering processes in RT digesters. However, there were no conclusions that could be made in regards to predicting dewaterability outcomes based off of the prior knowledge of the polymer flocculants specific properties such as charge density or molecular weight. This only furthers the need for a lab-scale, parallel testing format to evaluate the dewatering performance of various polymer flocculants as

reducing their overall consumption will ultimately lead to improved feasibility due to lowered operating costs.

In regards to the biogas properties measured during the study, there were no significant changes in range of the biogas methane composition for any of the RT digesters and the control digester. Also, the RT digester in first cycle had the same biogas yield as the control digester and therefore had a significant improvement in overall biogas productivity; this enhancement in biogas productivity was also observed in both RT digesters during the second 40-day cycle. This is most likely due to an improvement in the fed VS destruction from recycled solids in the RT technique leading to an improved SRT. It also indicates that the short term oxygen exposure during the thickening process had no discernable inhibitory effect on methanogenic activity and also the recycled polymer does not appear to be toxic to the anaerobic microorganisms. Finally, from the arguments presented above it can be concluded that this lab-scale RT technique is an effective tool to study RT processes and thus is ideal for addressing the current knowledge gaps in the field.

5.2 Recommendations for future designs and work

One of the disappointing aspects of the lab-scale setup was the mixing apparatus. During the initial design phase the Quad-mag magnetic stirrer seemed ideal as it would be able to stir multiple elements without having to worry about maintain the air tightness of the digester. However, the Quad-mag stirrer had several flaws in the end such as its poor quality motor. We had to replace the entire unit once due to motor failure and by the end of the second 40-day cycle the motor again had begun to wear down and was nearing

failure. This motor in my opinion may be suitable for occasional lab use, but is not suitable for constant operation or for extended periods of time. Another flaw was this motor is incapable of applying large amounts of low end torque; when mixing at low speeds, in a high solids environment, and with a relatively large magnet the motor was required to provide a fairly large amount of torque, but this motor was often unable to stop and start easily and at times struggled to overcome the large increase in viscosity which occurred with polymer addition during recuperative thickening. In my opinion, a better solution would have been to design an overhead stainless steel paddle mixer installed through an air-tight bearing through the top lid of the digester. Several of these overhead mixers could have been linked on the same timing belt to accommodate a large setup with more digesters in parallel. The overhead mixers should be driven by a motor capable of operating a low RPM's (60-200) and be large enough to supply sufficient torque even at low RPM's; a medium sized electric geared motor or a brushless DC electric motor would likely be adequate. The magnetic stir bars themselves also proved to be an issue themselves because of the grit that collects at the bottom of digesters. The grit wore the Teflon coating off of the stir bars which likely led to increased resistance to mixing and required replacement several times over the course of the experiments. The magnetic stir bars combined with the grit also wore the bottom of the digesters causing an uneven mixing surface which likely increased the chatter of the stir bars and increased the frequency of spin out (where the magnet jumps out of the magnetic field and loses its movement pattern). In order to combat this issue, a second pair of identical digesters was prepared for the second cycle of testing.

Another design flaw which was addressed before the testing began was the temperature control in and around the digesters. The digesters were originally designed to occupy an incubator however several problems arose from this setup. The mixer produced its own waste heat which was not being expelled originally causing the minimum temperature without the incubator even on to reach as high as 45 ^{o}C. A small hole was cut in the incubator door to insert a fan to expel waste heat, with this setup the digesters were able to approach mesophilic temperatures; however, the heat distribution in the incubator was inconsistent even with the fan resulting in hotspots which created a temperature discrepancy of 2-3 ^{o}C between the digesters. To overcome this issue an immersion heater and water bath design was adopted which resulted in consistent and controllable temperatures with little to no discrepancy in temperatures between the digesters.

As was concluded, this lab-scale RT technique is an effective tool to study RT processes and thus is ideal for addressing the current knowledge gaps in the field. One obvious area would be to continue to study the effect of various polymer flocculants the dewaterability. However the feed sludge source could be changed depending on the project location or taken from a potential future plant site as changing the feed may affect the polymer flocculant's performance. Moreover, since there are a seemingly endless number of sources available for co-digestion, this parallel lab-scale digester setup would be ideal for testing several co-digestion materials at one time. Conversely, the ratio of a single co-digestion waste to feed sludge could be manipulated in order to obtain the optimal biogas production and quality while also monitoring the change in dewatering performance. In regards to co-digestion, there are concerns over the increased presence of

H_2S in the biogas during co-digestion with high strength organics. Improved biogas monitoring for H_2S would be considered an asset when designing biogas upgrading scrubbers on the full scale. The digestate viscosity during RT is another concern in terms of increased operating cost of RT but was not considered in this experiment; the cost of mixing on the plant scale is dependent on the power required by the motors which are directly proportional to the viscosity of the digestate. In future works the effect of various polymer flocculants on the RT digestate viscosity could be used in a cost-benefit analysis regarding the overall RT operation as a whole. Additionally, little is known about the true microbial kinetics experienced during RT. The lab-scale setup provides a highly customizable and controllable environment to perform studies determining kinetic parameters such as the specific growth rate and substrate utilization rate. Additionally, the bacterial diversity could be monitored under various operating conditions during RT such as with or without co-digestion or at longer SRTs in order to determine if there is a variation in bacterial content from the common bacterial flora found in CAD.

Appendix

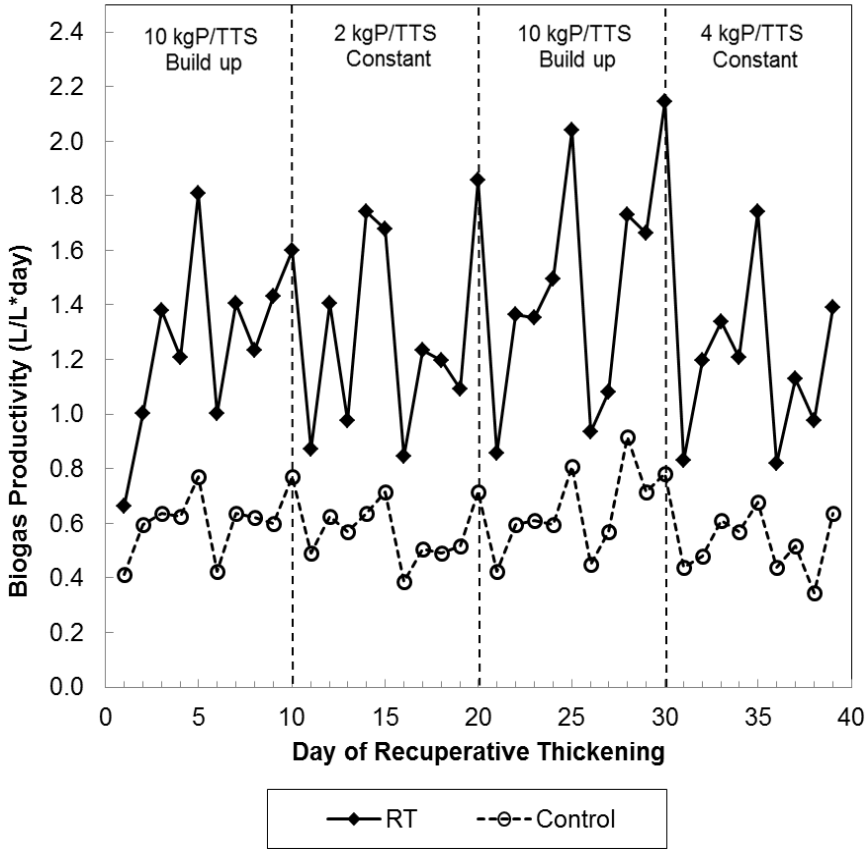


Fig. A.1 – Biogas productivity during the first 40-day test cycle for the RT (♦) and control (○) digesters. The vertical dashed lines are shown to identify the different stages of the polymer flocculant dosing cycle as shown in Fig. 18.

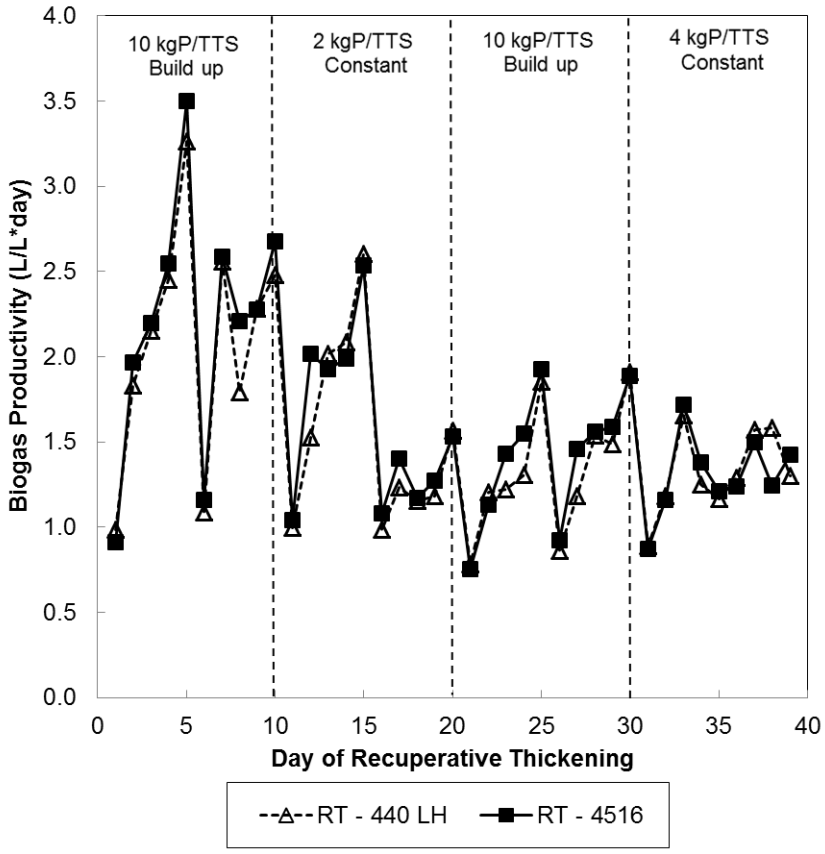


Fig. A.2 – Biogas productivity during the second 40-day test cycle for the RT - 440 LH (Δ) and RT - 4516 (■) digesters. The vertical dashed lines are shown to identify the different stages of the polymer flocculant dosing cycle as shown in Fig. 18.

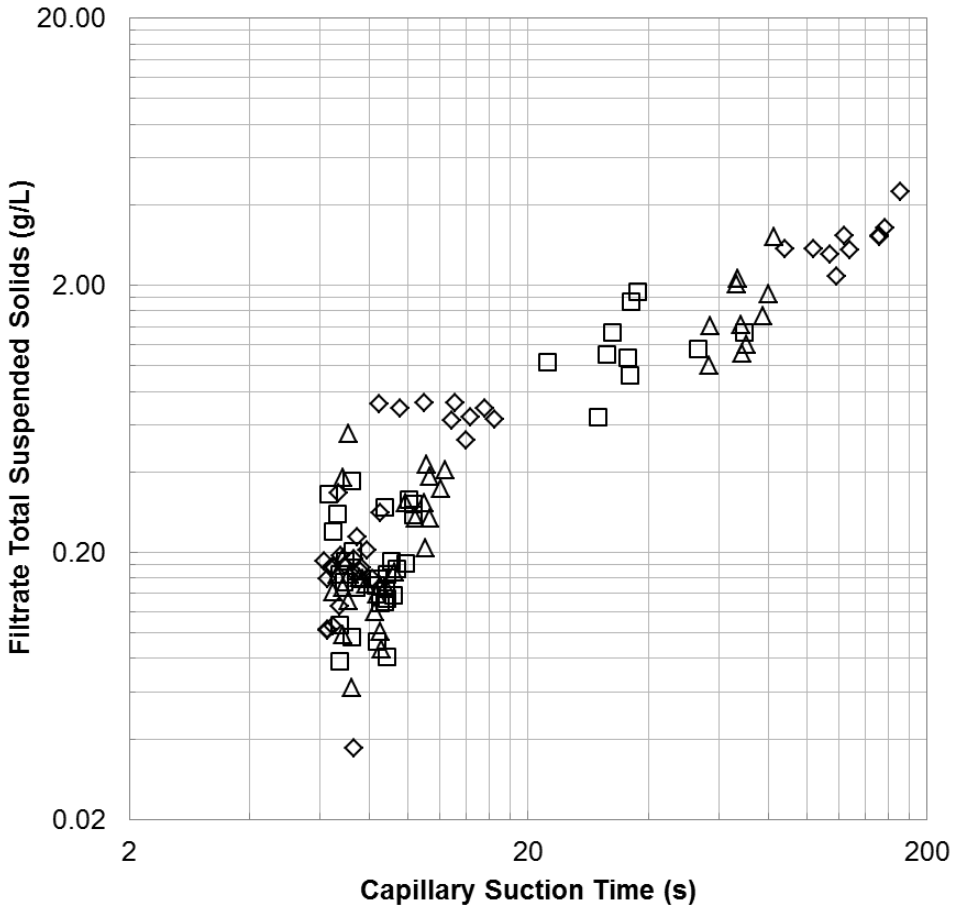


Fig. A.3 – A comparison of the filtrate TSS to the flocculated digestate average CST for the both 40-day cycles of operation of all RT digester as shown in Figure 24. The C-6267, 440 LH, and 4516 RT results are denoted by the ◊, Δ, and □ respectively.

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