An Evaluation of Alternatives for Enhancing Anaerobic Digestion of Waste Activated Sludge

by

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Author's Declaration

I hereby declare that I am the sole author of this thesis. This is a true copy of the thesis, including any required final revisions, as accepted by my examiners. I understand that my thesis may be made electronically available to the public.

Abstract

Waste activated sludge (WAS) is one of the largest by-products of biological wastewater treatment. Anaerobic digestion of WAS is beneficial for several reasons. In an ever increasingly energy conscientious world the production of renewable energy resources is becoming more important, and thus the production of methane has been seen as a valuable product. To achieve efficient conversion of organic matter to methane, the biomass in the digester must be provided optimal operating conditions, as well as adequate retention times, that will allow for substrate metabolism and prevent bacteria washout. Two approaches have been taken in this research to achieve improved biodegradation. Initially microwave pretreatment was employed to improve the biodegradability of the sludge, then the addition of a submerged hollow fibre membrane separation unit was used to allow for a longer SRT while maintaining the hydraulic residence time (HRT).

The impact of microwave pretreatment on WAS characteristics was assessed for both the low temperature operations and the high temperature operations. An increase due to pretreatment on the filtered to total COD ratio when comparing the feed to the microwaved feed was established to be 200 % for low temperature operations and 254 % for high temperature operations.

For the low temperature operations, COD_T destruction, VS destruction, and organic nitrogen destruction were all higher for the test digester than the control digester indicating that the microwaving of the WAS increased the biodegradation in the anaerobic digester. For the high temperature operation, COD_T destruction and organic nitrogen destruction were improved with microwave application, however VS destruction did not support this. The measured biogas data indicated that microwaving did influence the volume of biogas produced during anaerobic digestion of WAS for both the low and high temperature operations, and hence the VS destruction data for the high temperature operations was determined to be incorrect.

For the membrane operations both the COD_T and the VS destruction calculations indicated that at the same SRT the test digester was capable of more biodegradation than the control digester. The control digester organic nitrogen reduction was calculated to be higher than for the test digester, suggesting that the control digester removed more organic nitrogen than the test digester, however, these results were likely due to the lower HRT of the test digester compared to those of the control digester.

A greater volume of biogas was produced by the test digester than the control digester; however, the composition of the gas from both digesters was similar, although the percentage of methane produced by the test digester was higher than that produced by the control digester. The higher destruction by the test digester indicated that the presence of the membrane unit and the decoupling of the HRT and SRT improved the biodegradation capability of the digesters.

The results of the membrane performance study indicated that for a hollow fibre anaerobic membrane bioreactor, stable operations could be achieved with a total solids concentration of 2.01 %+/-0.34, an HRT of 15 days and an SRT of 30 days. With a constant flux of 14 L/m^2-h +/-0.68 the average TMP was 0.079 kPa/min+/-0.08. No cleaning was required to achieve this, however the operations consisted of 20 minutes of permeation followed by 5 hours and 40 minutes of relaxation. The critical flux was determined to be in the range of 18 to 22 L/m^2-h.

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Definitions

AnMBR	Anaerobic Membrane
COD	Chemical Oxygen Demand
COD_T	Total Chemical Oxygen Demand
COD_F	Filtered Chemical Oxygen Demand
sCOD	Soluble Chemical Oxygen Demand
CSTR	Continuous Stirred Tank Reactor
EPS	Extracellular Polymeric Substances
HRT	Hydraulic Residence Time
MF	Microfiltration
MLSS	Mixed Liquor Suspended Solids
MW	Microwave (or microwaved)
OLR	Organic Loading Rate
ON_T	Total Organic Nitrogen
PS	Primary Sludge
SRT	Solids Residence Time
TS	Total Solids
TSS	Total Suspended Solids
TWAS	Thickened Waste Activated Sludge
UASB	Upflow Anaerobic Sludge Blanket
UF	Ultrafiltration
VS	Volatile Solids
VSS	Volatile Suspended Solids
WAS	Waste Activated Sludge
WW	Wastewater

Chapter 1
Introduction

1.1 Objectives

Waste activated sludge (WAS) is one of the largest by-products of biological wastewater treatment. Anaerobic digestion of WAS is beneficial for several reasons. It reduces the organic content of WAS which results in a more benign end product, it improves dewaterability which allows for less energy intensive methods of water-solid separation, it is capable of the destruction of most pathogens which protects the health of those exposed to the end result, and reduces the volume of sludge to be eliminated (Taricska et al. 2006). In an ever increasingly energy conscientious world the production of renewable energy resources is becoming more important, and thus the production of methane has been seen as a valuable product.

There are some limitations to the anaerobic digestion of WAS. To achieve efficient conversion of organic matter to methane, the biomass in the digester must be provided optimal operating conditions, as well as adequate retention times, that will allow for substrate metabolism and prevent bacteria washout (Parkin and Owen 1986). Subsequent to urban population growth an increase in the production of wastewater and ultimately increased volumes of WAS is incurred. Increasing WAS production requires treatment plants to increase their treatment capacity yet still provide adequate solids retention time (SRT) and biodegradation. An adequate SRT is based on the ability of the anaerobic digestion process to achieve hydrolysis, which is the rate limiting step in converting complex organics into methane (Tchobanoglous and Burton 2003). Two approaches have been taken in this research to achieve this. Initially microwave pretreatment was employed to improve the biodegradability of the sludge and then the addition of a submerged hollow fibre membrane separation unit was used to allow for a longer SRT while increasing the hydraulic residence time (HRT).

For the microwave pretreatment the influence of the treatment was assessed based on the characteristics of the WAS. For both the pretreatment and the membrane operations the digester performance was assessed, based on degradation of total chemical oxygen demand (COD_T) and VSS and biogas production. Finally, the hollow fibre membrane was assessed based on its performance over the course of the research.

1.2 Scope

This project investigated both microwave pretreatment and membrane operations for a pilot scale anaerobic digestion system. The scope of this thesis included:

- Assessment of COD solubilization at low and high microwave temperatures
- Assessment of biodegradation with respect to COD_T, VSS, and organic nitrogen
- A comparison of Measured and Theoretical Biogas Production
- Characterization of the Biogas
- Assessment of Membrane Performance
    - Flux and Transmembrane Pressure
    - Fouling

Chapter 2
Background

2.1 Anaerobic Digestion

Anaerobic digestion is a treatment method that has been shown to effectively reduce the organic content in sludge, improve the dewaterability of sludge, achieve high pathogen reductions, reduce sludge volume, and consequently, produce methane, a valuable product(WEF and ASCE 2009). For this study the primary aspect considered was the reduction of organic content and the associated biogas production.

2.1.1 Purpose

Anaerobic digestion is a biological treatment process which degrades biological material typically between the temperature of 35 ºC and 55 ºC in an environment void of oxygen (Tchobanoglous and Burton 2003). Anaerobic digesters are commonly used to treat the sludge that has been produced through the biological wastewater treatment process.

The quantity and quality of the sludge at the end of the wastewater treatment process is a concern for operators of the plant. When sludge is removed from the plant for disposal, hauling and disposal are typically charged on a volume or mass basis. To reduce these associated costs it is in the best interest of the treatment plant to reduce the water content, thereby lowering both the volume and the weight of the sludge to be disposed of. To improve the quality of the sludge there should be a reduction in the number of pathogens and stabilization to eliminate odours and putrefaction and decomposition in an uncontrolled environment. Anaerobic digestion is a common method to achieve the quality improvements on the sludge.

More recently an added benefit of anaerobic digestion has been utilized. The methane produced during the biodegradation of biological matter has been captured and used as an energy source. With improved digestion there is an increase in the quantity of biogas produced.

2.1.2 Biodegradation and Organic Stabilization

In order to evaluate the performance of an anaerobic digester the type of material being digested must be considered. A description of waste activated sludge and the characteristics used to describe the biodegradation and stabilization of this type of sludge are presented in the following sections. The anaerobic digestion process is also described as the method to provide both stabilization and biodegradation.

2.1.2.1 Waste Activated Sludge

Secondary treatment converts soluble wastes into microorganisms, more commonly referred to as a conversion of substrates into biomass. Waste activated sludge (WAS) is composed of the secondary solids that are generated in the biological treatment process. Some of cellular material cannot be degraded due to the recalcitrant physical and chemical properties of that material (Baier and Schmidheiny 1997). Also included in WAS are particulates that have not been removed during primary sedimentation, which then become incorporated with the biomass in the secondary sludge.

The quality and quantity of the WAS produced by a treatment plant are dictated by the upstream operations. The efficiency of the primary treatment, the ratio of total suspended solids to biochemical oxygen demand, the influent soluble chemical oxygen demand along with the secondary treatment design parameters, the activated sludge treatment solids retention time (SRT) and the temperature of the secondary treatment influence the WAS. For example, a higher SRT in the secondary treatment stage results in more endogenous decay, and larger fraction of dead and disrupted cells in the WAS (WEF and ASCE 2009).

WAS and other biological sludges tend to be more difficult to thicken and dewater, compared to the sludge that results from primary treatment. Typical characteristics of secondary sludge are shown in Table 1.

Table 1 Typical Waste Activated Sludge Characteristics

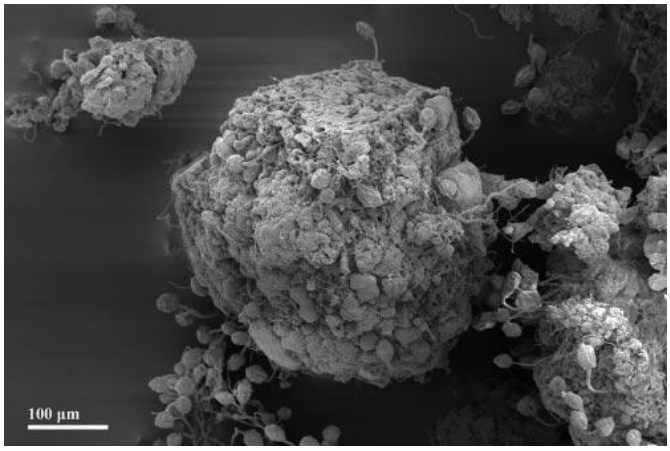
Characteristic	Range
Total Solids	0.4 - 1.2 %
Total Volatile Solids	60 – 85 % of TS
Grease	5 – 12 % of TS
Phosphorus	1.5 – 3.0 % of TS
Protein	32 – 41 % of TS
Nitrogen	2.4 – 7.0 % of TS
pH	6.5 – 8.0

(WEF and ASCE 2009)

In addition to the typical characteristics presented in Table 1, the ratio of volatile suspended solids to total suspended solids is typically between 0.7 and 0.8, which is indicative of the large percentage of biological material in WAS (WEF and ASCE 2009).

The majority of bacterial life existing in natural systems is found in surface-bound communities called biofilms (Xavier and Foster 2007). Biofilms are systems that allow cells to share secreted molecules, including enzymes, and extracellular polymers, and physically appear as a slime or matrix around the cells. The extracellular polymeric substances (EPS) are present in many biofilms as almost all bacteria are able to produce them, including those present in WAS(Geesey 1982). EPS is composed of proteins, polysaccharides, lipids, and nucleic acids in a composition that depends on the bacteria and environment (Beech et al. 2005), and originates from both the metabolism and cell autolysis in the activated sludge process and also from the raw wastewater (Eskicioglu et al. 2007c).

An analysis of the contents of an aerobic sequencing batch reactor, in a study completed by Weber et al.(2008), indicated that the formation of granules that were comprised of core and fringe zones, with a dense mixture of bacteria and EPS in the core. A photograph of the microscopic analysis of the SBR contents, Figure 1, shows a mature granule with a spherical shape, containing bacteria and EPS.



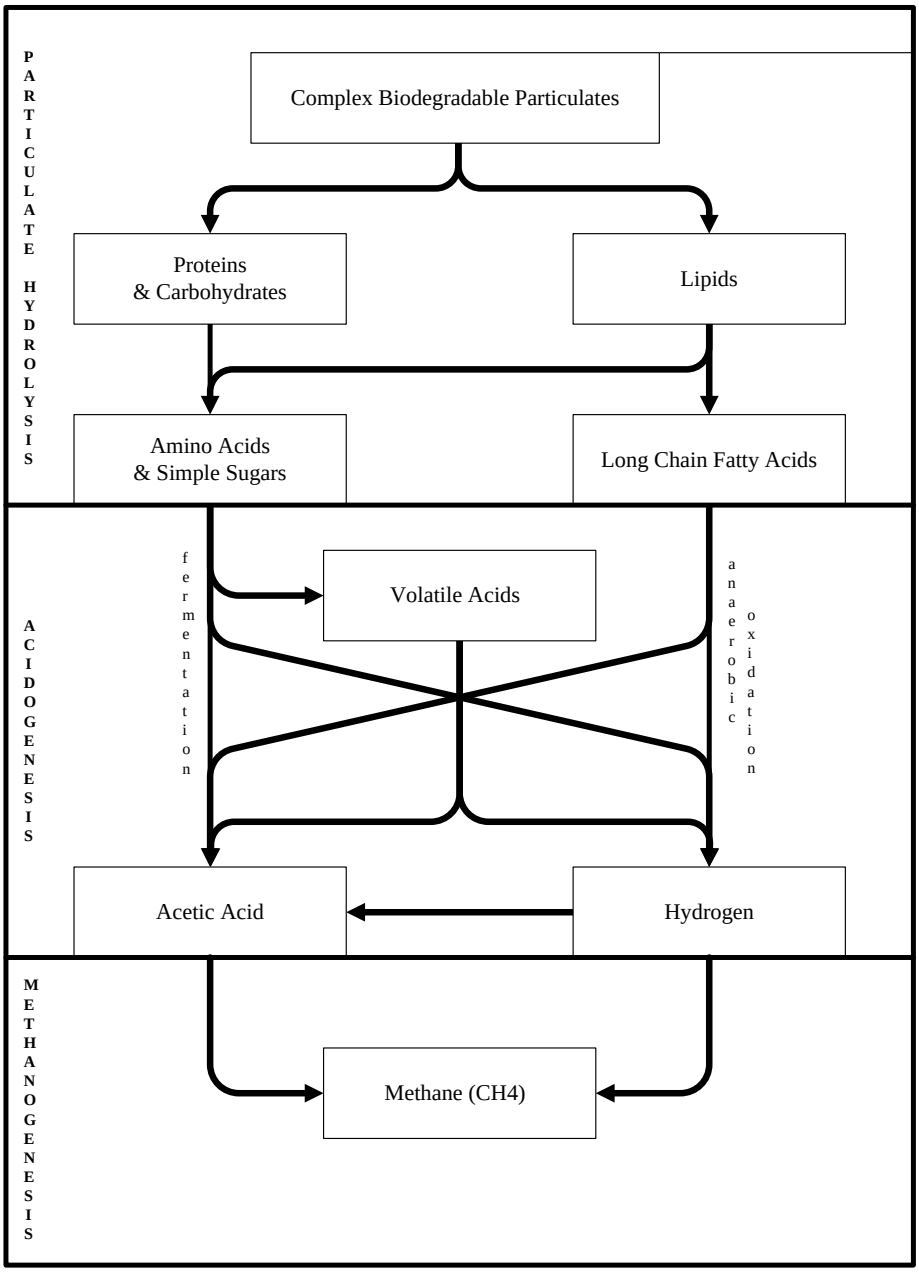
(Weber et al. 2008)

Figure 1 Mature granule from an SBR with synthetic wastewater

Although the biofilms produced in different processes are different, due to the nature of their environment and feed characteristics, the granules shown in Figure 1 are an example of an activated sludge biofilm. The aerobic bacteria in the secondary treatment embedded within the EPS share the polymers as a resource that provides a maintained structure, protection against dehydration, ultraviolet radiation, and predator grazing, and the facilitation of extracellular enzymatic activity (Xavier and Foster 2007). It is this biological material that is removed as WAS and which is often treated in anaerobic digestion, and the protection that the EPS provides could become an obstacle for further biodegradation. The concentration of EPS has been reported to be reduced from 70-90 mg-EPS/g-SS in the feed WAS to 10-20 mg-EPS/g-SS in anaerobic sludge (Bowen and Keinath 1985; Forster 1982; Morgan et al. 1991)through anaerobic treatment. The disruption of the EPS and the associated divalent cation network may allow for an enhanced rate of WAS biodegradation and the conversion of these organics into more readily biodegradable forms, resulting in shorter retention times required in the digester to achieve biodegradation (Hong et al. 2004; Park et al. 2003).

2.1.2.2 Anaerobic Digestion Process

The biodegradation that occurs within an anaerobic digester involves the breaking down of carbohydrates, lipids, and proteins that make up the biofilms in WAS and the production of a valuable product, methane. Figure 2 shows the intermediary steps between the complex biodegradable particulates found in WAS and methane.



(Grady Jr. et al. 1999)

Figure 2 Sequence of anaerobic biodegradation processes

The bacteria which are responsible for anaerobic digestion are classified as hydrolytic bacteria, fermentative acidogenic bacteria, acetogenic bacteria, and methanogens. Hydrolytic bacteria are responsible for breaking down complex molecules into soluble ones, through a process called hydrolysis, the first process shown in Figure 2. Hydrolysis is typically the rate limiting step in

anaerobic digestion of WAS, and is the primary method for changing particulate matter to soluble matter.

Once hydrolysis has been accomplished fermentative acidogenic bacteria are responsible for converting sugars, amino acids, and fatty acids into organic acids (acetic, propionic, and butyric), alcohols and ketones, carbon dioxide, and hydrogen gas. Acetogenic bacteria are then responsible for converting propionic acid, butyric acid and alcohol into acetate, hydrogen gas, and carbon dioxide.

The final step in anaerobic biodegradation is methanogenesis, which is the conversion of hydrogen and acetic acid into methane gas. Methanogens can be divided into two categories, H_2-oxidizing methanogens and aceticlastic methanogens, but the commonality between these categories is that both types of bacteria are capable of creating methane. H_2-oxidizing methanogens can create methane from hydrogen gas and carbon dioxide, while aceticlastic methanogens split acetate to produce methane and carbon dioxide (Grady Jr. et al. 1999).

Changes in pH can have a large influence on the function and growth rate of methanogens. A decline in the growth rate of methanogens can result in a reduction in the consumption of acetate and an accumulation of acids, further decreasing the pH. In order to buffer the acids that are present and generated in the anaerobic digestion process there must be an adequate amount of alkalinity. Alkalinity is present in anaerobic digesters because of the pre-existence in the feed, VFA alkalinity, and ammonium released during nitrogen decomposition (Parkin and Owen 1986).

2.1.2.3 Assessment of Biodegradation

To evaluate the ability of an anaerobic digester to accomplish the aims of this study, that is reduce organic content and produce biogas in the form of methane, two characteristics are of primary concern. Both chemical oxygen demand and volatile solids concentrations measured in the feed, digester contents, and effluent can be used to evaluate the extent of biodegradation.

2.1.2.3.1 Chemical Oxygen Demand

Chemical oxygen demand (COD) measures the oxygen equivalent of the material in a wastewater sample that is subject to oxidation and thus gives a measurement of the oxygen depletion potential. The importance of reporting the fractions of COD in a sample is particularly relevant for anaerobic sludge digestion because each component is affected differently by the nature of the digester operations, such as residence times, temperature, and substrate concentrations. Figure 3 presents the typical fractionation of COD for wastewater applications.

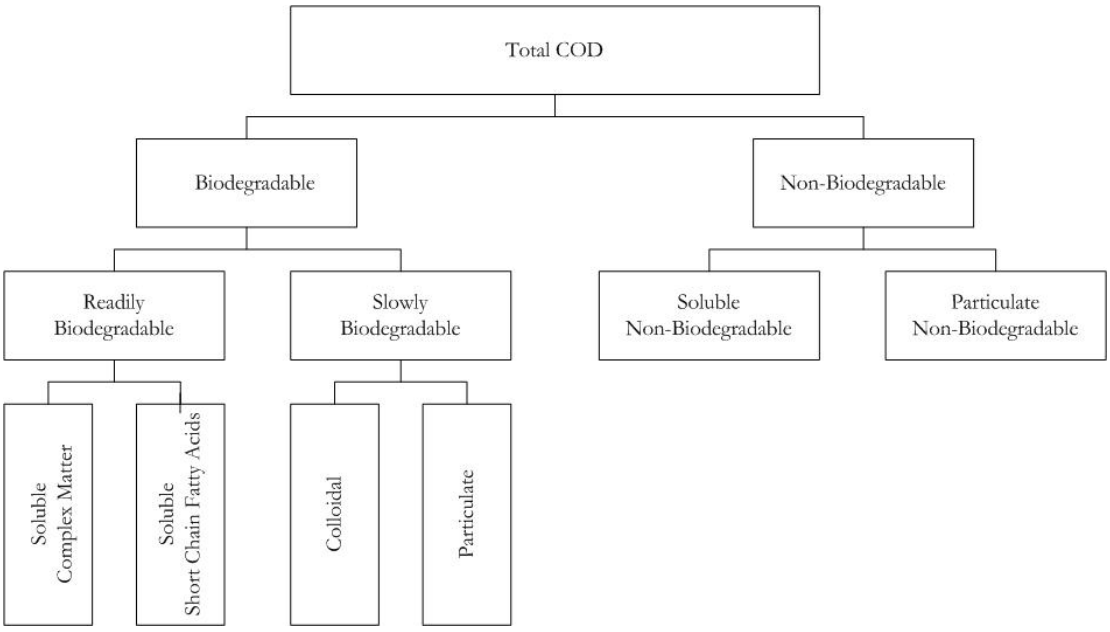


Figure 3. Chemical Oxygen Demand characteristic fractionation

Figure 3 shows the divisions of COD; in biological wastewater treatment and anaerobic sludge digestion the largest of these is biodegradable COD, due to the presence of the biological substrate and biomass. Biodegradable COD is composed of both readily biodegradable COD and slowly biodegradable COD, and contains particulate organic matter and active biomass.

Readily biodegradable COD, as the name indicates, can be quickly consumed in conventional biological treatment processes as it is comprised of low molecular weight substances and is soluble in nature. Readily biodegradable COD can be further subdivided into two categories, complex matter and short chain fatty acids.

Slowly biodegradable COD consists of materials that are more complex and thus require extra-cellular degradation prior to their utilization in biological treatment processes. Slowly biodegradable COD consists of both colloidal and particulate components. The colloidal components of the slowly biodegradable COD range in size from 0.01 µm to 1 µm (Tchobanoglous and Burton 2003), do not settle, and are considered particulate rather than soluble components. However, due to their size, colloidal material is often included in soluble COD measurements. WAS has a larger percentage of slowly biodegradable COD, which by its nature requires longer SRTs to achieve the biodegradation that a sludge with a higher percentage of readily biodegradable COD would require.

The non-biodegradable COD component is considerably smaller than the biodegradable component; however, this component is a concern in wastewater treatment plant operations since it contributes to the COD concentrations in the end products of treatment. Non-biodegradable COD consists of both a soluble component and a particulate component, and is not just a result of the influent wastewater but also biomass debris which is a result of the decay of active biomass.

The COD characterization for the pilot study was required to assess the biodegradation occurring in the anaerobic digesters to predict the volume of methane produced. Figure 4 shows the division of biological material and the relationship of that material to the fractionation of COD.

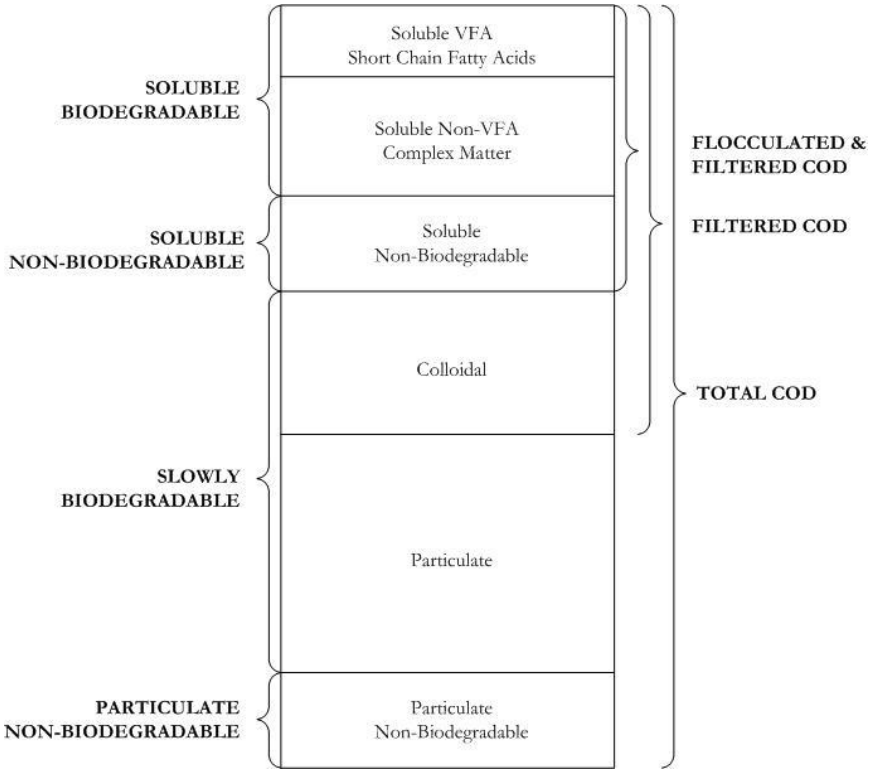


Figure 4. Chemical Oxygen Demand fractions

Figure 4 shows the biological meaning of the COD measurement. Total COD encompasses all of the biological material in the WAS, while Filtered COD, which for this study will be described as anything smaller than 1.5 µm, included both soluble and colloidal component as well as soluble biodegradable and non-biodegradable and some slowly biodegradable elements. The measurement of Filtered COD thus becomes more important when it is presented in a comparative way, and changes in the filtered component indicate an addition or removal of these elements.

2.1.2.3.2 Volatile Solids

Similarly to the measurement of COD, the concentration of total volatile solids (VS) is a standard characteristic when reporting or describing the physical nature of a sludge. The VS present in a sludge represents the sum of the volatile suspended solids and the volatile dissolved solids, and are usually established by combusting a solids sample in an oven at 500 ºC +/- 50 ºC.

The VS component of a sludge is considered to be the organic component of the sludge, and can be used to approximate the biodegradable component of the sludge. The ratio of VS to the total solids TS is an indicator of the biodegradability of a sludge.

2.1.2.3.3 Nitrogen

Total organic nitrogen (ON_T) is the difference between total Kjeldahl nitrogen (TKN) and ammonia (NH_3). The biodegradation within a digester can be estimated by analyzing the organic nitrogen destruction, through the measurement of the TKN mass loading versus the quantity of NH_3 produced in the digester. NH_3 is a product of the destruction of the proteins contained within biomass. Equation 1 is a simplified explanation of the presence of NH_3 in an anaerobic digester, showing that the biodegradation of biomass results in the creation of both NH_3 and biomass debris which also may have nitrogen content.

Equation 1 $$\boldsymbol{Biomass\ N \xrightarrow{biodegradation} NH_3 - N + biomass\ debris\ N}$$

2.1.2.3.4 Typical Performance

The typical performance for high-rate anaerobic digestion is summarized in Table 2.

Table 2 Typical High-Rate Anaerobic Process Performance

Parameter	Value
BOD_5 removal, percent	80 to 90%
COD removal, mass	1.5 x BOD_5 removed
Biogas production	0.5 m^3/kg COD removed
Methane production	0.35 m^3/kg COD removed
Biomass production	0.05-0.10 g VSS/g COD removed

(Grady Jr. et al. 1999)

The removal of biological material is typically high, as indicated by the 80 to 90% removal of BOD_5, and consequently COD. The solids production is typically low, with only 0.05-0.10 g-VSS produced for every gram of COD removed, and ultimately a reduction in VSS should be observable. The performance indicated in Table 2 is general summary for all high-rate anaerobic digestion processes and the digesters used in this study are expected to show similar performance.

2.2 Microwave Literature Review

Microwaves have been used in a variety of applications since their conception. Information regarding the history of the microwave as well as the operation and design of microwave equipment is presented in the following sections. The research regarding the effects of microwaving WAS were also considered, and the influence on solubilization and biodegradation will be discussed.

2.2.1 Microwave Background

The use of microwave energy in heating applications is often considered to be attractive because it provides for uniform and thorough heating. In addition, microwave units have instantaneous start-up, and can therefore provide rapid heating and thus energy savings (Decareau 1985).

Microwaves have been used in a variety of applications in both municipal and industrial settings. Studies of the application of microwaves have been conducted in areas such as organic decomposition, pathogen reduction in food, and the degradation of animal manure. In the 1960's microwaves were studied with respect to heating, biocidal effects, dielectric dispersion, and mutagenic effects to determine their impact on bacteria, viruses, and DNA (Oliner 1984).

However, the use of microwaves as a pretreatment method for WAS is a relatively new application of the technology. The potential for microwaves to destroy pathogens has been evaluated by Eskicioglu (2007), and although it has not been widely accepted that microwaves can reduce total coliform or fecal coliform counts in the waste from anaerobic digesters, other pretreatment methods, including thermal treatment have been found to improve total pathogen reduction (Eskicioglu et al. 2007b). These previous results would suggest that microwaves could provide a similar result.

2.2.2 Operation of Microwaves

Microwave heating is accomplished in nonconductors by dielectric heating. The heat generation is caused by polarization effects that occur at frequencies between 300 MHz and 300 GHz, which correspond to wavelengths between 1 m and 1 mm. Conversely, (Plazl et al. 1995) convection heating requires that heat be transferred from a heating element to the material. In convection heating, the speed at which a material is heated depends on the thermal conductivity, the temperature difference between the element and the sludge, and the convection currents within the sludge (Plazl et al. 1995). Microwave heating, on the other hand, heats the entire material at the same time.

Microwaves are used in many applications, including radar, navigational equipment, and communication equipment. In the United States, the Federal Communications Commission has indicated that for industrial, scientific, and medical functions the microwave ranges of 915 +/- 13 MHz and 2450 +/- 50 MHz may be used (Singh and Heldman 2009). It is within this range that both household and the industrial microwaves, including those used in this study, reside.

Microwaves are absorbed by materials resulting in the heating of those materials. There are materials that allow microwaves to pass through them and do not absorb any of the energy. The dielectric properties of materials dictate whether or not the energy is absorbed. There are two principles which govern the heating of material in a microwave, ionic polarization and dipole rotation.

Materials that contain ions, a material with a high salt content, will increase in temperature with the application of an electric field (Decareau 1985). This is due to the increased speed at which the ions move due to their inherent charge. When the ions collide with one another kinetic energy is converted to thermal energy. If a material had a higher concentration of ions there would be more ion collisions resulting in more thermal energy generated.

Dipole rotation occurs because water is a polar molecule which typically has a random orientation until an electric field is applied to it. With the application of an electric field the water molecules orient themselves according to the field. Within microwaves the polarity changes quickly and for a microwave with a frequency of 2450 MHz, the polarity changes at a rate of 2.45×10^9 cycles per

second. The rapid rotation of the molecules causes friction with the surrounding material and heat is generated (Singh and Heldman 2009).

Thus, to determine how microwaves will interact with a material being microwaved, the electrical characteristics of that material are important, and these are described by the relative dielectric constant ε' and the relative dielectric loss ε" (Singh and Heldman 2009). These values describe the electrical insulating ability of a material, and the relative dielectric constant is a measurement of a materials ability to store electrical energy, while the relative dielectic loss is the ability of that material to convert the electrical energy into heat.

The energy a microwave provides is not thermal energy, but because of the nature of the material within the microwave the electrical energy is converted to thermal energy. Equation 2 (Singh and Heldman 2009) describes this conversion.

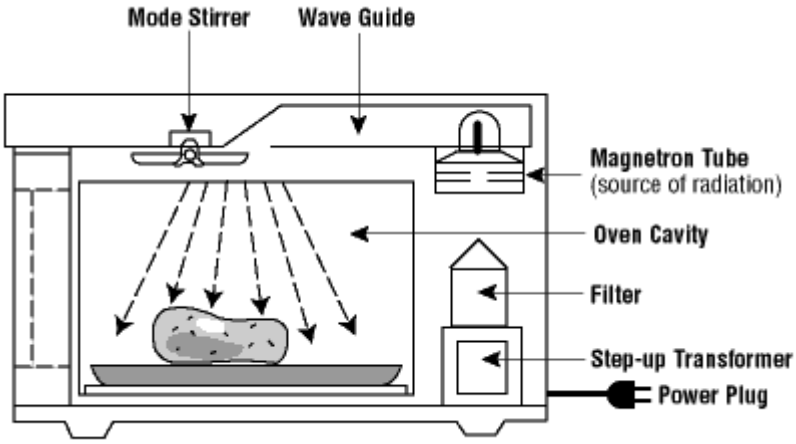
Equation 2 $$P_D = 55.61 \times 10^{-14} E^2 f' \varepsilon' tan\delta$$

In Equation 2, P_D is the power dissipation measured in W/cm^3, E is the electrical field strength measure in V/cm, *f*' is the frequency measured in Hz, and ε' is the relative dielectric constant, and tan δ is the loss tangent. The electrical field strength and the frequency are both related to the power source; however, the dielectric constant and the loss tangent are properties of the material indicating that the amount of heat produced is directly related to the material being microwaved.

For the microwave treatment of WAS, the characteristics of the sludge will indicate the influence of the pretreatment. WAS has a large water content which corresponds to a large dielectric loss factor, ε", indicating good microwave heating capabilities and if the concentrations of salts in the sludge is high, it is also expected that this would increase the rate of microwave heating (Singh and Heldman 2009).

2.2.2.1 Microwave Design

A typical household microwave consists of a power supply, a magnetron, a wave guide or transmission section, a stirrer, and an oven cavity. A cross-section of a typical household microwave is shown in Figure 5.



(Canadian Centre for Occupational Health & Safety 2004)

Figure 5 Household microwave cross-section

Although this figure only displays a household unit, it can be used to describe the components of microwaves. The power supply provides electricity to the magnetron. The magnetron is an oscillator which is capable of converting electricity into high-frequency radiant energy. The polarity of the radiation switches from negative to positive depending on the frequency of the microwave. The wave guide directs the energy away from the magnetron with little loss of energy. The stirrer distributes the energy throughout the oven by fanning the standing waves to provide more even distribution of energy (Osepchuk 2002). The oven cavity of most microwave units, commonly called the applicator, is typically a closed metal structure and has either an access door or open ports to allow the workload to pass through in a continuous flow (Meredith 1998). The oven cavity may vary and use conveyors, or have multiple cavities each with different power (Osepchuk 2002).

There are several issues relating to the use of microwaves. Heating uniformity is not always consistent, and there can be intermittent hot and cold zones in the material being microwaved,

resulting from the coupling of the magnetrons in the microwave (Osepchuk 2002), which could cause variability of the characteristics within the microwaved material.

2.2.3 Microwave Effects

There has been a great deal of research in the food industry to assess the ability of microwaves to heat materials of differing characteristics. The effect of microwave heating on foods has been shown to vary depending on the frequency, temperature, moisture content, salt content, and physical state (Decareau 1985). It is reasonable to assume that the varying characteristics of sludge will provide a similar variability. Table 3 is a summary of the studies that have been reported on investigations of the influence of microwaving on sludge, and the subsequent biodegradation of that sludge by anaerobic digestion. .

Table 3 Anaerobic Digestion Microwave WAS Pretreatment Studies

Type of Feed	Reactor Scale	MW Pretreatment	Reactor Type	Volume (L)	Number of Reactors	Dig.Temp (°C)	HRT (d)	SRT (d)	Reference
WAS	Batch Bench Scale	700kW 2450 MHz Household Batch MW for 0, 3, 5, 7, 9, 11, and 15 min	CSTR	5	3	35	15	15	(Park et al. 2004)
WAS	Batch Bench Scale	1250kW 2450 MHz Industrial Batch MW for 5 min	CSTR	0.125	50	33	23	23	(Eskicioglu et al. 2006)
WAS	Batch Bench Scale	1kW 2450 MHz Household Batch MW for 30, 60, 90secs Batch MW for 0, 3, 6, 9, 12, 15, and 18 sec	CSTR	6	3	35	7.5-20	7.5-20	(Hong et al. 2006)
PS:WAS (1:1)	Batch Bench Scale	1kW 2450 MHz Household Batch MW for 110 sec	CSTR	4	3	35	25	25	(Pino-Jelcic et al. 2006)
WAS	Batch Bench Scale	1250kW 2450 MHz Household Batch MW timed	CSTR	0.5	54	33	19	19	(Eskicioglu et al. 2007b)
WAS	Batch Bench Scale	1250kW 2450 MHz Household Batch MW timed	CSTR	0.5	54	33	19	19	(Eskicioglu et al. 2007a)
	Semi-Continuous Bench Scale	1250kW 2450 MHz Household Batch MW timed	CSTR	0.5	10	33		5, 10, 20	
WAS	Batch Bench Scale	1250kW 2450 MHz Industrial Batch MW timed	CSTR	0.125	32	35	15	15	(Eskicioglu et al. 2007c)
WAS	Batch Bench Scale	1250kW 2450 MHz Industrial Batch MW timed	CSTR	0.5L, 1L	2	35			(Eskicioglu et al. 2008)

Table 3 only summarizes the studies that have been completed using microwaves as a method of pretreatment. There are other pretreatment studies which consider thermal treatment, however, the purpose of this research was to only assess microwave treatment. From Table 3 it can be seen that the previous research has focused mostly on WAS, with household style microwaves, and in batch scale reactors.

Batch style microwaving is different from the continuous industrial scale microwave treatment, mostly with respect to the time it takes for the microwaved material to be heated. The column entitled 'MW Pretreatment' in Table 3 indicates the type of pretreatment the WAS received. The first four studies listed in the table used a predetermined length of time to analyze microwaving impacts. The last four listed used a predetermined temperature which was based on a calibration curve that predicted the length of time required to achieve a predetermined temperature. The times in the last four studies were in the range of 1.5 min to 3 min to achieve temperatures of 50 °C to 90 °C, when the microwave was operated at full intensity. When the microwave was operated at 50 % intensity in these studies the time range required to reach the aforementioned temperature range increased to 2 min to 5 min (Eskicioglu et al. 2007b). The batch scale microwaving, presented in Table 3, required exposure of the WAS to microwaves for lengths of time in the order of minutes, whereas continuous flow microwave units, only apply treatment for lengths of time in the order of seconds (Meredith 1998), although both can accomplish temperatures as high as 90 °C. It can be concluded that the WAS in the batch microwaving conditions would thus have longer exposure as the temperature of the WAS slowly ramps up to the desired temperature, and experience more thermal effects than the WAS treated with a continuous flow microwave.

The studies, completed by Eskicioglu (2006, 2007 and 2008), which compared the results with different microwave intensity further blur the line between microwave effects and thermal effects. In these studies, the microwaves were operated at half the intensity and they required longer to heat the WAS to the desired temperature. Meredith (1998) indicates that 'the duty-cycle of actual heating time to total process time, i.e. heating time plus loading/unloading time, may be unacceptable in a batch process; the peak microwave heating capacity becomes greater than for the corresponding continuous process, because the mean power is reduced by the duty cycle, resulting in a higher capital cost of the generator equipment' (Meredith 1998). The duty cycle employed by Eskicioglu was not indicated, however, with a 50 % intensity it can be deduced that the magnetron was only operating for half of the time. It would be expected that the time required to heat the WAS at 50 % intensity would

be twice the time of the 100 % intensity, although this was not the case. Due to the operational nature of the batch microwave used in the study the heating of the WAS also relied on conduction heating from the hot portions of the WAS to the cooler portions.

Thermal effects and conductive heating should be reduced in continuous microwave units as the microwaving occurs in a considerably shorter amount of time. Both the solubilization resulting from microwaves and the biodegradability of microwaved sludge will be evaluated in this study, but with the use of a continuous- flow microwave rather than a batch scenario.

2.2.3.1 Solubilization

To evaluate the effects caused by microwaving WAS, the soluble components of the sludge are typically measured prior to and after microwaving. As was discussed in Section 2.1.2.3.1 an increase of the soluble components, which are typically more easily biodegraded, is an indication of effective pretreatment. The measurement of filtered COD prior to and after pretreatment can indicate the amount of solubilization that has occurred. There has been a great deal of discussion as to the nature of the solubilization caused by microwaves (Banik et al. 2003; Hong et al. 2004; Osepchuk 2002; Singh and Heldman 2009). Although there are thermal effects caused by microwave technology, the non-thermal are of particular interest.

The non-thermal effects caused by a microwave are those effects not relating to an increase in temperature. The effects are suggested to be a result of the polarized side chains of macro-molecules lining up with the direction of the electric field, which causes the hydrogen bonds of the molecules to break (Hong et al. 2004). Ospechuk et al (2002) suggested that only effects derived from heating have resulted in practical applications, and that non-thermal effects are not detectable, however, in more recent articles it has been established that various molecular transformations and alterations are due to non-thermal effects (Banik et al. 2003). This has been further supported by the fact that the studies which compared microwave treatment to conventional heating have indicated that the microwave was superior as it related to pathogen destruction, stimulation of methane generation, and energy requirements(Hong et al. 2004) Banik et al.(2003) indicated that different waves and different modes produced significantly different responses in algae, suggesting that the microwaves did have a non-thermal influence. Their study suggested that the biological effects were caused by differentially

partitioning the ions, altering that rate and direction of biochemical reactions and ultimately inducing different responses depending on the filament length, turbidity of the cell suspension and the presence of protein, carbohydrate, chlorophyll a, caratenoids, and phycocyanin.

Microwaves can affect the chemical bonds in materials under certain circumstances. When a microwave is used to apply electromagnetic fields to sludge the induced rotation of water molecules causes reduction in the size of organic molecules as the weak hydrogen bonds are broken. Smaller organic molecules are generally more easily biodegraded, and contribute to the soluble COD value (Eskicioglu et al. 2007b). Factors that have been shown to influence the dielectric response are the frequency of the applied energy, the duration of radiation application, the concentration of the material being radiated, the particle size distribution, the viscosity of the material, and the penetration depth of the microwaves (Hong et al. 2004). Table 4 summarizes the research that has measured the increased solubilization due to microwaving. Only those studies which compared the soluble components of non-microwaved WAS to those of the microwaved WAS were shown, as a soluble value on its own does not indicate solubilization.

Table 4 Solubilization Studies for Microwave Preatreatment of WAS

Type of Feed	Scale	Type of Pretreatment	MW Temp (°C)	Pore size for soluble	COD_S/COD_T	Feed COD (mg/L)		Feed COD_S (mg/L)		Feed TS (g/L)		Feed VS (g/L)		Reference
						CTRL	MW	CTRL	MW	CTRL	MW	CTRL	MW	
WAS	Batch Bench Scale	MW 1250kW 2450 MHz Industrial	96(2)	0.45µm	0.15				143±34% increase					(Eskicioglu et al. 2006)
WAS	Batch Bench Scale	MW 1kW 2450 MHz Household	72.5	Not indicated	5% increase			2340	3400					(Hong et al. 2006)
PS:WAS (1:1)	Batch Bench Scale	MW 1kW 2450 MHz Household	60-65	1.5µm			46.3%[1]				43.9±6.7		31.3±6.9	(Pino-Jelcic et al. 2006)
WAS	Batch Bench Scale	MW 1250kW 2450 MHz Household	75	0.45µm	3.23±0.1 fold increase	41667±1190		2357±71		1.4%				(Eskicioglu et al. 2007b)
			75	0.45µm	3.66±0.1 fold increase					5.4%				

[1]Percent of nonsoluble COD fraction solubilized

The studies regarding the solubilization of WAS, shown in Table 4, analyzed the influence of microwaving at temperatures ranging from 60 °C to 96 °C. All of the research was completed using 2450 MHz household style microwaves with batch heating, and temperature measurement of the WAS after a certain length of microwaving time, which was described in Table 3. The results from these studies were not reported using the same measures. Eskicioglu (2006) reported the effects as a change in soluble COD, Hong (2006) and Eskicioglu (2007) reported effects as a change in the ratio of soluble COD to total COD, and Pino-Jelcic (2006) considered the percentage of the non-soluble COD that was solubilized. The definition of soluble COD varied from study to study. Hong (2006) did not indicate the filter size used, Pino-Jelcic (2006) used a filter with a nominal pore size of 1.5 µm, and for all the analyses done by Eskicioglu the WAS samples were centrifuged and put through a filter with a nominal pore size of 0.45 µm. Despite the different reporting methods all four studies indicated that there was either an increase in the soluble component or the volatile components of the WAS, even though for the studies using 1.5 µm filters colloidal matter would have been included in the 'soluble' measure.

Two separate data sets describing solubilization were reported by Eskicioglu (Eskicioglu et al. 2006; Eskicioglu et al. 2007b). The results presented in Eskicioglu et al.(2006) indicated that microwaving at 96 °C resulted in an increase of the soluble COD content by 143 % from pre to post microwaved samples. Eskicioglu et al. (2007) reported the solubilization of COD on the basis of the ratio of soluble COD to total COD. Eskicioglu et al (2007) found that at a microwave temperature of 75 °C, compared to the control, the ratios were found to be 3.6+/-0.6 times higher for WAS with a total solids concentration of 1.4% and 3.2+/-0.1 times higher for a WAS with a solids concentration of 5.4 %. Their study was repeated again at 96 °C and showed even higher solubilization ratios, however, these values were not reported. It was also shown that solubilization was higher at 50 % microwave intensity compared to 100 % intensity at the same temperature and with these different microwave intensities, however as was previously mentioned this could be a result of the longer exposure to a heated environment (Eskicioglu et al. 2007b).

The separation of the non-thermal microwave effects from the thermal microwave effects was not straightforward. Eskicioglu et al (2007) indicated that their ratios of soluble to total COD increased with the heat exposure time, suggesting that it was not only the temperature that was reached in the sludge that dictated the solubilization, but also the length of time that the sludge was exposed to heat.

The study completed by Hong showed a 5 % increase of soluble COD when a batch of WAS was microwaved until it reached 72.5 °C (Hong et al. 2006). The study reported by Pino-Jelcic et al. (2006), which used a PS:WAS combination with a total solids concentration of 43.9 g/L, showed a solubilization of 46.3 % of the nonsoluble COD fraction when exposed to a microwaving temperature of 60 °C.

None of the literature has reported the solubilization of WAS as a function of increased VS concentrations, and the definition of solubility was inconsistent. For this study the measurement of the soluble components and the influence of the microwave will be measured using a change in the concentration of both volatile solids and filtered COD, measured from the filtrate from a 1.5 µm filter paper. The change of the filtered component will be presented in a ratio of filtered to total COD concentrations, and the percent change of the volatile solids and filtered COD.

2.2.3.2 Biodegradation

In general terms, to improve the biodegradability within anaerobic digestion the sludge must be broken down so that extracellular and intracellular components of the sludge can be accessed by the microbes in the digester. Pretreatment methods have been shown to increase the access to both the extracellular and intracellular components, but they can also decrease the dewaterabilty following anaerobic digestion (Müller et al. 1998). The application of microwaves, as a form of pretreatment, to both primary sludge and waste activated sludge has been demonstrated to break down particulate organics into readily biodegradable organics.

Table 5 presents the results from the studies that have analyzed biodegradation of microwave treated sludges in anaerobic digesters. All of these studies employed batch microwaving, and bench scale digestion. The studies presented in Table 5 used 2450 MHz microwaves as previously shown in Table 3. The anaerobic digesters were all continually stirred reactors, and all were batch digesters with the exception of the research done by Eskicioglu (2007) which used semi-continuously fed digesters.

Table 5 Biodegradation Studies for Microwave Pretreatment of WAS

Vol (#Rcts)	Temp (°C)	HRT SRT (d)	MW Temp (°C)	COD_S/COD_T	Feed COD (mg/L)	Feed COD_S (mg/L)	Feed TS (g/L)		Feed VS (g/L)		Effluent COD_S (mg/L)		COD Removal Efficiency (%)		COD_S Removal Efficiency (%)		TS Removal Efficiency (%)	VS Removal Efficiency (%)		Increase in Biogas (MW compared to CTRL)	Reference
					CTRL	CTRL	CTRL	MW	CTRL	MW	CTRL	MW	CTRL	MW	CTRL	MW	MW	CTRL	MW	MW	
5 L (3)	35	15	77.2	0.17							414	516	14.4	23.6	14.4	23.6		25.9	23.0		(Park et al. 2004)
0.125L (50)	33	23	96	0.15																211%	(Eskicioglu et al. 2006)
4 L (3)	35	25	60					44		31.3								49.0	53.9	16.4%	(Pino-Jelcic et al. 2006)
0.5 L (54)	33	19	96				1.4%													15%	(Eskicioglu et al. 2007b)
		19	96				5.4%													20%	
0.5 L (54)	33	19	96				3%													15%	(Eskicioglu et al. 2007a)
0.5 L (10)		5	96	0.155			3%							16[1]		80[2]	29[1]		23[1]	28%	
		10	96	0.145			3%							3[1]		29[2]	13[1]		13[1]	4%	
		20	96	0.15			3%							<5[1]		5[2]	16[1]		11[1]	5%	
0.125L (32)	35	15	50	0.12	45714	4286	4.6%		3.1%											16%	(Eskicioglu et al. 2007c)
0.5L or 1L	35		175	0.35	41667	2357	3%		70%											31%	(Eskicioglu et al. 2008)

[1]Increase compared to control digester

[2]Decrease compared to control digester

[3]Percent of nonsoluble COD fraction solubilized

The most important information displayed in Table 5 is the increase in biogas produced in the digesters that were fed microwaved WAS as compared to the control digesters that were fed untreated WAS. All of the studies with the exception of the study completed by Park (2004) indicated an increase in biogas, however, that study showed an increase in the removal of both COD and soluble COD which is theoretically associated with an increase in biogas production. The reported increase in biogas ranged from 4 % to 211 %, but the average reported increase was approximately 12%.

In anaerobic digestion of WAS typically only 30-35% COD reduction is achievable in conventional sludge treatment (Grady Jr. et al. 1999). None of the literature reported their COD removal efficiencies alone, but rather compared to a control digester. For both the research completed by Eskicioglu and Park, the results indicated that the COD removal efficiency for the digester being fed microwaved WAS was higher than the control digester.

The comparative soluble COD removal showed improvements associated with the pretreatment of the WAS in the research completed by Park, however, there was a decrease in the soluble COD removal efficiency show in the research by Eskicioglu et al. (2007), which became more pronounced with shorter SRTs. This was postulated to be because of the increased concentration of soluble COD in the microwaved feed (Eskicioglu et al. 2007a).

Although not summarized in Table 5, the research completed by Eskicioglu et al. (2007) also considered the ammonia concentration in the WAS prior to and after the microwave pretreatment of the waste activated sludge, and it was observed that within the digesters higher ammonia concentrations were observed when the pretreatment temperature was increased, which was attributed to the higher anaerobic degradation efficiency of the nitrogenous organic matter in the digester (Eskicioglu et al. 2007a).

There was considerable variability from study to study with respect to the nature of the source of the feed sludge, solids concentrations and hydraulic retention times, and hence it was difficult to establish general conclusions that were true for all of the scenarios. It was however apparent that for bench scale batch anaerobic digestion of WAS, pretreatment using microwaves increased biogas production

and therefore this study will consider anaerobic digestion of WAS at a pilot scale with semi-continuous feeding.

2.3 Membrane Bioreactor Literature Review

The use of membranes in wastewater treatment is a relatively recent development. Their current use and background as well as their physical and operational characteristics will be discussed in this section.

2.3.1 Membrane Background

Membranes are selective barriers that are capable of separating different components of a feed stream. It has only been in the past twenty years that there has been a considerable amount of research into their applications for large scale industrial and municipal settings. As the interest grew, the acceptance of the use of membrane in wastewater treatment has also grown. Within North America the interest in membranes was slowed, since land acquisition for treatment has not been an issue. Throughout the world, Europe and Asia have more interest in the use of AnMBRs) than North America, although the interest in this field is growing (Jin et al. ; Wang et al. 2008; Yang et al. 2006).

Membrane research has included the study of membrane fouling, operational and design parameters, feed properties, microbiological characteristics, cost, and the modeling of the membrane processes. Few of these studies have included full-scale operations on a long term basis for high strength industrial or municipal wastes (Yang et al. 2006).

Membranes have been used to target the smaller constituents in wastewater, such as solids, bacteria, protozoan cysts, and oocysts. The membranes themselves can be classified depending on the type of filtration which can be microfiltration, ultrafiltration, reverse osmosis, dialysis, and electrodialysis.

Table 6 presents a summary of the research that has been done using membranes with anaerobic digesters, and in the following sections both the research regarding the digester performance and that regarding the membrane performance will be discussed. The research summarized only dealt with high strength wastewaters that had a high solids content.

Table 6 Previous Anaerobic Digester Membrane Studies

Type of Feed	Scale	Reactor Type	Membrane	Reference
PS	Pilot	CSTR	Tubular Inorganic	(Murata et al. 1994)
PS	Pilot	CSTR	MF Cross-Flow	(Pillay et al. 1994)
WAS	Bench	CSTR	UF	(Takashima et al. 1991)
PS	Batch Pilot	Upflow Mixed	MF Cross-flow Ceramic UF Tubular Polyether sulfone	(Ghyoot and Verstraete 1997)
Synthetic	Bench	CSTR	MF Inorganic Composite UF Inorganic Composite	(Elmaleh and Abdelmoumni 1998)
Synthetic	Bench	CSTR	MF Tubular Ceramic MF Tubular Polypropylene MF Plate & Frame Polyvinlyidene	(Choo et al. 2000)
Pig W/W	Pilot	UASB	Mixed Ester of Cellulose	(Lee et al. 2001)
WW	Bench	CSTR	Tubular Hydrophobic Polypropylene Tubular Zirconia skinned Inorganic	(Kang et al. 2002)
Chicken Slaughterhouse WW	Bench	CSTR	Ceramic Cross-flow	(Fuchs et al. 2003)
PS & WAS	Pilot	CSTR	UF Vibrating Polymeric Teflon UF Tubular Cross Flow	(Pierkiel and Lanting 2005)
Pig Manure	Pilot		UF Tubular Polysulfone	(du Preez et al. 2005)
Pig Manure	Bench	CSTR	UF Tubular Polyethersulfone	(Padmasiri et al. 2007)
WAS	Bench	CSTR	Tubular UF Polyvinylidene Fluoride	(Dagnew et al. 2008)
Kraft Condensate	Bench	UASB	MF Submerged Flat Sheet Polyvinylidene Fluoride	(Lin et al. 2009)

2.3.2 Anaerobic Treatment and Performance

In general terms, to achieve a high level of biodegradability within the anaerobic digester the sludge must be broken down so that extracellular and intracellular components of the sludge can be accessed by the microbes in the digester. The use of membranes as a method to decouple HRT and SRT allows for longer solids treatment and the breakdown of particulate organics into readily biodegradable organics. Membranes can also be applied to eliminate the need for thickening polymers and avoid their potentially inhibitory effect of anaerobic biomass (Pierkiel and Lanting 2005).

Table 1Table 7 presents the results from the studies, introduced in Table 6, which indicated the extent of biodegradation observed in anaerobic digesters, when used conjunctively with membranes. The studies presented in the table only have one commonality; that is that the feed for the anaerobic digesters was high strength with a high solids content. The source of the feed was varied, some of the research was bench scale, while others were pilot studies, and the type of reactor as well as the type of membrane varied.

Table 7 Summary of Performance for WAS Biodegradation with Membrane Treatment

Type of Feed	Scale	Reactor Type	Membrane	Vol (L)	Temp (°C)	HRT (d)	SRT (d)	OLR (kg COD/m^3d)	MLSS (g/L)	Feed COD (g/L)	Feed TS (g/L)	Permeate COD (mg/L)	Permeate sCOD (mg/L)	COD Removal Efficiency (%)	Reference
PS	Pilot	CSTR	Tubular Inorganic	500	35	8.4	335	0.93^{VSS}	55	0.24	0.16	30^{VSS}		79^{VSS}	(Murata et al. 1994)
				500	55	7.8	197	1.16^{VSS}		0.24	0.16	30^{VSS}		78^{VSS}	
PS	Pilot	CSTR	MF Cross-Flow	1800		14	26		55						(Pillay et al. 1994)
WAS	Bench	CSTR	UF	5	35	30	100				20.7	200			(Takashima et al. 1991)
PS	Batch Pilot	Upflow Mixed	MF Cross-flow Ceramic UF Tubular Polyether Sulfone	120	35	20		1.06	22-35	40.2	44.4	18 000		54	(Ghyoot and Verstraete 1997)
Synthetic	Bench	CSTR	MF Inorganic Composite		35				0.13					95	(Elmaleh and Abdelmoumni 1998)
			UF Inorganic Composite		35				0.13					95	
Synthetic	Bench	CSTR	MF Tubular Ceramic		54-56				2	27					(Choo et al. 2000)
			MF Tubular Polypropylene		54-56				2	27					
			MF Plate & Frame Polyvinlyidene		54-56				2	27					
Pig W/W	Pilot	UASB	Mixed Ester of Cellulose	3000	35	1-2				5.5	0.6	1000-1500		80	(Lee et al. 2001)
WW	Bench	CSTR	Tubular Hydrophobic Polypropylene		55	13	70	3-3.5	2	38.4				90	(Kang et al. 2002)
			Tubular Zirconia skinned Inorganic		55	13	70	3-3.5	2	38.4				90	
Chicken Slaughter WW	Bench	CSTR	Ceramic Cross-flow	7	30	1.2		4.3	22	5.2	2.4-4.7	<500		90	(Fuchs et al. 2003)
PS&WAS	Pilot	CSTR	UF Vibrating polymeric Teflon	550	35	1.7-11.8	4.2-70.5				0.5-2%			59^{VSS}	(Pierkiel and Lanting 2005)
			UF Tubular Cross Flow								1%			59^{VSS}	
Pig Manure	Pilot		UF Tubular Polysulfone	5000	37&51						4-8%				(du Preez et al. 2005)
Pig Manure	Bench	CSTR	UF Tubular Polyethersulfone	6	37	6		$1\text{-}3^{VS}$					1272	96	(Padmasiri et al. 2007)
WAS	Bench	CSTR	Tubular UF Polyvinylidene Fluoride	50		15	15			10-22	6-18				(Dagnew et al. 2008)
Kraft Condensate	Bench	UASB	MF Submerged Flat Sheet Polyvinylidene Fluoride	6.5	37		230	12.2	8.5	10		151		97-99	(Lin et al. 2009)
				6.5	55		230	3.1	8.5	10		197		97-99	

[1]final flux divided by starting flux

From Table 7 it can be observed that feed stocks have included PS, WAS, synthetic wastewater, piggery wastewater, raw wastewater, chicken slaughterhouse wastewater, Kraft condensate, and pig manure. With such diverse feed stocks, a comparison of biodegradability was challenging as it was highly dependent on the characteristics of each material.

The scale of the research has included bench and pilot scale studies, with both continuous feed and batch feed. Not only did the scale and operations differ, but a variety of sizes and digester types have been employed. The majority of the digesters were CSTRs, but there has also been research using upflow mixed digesters, and upflow sludge blanket digesters. The volumes of the digesters have varied from 5 L to 5000 L and were operated at either mesophilic temperatures or thermophilic temperatures. The subsequent sections discuss the results presented in Table 7 in more detail.

2.3.2.1 Solids and Hydraulic Retention Times

The most important benefit of using membranes in anaerobic treatment is the capability to separate the HRT from the SRT. This allows for the flexibility to treat the liquid and solid components of the sludge for optimal lengths of time, regardless of the loading rate and the influent solids concentrations.

In anaerobic digestion hydrolysis of the particulate matter is typically the limiting step of treatment. To achieve hydrolysis the residence time of the digester must be maximized. In typical operations this requires an increase to the volume of the digester, however, with the installation of a membrane, the hydraulic residence time can be decreased while the solids residence time can be increased using the same volume of digester. This is beneficial when there are increased flows to a treatment plant, but limited space (Yang et al. 2006).

The benefits associated with membrane technology not only include the reduced footprint, but also an increase in the treatment capability. The destruction of solids is improved and hence there is a reduction in the quantity of treated solids. The membrane bioreactor can be particularly useful when treating complex feed stocks that are high in slowly degradable solids, such as are present in WAS, which are often treated insufficiently by other standard anaerobic treatment systems (Fuchs et al. 2003). With respect to the permeate, the effluent is of very high quality and the permeate volume can be increased while maintaining appropriate solids treatment (Pillay et al. 1994).

For the research shown in Table 6, the HRT was reported, but for the majority of the studies the solids were never removed from the digesters, resulting in SRTs that were reported as high as 335 days. HRTs ranged from 1.2 days to 30 days. The exceptions to this trend was a pilot scale study done by Pierkiel (2005) using PS and WAS for feed. In this analysis an HRT of 1 to 3 days was examined while maintaining an SRT in the range of 8 to 12 days (Pierkiel and Lanting 2005). The second exception was the 15 day SRT reported for the research completed by Dagnew (2008).

2.3.2.2 Biodegradation

Unlike the research gathered for the microwave literature review the membrane literature review included feed sources that were not WAS. The increased scope was due to the limited literature that was available for anaerobic digestion of WAS with the aid of membranes, and the membrane performance of other high strength feeds was deemed to be pertinent. A challenge associated with the widened scope is that the biodegradation and digester performance was difficult to compare with such varied sources and operational conditions. The general trends of biodegradation will be described; however, the focus for these studies was typically on the membrane performance, so the reporting of biodegradation was often not complete.

All of the feed was deemed high strength. This definition was rather loose, and as is shown in Table 7 the total COD of the feeds ranged from 0.24 g/L to 40.2 g/L and the TS ranged from 0.16 g/L to 44.4 g/L. The resulting mixed liquor suspended solids in the digesters were no less varied and ranged from 0.13 g/L to 55 g/L.

In Table 7, the anaerobic performance was presented in terms of COD removal efficiency, and in several cases VSS removal efficiency. Padmasiri (2007) indicated that an increase to the cross-flow velocity would improve the membrane performance, but ultimately resulted in a reduction in anaerobic digestion performance. This was attributed to an increase in shear applied to the digester contents, ultimately increasing the rate of hydrolysis and a buildup of fermentation products (Padmasiri et al. 2007). Ghyoot and Verstraete (1997) also indicated that the transfer of anaerobic sludge through the membrane units disrupted the interactions between bacterial species due to the shear stress applied by the mechanical parts of membrane units, and further recommended that the attachment of a membrane to a digester would not be as beneficial as filtration to the waste sludge

prior to disposal of waste sludge (Ghyoot and Verstraete 1997). The results of Brockmann (1997), with a potato starch wastewater and a low-solids sludge, supported this recommendation in that the performance of the digester depended on the circulation rate of the contents. However, Padmasiri (2007), Ghyoot and Verstraete (1997), and Brockmann (1997) considered external tubular or cross-flow membranes, and there was no indication that this same effect would occur with submerged or hollow fibre membrane scenarios. Pierkiel (2005) indicated that the use of tubular membranes did not adversely affect biomass performance.

Despite the indication that membranes may negatively influence the anaerobic processes, as indicated in Table 6, for the most part the removal efficiencies were high. With the exception of the research completed by Ghyoot and Verstraete (1997) and Pierkiel (2005), the removal efficiencies were in the range of 79 % to 99%. The defining characteristics of those studies that were different from the studies with high removal efficiencies were that they both had either PS or WAS as a feed source. Dagnew (2008) also used WAS, but did not report removal efficiencies. The decreased biodegradability of PS and WAS as compared to other types of high strength wastewater would influence the quality of the sludge, however, the permeate quality was shown to be similar and much better, respectively, than the quality of the supernatant of a conventional anaerobic digester (Ghyoot and Verstraete 1997).

In the study completed by Lin (2009), in which thermophilic and mesophilic submerged AnMBRs were compared, it was concluded that the mesophilic digester had better filtration performance. The higher temperature digester promoted more EPS release and the sludge cake layer in the thermophilic digester was more compact and less porous, ultimately increasing fouling.

2.3.3 Physical and Operational Characteristics of Membranes

Typically either microfiltration or ultrafiltration membranes have been used in anaerobic biological environments. The classification of a membrane includes the type of material the membrane is made with, the nature of the driving force, the separation mechanism, and the nominal pore size of the separation. Metcalf and Eddy (2003) reported typical operating conditions for microfiltration membranes in wastewater treatment, and these are presented in Table 8.

Table 8 Typical Characteristic of Microfiltration Technologies in Wastewater Treatment

Characteristic	Value
Typical Operating Range	0.08 – 2.0 μm
Operating Pressure	7-100 kPa
Rate of Flux	405-1600 L/m^2-d
Type	Polypropylene, acrylonitrile, nylon, and polytetrafluoroethylene
Configuration	Spiral wound, hollow fibre, plate and frame

(Tchobanoglous and Burton 2003)

In the following sections the characteristics indicated in Table 8 will be discussed as they relate to the operations of membranes in anaerobic digestion. More specifically, the characteristics discussed in the following section are the fouling, configuration of the membrane, the pore sizes, trans-membrane pressure, flux, the materials used in membranes, the solid and hydraulic retention times, and the cost.

Table 9 presents the results from the studies, introduced in Table 6 that indicated the membrane performance when used conjunctively with anaerobic digestion. The studies presented in the table do not have comparable operational parameters. As was indicated by the summary table the type of membrane, the pore sizes, the operational parameters, the configuration, and the materials differed. However, the performance will be presented and discussed in the following sections.

Table 9 Summary of Membrane Performance for WAS Digestion

Type of Feed	Scale	Reactor Type	Membrane	Pore Size	Membrane Area (m^2)	TMP (kPa)	Linear Velocity (m/s)	Initial Flux (L/m^2h)	Final Flux (L/m^2h)	Reference
PS	Pilot	CSTR	Tubular Inorganic	0.1µm	0.88				25-42	(Murata et al. 1994)
PS	Pilot	CSTR	MF Cross-Flow			200			50	(Pillay et al. 1994)
WAS	Bench	CSTR	UF	30 000 Da	0.0177					(Takashima et al. 1991)
PS	Batch Pilot	Upflow Mixed	MF Cross-flow Ceramic	0.1 µm	0.05	200	4.5		200-250	(Ghyoot and Verstraete 1997)
			UF Tubular Polyether sulfone	60 000 Da	0.3	375	0.75	31	19	
Synthetic	Bench	CSTR	MF Inorganic Composite	0.14, 0.2 µm		50-100	3.5	72-120		(Elmaleh and Abdelmoumni 1998)
			UF Inorganic Composite	0.05, 0.08 µm						
Synthetic	Bench	CSTR	MF Tubular Ceramic	0.14µm			0.3		0.3[1]	(Choo et al. 2000)
			MF Tubular polypropylene	0.2µm			0.3		0.35[1]	
			MF Plate & Frame polyvinlyidene	0.1µm			0.3			
Pig W/W	Pilot	UASB	Mixed Ester of Cellulose	0.5µm						(Lee et al. 2001)
WW	Bench	CSTR	Tubular hydrophobic polypropylene	0.2 µm	0.0129	60	<3	300-400	0.48[1]	(Kang et al. 2002)
			Tubular zirconia skinned inorganic	0.14 µm	0.0113	60	<3	140-180	0.34[1]	
Chicken Slaughter WW	Bench	CSTR	Ceramic Cross-flow		0.126		2-3		5-10	(Fuchs et al. 2003)
PS&WAS	Pilot	CSTR	UF Vibrating polymeric Teflon	0.05µm	1.6				1600-2000	(Pierkiel and Lanting 2005)
			UF Tubular Cross Flow	0.1µm	1.4		5		3500	
Pig Manure	Pilot		UF Tubular Polysulfone	40 000 Da	1.7		1.5-3.5			(du Preez et al. 2005)
Pig Manure	Bench	CSTR	UF Tubular polyethersulfone	20 000 Da	0.0377	30-70	1.1-2	100	5-10	(Padmasiri et al. 2007)
WAS	Bench	CSTR	Tubular UF polyvinylidene fluoride	120 000 Da		80	1.4		43	(Dagnew et al. 2008)
Kraft Condensate	Bench	UASB	MF Submerged Flat Sheet polyvinylidene fluoride	70 000 Da	0.03	<30		7.2	7.2	(Lin et al. 2009)
						<30		2.4	2.4	

[1]final flux divided by starting flux

2.3.3.1 Fouling

There are several disadvantages to the use of membrane technologies. These include foaming within the bioreactor, fouling of the membrane, and lower performance capabilities than anticipated due to the nature of the feed source (Yang et al. 2006). Fouling, however, is the disadvantage that has received the most attention, since it causes the most noticeable negative impacts, such as reduced productivity, shorted membrane life, and increased operational costs. It has been suggested that the lack of wide spread commercialization is in part due to the limited research regarding the mechanisms of membrane fouling and methods to protect against it (Yang et al. 2006), however, currently there is research investigating methods to control and minimize membrane fouling, and it has been suggested that compared to aerobic MBRs, which are widespread, anaerobic MBRs may perform better as they are less likely to be fouled (Fawehinmi et al. 2007).

Fouling is caused by means of three mechanisms, pore narrowing, pore plugging, and gel or cake formation caused by concentration polarization (Tchobanoglous and Burton 2003). The biological components of wastewater, which participate in the fouling mechanisms, are the primary foulant for organic membranes. The fouling on organic membranes is believed to be caused by the formation of a cake layer rather than adsorption of soluble or particular matter within the pore structure of the membrane (Bérubé et al. 2006). For inorganic membranes the cake layer typically does not form, and internal fouling is the overriding fouling mechanism. This internal fouling has been reported to be caused by the precipitation of struvite on the membrane. In addition to membrane characteristics, solids concentration, flux, and charge have been shown to have a significant influence on fouling (Dagnew et al. 2008).

Methods to prevent fouling have been developed. Prior to operation, the design of the membrane can reduce fouling. Packing density, aerator location for the recirculation of biogas, and the orientation and diameter of the fibres are design components that can prevent fouling (Yang et al. 2006). The design flux can also influence fouling. For the purpose of this research the critical flux of a membrane was the flux at which irreversible fouling occurred (Bacchin et al. 2006), and the operation below the critical flux allows for a constant TMP, while operation above causes a rapid increase in TMP (Liao et al. 2006). If the membrane unit is designed for an operating flux that is lower than critical flux, fouling can be reduced. By removing the colloidal and particulate material from the membrane, fouling can be prevented. The destruction or agglomeration of colloids can protect

against the formation of a more compact cake layer (compared to larger particles) that colloids can form (Bérubé et al. 2006).

Gas sparging can protect the membrane from fouling by causing shear stress through the application of bubbles to the membrane surface, thereby removing the cake layer. The effectiveness of sparging can also be improved through the application intermittent suction on the retentate side of the membrane, which allows for the diffusion of solids away from the membrane (Yang et al. 2006). For anaerobic applications gas from the digester head space can be used for sparging (Bérubé et al. 2006). Similarly to sparging, the cross-flow velocity can provide shear at the surface of the membrane. The removal of the cake layer by sparging can however allow for more internal fouling, so there must be an assessment of the wastewater characteristics (Bérubé et al. 2006).

Once a membrane has become fouled there are several methods to clean it so that it can be kept in operation. Backwashing, relaxation, and chemical applications are methods to accomplish this. Backwashing is the reversal of filtration, and occurs for a short amount of time over the course of permeation. For instance backwashing could occur for 1 minute after 15 minutes of permeation. Similarly to backwashing, relaxation is typically employed for a period of time after permeation through the removal of the TMP. Relaxation has been shown to allow a longer use of a membrane as compared to continuous operation (Dagnew et al. 2008). In this study TMP under 40 kPa was maintained when using relaxation as compared to continuous operation that resulted in a TMP of 80 kPa.

Unlike backwashing and relaxation, chemical cleaning is not a physical removal process, but a chemical one. The cleaners are matched with the fouling components in the wastewater, and can be acidic, basic, oxidants, or surfactants. Lee (2001) found that fouling could be managed with alkali and acidic solutions, and would allow for long-term operations and, when used in series, increased the flux to 89 % of the original flux of a new membrane (Lee et al. 2001). The removal of ammonium ions with advanced technology and the addition of powdered activated carbon have been shown to reduce struvite precipitation and colloids which are the finer materials which can clog membranes (Choo et al. 2000).

2.3.3.2 Configuration

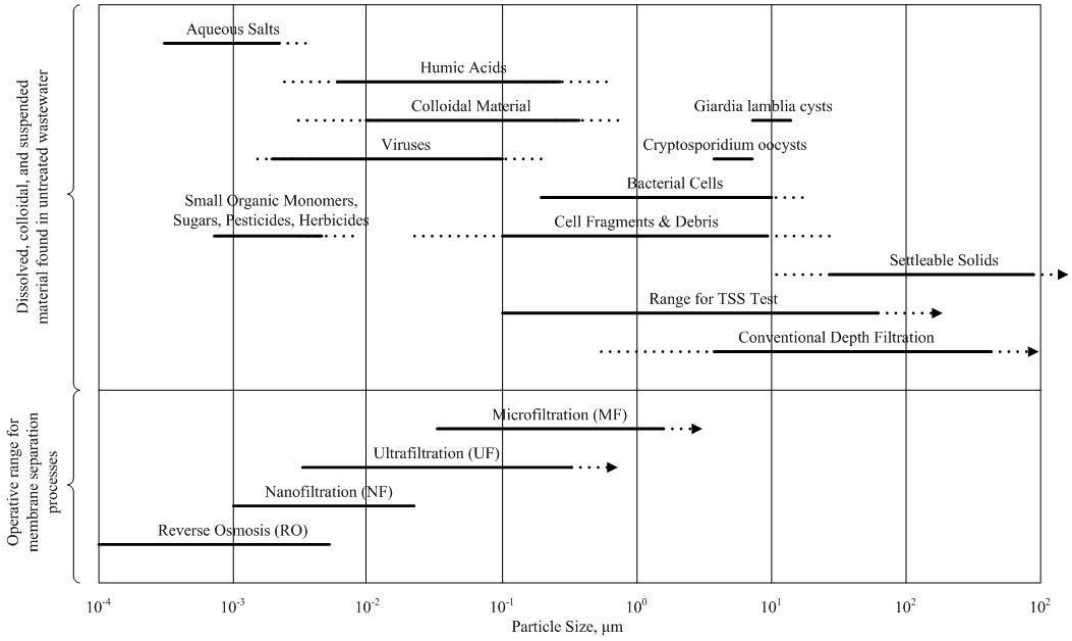
There are two operational configurations typical of membrane digesters, external and submerged membranes. For external membranes the circulation rate of the digester sludge and the transmembrane pressure are both relatively high. Berube et.al. (2006) reported that the cross-flow velocity was usually between 1 m/s and 5 m/s and the transmembrane pressure was typically between 207 kPa and 690 kPa. The submerged membrane is a more passive system. The cross-flow velocity is typically less than 0.6 m/s and the transmembrane pressure is less than that of the external membrane and has ranged between 21 kPa and 103 kPa.

The configurations employed in the research summarized in Table 6 were primarily external. Lin (2009) reported using submerged membranes in a 6.5 L bench scale digester and Lee (2001) used a submerged membrane in a pilot study. The initial and final fluxes for the submerged membranes were not reported by Lee (2001), however, the fluxes reported by Lin (2009) showed no difference between the initial and final flux values, 7.2 L/m^2-h for a microfiltration flat sheet membrane, and 2.4 L/m^2-h for a polyvinylidene fluoride membrane. These fluxes were considerably lower than the fluxes reported by the researchers using external membranes, however, comparatively the change in flux indicated less fouling than for the external membranes.

The research completed using submerged membranes in conjunction with high strength wastewater has been limited, and more research using submerged membranes, particularly at a pilot scale, would provide a better comparison of configurations. At pilot scale, submerged and external membranes could be compared over a longer life span, while maintaining similar feed characteristics, areas, and permeate volume passed.

2.3.3.3 Pore Size

The pore size of a membrane is important in determining what the membrane is capable of removing from the feed waters being applied, but it also influences the fluxes and energy required to achieve permeation. Membranes with the characteristics described in Table 8, are capable of accomplishing the removal of the dissolved, colloidal, and suspended material within the size ranges indicated in Figure 6.



(Tchobanoglous and Burton 2003)

Figure 6 Membrane filter sizes and associated wastewater component sizes

Figure 6 indicates that the operational range for filtration processes is between $10^{-1.5}$ and $10^{0.5}$ µm, and also presents the size ranges for typical wastewater components. Both ultrafiltration and microfiltration membranes are capable of removing some viruses, colloidal material and humic acids, and all cell fragments and debris, bacterial cells, Cryptosporidium, Giardia, and settleable solids. All of these components may be present in WAS and their removal may be required. Elmaleh and Abdelmoumni (1998) suggest that for anaerobic mixed liquor the optimal diameter of the nominal pore size is 0.45µm, or $10^{-0.35}$ µm.

The optimal range for a membrane pore size is a balance between fouling caused by pore sizes that are too small and too large, while accomplishing removal of the target components. If the pore sizes are too big the pores can be blogged by macro-colloids that can entirely block the pores. If the pore sizes are too small the micro-colloids can clog the pores by adhering to the membrane walls (Bérubé et al. 2006). Yang et al. (2006) found that microfiltration membranes with larger pore sizes experienced higher initial fouling than the ultrafiltration membranes (Yang et al. 2006).

The research summarized in Table 6 indicates a wide range of pore sizes, varying from 0.05 µm to 0.5 µm and 20000 Da to 120 000 Da. The pore size alone cannot be compared from study to study as

the operating characteristics, pore size reporting, and feed characteristics varied greatly and a common theme is not evident.

2.3.3.4 Transmembrane Pressure

The transmembrane pressure (TMP) is the force which pushes the liquid across the membrane. The pressure on the feed side of the membrane is higher than the pressure on the permeate side, and the TMP can be calculated using Equation 3.

Equation 3 $$\mathbf{P_{tm} = \left[\frac{P_f + P_c}{2}\right] - P_p}$$

In Equation 3, P_{tm} is the TMP gradient, P_f is in the inlet pressure of the feed stream, P_c is the pressure of the concentrate stream, and P_p is the pressure of the permeate stream (Tchobanoglous and Burton 2003). If the membranes are operated with a constant flux and a varying TMP, as the membrane becomes fouled the pressure required to provide a constant flux increases. Changing TMP is thus a good descriptor of the degree to which a membrane fouls.

Berube (2006) reported that the TMP was independent of the suspended solids concentration of the feed, but was more dependent on the cross-flow velocity. As the cross-flow velocity increased the cake layer was removed and fouling from micro-particles became the dominant fouling mechanism (Bérubé et al. 2006). Consequently, when cake layer formation caused the majority of the fouling the TMP was low; however, when this was not the case the TMP was higher (Bérubé et al. 2006).

When the TMP was low, the permeate flux was governed by the pressure and not the cross-flow velocity. At a high TMP the permeate flux was governed by the mass transfer of suspended solids away from the membrane. To accomplish the mass transfer, gas sparging was required, and the TMP required to maintain a constant permeate flux decreased as the gas sparging flow increased (Bérubé et al. 2006). Berube et.al found that the TMP required to maintain a constant flux was twice as high when the total solids concentration in the digester was 35 g/L compared to 7 g/L.

TMPs that have been reported in the literature are summarized in Table 6 and range from less than 30 kPa to 375 kPa. The highest TMP was associated with a relatively low cross-flow velocity of 0.75 m/s. Membranes may be operated with a constant TMP and a varying flux, or a constant flux with a varying TMP.

2.3.3.5 Flux

Flux is the measurement of mass or volume transfer through the membrane surface. Maintaining a constant and consistent flux is one of the most significant factors affecting capital and operating costs associated with membrane operations. The flow rate of permeate can be calculated using the flux values and Equation 4.

Equation 4 $\boldsymbol{Q_p = F_w A}$ (Tchobanoglous and Burton 2003)

In Equation 4, Q_p is the permeate stream flowrate measured in kg/s, F_w is the transmembrane water flux rate measured in kg/m^2-s, and A is the membrane area measured in m^2 (Tchobanoglous and Burton 2003). If a continuous high permeate flux can be maintained, operating and capital costs can also be maintained or reduced. However, at higher fluxes the fouling rate is also typically higher and cleaning is required more frequently, which ultimately increases operating costs. It is thus necessary to maintain a balance between the operating flux and the rate of fouling.

As the suspended material accumulates on the sludge side of the membrane, the pressure typically increases and the flux decreases. As the flux decreases the recovery rate also decreases. The recovery rate is the percentage of the feed stream flow that is recovered as permeate, and this can be calculated using Equation 5.

Equation 5 $\boldsymbol{r, \% = \frac{Q_p}{Q_f} \times 100}$ (Tchobanoglous and Burton 2003)

The recovery rate is reported as a percentage, and in Equation 5 Q_f is the feed stream flow, also measured in kg/s.

There has been considerable investigation into the parameters that influence permeate flux in aerobic MBRs, however, there has been much less research regarding the parameters affecting flux in anaerobic MBRs (Bérubé et al. 2006). The operating and physical conditions of the membrane affect the flux, and changes to the conditions can alter the flux. The permeate flux can be particularly influenced by the charge of the membrane, shear stress, cross-flow velocity, gas sparging, pore sizes, and TMPs.

Permeate flux has been shown to change depending on the membrane material characteristics. The flux has been shown to be higher when the surface of the membrane is hydrophilic not hydrophobic (Bérubé et al. 2006).

Shear stress can influence the flux. Bérubé (2006) found that an increase in shear stress, caused by an increase in the cross-flow velocity, can increase the permeate flux. Padmasiri (2007) found that with the anaerobic digestion of swine manure stable operations could be achieved using an external membrane with cross flow velocities of up to 2 m/s (Padmasiri et al. 2007). However, the shear stress can also be increased through the placement of baffles, which effectively draw the mass flux away from the membrane (Bérubé et al. 2006). Elmaleh (1998) supported the use of baffles to maintain flux in a situation in which flux decline was due to a reversible particle deposition. It was found that the deposition could be completely eliminated when operating at high cross-flow using a helical baffle and a flux of 180 L/m^2-h was reached without any increase in energy requirement (Elmaleh and Abdelmoumni 1998).

Somewhat counter-intuitively when higher gas sparging rates were applied by Imasaka et.al. (1989) the permeate flux decreased continuously over time, but with low gas sparging the flux reached a pseudo steady state. This was attributed to the reduction of the cake layer that was protecting against smaller foulants that clogged the pores (Imasaka et al. 1989).

The protective cake layer can create a smaller effective pore size than the original membrane, so it follows that with larger nominal pore sizes fouling occurs more rapidly and thus the permeate flux declines more rapidly than with a smaller nominal pore size (Yang et al. 2006).

During operation with low TMP, the pressure and not the cross-flow velocity govern the permeate flux. At high TMPs the permeate flux is governed by the mass transfer of suspended solids away from the membrane (Bérubé et al. 2006). The research by Dagnew (2008) supports this, as it was found that at a flux of 8 L/m^2-h an increase in the feed concentration from 6 g/L to 18 g/L showed no impact, but at a significantly higher flux (30 L/m^2-h) the fouling increased. The increase in flux corresponded with an increase in TMP, and the concentration of mass in the digester influenced the fouling rate (Dagnew et al. 2008).

The concentration of solids in the digester can influence the flux. It has been reported that the steady-state flux decreased log-linearly with an increase in suspended solids concentration of the sludge (Bérubé et al. 2006). The results supporting this conclusion showed that at the lower solids concentrations the mass transfer away from the membrane was more than then mass transfer toward the membrane. It was suggested that the reason for this was that as the suspended solids concentration increased the viscosity also increased causing a shift from turbulent to laminar flow conditions. Eddy diffusion, which moves mass away from the membrane, was much lower under laminar flow. Pierkiel (2005) showed that flux rates using a vibrating membrane could be maintained in range of 1.-2.0 m^3/m^2-d at concentrations of up to 2% solids and for a tubular membrane flux rates were maintained in the range 3.4-3.6 m^3/m^2-d at 1% solids concentration. At these reported fluxes the tubular membranes required more frequent cleaning than that achieved with vibrating membranes (Pierkiel and Lanting 2005).

Fawehinmi et al. (2007) reported that previous studies using submerged anaerobic MBRs had a sustainable maximum permeate flux of 5 L/m^2-hr, however, through their research they were able to maintain 10 to 20 L/m^2-hr by using gas sparging to prevent fouling. In this study fouling rates were deemed to be reasonable and gas sparging was 100 times lower than the gas to liquid ratio necessary in aerobic MBRs (Fawehinmi et al. 2007).

The flux values reported in the literature, shown in Table 6, were reported for both the initial flux that corresponded to the beginning of the experiment, and the final flux after the experiment or prior to cleaning. Both of the reported values had wide ranges. Of most interest was the flux that was reported as the final flux value divided by the initial value. These ranged between 0.3 and 0.48 for tubular membranes. The reported flux values are not comparable as the numbers do not indicate

whether or not this was for stable operation, the short duration of the test, or prior to failure of the membrane.

2.3.3.6 Membrane Materials

The material that a membrane is made from dictates the lifespan and mechanical and chemical stability of the technology. Membranes can either be formed with organic or inorganic materials, and in microfiltration the most common types of materials are often made of polypropylene, acrylonitrile, cellulose acetate, nylon, and polytetrafluoroethylene.

For both organic and inorganic anaerobic MBRs precipitates such as struvite can contribute to the cake layer (Bérubé et al. 2006). For organic membranes the majority of the fouling is governed by the biological and organic interactions at the membrane surface and the cake layer formation, however, for inorganic membranes the stuvite precipitation has been reported to dominate fouling (Choo et al. 2000).

The effect of acidic backwashing also can differ between organic and inorganic membranes. In organic membranes flux was doubled after an acidic backwash but for the inorganic membranes a negative effect was observed. An inorganic membrane had improved flux after alkaline backwashing (Kang et al. 2002). Choo et al. (2000) also observed this difference between an organic polymeric membrane and an inorganic ceramic membrane.

2.3.3.7 Cost

The implementation of membranes as a treatment technology has associated costs. Equipment, operations, and maintenance costs must be evaluated. Yang et al. (2006) reported a shorter start-up time of membrane units compared to the increase of capacity in traditional treatment, as well as lower operating and maintenance costs.

Microfiltration was reported to be cost effective as it can reduce the amount of treatment chemicals required for cleaning. However, these units may also require pretreatment units which can be expensive.

Membrane units also have a 50 % to 80 % smaller footprint than traditional plants. Membrane units can be automated quite easily and this reduces labour requirements. The units do require replacement every 3 to 5 years, which is an added expense (Tchobanoglous and Burton 2003).

With repect to energy requirements Fawehinmi et al. (2007) suggested that the energy required to scour an anaerobic MBR is 1 kW/m^3. This energy consumption estimate did not consider the energy requirements for the whole membrane unit, and it has been suggested that due to the high-flow recirculation pumps submerged membrane units use much less energy than external membrane units. Metcalf and Eddy (2003) suggest that microfiltration and ultrafiltration units use more energy as compared to reverse osmosis since they are high-pressure and energy intensive.

A comparative study showed that compared to external cross-flow, submerged membranes had a lower design flux, required a lower pressure, and required lower energy for filtration (Liao et al. 2006). The lower energy requirement would translate into lower costs required to operate the submerged membrane compared to the external membrane unit.

2.4 Literature Review Summary

For both the microwave pretreatment studies and the AnMBR studies, the largest issue with the body of research available is that the methods vary greatly in waste characteristics, operations, and equipment.

For the microwave pretreatment studies the previous research that has been completed has been done mostly using WAS, with household style microwaves in batch scale reactors. Like earlier research, this project was completed using WAS as feed. None of the microwave literature has reported the relationship between VS concentration and solubilization of WAS, and furthermore the definition of solubility was inconsistent. There was substantial variability from study to study with respect to the feed source, solids and hydraulic retention times, solids concentrations, and reporting of treatment and hence was difficult to establish conclusions that were consistent for all of the scenarios. It was, however, apparent that for bench scale batch anaerobic digestion of WAS, pretreatment using microwaves increases biogas production and therefore this study will consider anaerobic digestion of WAS at a pilot scale with semi-continuous feeding.

Research on the application of anaerobic membrane bioreactors to high strength feed stocks has included studies using PS, WAS, synthetic wastewater, piggery wastewater, raw wastewater, chicken slaughterhouse wastewater, Kraft condensate, and pig manure. A considerable range in biodegradability responses was observed and this was attributed to differences in the feed stock properties. The scale of the research completed has included bench and pilot scale studies, with both continuous and batch feeds. Not only did the scale and operations vary widely, but the size, operational conditions, and type of digesters differed. The difference in the characteristics typically associated with level of treatment was further complicated by the type of membrane used. The type of membrane, the pore sizes, the operational parameters, the configuration, and the materials have differed between studies.

2.5 Contribution to Existing Research

The research presented in this thesis is unique with respect to the equipment used for pretreatment and the scale of the equipment for the microwave operations as well as the equipment and operational design for the membrane operations.

Unlike earlier research, the use of a flow through, industrial scale microwave is novel. For this study the measurement of the soluble components and the influence of the microwave were measured using a change in the concentration of both volatile solids and filtered COD, which has not been done in past research. Also, the change of the filtered component is presented in a ratio of filtered to total COD concentrations, and the percent change of the volatile solids and filtered COD.

The conclusions for the membrane research presented in this thesis will be unique to the feed and operational characteristics of this study, but will build on the already documented research. The use of a submerged hollow fibre membrane in the research described in this thesis has not previously been reported for the pilot scale anaerobic digestion of WAS.

Chapter 3
Operations

3.1 Anaerobic Digestion Operations

For both the microwave studies and the membrane studies, two 550 L anaerobic digesters were operated in parallel. The digesters were maintained at a temperature between 34 ºC and 36 ºC, and an operating volume of 500 L. Both of the digesters were fed waste activated sludge (WAS) combined with thickened WAS (TWAS) from the same feed tank. The 1000 L feed tank was filled twice a week, on Tuesday and Friday mornings. The WAS used was from the Burlington Skyway Wastewater Treatment Plant. The target feed total solids content was 2 +/- 0.5 %, and if the solids content appeared too low from a visual inspection, the sludge was decanted and the feed tank was topped with additional WAS. The feed tank was kept at approximately 5 °C, by cooling with a Frigid chiller unit model 1098-3. The digesters were both fed 4 times daily, with a Moyno 2L2 feed pump.

Both the test digester and the control digester were continually mixed, This was accomplished through the use of a circulation pump (Waukesha Cherry-Burrell C-Series) that pumped the contents of the digesters from the bottom to the top third of the digester.

Key operational parameters were measured and recorded continuously and were downloaded to computers attached to the digesters. Both digesters had continuous temperature, gas production, and digester weight monitoring. The gas flow, from both digesters, was measured with a Fluid Components International ST98L gas flow meter. The mass of the digesters was used to control the feeding and wasting of the digesters, and thus the SRT and the HRT of the digesters. The digesters were placed on top of four load cells that recorded the mass. Due to the movement of the liquid inside the digesters the mass shown on the output from the load cells fluctuated around the actual mass of the digester. This fluctuation made it difficult to maintain a precise HRT and SRT in the digesters. Starting in January, digester calibrations were completed every week, comparing the actual mass wasted from the digesters with the recorded mass wasted. The results from the digester calibrations are shown in Appendix A.

The operation of the digesters began in January of 2008, however, this research project and the subsequent sampling began on June 10th, 2008. Table 10 provides a timeline of the major components of the study with the operation times relative to the starting date.

Table 10 Project Operations Dates

Operations	Start Date	End Date
Sampling	Day 0	Day 475
Low Temperature MW	Day 0	Day 128
Low Temperature MW Steady State	Day 52	Day 128
High Temperature MW	Day 210	Day 282
High Temperature MW Steady State	Day 240	Day 282
Membrane	Day 324	Day 475
Membrane Steady State	Day 405	Day 475

3.2 Microwave Operations

The microwave operations for this study began on Day 0. For the microwave operations, both of the digesters were maintained at an SRT and HRT of 15 days, by feeding 8.8 kg of WAS every 6 hours. The program managing the operations of the control digester provided mixing of the feed tank for 2 minutes prior to feeding. The digesters wasted 8.8 kg prior to the feeding of that same mass into the digester.

The test digester feed cycle included a period of 6 hours from the end of a cycle to the beginning of the next cycle. However, due to the length of time required for the microwave to heat the feed to the desired temperature, the test digester cycle became offset from the control digester by several minutes each cycle. Twice a week the starting time of the test digester was adjusted to realign the cycle start times. The synchronized start time of the digesters was important because the mixer for the feed tank was controlled by the program for the control digester. If the digesters cycles were offset, proper mixing did not occur prior to the test digester feeding, resulting in a discrepancy between the characteristics of the feed between the test and control digesters.

At the beginning of the microwave cycle, the pre-treated WAS was diverted from the test digester until it reached the target temperature. The temperature was measured by a resistance temperature detector (RTD) which was located at the digester input valve which was located at the end of an 8.2 m pipe that led from microwave unit. Once the target temperature was reached the feed was directed into the digester until the weight of the digester reached the desired constant weight.

The microwave used to heat the sludge was a 6 kW Sairem France industrial flow-through microwave which operated at a frequency of 2450 MHz. The Sairem GMP 60 KE/DC Microwave consisted of an adjustable stub and a circulator between generator output and load. The generator ran with constant power, and hence constant frequency. The power was adjusted through the positioning of the stub, which enabled the redirection of waves back to the circulator of the generator, and allowed for the reduction of power. This avoided adjustments to the magnetron, the frequency, and the spectrum which led to less variation and instability. The microwave head was made of stainless steel and included the following components as indicated by the Sairem Operations Manual:

- Magnetron mounted on a magnetron/guide transition with arc detection system
- Isolator protecting the magnetron from excessive reflected power
- Reflected power, measuring circuit
- Filament transformer
- Fan for antenna cooling
- Water Circuit cooling circuit

The schematic for the microwave operations is presented in the following figure.

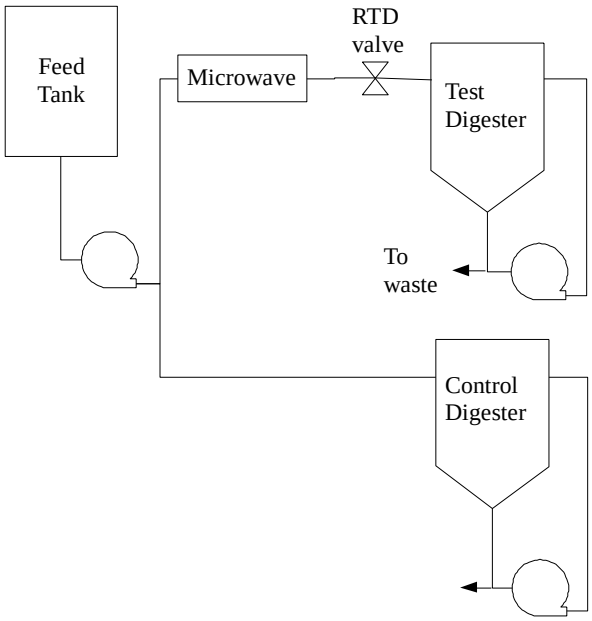


Figure 7 Process Layout for Microwave Operations

3.2.1 Sampling Schedule

Two rounds of sampling were done each week, corresponding to the refills of the feed tank. Samples were taken from the feed tank, the feed line after the microwave, and the recycle lines of both of the digesters. Samples were taken on Monday and Thursday, which was midway through the feed sludge storage time and when the surface elevation of the feed in the tank was above the mixer. A 500 mL feed sample was taken from the feed tank after the mixer had been operating for 2 minutes. Prior to sampling 4 L of the feed sludge was wasted from the feed sampling valve to eliminate solids that had settled in the base of the feed tank. A 500 mL sample of the microwaved feed was taken when the temperature of the sludge was 2 °C lower than the temperature set on the valve to allow the feed to enter the digester. The samples for the digesters were taken immediately prior to wasting. These 500 mL samples were taken from the recycle lines on the digesters.

The feed, microwaved feed, control digester and test digester samples were analyzed according to the sampling schedule detailed in Table 11.

Table 11 Microwave Operations Sampling Schedule

Characteristic	Mon	Tues	Wed	Thurs	Fri
TS	X			X	
VS	X			X	
TSS	X			X	
VSS	X			X	
COD – total	X			X	
COD - filtered	X			X	
VFA Total				X	
Acetic Acid				X	
Propionic Acid				X	
Isobutyric Acid				X	
Butyric Acid				X	
Isovaleric Acid				X	
Valeric Acid				X	
Total Alkalinity				X	
Ammonia as N				X	
Total Kjeldahl Nitrogen				X	
Soluble Total Kjeldal Nitrogen				X	

The samples were analyzed for solids and COD immediately after they were taken. The remaining analyses were conducted by the Wastewater Technology Centre Lab, and samples were immediately refrigerated and analyzed within one month. The samples submitted for acid analysis were first preserved using sulfuric acid, until the pH was below 2. The other samples were not preserved.

3.2.2 Low Temperature Operations

The sampling for the low temperature operations began on Day 0 and lasted until Day 128. For the low temperature operations the microwave temperature set point was 80 ºC. The valve that allowed the microwaved WAS to enter the test digester was opened once the WAS had reached a temperature of 67.5 ºC at the RTD sensor that was located immediately upstream of the digester at the RTD sensor that was located immediately upstream of the digester.

3.2.3 High Temperature Operations

Operation of the test digester with high temperature microwave conditions began on Day 174, and sampling began on Day 210 and lasted until Day 282. For the high temperature operations the microwave temperature set point was 95 ºC. The valve that allowed the microwaved WAS to enter the test digester was opened once the WAS had reached a temperature of 77.5 ºC.

3.3 Membrane Operations

The membrane operations began on Day 324, and the sampling of these operations lasted from Day 324 until Day 475. For the membrane operation, both the digesters were maintained at an SRT of 30 days. However, the HRT of the test digester was 15 days, while the HRT of the control digester was 30 days. The digesters were the same 500 L digesters operated in the microwave operations, however, for the test digester an 80 L ZeeWeed membrane tank from General Electric (GE) was added as a side stream unit.

The control digester was fed 4.4 kg every 6 hours. The program managing the operations of the control digester provided mixing of the feed tank for 2 minutes prior to feeding. The control digester wasted 4.4 kg prior to the feeding of that same mass into the digester. The total mass of WAS fed to the control digester was 17.4 kg per day.

The test digester was fed on a cycle with an interval of 6 hours between the end of the cycle to the beginning of the next cycle. Twice a week the starting time of the test digester was adjusted to realign the cycle start times of the two digesters to achieve proper mixing of the feed.

Prior to the wasting of the mixed liquor from the test digester, a permeation cycle occurred. In each cycle approximately 4.6 L of permeate were drawn through the membrane. Each day this resulted in 18.4 L of permeate. After permeation, the wasting cycle removed mixed liquor from the digester until the lower target mass was reached. The average mass of wasted sludge was 5.07 kg a cycle which resulted in 20.3 kg of sludge being wasted. The feed sludge was then fed into the test digester until the mass of the digester reached upper target mass. The total mass of WAS fed to the test digester per day was 38.7 kg.

The ZeeWeed unit contained a submerged hollow-fibre membrane with a total surface area of 1.07 m^2, and was controlled by a programmable logic controller (PLC). A process flow diagram is presented in Figure 8. The PLC controlled all operations of the membrane unit, including actuation of valves and pumps, and allowed for operational flexibility and continual monitoring. In addition to the continual measurement and recording of temperature, gas production and weights, the transmembrane pressure and the permeate flux were also recorded on a continual basis by the PLC.

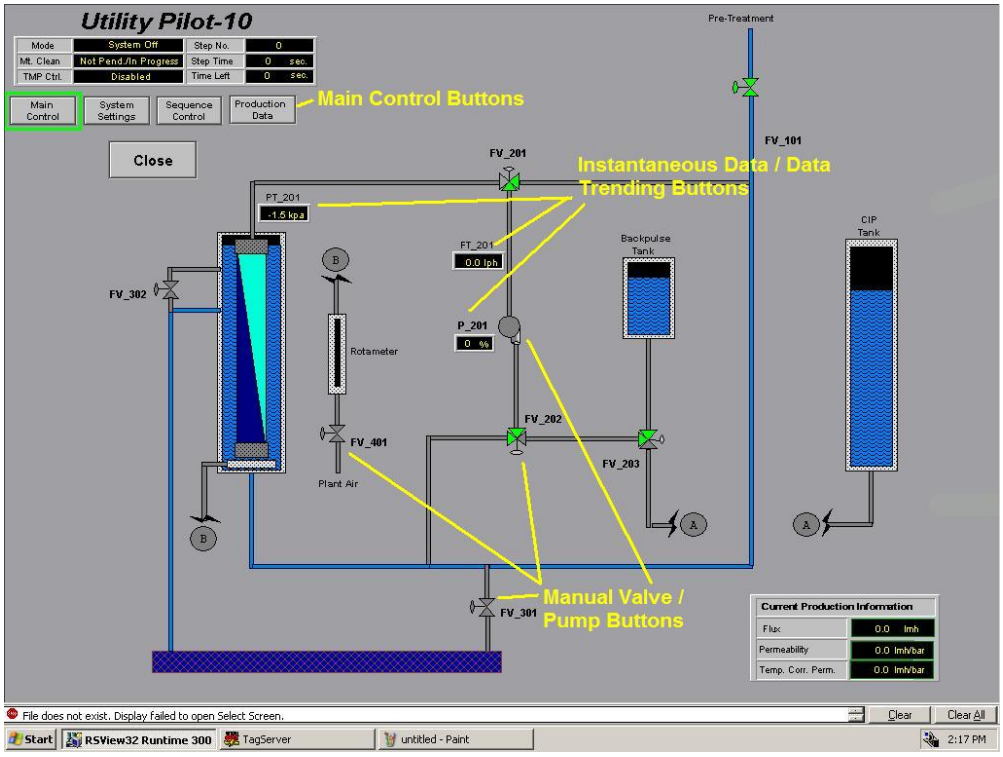


Figure 8 PLC control panel and monitoring screen

Mixed liquor from the digester was pumped to the base of the e ZeeWeed membrane tank, passed by the membrane fibres and then flowed out of the top of the membrane unit and back to the digester. The permeation cycle of the digester occurred 3 hours after the digester was fed, which was approximately half way through each cycle. This ensured that the program controlling the sludge wasting and feeding of the digester did not conflict with the permeation program. The permeation lasted for 20 mins at a permeate flow rate of 15 L/hr, and was followed by a relaxation period of 340 mins.

Gas from the digester was used to sparge the membrane. The gas was pumped from the headspace of the digester tank to the base of the membrane tank. The gas was re-circulated for 10 mins then turned off for 10 mins. The gas flow was maintained at 3 scfm. The schematic for the membrane operations is presented in the following figure.

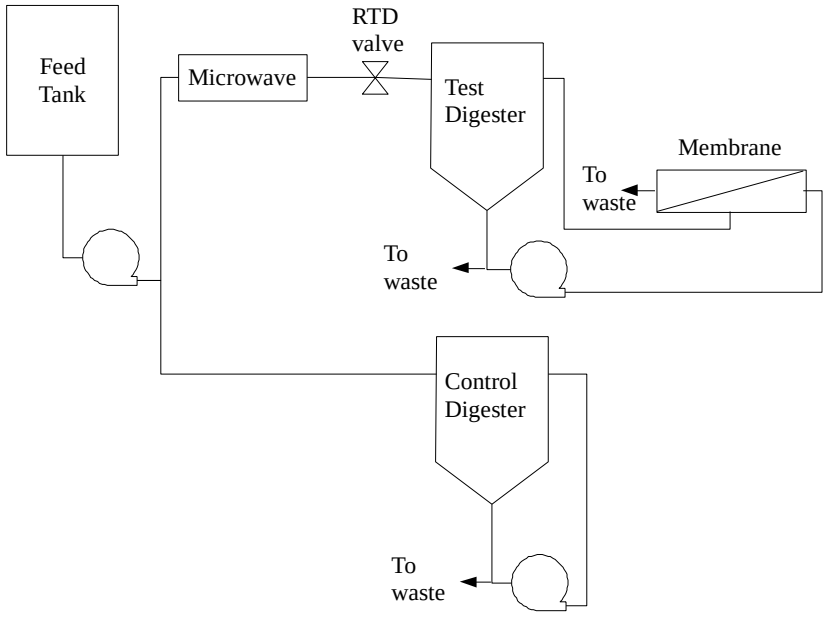


Figure 9 Process Layout for Membrane Operations

3.3.1 Sampling Schedule

Similar to the microwave operation, two rounds of sampling, corresponding to the two refills of the feed tank, were performed each week. Samples were taken from the feed tank, both of the digesters recycle lines, and from the permeate line. The sampling techniques were identical to those conducted in the microwave operations. All were 500 mL samples, and the permeate samples was retrieved immediately after the permeate was drawn from the digester. The sample from the test digester was taken after the permeate was wasted and prior to the mixed liquor wasting. The feed, control digester and test digester samples were analyzed according to the sampling schedule detailed in Table 12.

Table 12 Membrane Operations Sampling Schedule

Characteristic	Mon	Tues	Wed	Thurs	Fri
TS	X			X	
VS	X			X	
TSS	X			X	
VSS	X			X	
COD – total	X			X	
COD – filtered	X			X	
VFA Total	X			X	
Acetic Acid	X			X	
Propionic Acid	X			X	
Isobutyric Acid	X			X	
Butyric Acid	X			X	
Isovaleric Acid	X			X	
Valeric Acid	X			X	
Total Alkalinity	X			X	
Ammonia as N	X			X	
Total Kjeldahl Nitrogen	X			X	
Soluble Total Kjeldal Nitrogen	X				

The samples were analyzed for solids and COD immediately after they were sampled. Samples for analysis of the other characteristics were submitted to the Wastewater Technology Centre Lab where they were refrigerated and analyzed within one month. The samples submitted for volatile fatty acid analysis were first preserved using sulfuric acid, until the pH was below 3, while the other samples were not preserved. The Thursday samples were analyzed for solids and COD by the GE/Zenon laboratory.

3.3.2 Biogas Composition Analysis

The biogas composition analysis was completed on five separate days during the membrane operations. The analysis was completed for both the control digester and the test digester.

3.4 Analytical Methods

Samples were taken according to the sampling schedules presented in Sections 3.2 and 3.3. The samples were analyzed using the Environment Canada Wastewater Technology Analytical Laboratory guidelines, which are based on Standard Methods (Eaton et al. 2005), however, the dilutions required for the samples were determined from previous studies that used a similar lab set-up.

3.4.1 COD

Total and soluble chemical oxygen demand (COD) were measured following the Standard Methods for the Examination of Water and Wastewater (Greenberg et al. 2005). HACH COD Digestion Reagent Vials in the range of 0-1500 mg/L were used, and samples were measured on the subsequent sampling day using a HACH DR/2000 Spectrophotometer and the HACH program entitled 435 COD HR 1500 mg/L. Both the total COD samples and the filtered COD samples were completed in duplicate, to facilitate a characterization of the variability of the analyses.

3.4.1.1 Total COD

For the Total COD analysis the samples required dilution to be in the measureable range. A volume of 1 mL of sample was added to 24 mL of deionized water and then emulsified with a Brinkmann Instruments emulsifier for 1 minute. A volume of 2mL of the emulsified sample was then added to the COD vial and was mixed by rotating the vial from an upright to an upturned position several times. The vial was placed in the preheated HACH COD Reactor for 2 hours at 150°C.

3.4.1.2 Filtered COD

To measure the filtered COD the samples were also diluted. A volume of 2 mL of sample was added to 18 mL of deionized water. The diluted samples were filtered through Whatman Glass Microfibre Filters, (934-AH Circles) that had a pore size of 1.5 µm. A volume of 2 mL of the filtrate was placed into the COD vials and the contents were mixed by rotating the vial from an upright to an upturned position several times. The vial was placed in a preheated HACH COD Reactor for 2 hours at 150°C.

3.4.2 Solids

The total solids (TS), volatile solids (VS), total suspended solids (TSS), and volatile suspended solids (VSS) concentrations were measured using well documented and widely used methods (Greenberg et al. 2005).

The measurement of the mass of a sample at each stage was conducted with an Ohaus Analytical Plus balance. The aluminum tins were weighed prior to being filled with the samples. For the TSS and the VSS samples the tins were weighed with the filter paper folded and placed within them. Volumes of 10 mL of sample were placed in the tins for the TS and VS analysis. For the TSS and VSS analysis 2 mL of sample was diluted with 18 mL of deionized water, and filtered through Whatman Glass Microfibre Filters, (934-AH Circles) that had a pore size of 1.5 µm. The filter paper was placed back inside the tin. Duplicates were completed for all of the samples.

The tins were dried for 24 hours in a drying oven that was maintained at a temperature of 103-105 °C. The tins were removed and placed in a desiccator until the next sampling day, at which point they were weighed. The tins were then placed in a Fisher Scientific Isotemp Muffle Oven which had been preheated to 550 °C. The samples were left in the muffle furnace for 2 hours to burn off all of the organic material. The tins were removed from the muffle furnace and allowed to cool for 10 minutes prior to weighing.

3.4.3 Biogas

The production and the composition of biogas were determined according to the sampling schedule presented in Section 3.3. The samples were analyzed using the following methods that had been developed at Environment Canada Wastewater Technology Centre.

3.4.3.1 Production

The gas leaving of the digesters was measured using Key Instruments flow meters, that were originally designed for the measurement of air flows. The measured gas flow was recorded in mL/min, and a conversion was required for the measurement of biogas. Equation 6 presents the calculation to determine a relative K factor to calibrate the actual gas to the reference gas.

Equation 6 $$K = \frac{Q_a}{Q_r} = \frac{K_a}{K_r}$$

In Equation 6, Q_a is the flow of the biogas, Q_r is the flow of the reference gas (measured by the flow meter), K_a is the K of the biogas, and K_r is the K value for the reference gas (air) which had a value of 1. Assuming that biogas contained approximately 70 % methane, and 30 % carbon dioxide, and using the tables provided with the flow meter the resulting K value for conversion was estimated to be 0.724. Hence, Equation 7 provides the resulting equation that was employed to measure biogas.

Equation 7 $$\boldsymbol{Q_a = 0.724 \times Q_r}$$

Equation 7 was the calibration method given by the flow meter supplier to convert from air to biogas, however, further calibration was required to fine tune the flow meters and ensure that the results obtained were accurate. To fine tune the calibration of the flow meters, calibration curves were created using an additional three flow meters and the flow meter already in place. Equation 7 was combined with these equations, and the resulting calibration curves for the control and test digester were established as Equation 8 and Equation 9, respectively

Equation 8 $$\boldsymbol{Q_a = 0.724 \cdot (-0.3079{Q_r}^2 + 1.8941Q_r - 0.2326)}$$

Equation 9 $$\boldsymbol{Q_a = 0.724 \cdot (1.0852Q_r - 0.187)}$$

In these equations both Q_a and Q_r were measured in LPM. After this calibration was achieved, an adjustment to account for the inflation of the values due to the pressure from the biogas within the digester was completed. Baseline values were determined by indentifying the measured gas flow during the wasting period, when the pressure applied by the gas in the headspace was reduced. The baseline values were subtracted from the original biogas value, until the calibrated value during the wasting period reached zero. The baseline values used are presented in Table 13.

The change in baseline corresponded to the change in digester operations, as well as adjustments that were made to the mass contained in the digester. The total volume of biogas produced each cycle was the sum of the rate of gas produced per minute, multiplied by time, over the length of each cycle.

Table 13 Baseline Values for Measured Biogas Production

Control	Baseline Value (mLPM)
Day 1 - 263	85
Day 263 - 295	30
Day 356 - 385	40
Day 386 - 478	80
Test	**Baseline Value (mLPM)**
Day 1 - 51	0
Day 52 - 112	43
Day 113 - 204	70
Day 205 - 385	10
Day 386 - 478	75

3.4.3.2 Characterization

To analyze the composition of the biogas produced in the digesters, analysis was conducted with an Agilent 3000A Micro Gas Chromatograph. Table 14 and Table 15 present the operational parameters of the chromatograph.

Column A was used to detect oxygen, nitrogen and methane, while column B was used to detect carbon dioxide. A Teflon line was connected from the control and test digesters gas exhaust to the gas chromatograph. Either method A or B was selected and 6 samples were added to the queue. The analysis was initialized, and the first three samples were rejected to remove interference from the air in the sample line. The results for the last three samples were recorded.

Table 14 Gas Chromatography Column Descriptions

Channel	A	B
Injector Type	Backflush	Timed
Carrier Gas	Argon	Helium
Column Type	Molecular Sieve	Plot U
Detector Type	Thermal conductivity detector	Thermal conductivity detector
Inlet Type	heated	Heated

Table 15 Gas Chromatography Method Descriptions

Channel	A	B
Sample inlet temp. (°C)	95	95
Injector temp. (°C)	95	70
Column temp. (°C)	100	70
Sample pump	120s	120s
Inject time	30ms	30ms
Run time	120s	120s
Pressure equilibrium time	15s	15s
Column pressure	40psi	15psi
Backflush time	9.5s	-

3.4.4 Acids

The samples were analyzed for the acids, listed in Table 12, by the Environment Canada Wastewater Technology Analytical Laboratory. The results for acids were given in duplicate. The ion chromatographic method used for the measurement of VFA was Dionex Application Method 5.17, "Determination of Inorganic Anions and Low Molecular Weight Organic Acids using an IONPAC AS15-5um Column" The weakly retained anions were resolved using a 10 mM KOH solution, while the highly retained anions were eluted using a KOH gradient.

3.4.5 Nitrogen

The samples were analyzed for nitrogen by the Environment Canada Wastewater Technology Analytical Laboratory. The results for ammonia and TKN were given in duplicate, and from Day 121 onward filtered samples were submitted to obtain filtered TKN values.

3.4.5.1 Ammonia

The concentration of ammonia in the samples was measured colorimetrically using a Technicon (TRAACS 800) analyzer which had a 660 nm filter(*Technicon TRAACS 800 Method Industrial Manual no. 780-86T,* 1986) The samples were allowed to settle so that a clear aliquot could be removed for analysis. The ammonia concentrations were reported in mg/L NH_3 as N.

3.4.5.2 Total Kjeldahl Nitrogen

Both Total Kjeldahl Nitrogen (TKN) and filtered TKN concentrations were determined. The filtered samples were taken from the filtrate that had been collected for the filtered COD samples. The filters used in the analysis had 1.5 µm pore sizes. TKN was determined using a Technicon BD-40 Block Digester and a TRAACS 800 Continuous Flow Analytical System. Similarly to the ammonia analysis, the digested samples were allowed to settle prior to analysis.

3.4.6 Flux and Transmembrane Pressure

Both flux and TMP were measured on a continual basis, through the use of the PLC. A value for each characteristic was recorded every minute. The flux was measured in LMH and the TMP was measured in kPa.

Chapter 4
Results

4.1 Microwave Operations

The influence that microwaving WAS had on stability and biodegradation was assessed by analyzing the effect of the pretreatment on WAS characteristics, as well as through the destruction of organic material, the theoretical biogas production estimates, and the measured biogas production in anaerobic digestion. The theoretical biogas production estimates were based on the COD and solids destruction within the digesters to predict what the volume of biogas produced ideally would be. The influence of the microwave pretreatment was also considered as it related to the destruction of organic material and biogas production within the digesters. The concentrations of acids and nitrogen species were measured to ensure well functioning digesters.

4.1.1 Pretreatment Effects

To evaluate the influence of pretreatment on WAS properties, COD and SS were considered. Initially the total COD (COD_T) and the total suspended solids (TSS) concentrations were compared prior to and after microwave pretreatment. Filtered COD (COD_F) and VSS concentrations were then compared, for both low and high temperature conditions. Finally the COD_T and TSS concentrations were compared between the low temperature operations and the high temperature operations to determine if they were significantly different, or if the results from the two conditions could be compared directly.

4.1.1.1 COD

Samples were collected prior to and after microwaving for analysis of COD_F to permit and evaluation of the effect of microwaving on WAS. Initially consideration was given to whether the COD_T measurements for the raw feed and the microwaved feed were significantly different. If the feed and the microwaved feed were not significantly different then the COD_F values could be compared directly, however, if they were significantly different the comparison might require data adjustment. Table 16 presents a summary of the characteristics of the samples analyzed for COD.

COD values were collected 16 times during the low temperature operations. Duplicate COD analyses were taken for 10 of the 16 days, and single COD analyses were completed on the remaining 6 days. Duplicate COD analyses were taken for all 22 sampling days during the high temperature operations.

Table 16 COD Sampling Results

		Low Temperature		High Temperature	
		Raw Feed	**MW Feed**	**Raw Feed**	**MW Feed**
Samples	**Start Date**	Day 52	Day 52	Day 210	Day 210
	End Date	Day 128	Day 128	Day 282	Day 282
	Number	16	16	22	22
COD_T	**Mean**	21789 mg/L	21525 mg/L	18890 mg/L	20744 mg/L
	Std Dev	7343 mg/L	6613 mg/L	4719 mg/L	5662 mg/L
COD_F	**Mean**	775 mg/L	2307 mg/L	506 mg/L	2051 mg/L
	Std Dev	445 mg/L	645 mg/L	355 mg/L	721 mg/L

The intended method for COD sampling included duplicates for the COD samples, however, due to the occasional unavailability of COD vials there were several dates with only singular samples. The duplicate COD results were averaged to obtain a single value for each sampling day. The difference between the replicates for each COD_F sample and each COD_T sample were calculated and statistically analyzed, and it was found that the analysis for COD resulted in consistent data. The calculations for these results are presented in Appendix B.1.

The low temperature study had 16 values to analyze due to the instability of the pilot plant operations and the variability in the WAS supplied to the plant during this period. It was possible to collect more data during the higher temperature analysis because of more stable operations and 22 samples were taken.

4.1.1.1.1 Total COD Comparison for Feed and Microwaved Feed

A paired t-test was completed to compare the COD_T values of the raw and microwaved feeds. This test assessed the difference between the two sets of data. In this method the feed and microwaved samples, taken on the same day, were paired and the difference between the two values was determined. This was done for both the low temperature operation and the high temperature operation. In Appendix B.2 the raw data and the results from the t-test are presented.

The $t_{calculated}$ value for the low temperature COD_T comparison was less than the $t_{critical}$ value indicating that for the low temperature operations the COD_T sample sets of the feed and the microwaved feed were not significantly different. This was what was expected since the feed sample and the microwaved sample were taken from the same source, with the only difference being the microwave.

For the high temperature operations the COD_T sample sets for the feed and the microwaved feed were compared. The $t_{calculated}$ value was determined to be greater than the $t_{critical}$ value indicating that the feed and the microwaved feed COD_T were significantly different. It was anticipated that the high temperature comparison would have similar results to the low temperature experiment, in that the feed and the microwaved feed COD_T would not be significantly different, however this was not the case. To determine if this was sampling error a significant effect, the VS and TS were compared prior to, and after treatment. The results for this are located in Appendix B.2. These results showed that there was no significant change in the ratio of VS to TS due to microwaving, hence, it was assumed that the difference between the feed and the microwaved feed was due to analytical and sampling error, rather than an effect of the treatment.

For the low temperature operations the results of this comparison showed that the COD_T values for the feed and the microwaved feed were not significantly different, so the COD_F values for the feed and the microwaved feed samples could be compared directly, as is shown in 4.1.1.1.2. Also to compare the filtered component, the ratio of COD_F to COD_T was analyzed. Since the feed and the microwaved feed COD_T samples from the high temperature operations were significantly different the COD_F values could not be compared directly. For the high temperature operations COD_F comparison only the ratio of COD_F to COD_T was considered and is presented in 4.1.1.1.3.

4.1.1.1.2 Filtered COD Comparison for Low Temperature Operations

The average values for COD_F measurements were presented in Table 16 and from this table it can be seen that the microwaved feel samples were substantially higher than the raw feed samples. A statistical comparison was completed to determine whether microwaving had a significant effect on the filtered component of the WAS COD. A paired t-test was completed to determine whether the feed and the microwaved feed were significantly different. The details of the paired t-test are located

in Appendix B.3. The results of the paired t-test indicated that the feed and the microwaved feed COD_F values were significantly different.

To determine the difference between the mean value of the feed sample and the mean value of the microwaved feed sample an estimate of the difference between the two means was carried out. Using the Smith Satterthwaite Approximation the degrees of freedom were determined to be 27(Johnson 2000). Using a t-table and a 95% confidence interval the mean of the COD_F in the microwaved feed was found to be 1533 mg/L +/- 379 mg/L greater than that of the mean of the COD_F in the feed. The average COD_T concentration for both the raw feed and the microwaved feed was around 21000mg/L. Thus the increase of the solubilized component of the COD was approximately 7.3 % of the total.

Figure 10 displays the ratio of filtered to total COD values for the raw and microwaved feeds that were collected over the duration of the low temperature test, and the raw data is located in Appendix B.3. From Figure 10 it can be seen that there was an increase in the filtered to total COD ratio when comparing the feed to the microwaved feed. The average ratio for the feed was 0.0365+/-0.02 while the average ratio for the microwaved feed was 0.1091+/-0.02. This suggests that the soluble component of the WAS increased by 200 % due to pretreatment, during the low temperature operations.

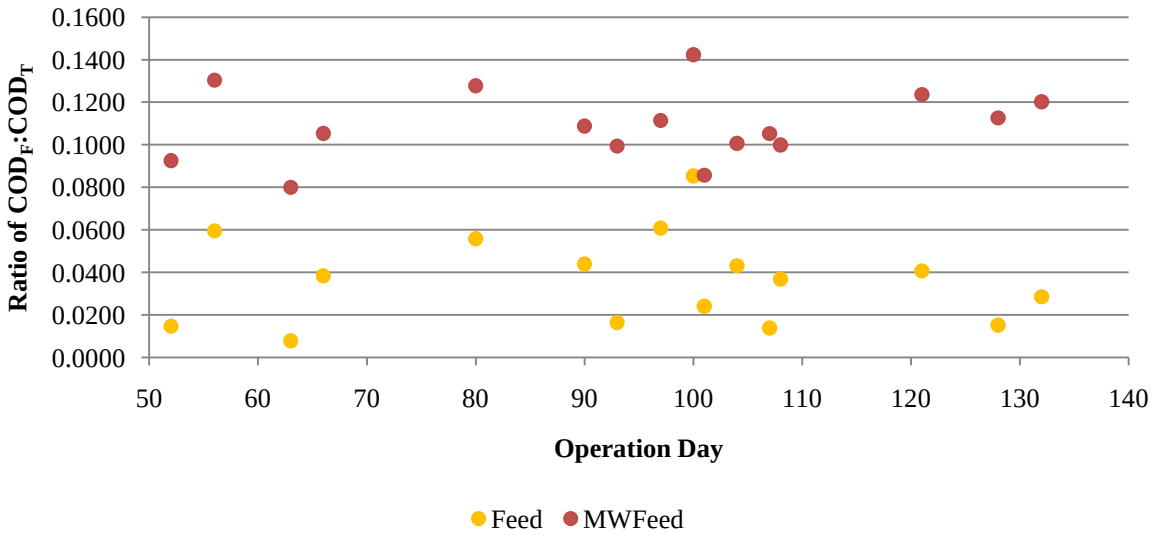


Figure 10 Ratio of Filtered COD to Total COD for Low Temperature Operations

4.1.1.1.3 Filtered COD Comparison for High Temperature Operations

The average values for COD_F measurements were presented in Table 16 and from this table it can be seen that the microwaved feel samples were substantially higher than the raw feed samples. A statistical comparison was completed to determine whether microwaving had a significant effect on the filtered component of the WAS COD. A paired t-test was completed to determine whether the feed and the microwaved feed were significantly different. The details of the paired t-test are located in Appendix B.4. The results of the paired t-test indicated that the feed and the microwaved feed COD_F values were significantly different.

To determine the difference between the mean value of the feed sample and the mean value of the microwaved feed sample an estimate of the difference between the two means was carried out. Using the Smith Satterthwaite Approximation the degrees of freedom were determined to be 31(Johnson 2000). Using a t-table and a 95% confidence interval the mean of the COD_F in the microwaved feed was found to be 1545 mg/L +/- 350 mg/L greater than that of the mean of the COD_F in the raw feed. The average COD_T concentration for both the raw feed and the microwaved feed was approximately 20000 mg/L. Thus the increase of the solubilized component of the COD was approximately 7 % of the total.

Figure 11 displays the ratio of filtered to total COD values for the raw and microwaved feeds that were collected over the duration of the low temperature test, and the raw data is located in Appendix B.3. Similar to the low temperature operations, for the high temperature operations there was also visible increase in the filtered to total COD ratio when comparing the raw feed to the microwaved feed. The average ratio for the feed was 0.0291+/-0.02 while the average ratio for the microwaved feed was 0.1032+/-0.03. This suggests that the soluble component of the WAS was increased by 254 % due to pretreatment, during the high temperature operations.

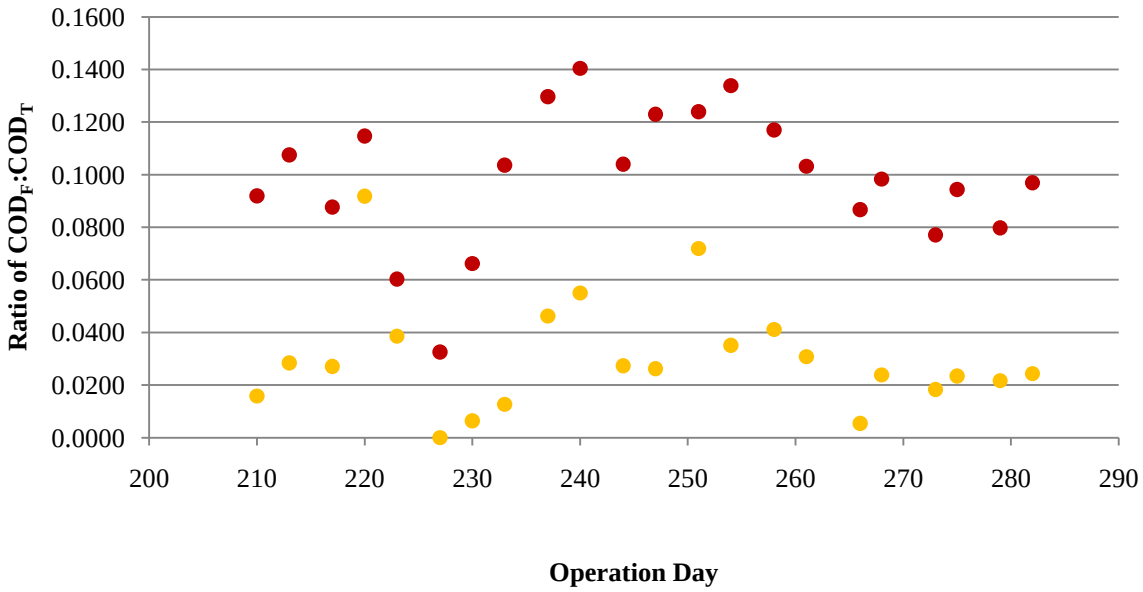


Figure 11 Ratio of Filtered COD to Total COD for High Temperature Operations

4.1.2 Low Temperature Operations

To establish and compare the control and test digesters with respect to biodegradation the destruction of COD, solids, and organic nitrogen were analyzed. The methane produced by both the control and test digester was compared to confirm the biodegradation that was exhibited within the digesters.

4.1.2.1 COD Destruction

The concentration of COD within the digesters was compared to the concentration of the feed giving an indication of the performance of the digesters. Shown in the following sections the COD_T concentrations of the feed and the control digester were paired together and the concentrations of COD_T in the microwaved feed and the test digester were paired.

The COD_T concentration of the feed and the digester contents, along with the feeding rates and digester volumes were used to determine the destruction of organic material in both the control and test digesters. Initially the COD_T mass loadings and removals were calculated, then they were graphed and the difference between them was calculated to give COD_T removal.

The daily mass flow of COD_T, entering into the digesters ($COD_{T\text{-}IN}$), was estimated as the product of the average of the bi-weekly COD_T concentrations and the daily feed flow to give a daily mass loading (Equation 10).

Equation 10 $$\boldsymbol{COD_{T-IN} = [Feed\ COD_T] \cdot Feed\ Rate \cdot \frac{1kg}{10^6 mg} \cdot \frac{1kgWAS}{1LWAS}}$$

The feed rate during the low and high temperature operations was 35.2 L/d. Similarly the mass flow exiting the digester each day ($COD_{T\text{-}OUT}$) was using Equation 11.

Equation 11 $$\boldsymbol{COD_{T-OUT} = [Digester\ COD_T] \cdot Wasting\ Rate \cdot \frac{1kg}{10^6 mg} \cdot \frac{1kgWAS}{1LWAS}}$$

The wasting rate during the low and high temperature operations was also 35.2 L/d. The removal of COD_T was determined by comparing the cumulative mass flow of feed and the cumulative mass wasting from the digesters.

Figure 12 and Figure 13, present the cumulative values of $COD_{T\text{-}IN}$ and $COD_{T\text{-}OUT}$ plotted against the operation day for the low temperature operation for the control and test digesters respectively. Linear regression was employed to estimate the slopes of the cumulative lines and these represented the average mass flows in and out of the digesters over the duration of the steady state period. All four of the lines of best fit shown in the previous two figures have high R^2 values, indicating that the lines of best fit were appropriate for the data. The destruction of COD_T in the digesters, was calculated by determining the difference between the slope of the $COD_{T\text{-}IN}$ line and the slope of the $COD_{T\text{-}OUT}$ line, then dividing by the $COD_{T\text{-}IN}$ slope. For the low temperature operations the control digester COD_T reduction was calculated to be 32.2% and for the test digester, being fed microwaved sludge, the COD_T reduction was calculated to be 38.2%, indicating that the microwaving of the WAS increased the biodegradation in the anaerobic digester.

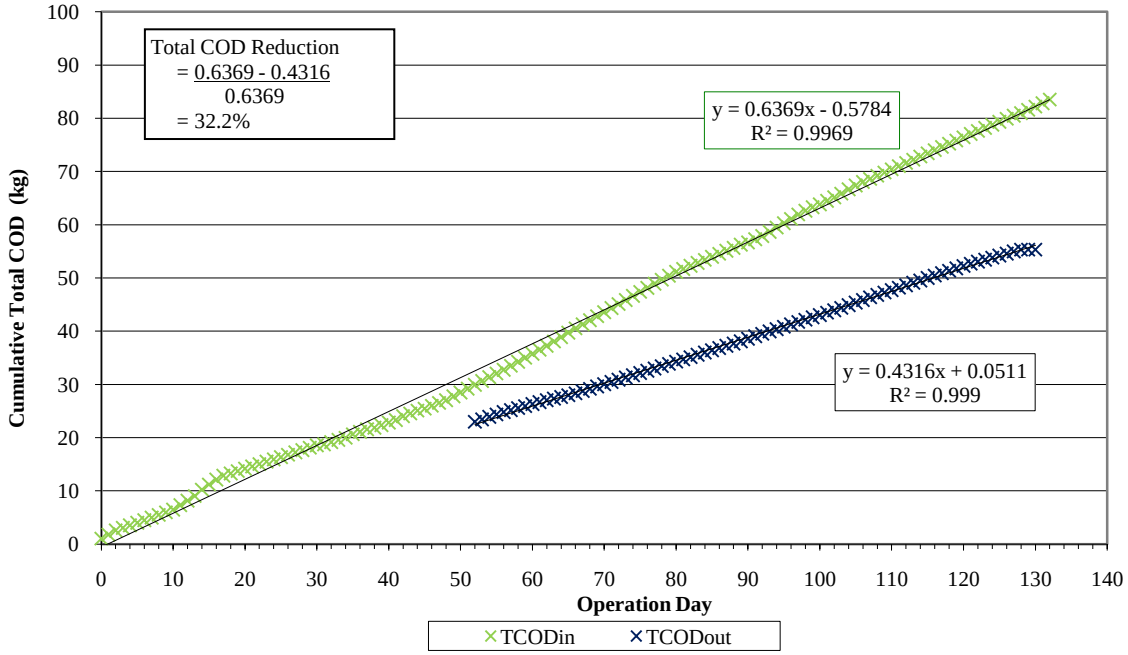


Figure 12 Total COD Destruction for Control Digester

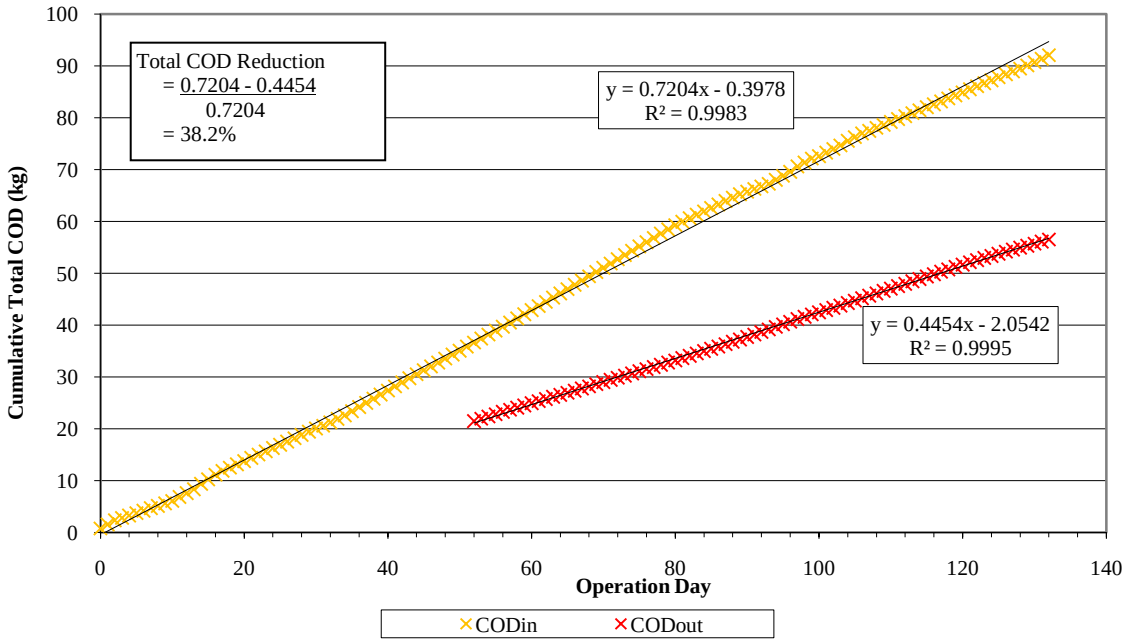


Figure 13 Total COD Destruction for Test Digester

4.1.2.2 Solids Destruction

The solids concentration gave an alternative indication of the performance of the digester. In the following graphs the solids concentrations of the feed and the control digester were paired together and the concentrations of the microwaved feed and the test digester were paired.

The volatile solids concentration of the feed and the digester contents, along with the feeding rates and digester volumes were used to determine the destruction of organic material in both the control and test digesters. Initially the volatile mass loadings and removals were calculated, then they were graphed and the difference between them was calculated to give volatile solids destruction.

The daily mass flow of volatile solids, entering the digesters (VS_{IN}), was estimated as the product of the average of the bi-weekly volatile solids concentrations and the daily feed flow to give a daily mass loading.

Equation 12 $$\boldsymbol{VS_{IN} = [Feed\ VS] \cdot Feed\ Rate \cdot \frac{1kg}{10^6 mg} \cdot \frac{1kgWAS}{1LWAS}}$$

The feed rate during the low and high temperature operations was 35.2 L/d. Similarly the mass flow exiting the digester each day (VS_{OUT}) was determined using Equation 13.

Equation 13 $$\boldsymbol{VS_{OUT} = [Digester\ VS] \cdot Wasting\ Rate \cdot \frac{1kg}{10^6 mg} \cdot \frac{1kgWAS}{1LWAS}}$$

The wasting rate during the low and high temperature operations was also 35.2 L/d.

Figure 14 and Figure 15, present the cumulative values of VS_{IN} and VS_{OUT} plotted against the operation day for the low temperature operations for the control and test digesters respectively. Linear regression was employed to estimate the slopes of the cumulative lines and these represented the average mass flows in and out of the digesters over the duration of the steady state operations. All four of the lines of best fit shown in the previous two figures have high R^2 values, indicating that the lines of best were appropriate for the data. The destruction of volatile solids in the digesters was calculated by determining the difference between the slope of the VS_{IN} line and the slope of the

VS_{OUT} line, then dividing by the VS_{IN} slope. For the low temperature operations the control digester volatile solids reduction was calculated to be 27.4% and for the test digester, being fed microwaved sludge, the volatile solids reduction was calculated to be 35.4%, and hence the digesters being fed microwaved sludge showed more biodegradation, than the control digester that was fed untreated sludge.

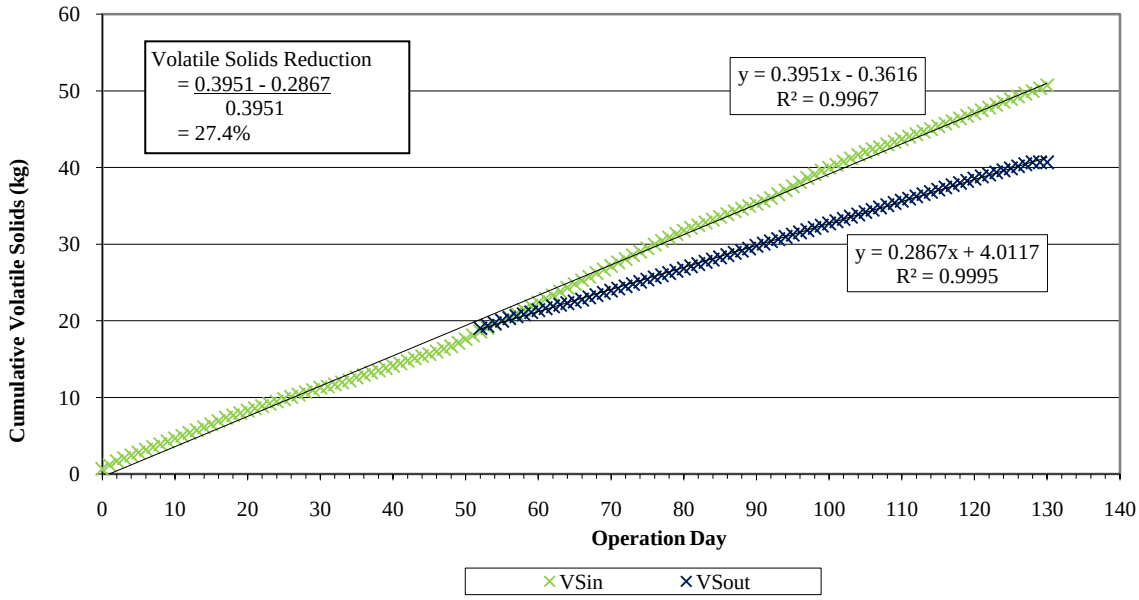


Figure 14 Volatile Solids Destruction for Control Digester

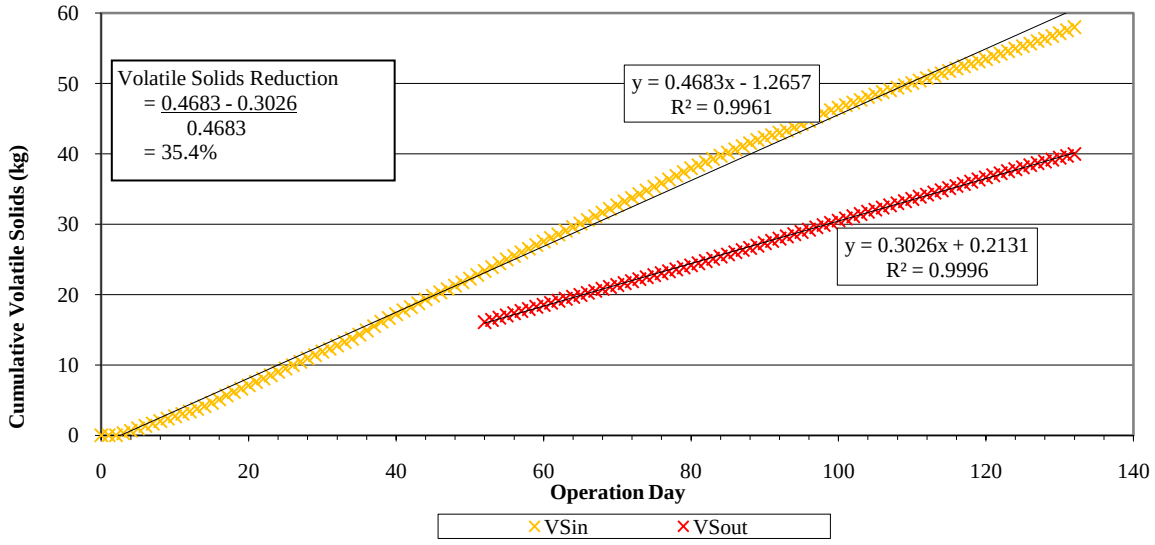


Figure 15 Volatile Solids Destruction for Test Digester

4.1.2.3 Organic Nitrogen Destruction

The concentration of ammonia (NH_3) produced within the digesters was compared to the concentration of the feed and gave an indication of the performance of the digesters. Shown in the following sections the TKN and NH_3 concentrations of the feed and the control digester were paired together and the concentrations of the TKN and NH_3 in the microwaved feed and the test digester were paired.

The TKN and NH_3 concentrations, measured in mg/L of nitrogen, of the feed and the digester contents, along with the feeding rates and digester volumes were used to determine the destruction of organic nitrogen in both the control and test digesters. Initially the TKN and NH_3 mass loadings and the NH_3 removals were calculated, and then graphed. The difference between the slopes of the $NH_{3\text{-}IN}$ and $NH_{3\text{-}OUT}$ was determined to assess the destruction of protein, and thus the creation of NH_3 within the digester. The aforementioned was divided by the difference between the TKN_{IN} and the $NH_{3\text{-}IN}$, which was the organic nitrogen removal.

The daily mass flow of NH_3, entering into the digesters ($NH_{3\text{-}IN}$), was estimated as the product of the average of the bi-weekly NH_3 concentrations and the daily feed flow to give a daily mass loading (Equation 14).

Equation 14 $$\boldsymbol{NH_{3-IN} = [Feed\ NH_3] \cdot Feed\ Rate \cdot \frac{1kg}{10^6 mg} \cdot \frac{1kgWAS}{1LWAS}}$$

The daily mass flow of TKN, entering into the digesters (TKN_{IN}), was estimated as the product of the average of the bi-weekly TKN concentrations and the daily feed flow to give a daily mass loading (Equation 15).

Equation 15 $$\boldsymbol{TKN_{IN} = [Feed\ TKN] \cdot Feed\ Rate \cdot \frac{1kg}{10^6 mg} \cdot \frac{1kgWAS}{1LWAS}}$$

The feed rate during the low and high temperature operations was 35.2 L/d. Similarly the mass flow exiting the digester each day ($NH_{3\text{-}OUT}$) was using Equation 16.

Equation 16 $$\boldsymbol{NH_{3-OUT} = [Digester\ NH_3] \cdot Wasting\ Rate \cdot \frac{1kg}{10^6 mg} \cdot \frac{1kgWAS}{1LWAS}}$$

The wasting rate during the low and high temperature operations was also 35.2 L/d. The removal of NH_3 was determined by comparing the cumulative mass flow of feed and the cumulative mass wasting from the digesters.

Figure 16 and Figure 17, present the cumulative values of TKN_{IN}, $NH_{3\text{-}IN}$, and $NH_{3\text{-}OUT}$ plotted against the operation day for the low temperature operation for the control and test digesters respectively. Linear regression was employed to estimate the slopes of the cumulative lines and these represented the average mass flows in and out of the digesters over the duration of the steady state period. All of the lines of best fit shown in the two figures have high R^2 values, indicating that the lines of best fit were appropriate for the data. The destruction of organic nitrogen in the digesters, was calculated by determining the difference between the slope of the $NH_{3\text{-}OUT}$ line and the slope of the $NH_{3\text{-}IN}$ line, then dividing the difference between the slope of the TKN_{IN} line and the slope of the $NH_{3\text{-}IN}$ line. For the low temperature operations the control digester organic nitrogen reduction was calculated to be

28.2% and for the test digester, being fed microwaved sludge, the organic nitrogen reduction was calculated to be 67.9%, indicating that the microwaving of the WAS increased the biodegradation in the anaerobic digester.

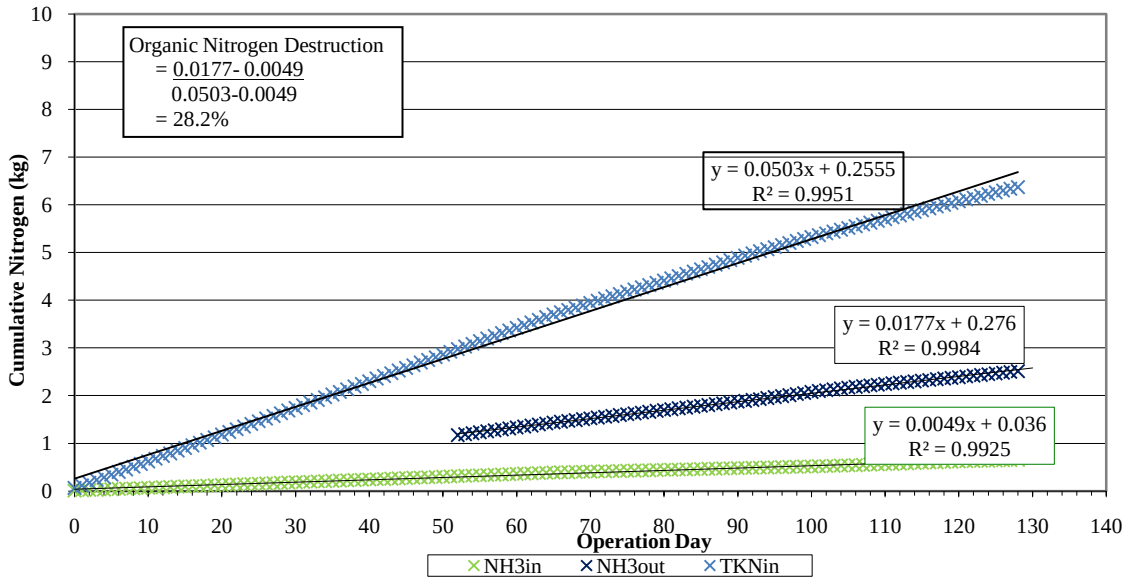


Figure 16 Organic Nitrogen Destruction for Control Digester

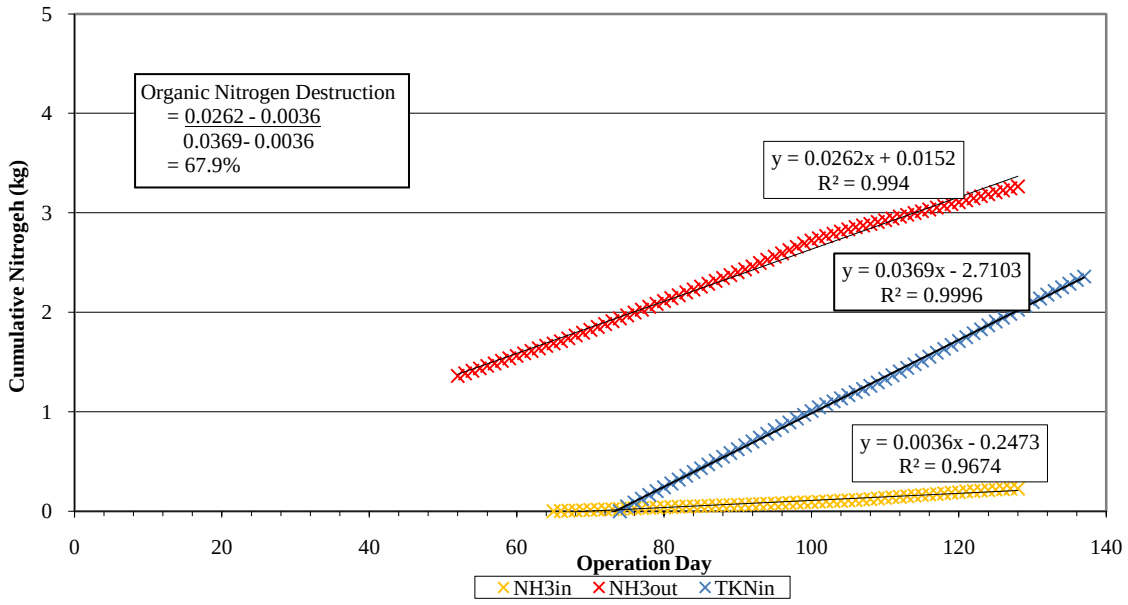


Figure 17 Organic Nitrogen Destruction for Test Digester

4.1.2.4 Measured Biogas Production

The measured biogas produced during the low temperature microwave operations was measured as a flow, however in Figure 18 the biogas production is shown as a total volume of biogas produced per cycle. The total volume of biogas produced by both the control digester and the test digester is shown.

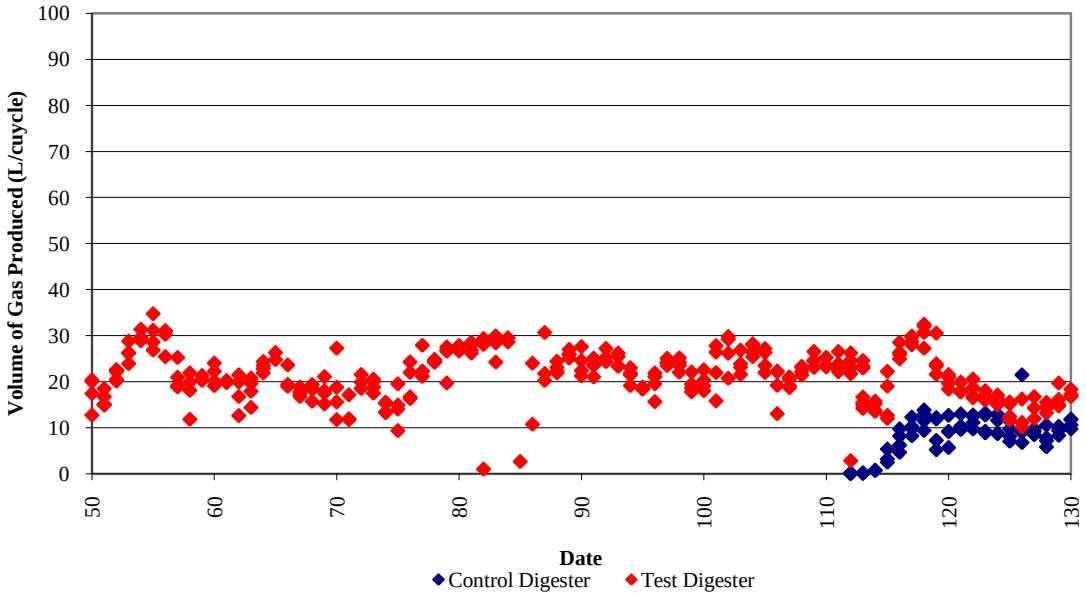


Figure 18 Measured Biogas Production

In the previous figure data was available for the test digester for low temperature operations for the entire operation; however, due to file corruption the results for the control digester were only available for the latter portion of the test. A statistical summary of the biogas values, presented in the previous figures, is shown in Table 17.

The values presented in Table 17, suggest that for the low temperature operations the test digester showed higher biogas production. The digesters showed similar variability, despite that the control digester only had 64 values to use. These results indicated that for the low temperature operations microwaving did influence the volume of biogas produced during anaerobic digestion of WAS.

Table 17 Measured Gas Production Statistics

Biogas Production (L/cycle)	Low	
	Control	Test
Mean	8.00	21.61
Std Dev	4.56	5.36
Number	64	306

4.1.3 High Temperature Operations

To establish and compare the control and test digesters with respect to biodegradation the destruction of COD, solids, and organic nitrogen were analyzed. The methane produced by both the control and test digester was compared to confirm the biodegradation that was exhibited within the digesters.

4.1.3.1 COD Destruction

The COD_T concentration of the feed and the digester contents, along with the feeding rates and digester volumes were used to determine the destruction of organic material in both the control and test digesters. Initially the COD_T mass loadings and removals were calculated, then they were graphed and the difference between them was calculated to give COD_T removal.

The calculations for the high temperature operations were the same as those completed for the low temperature microwave operations. To calculate the mass of COD_T entering the digester Equation 10 was used. The feed rate and the wasting rate during the high temperature microwave operations was 35.2 L/d for both the control digester and the test digester. The calculation to determine the COD_T leaving the digester Equation 11 was used.

Figure 19 and Figure 20, show the cumulative $COD_{T\text{-}IN}$ and $COD_{T\text{-}OUT}$ values plotted against the operation time for the high temperature operations, for the control digester and the test digesters, respectively. Similar to the low temperature operation linear regression was employed to estimate the average loadings in and out of the digesters over the test period. The best fit lines shown in the previous two figures had high R^2 values, indicating that these lines appropriately described the data.

As shown in Figure 19 and Figure 20 the COD_T destruction was calculated in the same manner that was employed for the low temperature operations. For the high temperature operations the control digester COD_T reduction was calculated to be 44.1% and for the test digester, being fed microwaved sludge, the COD_T reduction was calculated to be 48.2%, hence indicating that the microwaving pretreatment increased the biodegradability. .

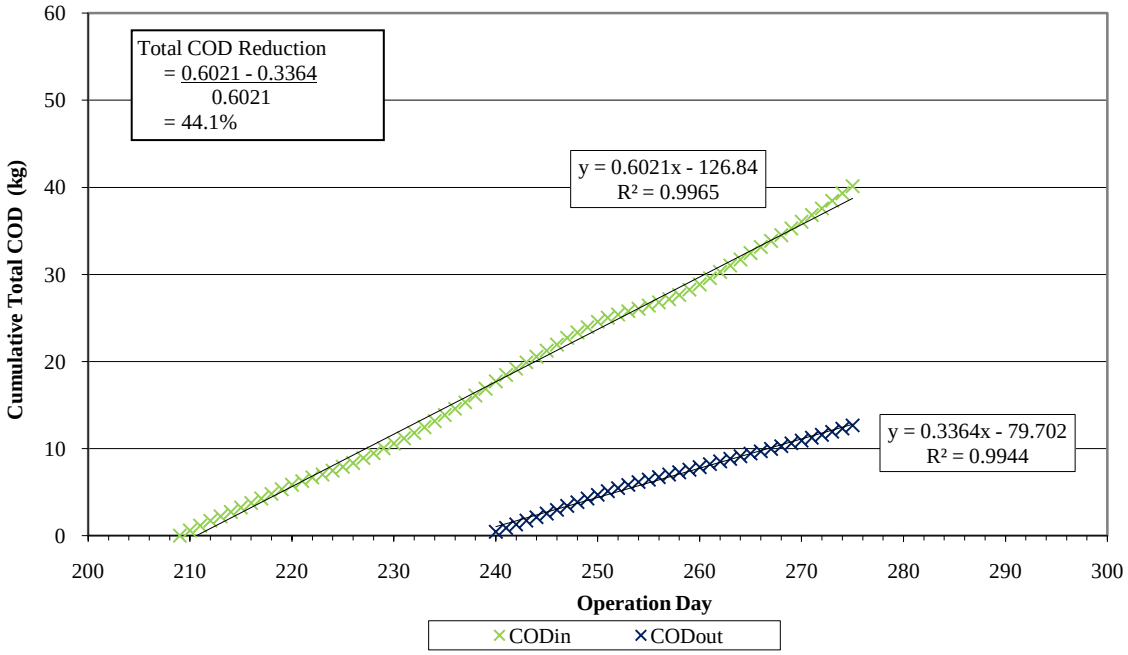


Figure 19 Total COD Destruction for Control Digester - High Temperature

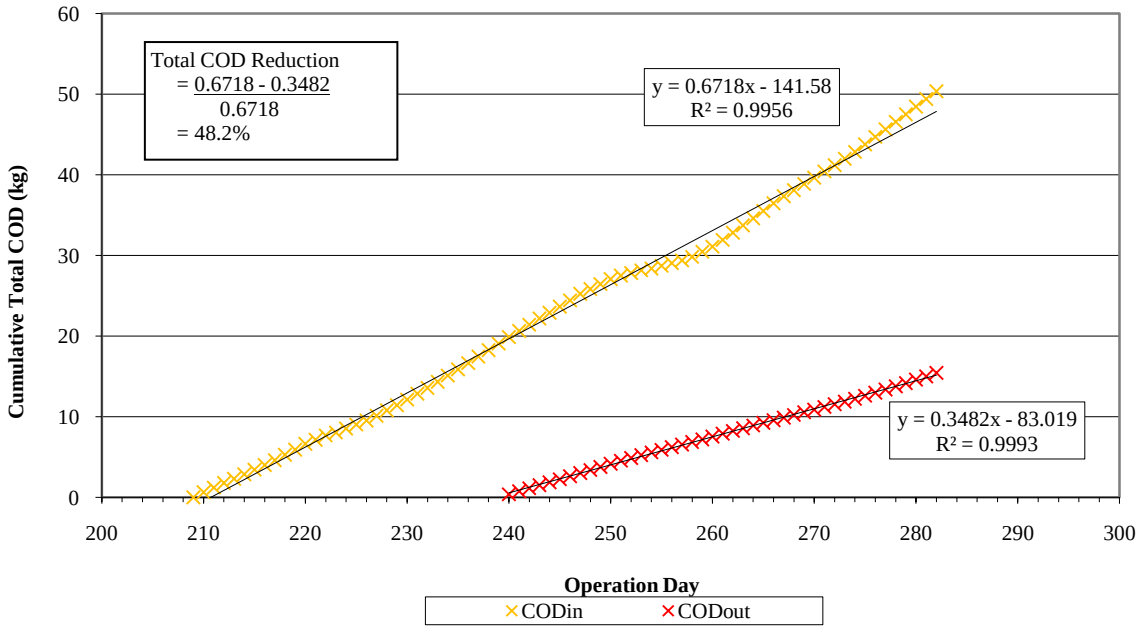


Figure 20 Total COD Destruction for Test Digester – High Temperature

4.1.3.2 Solids Destruction

The solids concentrations gave an alternative indication of the performance of the digester. In the following graphs the solids concentrations of the feed and the control digester were paired together and the concentrations of the microwaved feed and the test digester were paired. The volatile solids concentration of the feed and the digester contents, along with the feeding rates and digester volumes were used to determine the destruction of organic material in both the control and test digesters. Initially the volatile mass loadings and removals were calculated, then they were graphed and the difference between them was calculated to give volatile solids destruction.

The calculations for the high temperature operations were the same as those completed for the low temperature microwave operations. The mass of VS entering the digester was determined using Equation 12. The feed rate and wasting rate during the high temperature microwave operations were 35.2 L/d for both the control digester and the test digester. The VS leaving the digester was calculated using Equation 13.

Figure 21 and Figure 22, show the cumulative VS_{IN} and VS_{OUT} values plotted against the operation time for the high temperature operations for the control and test digesters, respectively. Similar to the low temperature operation linear regression was employed to estimate the average loadings in and out of the digesters over the test period. The best fit lines shown in the previous two figures had high R^2 values, indicating that these lines appropriately described the data. As shown in Figure 21 and Figure 22 the VS destruction was calculated in the same manner that was employed for the low temperature operations. For the high temperature operations the control digester volatile solids reduction was calculated to be 47.2% and for the test digester, being fed microwaved sludge, the volatile solids reduction was calculated to be 47.5%. These results suggest that there was no difference between the control digest and the test digester with respect to biodegradation.

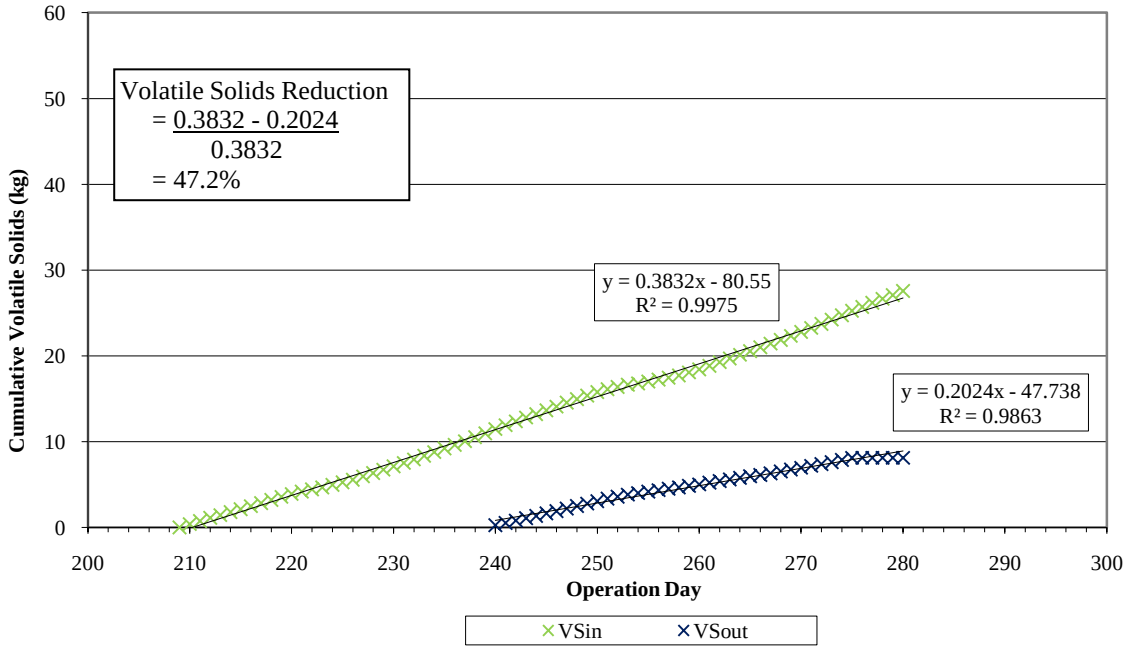


Figure 21 Volatile Solids Destruction for Control Digester - High Temperature

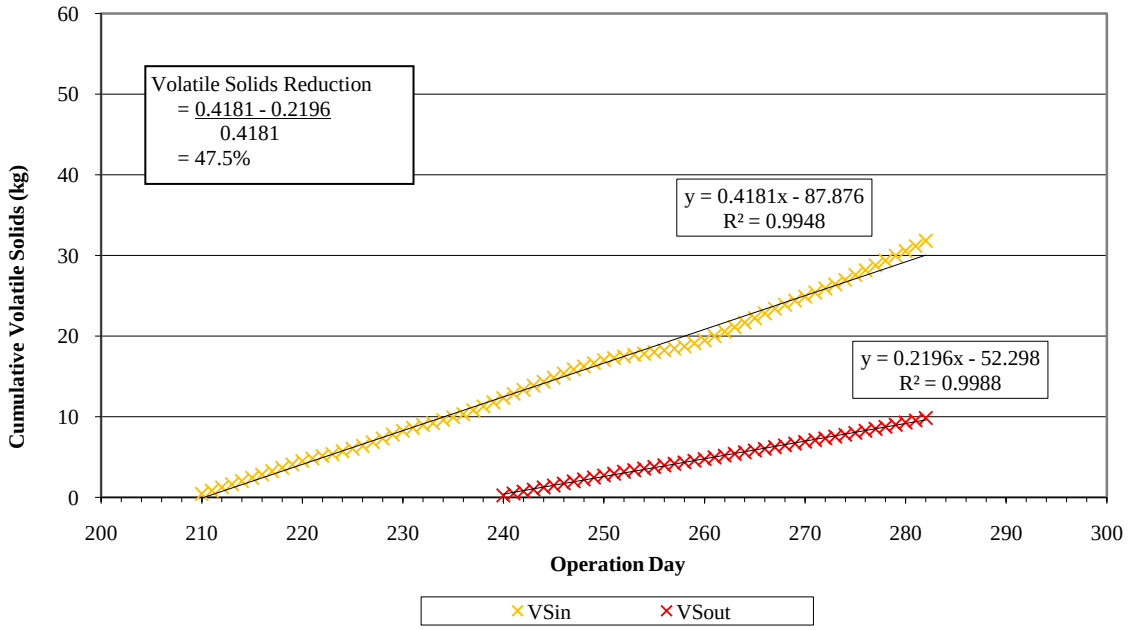


Figure 22 Volatile Solids Destruction for Test Digester – High Temperature

4.1.3.3 Organic Nitrogen Destruction

The nitrogen content gave an alternative indication of the performance of the digester. In the following graphs the NH_3 and TKN concentrations of the feed and the control digester were paired together and the concentrations of the microwaved feed and the test digester were paired. The NH_3 and TKN concentrations of the feed and the digester contents, along with the feeding rates and digester volumes were used to determine the destruction of organic nitrogen in both the control and test digesters. Initially the loadings and removals were calculated, then they were graphed and the difference between them was calculated to give organic nitrogen destruction.

The calculations for the high temperature operations were the same as those completed for the low temperature microwave operations. The masses of NH_3 and TKN entering the digesters were determined using Equation 14 and Equation 15, respectively. The feed rate and wasting rate during the high temperature microwave operations were 35.2 L/d for both the control digester and the test digester. The NH_3 leaving the digester was calculated using Equation 16.

Figure 23 and Figure 24, present the cumulative values of TKN_{IN}, $NH_{3\text{-}IN}$, and $NH_{3\text{-}OUT}$ plotted against the operation day for the low temperature operation for the control and test digesters respectively. Linear regression was employed to estimate the slopes of the cumulative lines and these represented the average mass flows in and out of the digesters over the duration of the steady state period. All of the lines of best fit shown in the two figures have high R^2 values, indicating that the lines of best fit were appropriate for the data. The destruction of organic nitrogen in the digesters, was calculated by determining the difference between the slope of the $NH_{3\text{-}OUT}$ line and the slope of the $NH_{3\text{-}IN}$ line, then dividing the difference between the slope of the TKN_{IN} line and the slope of the $NH_{3\text{-}IN}$ line. For the high temperature operations the control digester organic nitrogen reduction was calculated to be 41% and for the test digester, being fed microwaved sludge, the organic nitrogen reduction was calculated to be 47.3%, indicating that the microwaving of the WAS increased the biodegradation in the anaerobic digester.

The VS destruction comparison between the control and test digesters for high temperature operation differed from the COD results, as the COD data showed an improvement with the implementation of microwaving. It was expected that the results would be comparable for COD and VS. The organic nitrogen destruction was used to further compare the results, and the organic nitrogen results suggest that the solids destruction results could have been unreliable, as both the COD and organic nitrogen indicate that microwaving improved biodegradation.

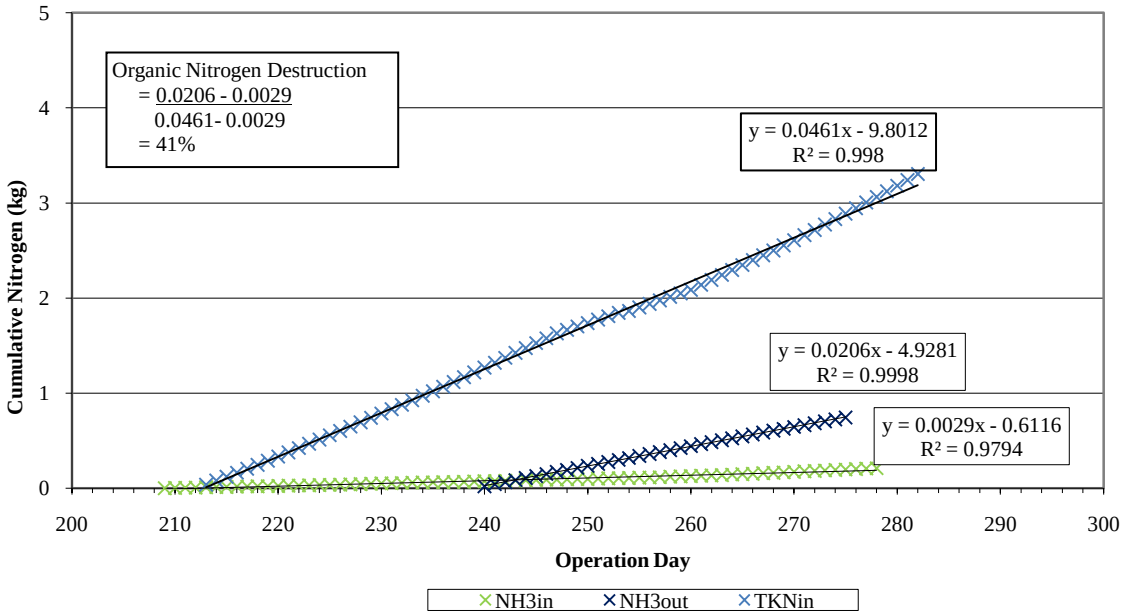


Figure 23 Organic Nitrogen Destruction Control Digester

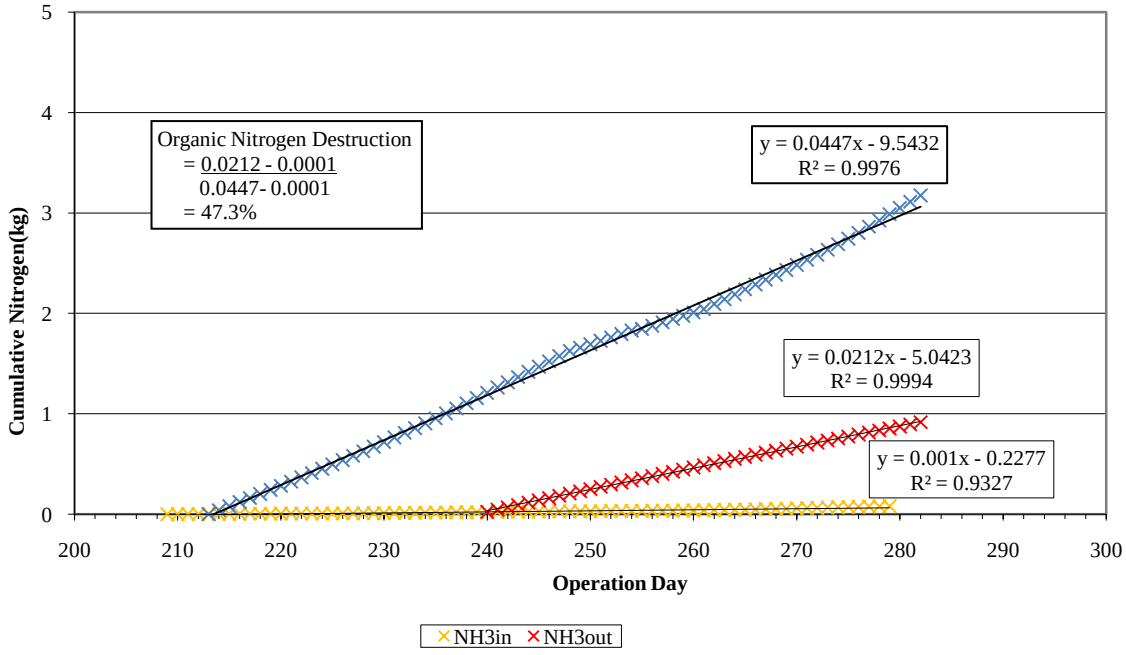


Figure 24 Organic Nitrogen Destruction Test Digester

4.1.3.4 Measured Biogas Production

The measured biogas produced during the high temperature microwave operations was measured as a flow, however in Figure 25 the biogas production is shown as a total volume of biogas produced per cycle. The total volume of biogas produced by both the control digester and the test digester are shown.

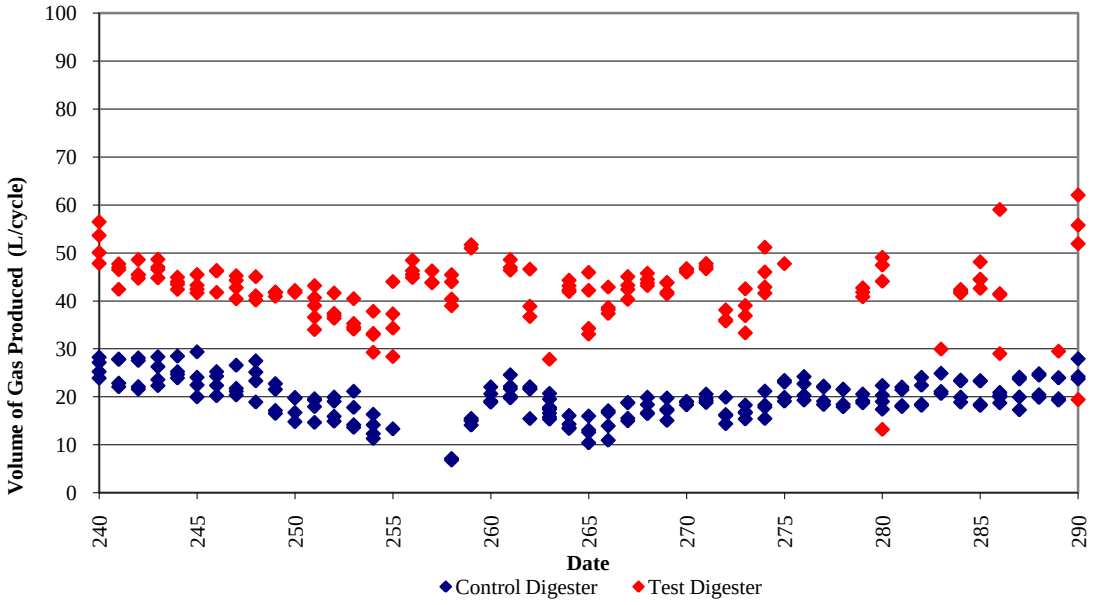


Figure 25 Measured Biogas Production

For high temperature operations, there were two points that were above 100, that were not consistent with the other data. These two data points were not shown since they appeared to be outliers. These values were not included in the statistical summary of the biogas production.

A statistical summary of the biogas values, presented in the previous figure, is shown in Table 18. The values presented in Table 18, suggest that for the high temperature operations the test digester showed higher biogas production, again indicating that the solids destruction data was not consistent with the other biodegradation indicators. The test digester performed much better, and also had less variability. These results indicated that microwaving did influence the volume of biogas produced during anaerobic digestion of WAS at the high microwave temperature.

Table 18 Measured Gas Production Statistics

Biogas Production (L/cycle)	High	
	Control	Test
Mean	20.17	42.33
Std Dev	10.66	5.55
Number	164	133

4.1.4 Theoretical Biogas

The theoretical biogas measurements were used to predict the biogas produced by the digesters, as well as compare trends between the measured biogas production and the theoretical biogas production which was based on the destruction of organics within the digesters. The theoretical biogas information was used to evaluate the validity of the measured biogas production.

4.1.4.1 Theoretical Gas Production Based on COD

To determine the production of biogas caused by the destruction of organic material in both the control and test digesters, the COD_T concentration of the feed and the digester contents, along with the feeding rates and the typical yield coefficients were used.

The COD mass loadings and removals were calculated, and they were used to establish the COD destruction in the digesters. Equation 17, Equation 18 and Equation 19 were used to calculate the theoretical gas production.

Equation 17 $$V = \frac{(1\ mole)\cdot(0.082057\ atm\cdot L/mole\cdot K)\left((273.15+35)K\right)}{1.0\ atm} = 25.29\ L$$

Equation 18 $$CH_4 Equivalent = \frac{(25.29L/mole)}{\left(\frac{64gCOD}{moleCH_4}\right)} = \frac{0.4L\ CH_4}{gCOD}$$

Equation 19 $$Vol_{CH4} = (COD_{T-IN} - COD_{T-OUT}) \cdot \frac{0.4LCH_4}{gCOD}$$

Equation 17 was used to calculate the volume occupied by 1 mole of gas at 35 °C, and Equation 18 was used to calculate the equivalent volume of methane produced for each gram of COD removed. Equation 19 was used to calculate the theoretical volume of methane produced by the digesters. $COD_{T\text{-}IN}$ and $COD_{T\text{-}OUT}$ were offset by the length of the SRT, to estimate the COD_T destruction over that time. To estimate the total volume of biogas produced by the digesters Equation 20 was used.

Equation 20 $$Vol_{Biogas} = Vol_{CH4} \cdot \frac{1LBiogas}{0.65LCH_4}$$

Equation 20 was used to calculate the volume of biogas. The estimate that the percentage of methane in the gas was 65 % of the total came from Metcalf and Eddy (Tchobanoglous and Burton 2003). The volume of biogas produced per day was divided by 4 to estimate the theoretical biogas produced per cycle. Figure 26 and Figure 27 show the theoretical biogas production per cycle for the low and high temperature microwave operations, respectively.

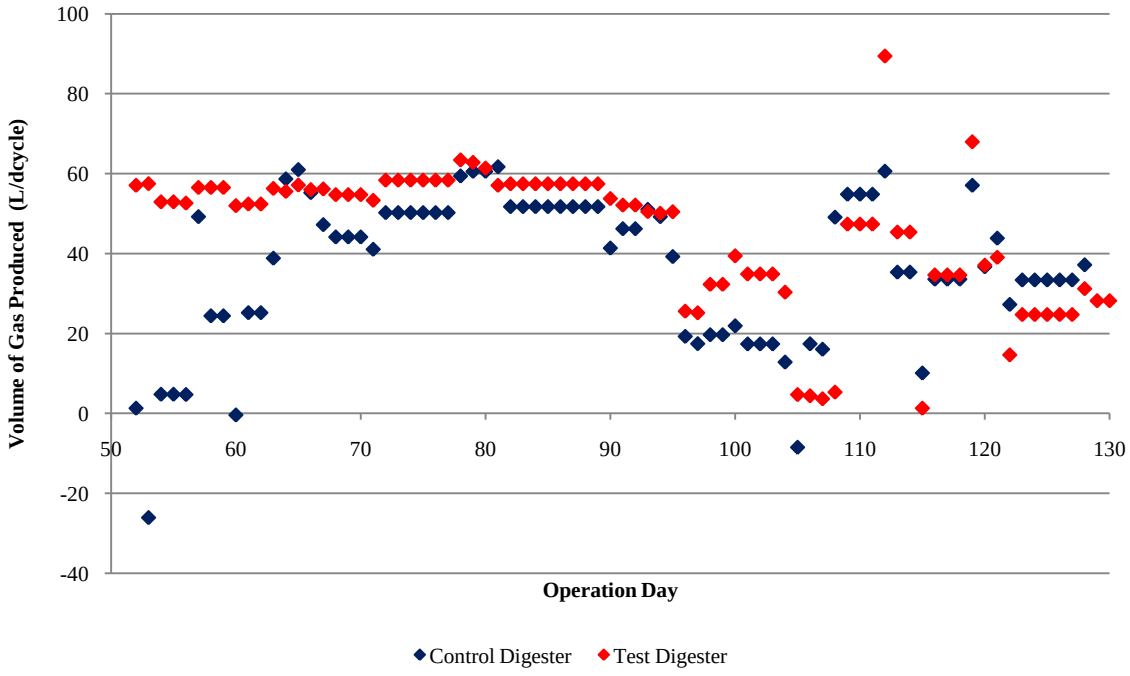


Figure 26 Theoretical Biogas Production based on COD destruction – Low Temperature

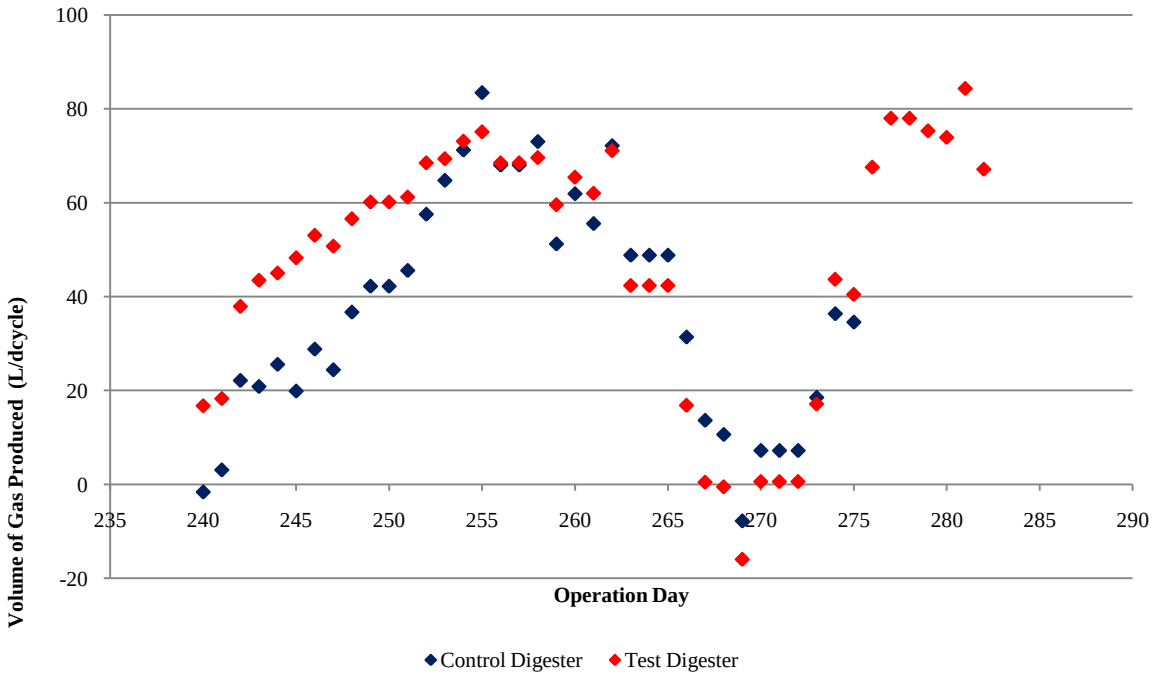


Figure 27 Theoretical Biogas Production based on COD destruction – High Temperature

The theoretical biogas production data shown in the previous figures show scatter. The variability of the feed COD_T values resulted in variability of the biogas production. The negative values of biogas production shown in the previous figures, suggest that the results of the estimation method could be flawed. When the influent concentrations of COD were lower than the effluent concentrations the nature of Equation 19 resulted in negative results, or a creation of COD within the digester, which in fact was not the case. The variability of the feed greatly influenced the ability to produce consistent and reliable estimates. The values presented in the previous figures for both low and high operations are summarized in Table 19.

Table 19 Theoretical Gas Production Statistics (COD)

Biogas Production (L/cycle)	**Low**		**High**	
	Control	**Test**	**Control**	**Test**
Mean	36.59	44.68	37.24	47.85
Std Dev	18.81	17.00	24.34	26.78

For both the low temperature operations and the high temperature microwave operations the test digester has a higher average biogas production per cycle than the control digester. The standard deviations were similar between the control and test digesters. These theoretical estimations supported the conclusions made through the analysis of measured biogas values.

4.1.4.2 Theoretical Gas Production Based on Solids

To determine the production of biogas caused by the destruction of organic material in both the control and test digesters, the VSS concentration of the feed and the digester contents, along with the feeding rates and the typical yield coefficients were used.

The VSS mass loadings and removals were calculated, and they were used to establish the destruction in the digesters. Equation 17 and Equation 18 were used to calculate the volume of 1 mole of gas at 35 °C and the methane equivalent for each gram of COD removed. The estimation of the theoretical biogas produced required the conversion of VSS removed to COD removed.

Equation 21 $$\boldsymbol{Vol_{CH4} = (VSS_{IN} - VSS_{OUT}) \cdot \frac{0.4 L CH_4}{gCOD} \cdot \frac{1.42 gCOD}{gVSS}}$$

Equation 21 was used to calculate the volume of methane produced by the digesters. VSS_{IN} and VSS_{OUT} were offset by the length of the SRT, to estimate the VSS destruction over that time. To estimate the total volume of biogas produced by the digesters Equation 20 was used. The estimate that the percentage of methane in the gas was 65 % of the total came from Metcalf and Eddy (Tchobanoglous and Burton 2003). The volume of biogas produced per day was divided by 4 to estimate the theoretical biogas produced per cycle. Figure 28 and Figure 29 show the theoretical biogas production per cycle for the low and high temperature microwave operations, respectively.

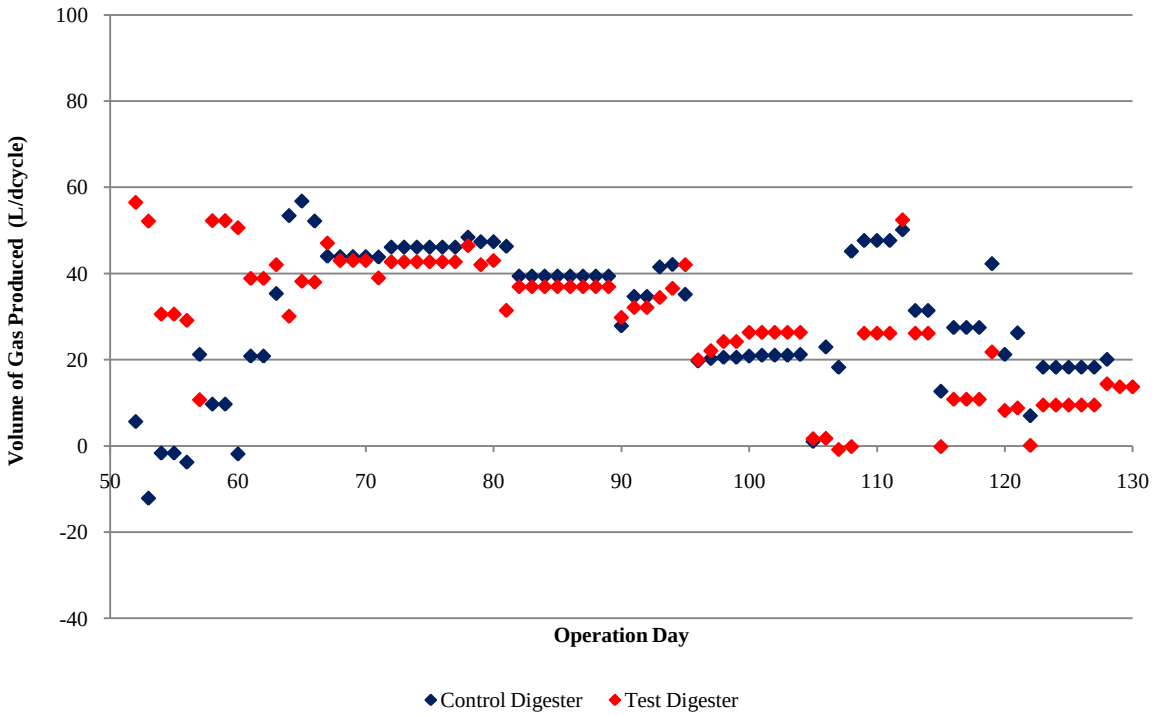


Figure 28 Theoretical Biogas Production based on VSS destruction – Low Temperature

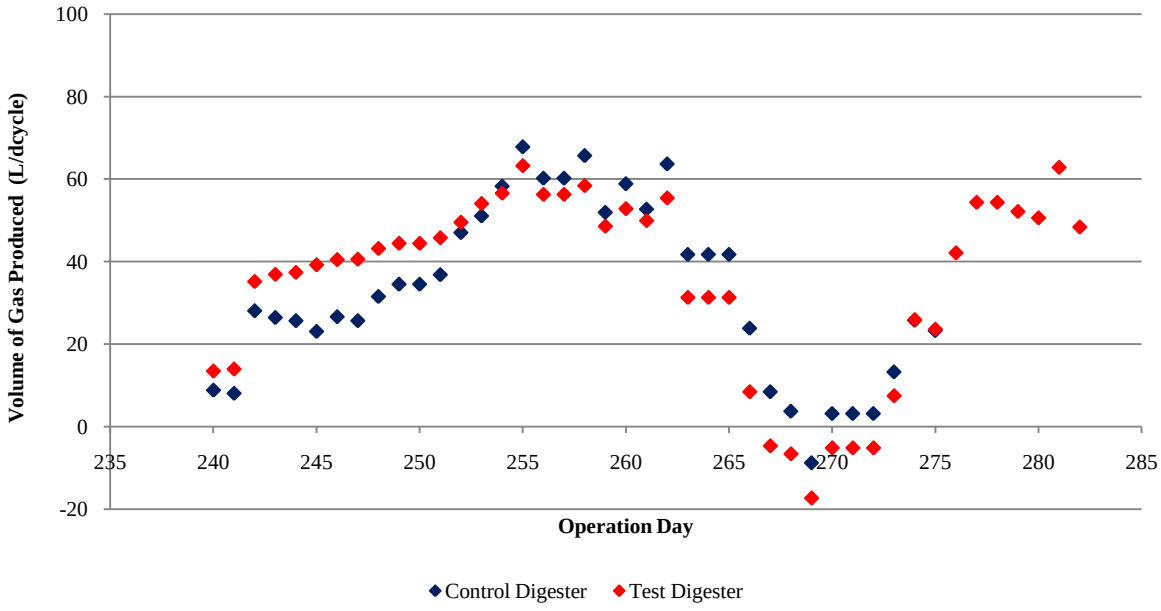


Figure 29 Theoretical Biogas Production based on VSS destruction – High Temperature

The theoretical biogas production data shown in the previous figures show less variability than the biogas estimated with the COD removal. The theoretical biogas production estimates for the low temperature operations showed less variability than the high temperature operations. The variation in the VS concentration of the feed resulted in a decrease in the VS within the digester, as shown in Appendix D, and lower theoretical biogas production. The negative values of biogas production shown in the previous figures, suggest that the results of the estimation method could be flawed. When the influent concentrations of VSS were lower than the effluent concentrations the nature of Equation 21 resulted in negative results, or a creation of VSS within the digester, which in fact was not the case. The variability of the feed greatly influenced the ability to produce consistent and reliable estimates. The values presented in the previous figures for both low and high operations are summarized in Table 20.

Table 20 Theoretical Gas Production Statistics (VSS)

Biogas Production (L/cycle)	Low		High	
	Control	Test	Control	Test
Mean	30.07	28.66	32.53	35.28
Std Dev	15.99	15.00	21.05	22.17

For the low temperature operations the control digester had a greater theoretical biogas production per cycle. For the high temperature microwave operations the test digester had a higher average biogas production per cycle than the control digester. The standard deviations were similar between the control and test digesters for both the low and high microwave operations.

4.1.4.3 Comparative Theoretical to Measured Biogas Production

Compared to the theoretical biogas production, the measured gas production results during both the low and high temperature operations were lower. The measured biogas results clearly indicated that the test digester was producing more biogas, compared to the control digester. The trends between the theoretical and measured data were not similar. The measured results were more consistent, showing a more linear pattern than the theoretical results. This was further supported by the statistical analysis. For the theoretical biogas production estimates the standard deviations of each set

of data ranged between 15 and 28 L/cycle, where as for the measured production three of the 4 standard deviations were below 6 L/cycle and the fourth was lower than 11 L/cycle.

4.1.5 Volatile Fatty Acids

The results for the measurement of the VFA in the digesters is presented in Appendix F. Poor operations can lead to a system imbalance, which is indicated by high volatile acids concentrations (Parkin and Owen 1986). VFA concentrations that are above 2000 mg/L have been shown to be inhibitory to methanogens (Grady Jr. et al. 1999). The results indicated in Table 21 show that appropriate concentrations were always far below that inhibitory level for both the control and the test digester.

Table 21 VFA Concentrations Statistics – Microwave Operations

Concentration	Low		High	
(µg/mL)	Control	Test	Control	Test
Mean	27	51	109	128
Std Dev	7	37	43	71
Min	18	18	39	31
Max	41	143	175	213
Number	8	10	8	8

4.1.6 Nitrogen

The results for the measurement of the ammonia in the digesters is presented in Appendix G. The results indicated appropriate concentrations within the both the control and the test digester. The summary of these results is shown in Table 22. If the concentration of NH_3-N was between 1500 mg/L and 3000 mg/L within the digesters the fermentation could be inhibited, reducing the functionality of the digesters (Parkin and Owen 1986). During the length of the microwave operations none of the of digesters had ammonia-N concentrations greater than 1010 mg/L, and the averages, which are shown in Table 22, ranged between 540 and 735 mg/L.

Table 22 Ammonia Concentration Statistics – Microwave Operations

Concentration	Low		High	
(mg/L-N)	Control	Test	Control	Test
Mean	540	735	591	601
Std Dev	132	185	27	48
Min	370	442	547	518
Max	711	1010	627	671
Number	8	10	8	8

4.1.7 Low versus High Temperature Operations

For the low temperature operations the COD_T destruction was 6% higher for the test digester, indicating that with low temperature microwaving the digester was capable of more biodegradation. For the high temperature operations the percent destruction was also higher for the test digester, but only by 4.1%. The difference in destruction between the control digester and the test digester was not considerably different between the low temperature operations and the high temperature operations, however, the percentage COD_T destruction at the higher temperature operations were higher than those at the lower temperature operations. During the low temperature operations, the continuity of the feed WAS characteristics was not optimal and the TS concentration of the sludge had a larger standard deviation than during the high temperature operations. Compared to the feed during the high temperature operations, during the low temperature digester operations the feed to the control digester had a total solids concentration standard deviation that was 2700 mg/L higher than during the high temperature operations. This variability in the control digester feed could have accounted for non-ideal operating conditions within the digester, such as lower biomass concentrations and lower organic loading.

For the low temperature operations the volatile solids destruction was 8% higher for the test digester, indicating that with low temperature microwaving the digester was capable of more biodegradation. For the high temperature operations the percent destruction was almost identical for the control and test digesters suggesting that there was no difference in the solids destruction. This could be attributed to the shorter time period of the steady state operations, compared to the lengthier low temperature operations

For the low temperature operation the test digester showed organic nitrogen reduction that was 140 % higher than that of the control digester. For the high temperature operations an increase of 15 % from the control digester to the test digester was seen, suggesting that the low temperature operations showed better biodegradation, however, the inconsistency of the feed decreases the validity of this comparison.

The measured biogas production for the low temperature operations indicated that the test digester produced 13.61 L/cycle more biogas than the control digester. For the high temperature operations the test digester produced 22.16 L/cycle more than the control digester. These results are difficult to compare as the feed characteristics varied from low to high temperature, but both low and high operations showed an increase in biogas production with the application of microwave pretreatment.

4.2 Membrane Operations

The influence that the introduction of a submerged hollow fibre membrane, to convert the test digester to an anaerobic membrane bioreactor configuration, had on process stability and biodegradation was assessed by the destruction of organic material, theoretical biogas production, measured biogas production, as well as the characteristics of the biogas produced. The influence of the membrane bioreactor operation was also considered as it related to the biodegradation of organics and the concentrations of acids and nitrogen species. In addition, the membrane performance was assessed over the duration of the study to determine the critical flux and the influence of fouling on the unit.

4.2.1 COD Destruction

The concentration of COD_T within the digesters compared to the concentration of the feed to obtain an indication of the performance of the digesters. In the following sections the COD_T destruction achieved by both the control and test digesters, as well as the theoretical volume of biogas produced through that destruction will be presented and compared.

The COD_T concentrations of the feed and the digester contents, along with the feeding and wasting rates and digester volumes were used to determine the destruction of organic material in both the

control and test digesters. Initially the COD_T mass loadings and removals were calculated, then they were graphed and the difference between them was calculated to give COD removal.

The calculations for the membrane operations were the same as those completed for the microwave operations. To calculate the mass of COD_T entering the digester Equation 10 was used. The feed rate during the membrane operations was 17.6 L/d for the control digester and 38.7 L/d for the test digester. The calculation to determine the COD_T leaving the digester Equation 11 was used. The wasting rates used were 17.6 L/d for the control digester and 20.3 L/d for the test digester. The permeate from the test digester was also wasted at a rate of 18.4 L/d, however, it was assume that the COD_T concentration of the permeate was 0 mg/L, as sampling was completed for the permeate and the values measured for COD_T were only 1.4% of the total COD_T in the membrane digester.

Figure 30 and Figure 31, present the cumulative values of $COD_{T\text{-}IN}$ and $COD_{T\text{-}OUT}$ plotted against the operation day for the membrane operations for the control digester and the test digester, respectively. Linear regression was employed to estimate the slopes of the cumulative lines and these represented the average mass flows in and out of the digesters over the duration of the steady state period. All four of the lines of best fit shown in the previous two figures have high R^2 values, indicating that the lines of best fit are appropriate for the data. The destruction of COD_T in the digesters was determined as the difference between the slope of the $COD_{T\text{-}IN}$ line and the slope of the $COD_{T\text{-}OUT}$ line, divided by the $COD_{T\text{-}IN}$ slope. The control digester COD_T reduction was calculated to be 44.4% and for the test digester the COD_T reduction was calculated to be 48.2%. Hence, the COD_T removal accomplished by the test digester, which was connected to a membrane unit, achieved slightly higher COD_T removal. The membrane digester was capable of removing more COD_T than the control digester, while maintaining a higher throughput. The daily quantity of sludge fed to the test digester was almost 2.2 times great than that to the control digester, and yet the removal rate of the test digester exceeded the performance of the control digester.

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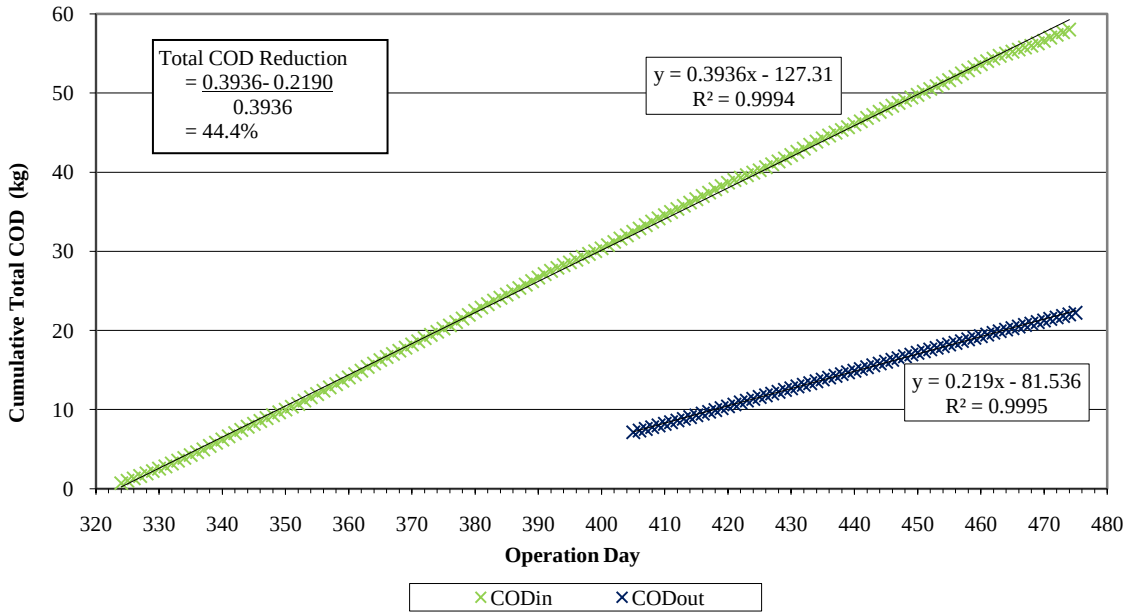

Figure 30 Total COD Destruction for Control Digester – Membrane Operations

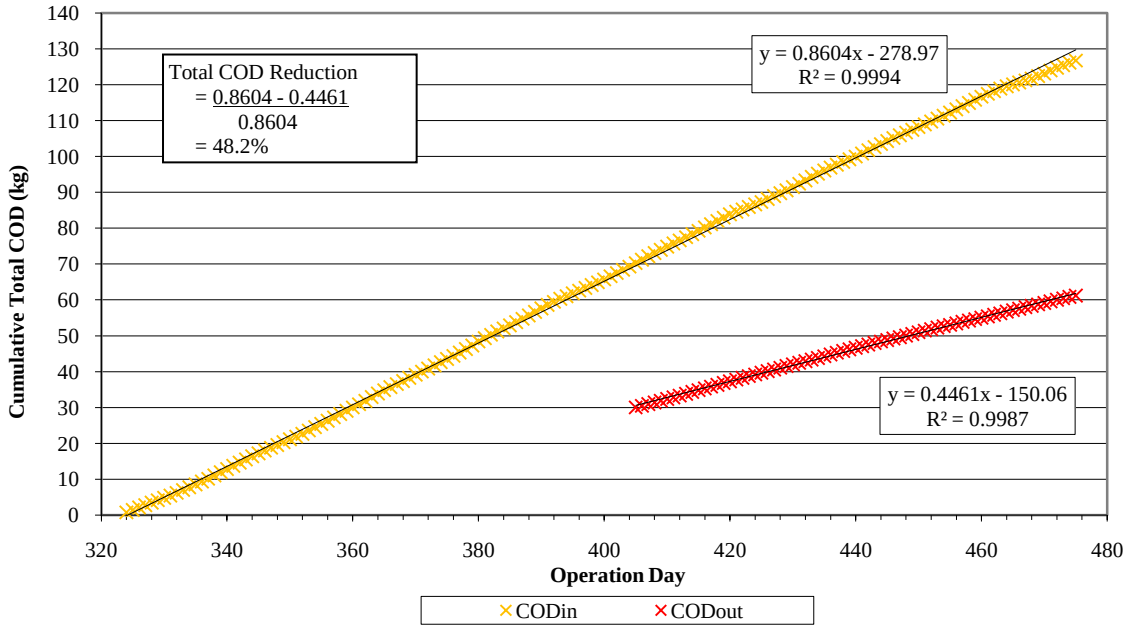

Figure 31 Total COD Destruction for Test Digester – Membrane Operations

4.2.2 Solids Destruction

In the following section the solids concentrations of the feed are compared with the solids concentrations in the digesters to determine the degree of biodegradation. Both the destruction of the solids and the theoretical biogas produced through this destruction will be calculated and compared for the control and test digesters.

The VS concentrations of the feed and the digester contents, along with the feeding rates and digester volumes were used to determine the destruction of organic material in both the control and test digesters. Initially the volatile mass loadings and removals were calculated, then they were graphed and the difference between them was calculated to give volatile solids destruction.

The calculations for the membrane operations were the same as those completed for the microwave operations. The mass of VS entering the digester was determined using Equation 12. The feed rate during the membrane operations was 17.6 L/d for the control digester and 38.7 L/d for the test digester. The VS leaving the digester was calculated using Equation 13. The sludge wasting rates used were 17.6 L/d for the control digester and 20.3 L/d for the test digester. The permeate from the test digester was also wasted at a rate of 18.4 L/d, however, it was assume that the VS concentration of the permeate was 0 mg/L, as the pore size of the membrane was small enough to eliminate the majority of the solids. Sampling, however, was not completed to support this assumption.

Figure 32 and Figure 33, present the cumulative values of VS_{IN} and VS_{OUT} plotted against the operation day for the low temperature operations for the control and test digester, respectively. Linear regression was employed to estimate the slopes of the cumulative lines and these represented the average mass flows in and out of the digesters over the duration of the steady state period. All four of the lines of best fit shown in the previous two figures have high R^2 values, indicating that the lines of best are appropriate for the data. The destruction of VS in the digesters, was calculated in the same manner that was done for the microwave operations. For the control digester volatile solids reduction was calculated to be 39.5% and for the test digester, with a connected membrane unit, the volatile solids reduction was calculated to be 44.6%. This increase was consistent with the COD_T destruction, which showed an increase of 5.1%, similar to the VS destruction improvement of 3.8%.

The membrane digester had 5 % higher volatile solids reduction. Both of the digesters had the same SRT of 30 days, however, the membrane allows for the retention of biomass within the digester and the digester contents. During the operations the control digester had a solids concentration around 15000 mg/L and the test digester had a solids concentration around 25000 mg/L. The test digester not only was capable of higher solids destruction, but a higher destruction with a larger volume throughput.

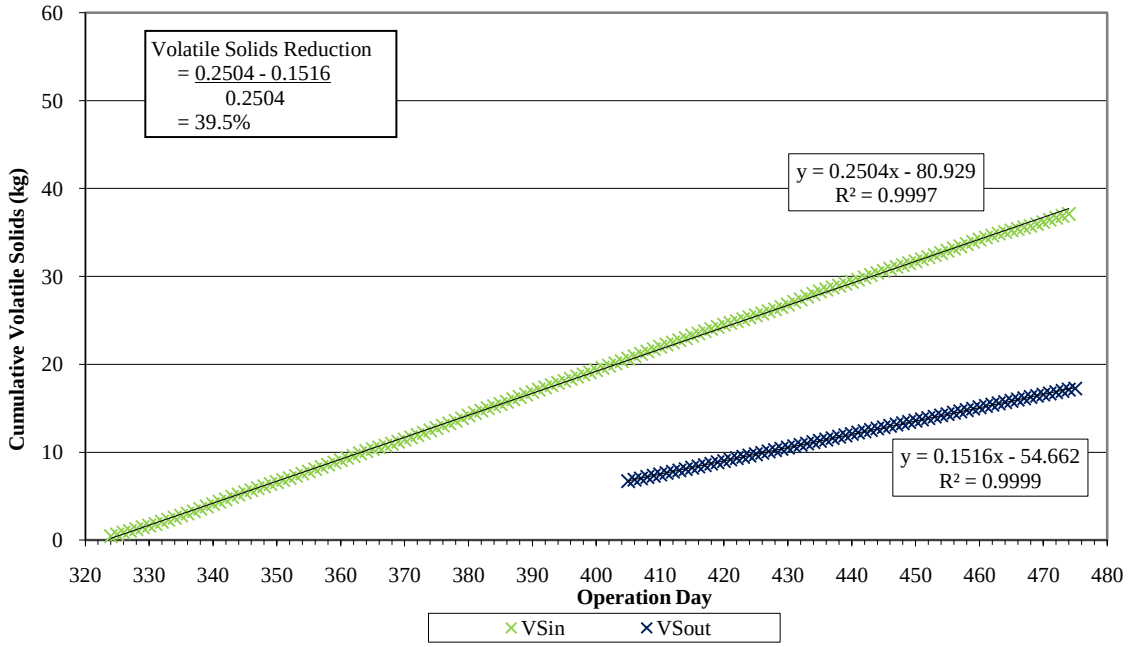

Figure 32 Volatile Solids Destruction for Control Digester – Membrane Operations

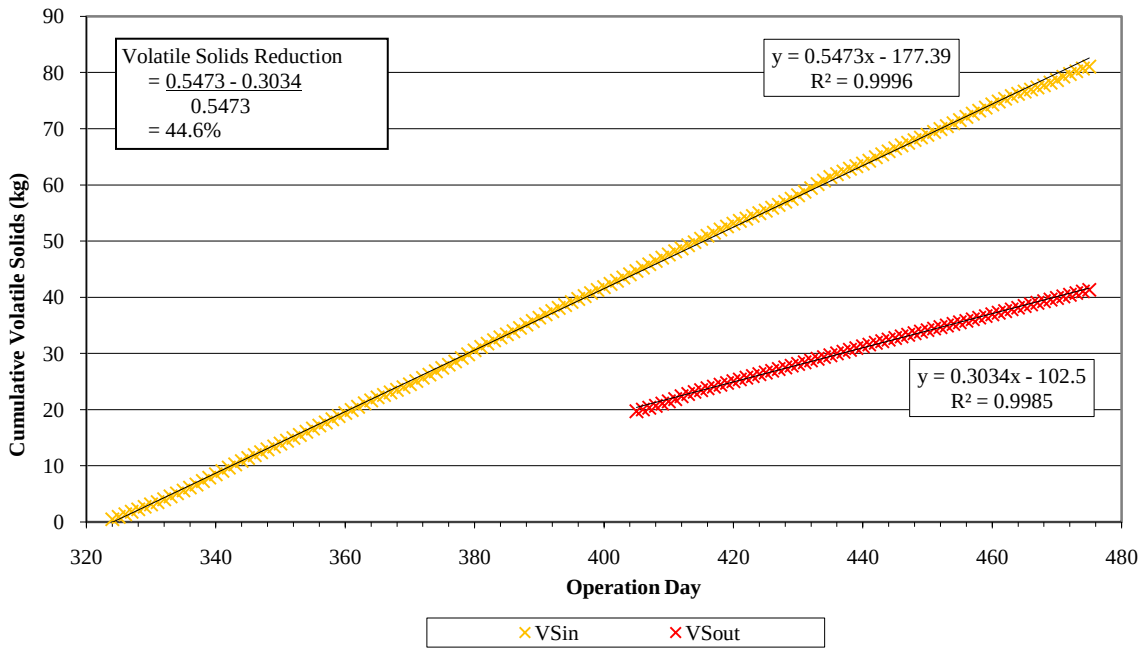

Figure 33 Volatile Solids Destruction for Test Digester – Membrane Operations

4.2.3 Organic Nitrogen Destruction

The nitrogen content gave an alternative indication of the performance of the digester. In the following graphs the ammonia and total Kjeldahl nitrogen concentrations of the feed and the control digester were paired together and the concentrations of the microwaved feed and the test digester were paired. The ammonia and total Kjeldahl nitrogen concentrations of the feed and the digester contents, along with the feeding rates and digester volumes were used to determine the destruction of organic nitrogen in both the control and test digesters. Initially the loadings and removals were calculated, then they were graphed and the difference between them was calculated to give organic nitrogen destruction.

The calculations for the membrane operations were the same as those completed for the low microwave operations. The masses of NH_3 and TKN entering the digesters were determined using Equation 14 and Equation 15, respectively. The feed rate during the membrane operations was

17.6 L/d for the control digester and 38.7 L/d for the test digester. The NH_3 leaving the digester was calculated using Equation 16, using both the concentration of NH_3 in the wasting sludge and the concentration of the permeate. The mass loading of in the wasting sludge and permeate were added together to give the total NH_3 removed from the test digester. The sludge wasting rates used were 17.6 L/d for the control digester and 20.3 L/d for the test digester. The permeate from the test digester was wasted at a rate of 18.4 L/d.

Figure 34 and Figure 35, present the cumulative values of TKN_{IN}, $NH_{3\text{-}IN}$, and $NH_{3\text{-}OUT}$ plotted against the operation day for the microwave operation for the control and test digesters respectively. Linear regression was employed to estimate the slopes of the cumulative lines and these represented the average mass flows in and out of the digesters over the duration of the steady state period. All of the lines of best fit shown in the two figures have high R^2 values, indicating that the lines of best fit were appropriate for the data. The destruction of organic nitrogen in the digesters, was calculated by determining the difference between the slope of the $NH_{3\text{-}OUT}$ line and the slope of the $NH_{3\text{-}IN}$ line, then dividing the difference between the slope of the TKN_{IN} line and the slope of the $NH_{3\text{-}IN}$ line. For the membrane operations the control digester organic nitrogen reduction was calculated to be 45.2% and for the test digester, with an associated membrane, the organic nitrogen reduction was calculated to be 40.8%. This comparison would suggest that the control digester removed more organic nitrogen than the test digester.

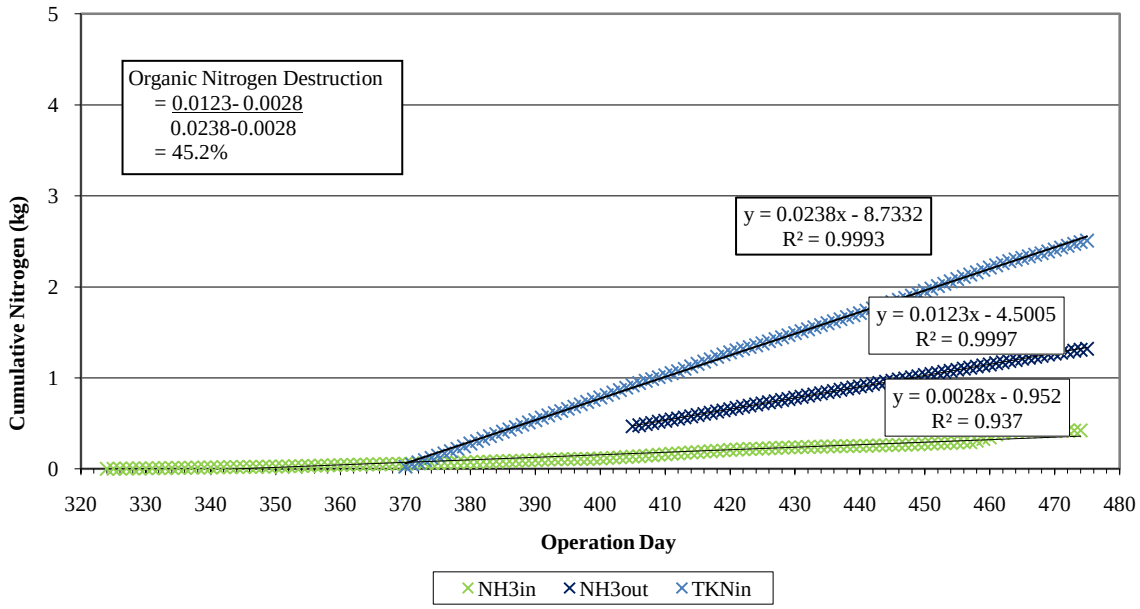

Figure 34 Organic Nitrogen Destruction - Control Digester

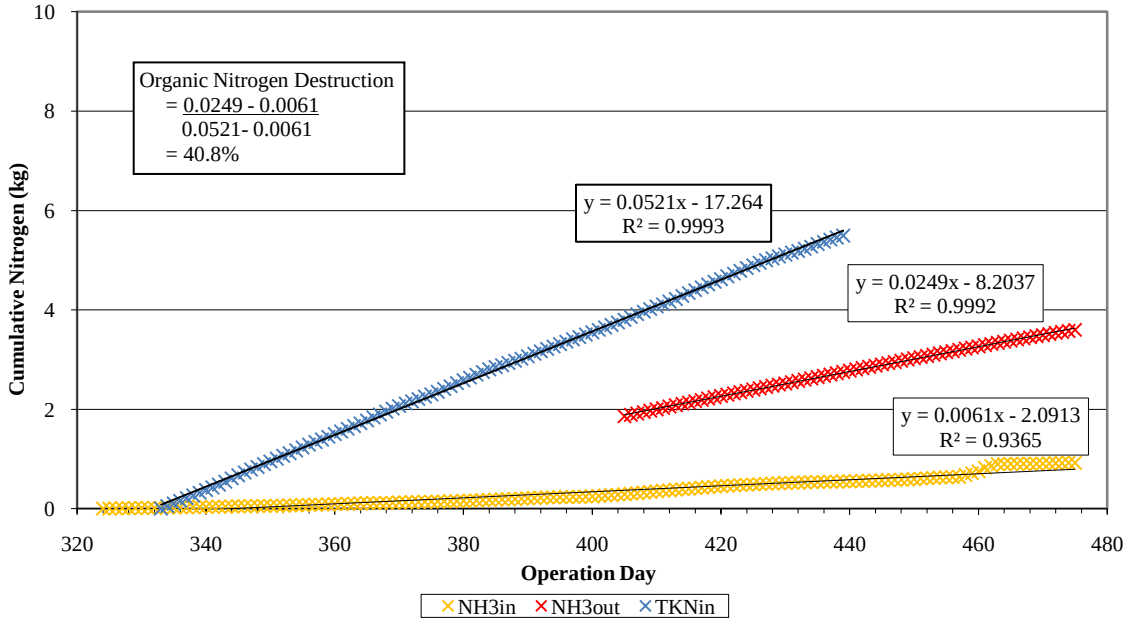

Figure 35 Organic Nitrogen Destruction - Test Digester

4.2.4 Measured Biogas Production

The biogas production during membrane operations was originally measured as a flow, however in Figure 36 the biogas production is shown as the total volume of biogas produced per cycle. The total volume of biogas produced by both the control digester and the test digester are shown. The membrane operations occurred between Day 405 and 475; however, due to file corruption caused by power outages the results were not available after Day 448. Over the duration of the membrane operations, shown in Figure 36, the volume of biogas produced per cycle is consistently higher for the test digester than the control digester, indicating that the addition of a membrane unit allowed for increase biogas production compared to the control operations.

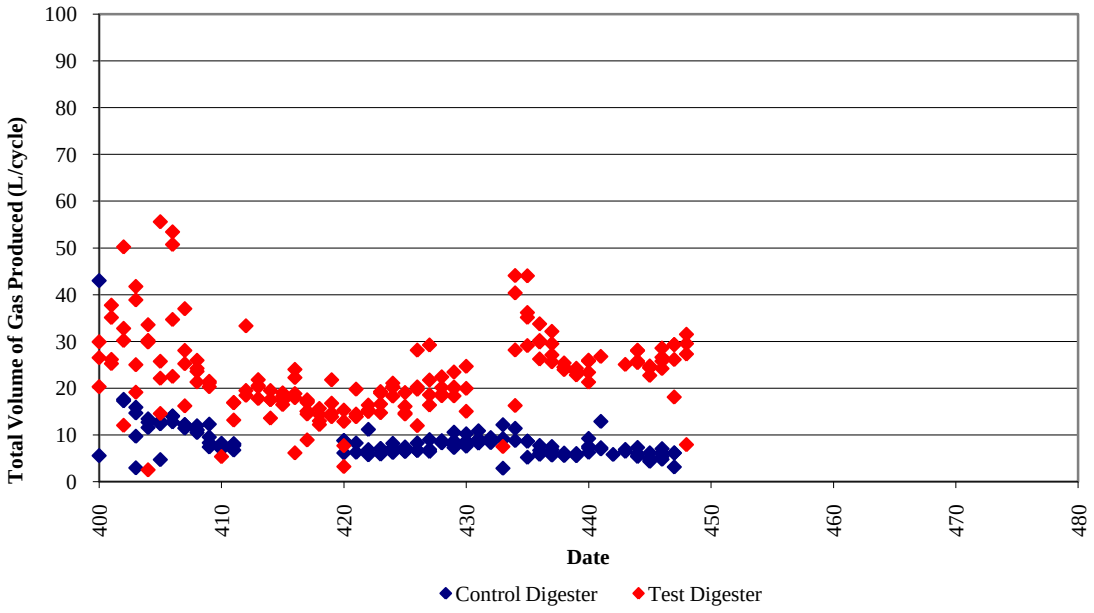

Figure 36 Measured Biogas Production

A statistical summary of the biogas values, presented in the previous figures, is shown in Table 23. The values presented in Table 23, suggest that for the membrane operations the test digester showed higher measured biogas production, however, the variability of the data was greater. The increased variability may have been due to the increased time spent removing mass from the digester, in the form of permeate. Withdrawing permeate from the digester created a suction within the digester, drawing biogas away from the flow meter. Despite the variability in the test digester, these results

indicated that having a membrane unit attached to the digester allowed for a greater volume of biogas produced during anaerobic digestion of WAS.

Table 23 Measured Biogas Production Statistics

Biogas Production (L/cycle)	Membrane	
	Control	Test
Mean	7.85	21.74
Std Dev	2.28	8.51
Number	124	154

4.2.5 Biogas Composition

An analysis of biogas composition was completed for both the control digester and the test digester during the membrane operations. This characterization was conducted to further compare the biodegradation within the digesters, and particularly evaluate the methane production from each digester. Five samples were taken over the course of the final 30 days of the membrane operations. The results from these gas chromatographic analysis are presented in Appendix E.1. The average results from the analysis are presented in Table 24.

Table 24. Average Percent Biogas Characteristics

	O_2	N_2	CH_4	CO_2
Control	**0.19**	**0.55**	**69.40**	**25.97**
Test	**0.17**	**0.50**	**71.51**	**24.06**

The results presented indicate that the percentage of methane in the biogas produced by the digesters is greater for the test digester that has a membrane unit, than the control digester.

4.2.6 Theoretical Biogas Production

The theoretical biogas measurements gave an indication of the performance of the digesters, but they were also used to predict validate the results of the measured biogas production.

4.2.6.1 Theoretical Gas Production Based on COD

To determine the production of biogas caused by the destruction of organic material in both the control and test digesters, the COD_T concentration of the feed and the digester contents, along with the feeding rates and the typical yield coefficients were used.

The COD mass loadings and removals were calculated, and they were used to establish the COD destruction in the digesters. Equation 17, Equation 18 and Equation 19 were used to calculate the theoretical gas production. Equation 19 was used to calculate the volume of methane produced by the digesters. $COD_{T\text{-}IN}$ and $COD_{T\text{-}OUT}$ were offset by the length of the SRT, 30 days, to estimate the COD destruction over that time. To estimate the total volume of biogas produced by the digesters Equation 20 was used. The estimate that the percentage of methane in the gas was 65 % of the total came from Metcalf and Eddy (Tchobanoglous and Burton 2003). The volume of biogas produced per day was divided by 4 to estimate the theoretical biogas produced per cycle. Figure 37 displays the theoretical biogas production per cycle for the control and test digesters during membrane operations.

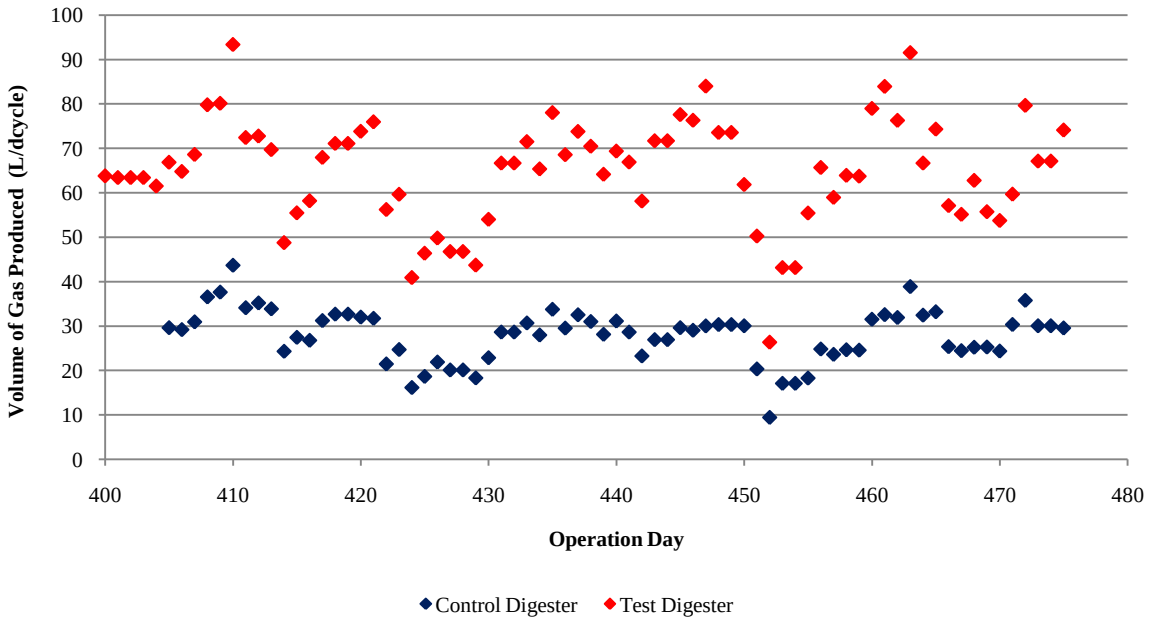

Figure 37 Theoretical Biogas Production based on COD destruction

The theoretical biogas production data shown in the previous figure shows more consistent estimations when compared to the microwave operations. The test digester showed higher theoretical biogas production throughout the entire membrane operation period. When the influent concentrations of COD were lower than the effluent concentrations the nature of Equation 19 resulted in lower than expected results, or what numerically appeared to be a creation of COD within the digester, which in fact was not the case. The variability of the feed greatly influenced the ability to produce consistent and reliable estimates. The data presented in the previous figure is summarized in Table 25.

Table 25 Theoretical Gas Production Statistics (COD)

Biogas Production (L/cycle)	**Membrane**	
	Control	**Test**
Mean	27.92	65.05
Std Dev	5.95	12.47

The standard deviation for the test digester was almost twice that of the control digester, however, the theoretical biogas production for the test digester was also greater than twice that of the control digester. Although the digesters operated at the same 30 day SRT, the HRT was less for the test digester, and as a result a greater volume of sludge passed through the test digester and thus a great volume of biogas was produced.

4.2.6.2 Theoretical Gas Production Based on Solids

To determine the production of biogas caused by the destruction of organic material in both the control and test digesters, the VSS concentration of the feed and the digester contents, along with the feeding and wasting rates and the typical yield coefficients were used.

The VSS mass loadings and removals were calculated, and they were used to establish the destruction in the digesters. Equation 17 and Equation 18 were used to calculate the volume of 1 mole of gas at 35 °C and the methane equivalent for each gram of COD removed, and Equation 21 was used to calculate the volume of methane produced by the digesters. VSS_{IN} and VSS_{OUT} were offset by the

length of the SRT, 30 days, to estimate the VSS destruction over that time. To estimate the total volume of biogas produced by the digesters Equation 20 was used. The estimate that the percentage of methane in the gas was 65 % of the total came from Metcalf and Eddy (Tchobanoglous and Burton 2003). The volume of biogas produced per day was divided by 4 to estimate the theoretical biogas produced per cycle. Figure 38 shows the theoretical biogas production per cycle for the control and test digesters over the membrane operations.

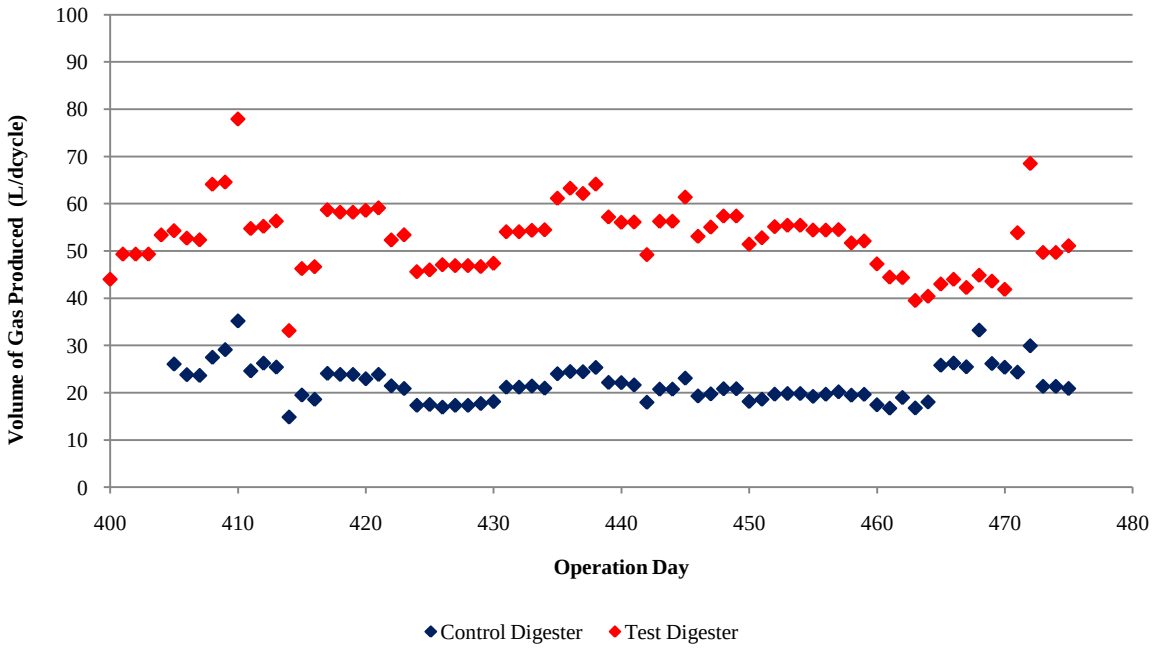

Figure 38 Theoretical Biogas Production based on VSS destruction

The theoretical biogas production data shown in the previous figure shows more consistent estimations when compared to the microwave operations. The test digester showed higher theoretical biogas production throughout the entire membrane operation period. The data presented in the previous figure is summarized in Table 26.

Table 26 Theoretical Gas Production Statistics (VSS)

Biogas Production (L/cycle)	Membrane	
	Control	Test
Mean	21.84	52.84
Std Dev	3.88	7.34

The standard deviation for the test digester is almost twice that of the control digester, however, the theoretical biogas production for the test digester is also greater than twice that of the control digester. The standard deviations for the biogas production based on VSS destruction was less than those based on the COD destruction. Similarly to the COD destruction measures, the theoretical biogas production for the test digester was higher than that of the control digester, indicating that the digester was capable of more biodegradation.

Compared to the theoretical biogas production, the measured gas production results for both the control and test digester were lower. However, the trends between the theoretical and measured data were similar. The difference between the observed and predicted gas production could have been attributed to quality of the actual biogas measurements and the resulting calibrations of the flow meters.

4.2.7 Volatile Fatty Acids

The results from the measurement of the VFAs in the digesters are presented in Appendix F. Poor operations can lead to a system imbalance, which is indicated by high volatile acids concentrations (Parkin and Owen 1986). VFA concentrations that are above 2000 mg/L have been shown to be inhibitory to methanogens (Grady Jr. et al. 1999). The results indicated in Table 27 show that appropriate concentrations were always far below that inhibitory level for both the control and the test digester.

Table 27 VFA Concentrations Statistics – Membrane Operations

Concentration (μg/mL)	Membrane	
	Control	Test
Mean	34	46
Std Dev	13	15
Min	5	14
Max	55	81
Number	27	28

4.2.8 Nitrogen

The results for the measurement of the ammonia in the digesters is presented in Appendix G. The results indicated appropriate concentrations within the both the control and the test digester. The summary of these results is shown in Table 28. If the concentration of ammonia-N was between 1500 mg/L and 3000 mg/L within the digesters the fermentation could be inhibited, reducing the functionality of the digesters (Parkin and Owen 1986). During the length of the membrane operations neither of the of digesters had ammonia-N concentrations greater than 744 mg/L, and the averages, which are shown in Table 28, were 645 and 589 mg/L for the control and test digesters, respectively.

Table 28 Ammonia Concentration Statistics – Membrane Operation

Concentration (mg/L-N)	Membrane	
	Control	Test
Mean	645	589
Std Dev	60	77
Min	496	448
Max	744	725
Number	43	51

4.2.9 Membrane Performance

The membrane performance gave an indication of the applicability of using submerged hollow fibre membranes in the anaerobic digestion of high strength WAS. The performance was assessed by analyzing the transmembrane pressure over the course of the test.

Over the 150 days of the operation of the membrane there was no significant increase in the TMP. Figure 39 presents the TMP values for the permeation of approximately 4.6 L on Day 0, Day 30, Day 90, and Day 150 of the operations. Figure 39 shows the average value and the minimum and maximum values for each permeation cycle.

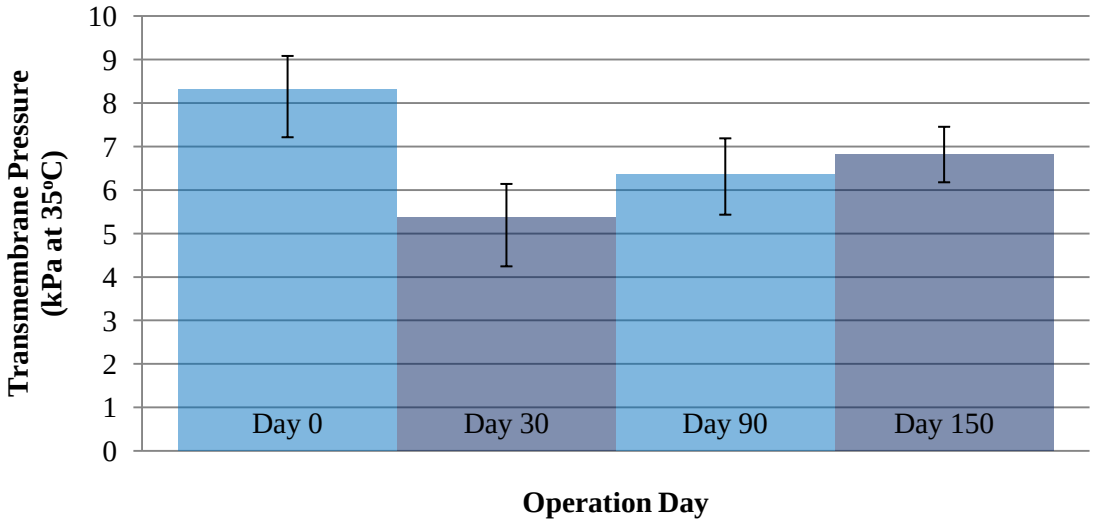

Figure 39 TMP for Permeations Over Membrane Life Span

From Figure 39 it can be seen that there was minimal difference in the membrane performance over the length of the operations. The flux was maintained at 14 L/m^2-h +/-0.68 and the TMP remained below 10 kPa throughout the 150 days of the study period. The high TMP at the beginning of the membrane operations was attributed to the initial start-up operations adjustments. From Day 30 to Day 150 a slight increase in the TMP was observed, however, the increase was less than 2 kPa.

The membrane performance was also assessed in terms of the fouling index, which was calculated using Equation 22.

Equation 22 $$Fouling\ Index = \frac{TMP_{end\ of\ permeation} - TMP_{beginning\ of\ permeation}}{Length\ of\ Permeation}$$

The fouling index was measured in kPa per minute, both the TMP values were measured in kPa, and the length of permeation was measured in minutes. The fouling index indicated the change in pressure across the membrane over time, and the value for each permeation cycle is presented in Figure 40.

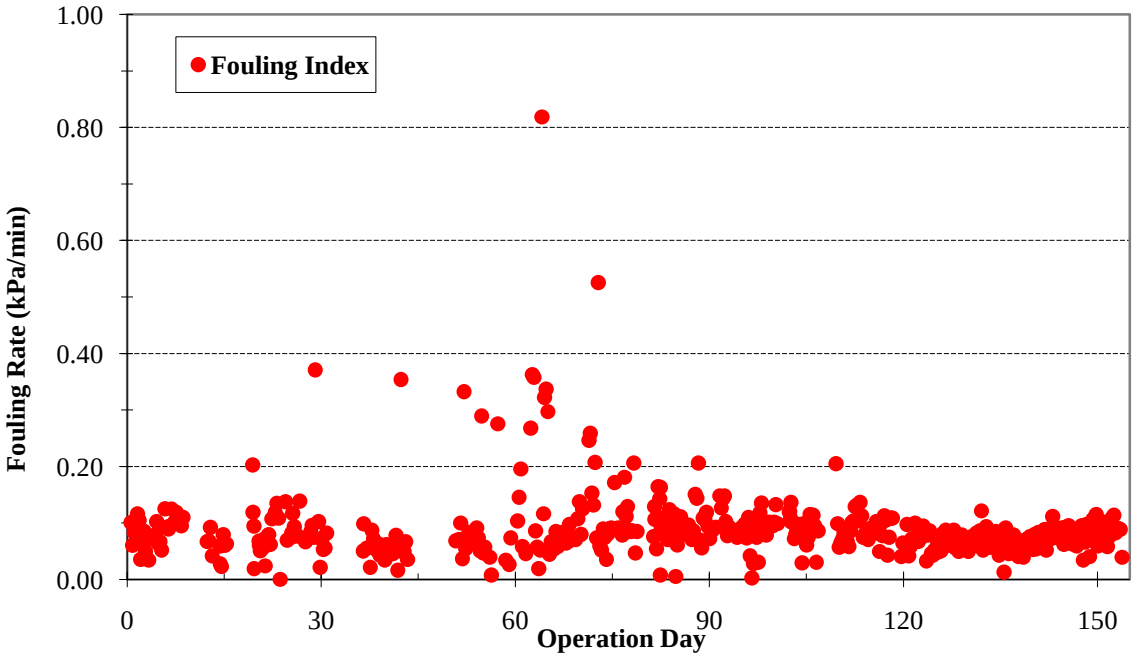

Figure 40 Fouling Index for Hollow Fibre Membrane

The significance of the fouling index indicates the sustainability of the membrane operations at the operational flux, hence the relatively consistent fouling index over the course of the study indicated good membrane operations. The majority of the values ranged between 0.03 and 0.08 kPa/min. The TMP fouling index average for the study was 0.079 kPa/min+/-0.08. .

4.2.9.1 Critical Flux Test

Upon completion of steady state operations, two critical flux texts were completed when the MLSS concentration was 22.4 g/L. The first test compared the TMP when the flux was set at values of 14

L/m^2-h, 20 L/m^2-h, and 26 L/m^2-h and the second test compared the TMP when the flux was set at values of 14 L/m^2-h, 18 L/m^2-h, and 22 L/m^2-h. Each permeation cycle was 30 minutes long, and there were 10 minute relaxation periods between each change in flux.

The first test in which the TMP was compared when the flux was 14 L/m^2-h, 20 L/m^2-h, and 26 L/m^2-h is presented in Figure 41. Prior to testing, membrane relaxation occurred for more than 1 hour. As Figure 41 shows, there were two 14 L/m^2-h cycles followed by a 20 L/m^2-h cycle, and then a return again to the 14 L/m^2-h cycle. A 26 L/m^2-h cycle was then followed by a 14 L/m^2-h. The 14 L/m^2-h and 20 L/m^2-h cycles were each run for 30 minutes with 10 minutes of relaxation between each cycle, however the 26 L/m^2-h cycle was only run for 22 minutes due to rapid increase in TMP. This initial test indicated that the critical flux occurred between 20 L/m^2-h and 26 L/m^2-h. At the flux of 14 L/m^2-h there was little increase in TMP until after the 26 L/m^2-h trial, suggesting that fouling had occurred during the 26 L/m^2-h trial.

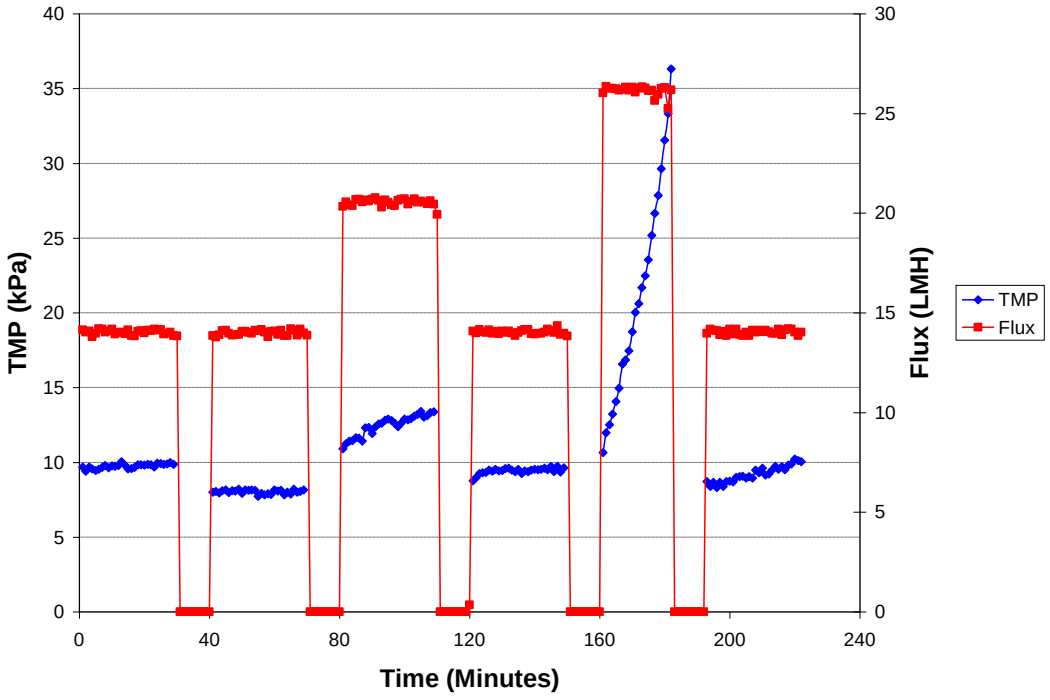

Figure 41 First Critical Flux determination using step wise method

The changing TMP was also compared for the 14, 20 and 26 L/m^2-h. As the flux increased, the TMP across the membrane also increased, indicating that at a higher flux rate, the resistance due to cake layer formation increased. The increase in TMP occurred much more rapidly for the 26 L/m^2-h flux, compared to the 14 L/m^2-h and 20 L/m^2-h flux levels, indicating that the critical flux had been reached by 26 L/m^2-h.

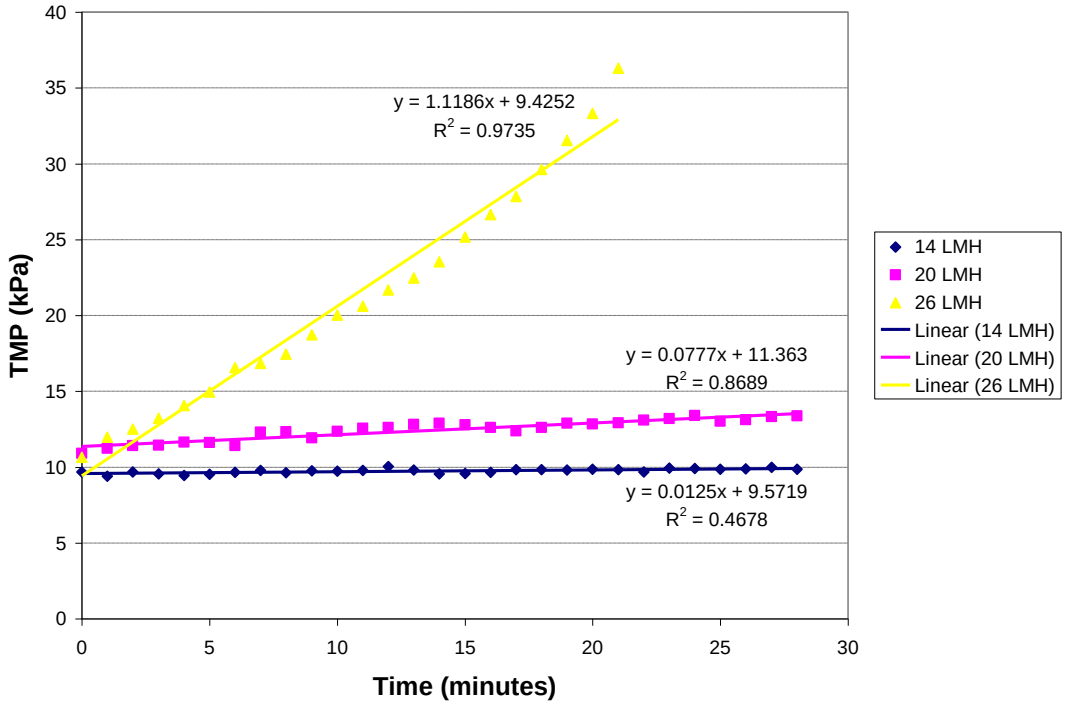

Figure 42 First test TMP versus operation time

The second test, which was conducted three days after the first test, compared the TMP when the flux was 14 L/m^2-h, 18 L/m^2-h, and 22 L/m^2-h. The step wise method results are presented in Figure 43. As was done for the first test, membrane relaxation occurred for more than 1 hour before testing. As Figure 43 shows, there was one 14 L/m^2-h cycle followed by an 18 L/m^2-h cycle, and then a return again to the 14 L/m^2-h cycle. A final 26 L/m^2-h cycle was then completed. The 14 L/m^2-h and 18 L/m^2-h cycles were each run for 30 minutes with 10 minutes of relaxation between each cycle, however the 26 L/m^2-h cycle was only run for 20 minutes due to rapid increase in TMP. This second test, narrowed the estimation range of the critical flux and indicated that the critical flux occurred between 18 L/m^2-h and 22 L/m^2-h.

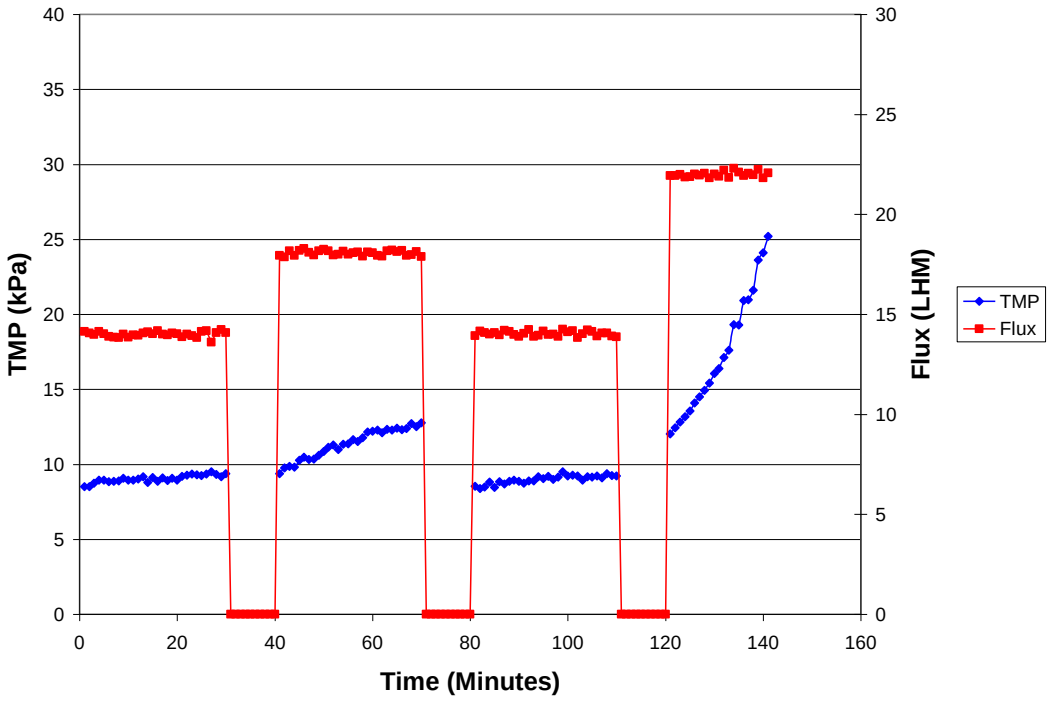

Figure 43 Second Critical Flux determination using step wise method

The changing TMP was also compared for the 14, 18 and 22 L/m^2-h. As the flux increased, the TMP across the membrane also increased, indicating that at a higher flux rate, the resistance due to cake layer formation increased. The increase in TMP occurred much more rapidly for the 22 L/m^2-h flux, compared to the 14 L/m^2-h and 18 L/m^2-h flux levels, indicating that the critical flux had been reached by 22 L/m^2-h.

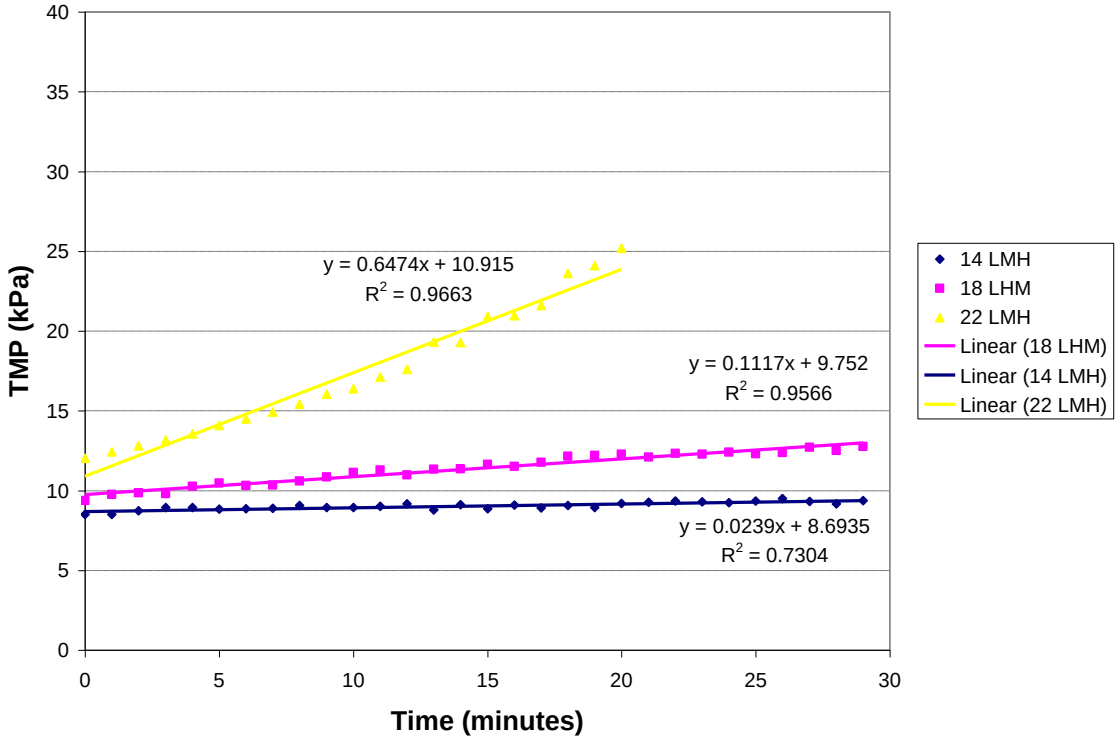

Figure 44 Second test TMP versus operation time

4.3 Comparison of Microwave Pre-treatment to Membrane Bioreactor Operation

A comparison of the microwave operations directly to the membrane operations was difficult, due to both the difference in feed and operational characteristics. However the percent increase of the characteristics in question is presented as a method to facilitate comparison of the two approaches to enhancing anaerobic digestion of WAS.

Table 29, Table 30, and Table 31 present the average performance of the digesters with respect to COD, VS, and organic nitrogen destruction, respectively. In most cases it can be observed that the test digester showed improved performance over the control digester. For both the COD and solids destruction the low temperature microwave operations showed the highest percent increase from the control to the test digesters, however, in both cases the low temperature microwave operation exhibited the lowest percent removal for both the control and the test digesters. This was likely due

to the low solids concentration in the feed, leading to non-ideal operating conditions within the digesters. The organic nitrogen destruction was increased for the low and high temperature operations when pretreatment was applied. The apparent organic nitrogen destruction for the membrane operations was lower for the test digester than the control digester. This could be due to the lower HRT in the test digester, which could have lead to lower destruction of some soluble proteins that were washed out of the digester in the permeate.

Table 29 Comparative COD Destruction for Digesters

% Removal	Control	Test	% Increase
Low MW	32.2	38.2	15.7%
High MW	44.1	48.2	8.5%
Membrane	44.4	48.2	7.9%

Table 30 Comparative Volatile Solids Destruction for Digesters

% Removal	Control	Test	% Increase
Low MW	27.4	35.4	22.6%
High MW	47.2	47.5	0.6%
Membrane	39.5	44.6	11.4%

Table 31 Organic Nitrogen Destruction for Digesters

% Removal	Control	Test	% Increase
Low MW	28.2	67.9	140.8%
High MW	41.0	47.3	15.4%
Membrane	45.2	40.8	-9.7%

The measured biogas results appeared to lack precision, since logic would suggest that the membrane operation which had twice the throughput and comparative COD and VS destruction rates when compared to the microwave operations, would also have twice the biogas production. However, this

was not the case, and more biogas was observed being produced for the microwave operations, further suggesting that the biogas data lacked complete reliability.

Table 32 Comparative Actual Biogas Production

Biogas L/Cycle	Control	Test	% Increase
Low MW	8.0	21.61	63.0%
High MW	20.17	42.33	52.4%
Membrane	7.85	21.74	63.9%

Both pretreatment using microwaves and membrane bioreactor operation showed an increase in the biodegradation capabilities of the digesters and ultimately the biogas produced. The membrane operations allowed for a greater throughput while maintaining improved biodegradation and biogas production. The comparison of the actual measured biogas indicated that there was a considerable increase in biogas production from the control digester to the test digester. The membrane digester showed the largest increase of biogas, however, the largest amount of biogas produced per cycle was for the high temperature microwave operations.

The theoretical biogas production based on both the COD and VSS destruction was higher for the test digester than the control digester in all cases with the exception of the low temperature microwave operations. The largest estimated volume of biogas produced per cycle, using both the destruction of COD and VSS as the estimation methods, was for the membrane digester. The membrane operations also showed a higher increase in biogas when compared to the control digester. In both Table 33 and Table 34 the estimation using COD indicated a higher increase in biogas production than compared to the estimation using solids. This method and estimation was suspect as the analysis was based on daily samples that could result in negative values. Although the trends for this data appeared similar to the measured biogas data, the validity of these results is questionable.

Table 33 Comparative Theoretical Biogas Production (COD)

Biogas L/Cycle	Control	Test	% Increase
Low MW	36.59	44.68	18.1%
High MW	37.24	47.85	22.2%
Membrane	27.92	65.05	57.1%

Table 34 Comparative Theoretical Biogas Production (VSS)

Biogas L/Cycle	Control	Test	% Increase
Low MW	30.07	28.66	-4.9%
High MW	32.53	35.28	7.8%
Membrane	21.84	52.84	58.7%

Chapter 5
Conclusions

5.1 Microwave Operations

The conclusions from the research completed for both the low and high temperature operations are presented in the following section. The conclusions from the pretreatment assessment, the biodegradation and destruction, the theoretical biogas production and the measured biogas are presented.

5.1.1 Pretreatment

The effect of microwave pretreatment on WAS characteristics were assessed for both the low temperature operations and the high temperature operations. For the low temperature operations there was a visible increase in the filtered to total COD ratio when comparing the feed to the microwaved feed. The average COD_F to COD_T ratio for the feed WAS increased by 200 % due to pretreatment, during the low temperature operations. At a 95 % confidence interval the mean value of the microwaved feed COD_F was found to be 1532 mg/L +/- 402 mg/L greater than for the raw feed.

For the high temperature operations there was also an increase in the COD_F to COD_T ratio when comparing the raw feed to the microwaved feed. The soluble component of the WAS increased by 254 % due to pretreatment, during the high temperature operations. At a 95% confidence interval the difference between the mean of the COD_F in the microwaved feed was found to be 1545 mg/L +/- 350 mg/L greater than that of the mean of the COD_F in the raw feed.

The high temperature operations showed a larger percent increase in the ratio of COD_F to COD_T, however, the low and high temperature operations showed similar differences in the mean concentrations of COD_F when comparing the raw feed and the microwaved feed.

5.1.2 Destruction

The COD_T destruction, for the low temperature operations indicated that the COD_T destruction for the control digester was 32.2% and for the test digester, being fed microwaved sludge, the COD_T reduction was 38.2%, indicating improved biodegradation with microwave pretreatment. The volatile

solids destruction supported the improved operation with microwave treatment as the volatile solids reduction in the control digester was 27.4% and the volatile solids reduction was 35.4% for the test digester receiving pretreated WAS. The organic nitrogen reduction in the control digester was 28.2% and for the test digester was calculated to be 67.9%, further indicating that the microwaving of the WAS increased the biodegradation in the anaerobic digester.

For the high temperature operations the control digester COD_T destruction was calculated to be 44.1% and for the test digester, being fed microwaved sludge, the COD_T destruction was calculated to be 48.2%. However, the VS reduction did not indicate this trend, and for the control digester the solids destruction was 47.2% and for the test digester, being fed microwaved sludge, the VS reduction was calculated to be 47.5%, suggesting no difference between the biodegradation of the pretreated WAS and the raw WAS. The organic nitrogen destruction within the test digester was higher than that of the control digester, 47.3 % versus 41 % respectively, giving suggesting that the VS destruction was not representative of the digester performance and that the microwaving of the WAS increased the biodegradation in the anaerobic digester.

5.1.3 Measured Biogas Production

The measured biogas data indicated that microwaving did influence the volume of biogas produced during anaerobic digestion of WAS. Compared to the theoretical biogas production, the measured gas production results for both the low and high temperature operations were lower than the predicted values. The exception to this was that the theoretical biogas volume based on COD removal during the high temperature operations was higher than that predicted. The measured biogas results indicated that the test digester produced more biogas, compared to the control digester.

5.1.4 Theoretical Biogas Production

With respect to a COD approximation of biogas production, for both the low temperature operations and the high temperature microwave operations the test digester has a higher average biogas production per cycle than the control digester, indicating the microwaving improved the biodegradation within the digesters, and indicated that the biogas produced by the test digester should be higher than the control digester. For the theoretical biogas production based on VSS removal, the

difference in estimated biogas production was less pronounced, and for the low temperature operations the control digester had higher theoretical yields than the test digester.

5.2 Membrane Operations

The conclusions from the research completed during the membrane operations are presented in the following section. The conclusions from the biodegradation and destruction, the theoretical biogas production and the measured biogas, characterization of the biogas, and the membrane performance are presented.

5.2.1 Destruction of Organics

The test digester achieved a higher COD_T removal with a throughput almost 2.2 times great than that to the control digester. The destruction of VS for the control digester was calculated to be 39.5% and for the test digester, with a connected membrane unit, the VS reduction was calculated to be 44.6%. Both the COD_T and the VS destruction calculations indicated that at the same SRT the test digester was capable of more biodegradation than the control digester. For the membrane operations the control digester organic nitrogen reduction was calculated to be 45.2% and for the test digester the organic nitrogen reduction was calculated to be 40.8%. This comparison would suggest that the control digester removed more organic nitrogen than the test digester, however, these results are likely due to the lower HRT of the test digester compared to those of the control digester.

5.2.2 Measured Biogas Production

The membrane unit attached to the digester allowed for a greater volume of biogas produced during anaerobic digestion of WAS, when compared to the control digester. The measured biogas compared to the theoretical biogas production was lower for both the control and test digester, however, the trends between the theoretical and measured data were similar. The composition of the gas from both digesters was similar, although the percentage of methane produced by the test digester was higher than that produced by the control digester.

5.2.3 Theoretical Biogas Production

The test digester showed higher theoretical biogas production throughout the entire membrane operation period. The higher destruction for the test digester indicated that the presence of the membrane unit and the decoupling of the HRT and SRT improved the biodegradation capability of the digesters.

5.2.4 Membrane Performance

The results of the membrane performance study indicated that for a hollow fibre AnMBR, stable operations could be achieved with a total solids concentration of 2.01 %=/-0.34, an HRT of 15 days and an SRT of 30 days. With a constant flux of 14 L/m^2-h +/-0.68 the average TMP was 0.079 kPa/min+/-0.08. No cleaning was required to achieve this, however the operations consisted of 20 minutes of permeation followed by 5 hours and 40 minutes of relaxation. The critical flux was determined to be in the range of 18 to 22 L/m^2-h.

5.3 Comparison of Microwave and Membrane Operations

The comparison of the microwave operations directly to the membrane operations was completed. For COD, solids, and organic nitrogen destruction and biodegradation the low temperature operations showed the highest percent increase from the control to the test digesters, however, in all cases the low temperature operations exhibited the lowest percent removal for both the control and the test digesters, with the exception of the organic nitrogen removal for the test digester.

The comparison of the actual measured biogas indicated that there was a considerable increase in biogas production from the control digester to the test digester. The membrane digester showed the largest increase of biogas, however, the largest amount of biogas produced per cycle was for the high temperature microwave operations.

The theoretical biogas production based on both the COD and VSS destruction was higher for the test digester than the control digester in all cases with the exception of the low temperature microwave operations. The largest estimated volume of biogas produced per cycle, using both the destruction of

COD and VSS as the estimation methods, was for the membrane digester. The membrane operations also showed a higher increase in biogas when compared to the control digester.

Both pretreatment using microwaves and membranes as a method of separating HRT and SRT showed an increase in the biodegradation capabilities of the digesters and ultimately the biogas produced. The membrane operations allowed for a greater throughput while maintaining improved biodegradation and biogas production.

Chapter 6
Recommendations

6.1 Microwave Operations

Several recommendations are suggested for further pretreatment operations for the anaerobic digestion of WAS.

The raw feed supplied in this study varied in quality, due to the operational conditions at the Burlington Skyway Treatment Plant, which resulted in feed supplies that were not predictable or stable ultimately causing difficulty reaching stable conditions in the digesters. Raw feed with constant solids concentrations would allow for more accurate quantification of the processes within the digesters.

For future pretreatment studies, a shorter distance and time interval between the raw feed and the microwaved feed would reduce the difference in the WAS characteristics that should ideally be constant.

Further pretreatment studies should focus on a larger temperature array, to evaluate whether higher and lower microwave temperatures further influence the solubilization of the WAS.

Further biogas measurement should be done with flow meters calibrated for biogas rather than air to give a more accurate reading. Over the entire length of the study the biogas should be characterized for methane content.

6.2 Membrane Operations

Recommendations for the membrane operations include inducing further critical flux tests over time to determine the environments submerged hollow fibre membranes are appropriate for.

Also recommended would be the decrease of the HRT to determine the minimum HRT that would still allow for effective treatment, but increase the capability of the WAS digestion.

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Appendix A
Digester Calibration Logs

Table 35 Control Digester Calibrations

Date	Avg Weight before waste (kg)	Avg Weight after waste (kg)	Avg Weight after feeding (kg)	Feed Weight/cycle (kg)	Waste weight/cycle (kg)	Actual sludge volume wasted (L)	Weight of Sludge (g)	Panel SRT	Actual SRT
01/19/09	529.99	520.96	529.77	8.81	9.03	11		14.67	12.04
01/26/09	529.44	521.88	529.77	7.89	7.56	8.6	8415.0	17.51	15.74
02/02/09	530.21	521.73	529.88	8.15	8.48	9.5	9287.0	15.62	14.26
02/09/09	527.79	521.29	529.88	8.59	6.50	8		20.38	16.56
02/11/09	529.77	521.88	529.59	7.71	7.89	8.8	8713.0	16.77	15.20
02/17/09	528.34	522.06	529.44	7.38	6.28	7.5	7542.0	21.08	17.55
02/19/09	527.09	521.51	529.00	7.49	5.58	8.4	8330.5	23.69	15.88
03/02/09	529.44	521.73	529.99	8.26	7.71	8.8	8700.0	17.19	15.23
03/11/09	530.10	521.58	529.59	8.00	8.52	9.2	9112.5	15.55	14.53
03/19/08	529.44	521.58	529.88	8.30	7.86	8.8	8729.5	16.86	15.17
03/26/09	530.10	521.73	529.77	8.04	8.37	9.2	9056.0	15.82	14.62
04/09/09	529.29	521.51	529.77	8.26	7.78	9	8926.5	17.02	14.84
04/16/09	530.69	521.73	529.88	8.15	8.96	9	8912.5	14.79	14.86
04/22/09	530.10	521.58	529.59	8.00	8.52	9.6	9479.5	15.55	13.97
04/30/09	530.39	521.58	529.88	8.30	8.81	9.6	9516.0	15.04	13.92
05/29/09	529.59	526.14	529.88	3.74	3.45	3.8	3818.0	38.43	34.70
06/08/09	529.99	525.59	529.88	4.29	4.40	4.8	4770.5	30.11	27.77
06/24/09	529.77	525.70	529.88	4.18	4.07	4.7	4661.0	32.55	28.42
07/09/09	529.44	526.29	529.77	3.48	3.15	3.85	3840.0	42.00	34.49
07/23/09	529.88	525.79	529.73	3.94	4.09	4.6	4568.5	32.38	28.99
07/29/09	530.10	525.99	529.88	3.89	4.11	4.75	4734.5	32.26	27.98
08/13/09						4.8	4853.5		27.30
08/21/09							4176.0		31.73
09/03/09						4.45	4464.0		29.68
09/10/09						4	4003.0		33.10

Table 36 Test Digester during Microwave Operation

Date	Avg Weight before waste (kg)	Avg Weight after waste (kg)	Avg Weight after feeding (kg)	Feed Weight/cycle (kg)	Waste weight/cycle (kg)	Actual sludge volume wasted (L)	Weight of Sludge (g)	Panel SRT	Actual SRT
01/19/09	514.52	503.78	515.02	11.24	10.74	11.60	11485.0	11.99	11.21
01/28/09	515.27	505.46	514.35	8.89	9.81	11.25	11043.0	13.12	11.66
02/02/09	515.02	505.46	514.52	9.06	9.56	10.70	10538.5	13.47	12.22
02/09/09	514.52	506.85	514.52	7.66	7.66	10.35	10302.0	16.80	12.50
02/09/09	514.52	504.45	514.52	10.07	10.07	10.35	10183.0	12.79	12.64
02/10/09	515.02	504.96	515.02	10.06	10.06	12.00	13661.0	12.80	9.42
02/11/09	514.52	504.96	514.52	9.56	9.56	9.60	9503.0	13.47	13.55
02/16/09	515.52	503.28	514.52	11.24	12.25	12.70	12534.5	10.51	10.27
03/04/09	515.52	503.95	514.52	10.57	11.57	12.40	12250.0	11.12	10.51
03/16/09	516.19	502.44	514.18	11.75	13.76	13.90	13703.0	9.36	9.40
03/23/09	515.86	504.45	515.02	10.57	11.41	14.00	13895.0	11.29	9.27

Table 37 Test Digester During Membrane Operation

Date	Avg Weight before waste (kg)	Avg Weight after filtration (kg)	Avg Weight after wasting (kg)	Avg Weight after feeding (kg)	Actual Volume Wasted (L)	Actual Sludge Wasted (kg)	Actual Permeate Filtered (L)	Sludge Waste Weight/Cycle (kg)	Permeate Weight/Cycle (kg)	Feed Weight/Cycle (kg)	Panel SRT	Panel HRT	Actual SRT	Actual HRT
04/27/09	520.27	515.69	509.76	520.27	7.10	7.089	4.90	5.93	4.58	10.51	25.31	14.27	21.16	12.51
05/04/09		514.35	512.67	520.27	4.15	4.119	4.90	1.68		7.60	89.29		36.42	16.63
05/04/09														
05/19/09	520.72	515.24	511.89	520.72	4.7	4.641	5.05	3.35	5.48	8.83	44.73	16.98	32.32	15.48
05/29/09	521.06	516.92	511.16	520.27	7.25	7.210	4.90	5.76	4.14	9.11	26.04	15.16	20.80	12.39
06/10/09	519.60	515.24	511.44		6.21	6.108	4.90	3.80	4.36		39.47	18.38	24.56	13.63
06/22/09	520.55	515.91	511.43	519.82		6.964	4.40	4.48	4.64	8.39	33.48	16.45	21.54	13.20
07/23/09	519.63	515.30	514.07	519.63	3.50	3.395	4.70	1.23	4.33	5.56	121.62	26.96	44.18	18.53
07/27/09	519.63	515.64	511.91	519.22	4.60	4.545	4.65	3.73	3.99	7.31	40.25	19.43	33.01	16.31
08/07/09	519.94	515.30	511.91	519.63	4.70	4.667	4.70	3.39	4.64	7.72	44.25	18.69	32.14	16.01
08/12/09						5.367	4.80						27.95	14.75
08/21/09						5.718	4.70						26.23	14.40
09/03/09					7.40	7.381	4.70						20.32	12.42
09/10/09					6.2	6.159	4.80						24.35	13.69
09/14/09					7.30	7.174	4.80						20.91	12.53
09/15/09					5.70	5.688	4.80						26.37	14.30
09/16/09					5.40	5.448	4.80						27.53	14.64

Appendix B

COD Pretreatment Data

Appendix B.1

Table 38 Filtered and Total COD Data for Low and High Temperature Operations

Date Sampled	FILTERED COD (mg/L)				TOTAL COD (mg/L)			
	Feed	Duplicate Difference Feed	MWFeed	Duplicate Difference MWFeed	Feed	Duplicate Difference Feed	MWFeed	Duplicate Difference MWFeed
52	390	170	2160	230	20900	200	22325	425
52	220		1930		20700		21900	
56	1190	40	2830	30	17700	3925	21050	1075
56	1150		2800		21625		22125	
63	210	60	1920	90	25000	3900	24025	1150
63	150		1830		21100		22875	
66	830	140	2550	210	24925	2900	24050	1650
66	970		2340		22025		22400	
80	1040	130	2910	150	20275	975	23000	725
80	1170		3060		19300		23725	
90	510	70	1680	270	11975	875	14700	1000
90	580		1410		12850		13700	
93	180	360	1510	220	22125	300	13900	375
93	540		1290		21825		14275	
97	1460	10	3250	90	24100		29575	29575
97	1470		3340					
100	1260		1900		14775		13350	
101	900		2710		37400		31625	
104	1010		2580		23450		25625	
107	220		1500		15925		14250	
108	1440		3460		39075		34625	
121	830	180	2300	160	19200	2025	18225	525
121	650		2140		17175		17700	
128	330	230	1600	80	14375	350	14750	375
128	100		1680		14025		14375	
132	580		2500		20350		20800	
210	350	160	1760	240	15050	4050	15900	3875
210	190		1520		19100		19775	
213	400	20	1450	270	14200	450	14475	525
213	420		1720		14650		15000	
217	550	300	1240	560	15050	575	17500	325
217	250		1800		14475		17175	
220	1760	780	2500	470	14875	100	19400	700
220	980		2030		14975		20100	
223	560	420	700	160	8850	450	10850	1150
223	140		540		9300		9700	
227	0	0	590	40	16700	1175	17700	375
227	0		550		15525		17325	
230	0	200	1080	430	15450	375	19050	1025
230	200		1510		15825		20075	
233	140	200	1820	780	18025	1825	21800	950
233	340		2600		19850		20850	
237	1050	160	2620	630	19675	2600	22750	225
237	890		3250		22275		22525	
240	1610	620	3070	310	21600	4075	22125	1675
240	990		3380		25675		23800	
244	550	120	2140	10	18025	175	20800	550
244	430		2130		17850		20250	
247	550	50	2760	60	21875	25	22725	75
247	600		2820		21900		22650	
251	900	110	1480	40	13550	550	11875	450
251	1010		1520		13000		12325	
254	260	30	860	0	8150	625	6525	200
254	290		860		7525		6325	
258	530	40	1520	110	13350	50	12425	200
258	570		1410		13400		12625	

Date Sampled	FILTERED COD (mg/L)				TOTAL COD (mg/L)			
	Feed	Duplicate Difference Feed	MWFeed	Duplicate Difference MWFeed	Feed	Duplicate Difference Feed	MWFeed	Duplicate Difference MWFeed
261	660	50	2390	70	20625	0	23175	650
261	610		2460		20625		23825	
266	70	80	2430	120	20225	150	27200	275
266	150		2310		20375		27475	
268	480	70	2060	110	19425	1550	21250	525
268	410		2170		17875		21775	
273	480	40	1740	40	25625	1100	22150	325
273	440		1700		24525		22475	
275	480	130	2490	20	23150	250	26675	375
275	610		2510		23400		26300	
279	450	60	2180	160	20025	1300	26500	325
279	390		2020		18725		26175	
282	650	80	2580	110	24350	1425	27300	250
282	570		2690		25775		27050	
MEAN =				**178**				**1432**
STDEV =				**182**				**3758**
VAR =				**33151**				**14120533**

Appendix B.2

Table 39 Low Temperature COD_T Data for Paired T-Test

Date Sampled	FEED		MW FEED		Difference COD_T AvgFeed-AvgMWFeed (mg/L)
	COD_T (mg/L)	Average COD_T (mg/L)	COD_T (mg/L)	Average COD_T (mg/L)	
52	20900	20800	22325	22113	-1313
52	20700		21900		
56	17700	19663	21050	21588	-1925
56	21625		22125		
63	25000	23050	24025	23450	-400
63	21100		22875		
66	24925	23475	24050	23225	250
66	22025		22400		
80	20275	19788	23000	23363	-3575
80	19300		23725		
90	11975	12413	14700	14200	-1788
90	12850		13700		
93	22125	21975	13900	14088	7888
93	21825		14275		
97	24100	24100	29575	29575	-5475
100	14775	14775	13350	13350	1425
101	37400	37400	31625	31625	5775
104	23450	23450	25625	25625	-2175
107	15925	15925	14250	14250	1675
108	39075	39075	34625	34625	4450
121	19200	18188	18225	17963	225
121	17175		17700		
128	14375	14200	14750	14563	-363
128	14025		14375		
132	20350	20350	20800	20800	-450
MEAN =	**20887**	**21789**	**20758**	**21525**	**264**
STDEV =	**6401**	**7343**	**5865**	**6613**	**3423**
VAR =	**40974225**	**53914539**	**34394204**	**43731688**	**11720247**

Table 40 High Temperature COD_T Data for Paired T-Test

Date Sampled	FEED		MW FEED		Difference COD_T AvgFeed-AvgMWFeed (mg/L)
	COD_T (mg/L)	Average COD_T (mg/L)	COD_T (mg/L)	Average COD_T (mg/L)	
210	15050	17075	15900	17837.5	-762.5
210	19100		19775		
213	14200	14425	14475	14737.5	-312.5
213	14650		15000		
217	15050	14762.5	17500	17337.5	-2575
217	14475		17175		
220	14875	14925	19400	19750	-4825
220	14975		20100		
223	8850	9075	10850	10275	-1200
223	9300		9700		
227	16700	16112.5	17700	17512.5	-1400
227	15525		17325		
230	15450	15637.5	19050	19562.5	-3925
230	15825		20075		
233	18025	18937.5	21800	21325	-2387.5
233	19850		20850		
237	19675	20975	22750	22637.5	-1662.5
237	22275		22525		
240	21600	23637.5	22125	22962.5	675
240	25675		23800		
244	18025	17937.5	20800	20525	-2587.5
244	17850		20250		
247	21875	21887.5	22725	22687.5	-800
247	21900		22650		
251	13550	13275	11875	12100	1175
251	13000		12325		
254	8150	7837.5	6525	6425	1412.5
254	7525		6325		
258	13350	13375	12425	12525	850
258	13400		12625		
261	20625	20625	23175	23500	-2875
261	20625		23825		
266	20225	20300	27200	27337.5	-7037.5
266	20375		27475		
268	19425	18650	21250	21512.5	-2862.5
268	17875		21775		
273	25625	25075	22150	22312.5	2762.5
273	24525		22475		
275	23150	23275	26675	26487.5	-3212.5
275	23400		26300		
279	20025	19375	26500	26337.5	-6962.5
279	18725		26175		
282	24350	25062.5	27300	27175	-2112.5
282	25775		27050		
MEAN =	**19161**	**18890**	**20866**	**20744**	**-1854**
STDEV =	**4730**	**4719**	**5620**	**5662**	**2503**
VAR =	**22715717**	**23350400**	**31993403**	**33481539**	**6518833**

Table 41 Total COD Paired T-Test Information

	Low Temperature Paired t-test	High Temperature Paired t-test
Number of Samples (n)	16	22
Mean COD_T Difference Between Feed and Microwaved Feed (d)	264 mg/L	-1847 mg/L
Standard Deviation of the COD_T Difference (s_d)	3423 mg^2/L^2	2503 mg^2/L^2
Standard Error of Mean COD_T Difference (SE_d)	856 mg/L	534 mg/L
$t_{calculated}$	0.3	-3.5
$t_{critical}$ (95% C.I.)	2.131	2.080

The results from the paired t-test displayed in the table above show that for the low temperature operations the COD_T sample sets for the feed and the microwaved feed are not significantly different. The $t_{calculated}$ value for the low temperature COD_T comparison was determined to be 0.3, which is less than the $t_{critical}$ value of 2.131 indicating that the feed and the microwaved feed are not significantly different. For the high temperature operations the total COD sample sets for the feed and the microwaved feed were found to be significantly different. The $t_{calculated}$ value for the high temperature COD_T comparison was determined to be -3.5, the absolute value of which is greater than the $t_{critical}$ value of 2.080 indicating that the feed and the microwaved feed are significantly different.

The following figures show the ratio of volatile solids to total solids for both the raw feed and the microwaved feed, to assess if the WAS sampling was a result of improper sampling, in which a loss of solids occurred.

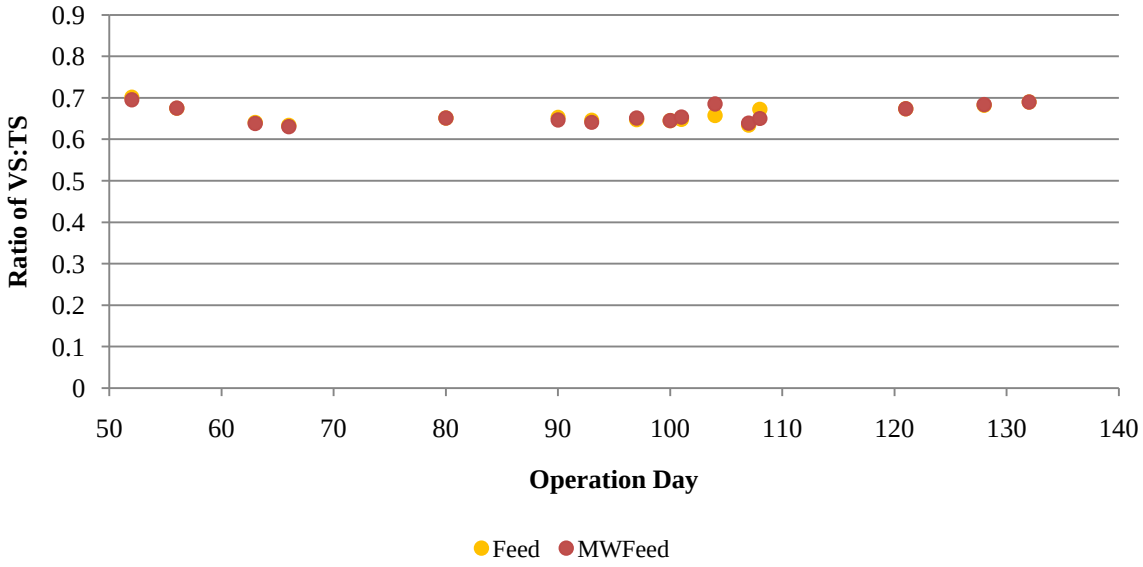

Figure 45. Ratio of VS to TS for Low Temperature Operations

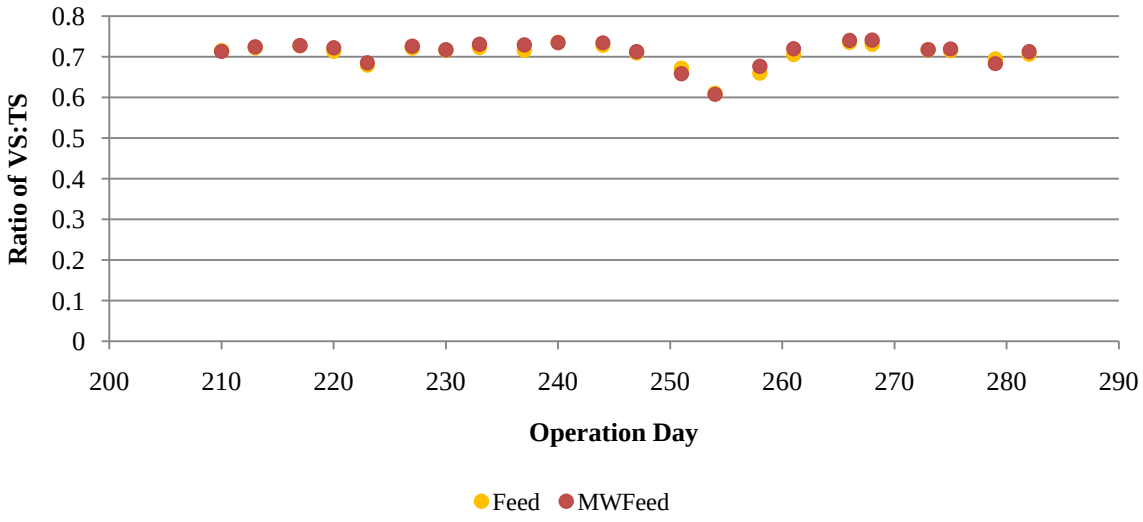

Figure 46. Ratio of VS to TS for High Temperature Operations

In both Figure 45and Figure 46 the ratio of VS to TS was almost identical when comparing the raw feed and the microwaved feed for both the low and high temperature operations. The consistency in WAS characteristics would suggest that the variance in the total COD results was more related to the analysis that the operating conditions.

Appendix B.3

Table 42 Low Temperature COD_F Data for Paired T-Test

Date Sampled	FEED		MW FEED		Difference COD_F AvgMWFeed-AvgFeed (mg/L)
	COD_F (mg/L)	Average COD_F (mg/L)	COD_F (mg/L)	Average COD_F (mg/L)	
52	390	305	2160	2045	1740
52	220		1930		
56	1190	1170	2830	2815	1645
56	1150		2800		
63	210	180	1920	1875	1695
63	150		1830		
66	830	900	2550	2445	1545
66	970		2340		
80	1040	1105	2910	2985	1880
80	1170		3060		
90	510	545	1680	1545	1000
90	580		1410		
93	180	360	1510	1400	1040
93	540		1290		
97	1460	1465	3250	3295	1830
97	1470		3340		
100	1260	1260	1900	1900	640
101	900	900	2710	2710	1810
104	1010	1010	2580	2580	1570
107	220	220	1500	1500	1280
108	1440	1440	3460	3460	2020
121	830	740	2300	2220	1480
121	650		2140		
128	330	215	1600	1640	1425
128	100		1680		
132	580	580	2500	2500	1920
MEAN =	**745**	**775**	**2276**	**2307**	**1533**
STDEV =	**445**	**445**	**638**	**645**	**379**
VAR =	**198394**	**197725**	**407545**	**416297**	**143900**

Table 43 Filtered COD Paired T-Test Information for Low Temperature

	Low Temperature Paired t-test
Number of Samples (n)	16
Mean COD_S Difference Between Feed and Microwaved Feed (d)	1533 mg/L
Standard Deviation of the COD_F Difference (s_d)	379 mg^2/L^2
Standard Error of Mean COD_F Difference (SE_d)	95 mg/L
$t_{calculated}$	16.2
$t_{critical}$ (95% C.I.)	2.131

The results of the paired t-test, presented above, indicate that the $t_{calculated}$ value of 16.2 is higher than the $t_{critical}$ value of 2.131 and thus the feed and the microwaved feed COD_F values are significantly different.

Table 44 Ratio of COD_F to COD_T for Low Temperature Operations

Date Sampled	FEED			MW FEED		
	Average COD_f (mg/L)	Average COD_T (mg/L)	$AvgCOD_F$/ $AvgCOD_T$	Average COD_f (mg/L)	Average COD_T (mg/L)	$AvgCOD_F$/ $AvgCOD_T$
52	305	20800	0.0147	2045	22112.5	0.0925
56	1170	19662.5	0.0595	2815	21587.5	0.1304
63	180	23050	0.0078	1875	23450	0.0800
66	900	23475	0.0383	2445	23225	0.1053
80	1105	19787.5	0.0558	2985	23362.5	0.1278
90	545	12412.5	0.0439	1545	14200	0.1088
93	360	21975	0.0164	1400	14087.5	0.0994
97	1465	24100	0.0608	3295	29575	0.1114
100	1260	14775	0.0853	1900	13350	0.1423
101	900	37400	0.0241	2710	31625	0.0857
104	1010	23450	0.0431	2580	25625	0.1007
107	220	15925	0.0138	1500	14250	0.1053
108	1440	39075	0.0369	3460	34625	0.0999
121	740	18187.5	0.0407	2220	17962.5	0.1236
128	215	14200	0.0151	1640	14562.5	0.1126
132	580	20350	0.0285	2500	20800	0.1202
MEAN =	**775**	**21789**	**0.0365**	**2307**	**21525**	**0.1091**
STDEV =	**445**	**7343**	**0.0214**	**645**	**6613**	**0.0168**
VAR =	**197725**	**53914539**	**0.0005**	**416297**	**43731688**	**0.0003**

% Increase from Feed to Microwave = (0.1091-0.0365)/0.0365*100 = 199%

Appendix B.4

Table 45 High Temperature COD_F Data for Paired T-Test

Date Sampled	FEED		MW FEED		Difference COD_F AvgMWFeed-AvgFeed (mg/L)
	COD_F (mg/L)	Average COD_F (mg/L)	COD_F (mg/L)	Average COD_F (mg/L)	
210	350	270	1760	1640	1370
210	190		1520		
213	400	410	1450	1585	1175
213	420		1720		
217	550	400	1240	1520	1120
217	250		1800		
220	1760	1370	2500	2265	895
220	980		2030		
223	560	350	700	620	270
223	140		540		
227	0	0	590	570	570
227	0		550		
230	0	100	1080	1295	1195
230	200		1510		
233	140	240	1820	2210	1970
233	340		2600		
237	1050	970	2620	2935	1965
237	890		3250		
240	1610	1300	3070	3225	1925
240	990		3380		
244	550	490	2140	2135	1645
244	430		2130		
247	550	575	2760	2790	2215
247	600		2820		
251	900	955	1480	1500	545
251	1010		1520		
254	260	275	860	860	585
254	290		860		
258	530	550	1520	1465	915
258	570		1410		
261	660	635	2390	2425	1790
261	610		2460		
266	70	110	2430	2370	2260
266	150		2310		
268	480	445	2060	2115	1670
268	410		2170		
273	480	460	1740	1720	1260
273	440		1700		
275	480	545	2490	2500	1955
275	610		2510		
279	450	420	2180	2100	1680
279	390		2020		
282	650	610	2580	2635	2025
282	570		2690		
MEAN =	**525**	**506**	**2104**	**2051**	**1409**
STDEV =	**374**	**355**	**729**	**721**	**595**
VAR =	**142420**	**128667**	**543573**	**541229**	**353652**

Table 46 Filtered COD Paired T-Test Information for High Temperature

	Low Temperature Paired t-test
Number of Samples (n)	22
Mean COD_S Difference Between Feed and Microwaved Feed (d)	1409 mg/L
Standard Deviation of the COD_F Difference (s_d)	595 mg^2/L^2
Standard Error of Mean COD_F Difference (SE_d)	126.8 mg/L
$t_{calculated}$	11.1
$t_{critical}$ (95% C.I.)	2.080

The results of the paired t-test, presented above, indicate that the $t_{calculated}$ value of 11.1 is higher than the $t_{critical}$ value of 2.831 and thus the feed and the microwaved feed COD_F values are significantly different.

Table 47 Ratio of COD_F to COD_T for High Temperature Operations

Date Sampled	Date Sampled	FEED			MW FEED		
		Average COD_f (mg/L)	Average COD_T (mg/L)	$AvgCOD_F$/ $AvgCOD_T$	Average COD_f (mg/L)	Average COD_T (mg/L)	$AvgCOD_F$/ $AvgCOD_T$
6-Jan-09	210	270	17075	0.0158	1640	17837.5	0.0919
9-Jan-09	213	410	14425	0.0284	1585	14737.5	0.1075
13-Jan-09	217	400	14762.5	0.0271	1520	17337.5	0.0877
16-Jan-09	220	1370	14925	0.0918	2265	19750	0.1147
19-Jan-09	223	350	9075	0.0386	620	10275	0.0603
23-Jan-09	227	0	16112.5	0.0000	570	17512.5	0.0325
26-Jan-09	230	100	15637.5	0.0064	1295	19562.5	0.0662
29-Jan-09	233	240	18937.5	0.0127	2210	21325	0.1036
2-Feb-09	237	970	20975	0.0462	2935	22637.5	0.1297
5-Feb-09	240	1300	23637.5	0.0550	3225	22962.5	0.1404
9-Feb-09	244	490	17937.5	0.0273	2135	20525	0.1040
12-Feb-09	247	575	21887.5	0.0263	2790	22687.5	0.1230
16-Feb-09	251	955	13275	0.0719	1500	12100	0.1240
19-Feb-09	254	275	7837.5	0.0351	860	6425	0.1339
23-Feb-09	258	550	13375	0.0411	1465	12525	0.1170
26-Feb-09	261	635	20625	0.0308	2425	23500	0.1032
3-Mar-09	266	110	20300	0.0054	2370	27337.5	0.0867
5-Mar-09	268	445	18650	0.0239	2115	21512.5	0.0983
10-Mar-09	273	460	25075	0.0183	1720	22312.5	0.0771
12-Mar-09	275	545	23275	0.0234	2500	26487.5	0.0944
16-Mar-09	279	420	19375	0.0217	2100	26337.5	0.0797
19-Mar-09	282	610	25062.5	0.0243	2635	27175	0.0970
MEAN =	**MEAN =**	**506**	**18890**	**0.0291**	**2051**	**20744**	**0.1032**
STDEV =	**STDEV =**	**355**	**4719**	**0.0214**	**721**	**5662**	**0.0258**
VAR =	**VAR =**	**128667**	**23350400**	**0.0005**	**541229**	**33481539**	**0.0007**

% Increase from Feed to Microwave = (0.1032-0.0291)/0.0291*100 = 254%

Appendix C
Solids Pretreatment Data

Appendix C.1

Table 48 Volatile and Total Solids Data for Low and High Temperature Operations

Date Sampled	VOLATILE SOLIDS (mg/L)				TOTAL SOLIDS (mg/L)			
	Feed	Duplicate Difference Feed	MW Feed	Duplicate Difference MWFeed	Feed	Duplicate Difference Feed	MW Feed	Duplicate Difference MWFeed
14	10290	-40	10890	-390	13810	-90	14630	-590
14	10330		11280		13900		15220	
17	13150	350	15690	-680	17950	460	21730	-570
17	12800		16370		17490		22300	
31	4170	-40	10740	-750	6520	2810	14000	-1400
31	4210		11490		3710		15400	
35	10790	-350	15240	-2120	15660	-450	21900	-3000
35	11140		17360		16110		24900	
38	7300	-40	17030	-1810	11120	-140	24760	-2610
38	7340		18840		11260		27370	
42	10200	-40	12970	760	15200	-110	19270	1390
42	10240		12210		15310		17880	
45	6870	-100	13480	-1080	10420	-70	19980	-1530
45	6970		14560		10490		21510	
49	12750	120	13080	-310	18810	170	19530	-460
49	12630		13390		18640		19990	
52	14310	-200	16590	180	20430	-240	23830	190
52	14510		16410		20670		23640	
56	13330	320	14160	-100	19700	370	20980	-140
56	13010		14260		19330		21120	
63	13450	-280	14620	-80	21000	-420	22940	-70
63	13730		14700		21420		23010	
66	14190	30	14270	-270	22400	60	22610	-490
66	14160		14540		22340		23100	
80	12340	220	15110	-260	18900	240	23190	-420
80	12120		15370		18660		23610	
90	8500	810	9900	-420	13000	1190	15300	-670
90	7690		10320		11810		15970	
93	13370	10	8680	70	20710	40	13540	100
93	13360		8610		20670		13440	
97	16470	200	17180	-100	25460	340	26410	-100
97	16270		17280		25120		26510	
100	10450	-90	9370	100	16260	-20	14520	140
100	10540		9270		16280		14380	
101	21290	30	20330	-240	32960	270	31130	-320
101	21260		20570		32690		31450	
104	12990	10	14450	5180	19780	50	22010	9400
104	12980		9270		19730		12610	
107	7960	170	8030	40	12490	140	12570	70
107	7790		7990		12350		12500	
108	21840	1880	18850	-10	31330	490	29000	-10
108	19960		18860		30840		29010	
121	11380	70	11260	80	16870	60	16730	140
121	11310		11180		16810		16590	
128	9340	30	9170	0	13680	20	13420	10
128	9310		9170		13660		13410	
132	11680	30	13310	-90	16910	-10	19170	-380
132	11650		13400		16920		19550	
210	11330	130	12040	-880	15840	170	16920	-1180
210	11200		12920		15670		18100	
213	9140	-120	10400	-610	12670	-150	14340	-880
213	9260		11010		12820		15220	
217	10610	150	12130	100	14560	130	16640	50
217	10460		12030		14430		16590	
220	9780	230	11400	-360	13680	260	15810	-470
220	9550		11760		13420		16280	
223	5870	-10	6350	10	8650	-10	9260	-10
223	5880		6340		8660		9270	

Date Sampled	VOLATILE SOLIDS (mg/L)				TOTAL SOLIDS (mg/L)			
	Feed	Duplicate Difference Feed	MW Feed	Duplicate Difference MWFeed	Feed	Duplicate Difference Feed	MW Feed	Duplicate Difference MWFeed
227	11200	-60	12860	-320	15500	-140	17730	-420
227	11260		13180		15640		18150	
230	11050	250	13140	-410	15400	270	18350	-530
230	10800		13550		15130		18880	
233	12230	190	14130		16940	280	19340	
233	12040				16660			
237	11890	-380			16690	-400	18640	270
237	12270		13490		17090		18370	
240	14120	320	15020	-110	19200	410	20440	-160
240	13800		15130		18790		20600	
244	11580	140	13620	-340	15930	230	18550	-470
244	11440		13960		15700		19020	
247	13250	90	14660	-170	18740	230	20590	-230
247	13160		14830		18510		20820	
251	10680	1550	7250	-10	15780	2070	10990	-70
251	9130		7260		13710		11060	
254	4520	0	3980	140	7440	60	6510	140
254	4520		3840		7380		6370	
258	7980	0	7910	190	12200	190	11730	340
258	7980		7720		12010		11390	
261	11890	340	13240	-1720	16880	500	18350	-2500
261	11550		14960		16380		20850	
266	12660	20	17220	-420	17200	-30	23270	-590
266	12640		17640		17230		23860	
268	12140	160	13420	-1790	16630	240	18140	-2380
268	11980		15210		16390		20520	
273	14630	400	14080	-240	20400	550	19630	-330
273	14230		14320		19850		19960	
275	14450	180	16990	-600	20240	270	23650	-800
275	14270		17590		19970		24450	
279	12380	180	16310	-400	17820	230	23840	-670
279	12200		16710		17590		24510	
282	15030	260	17760	-530	21290	360	24960	-690
282	14770		18290		20930		25650	
MEAN =				**-41**				**-22**
STDEV =				**786**				**1275**
VAR =				**618473**				**1626655**

Appendix C.2

Table 49 Low Temperature TS Data for Paired T-Test

Date Sampled	FEED		MW FEED		Difference TS AvgFeed-AvgMWFeed (mg/L)
	TS (mg/L)	Average TS (mg/L)	TS (mg/L)	Average TS (mg/L)	
14	13810	13855	14630	14925	-1070
14	13900		15220		
17	17950	17720	21730	22015	-4295
17	17490		22300		
31	6520	5115	14000	14700	-9585
31	3710		15400		
35	15660	15885	21900	23400	-7515
35	16110		24900		
38	11120	11190	24760	26065	-14875
38	11260		27370		
42	15200	15255	19270	18575	-3320
42	15310		17880		
45	10420	10455	19980	20745	-10290
45	10490		21510		
49	18810	18725	19530	19760	-1035
49	18640		19990		
52	20430	20550	23830	23735	-3185
52	20670		23640		
56	19700	19515	20980	21050	-1535
56	19330		21120		
63	21000	21210	22940	22975	-1765
63	21420		23010		
66	22400	22370	22610	22855	-485
66	22340		23100		
80	18900	18780	23190	23400	-4620
80	18660		23610		
90	13000	12405	15300	15635	-3230
90	11810		15970		
93	20710	20690	13540	13490	7200
93	20670		13440		
97	25460	25290	26410	26460	-1170
97	25120		26510		
100	16260	16270	14520	14450	1820
100	16280		14380		
101	32960	32825	31130	31290	1535
101	32690		31450		
104	19780	19755	22010	17310	2445
104	19730		12610		
107	12490	12420	12570	12535	-115
107	12350		12500		
108	31330	31085	29000	29005	2080
108	30840		29010		
121	16870	16840	16730	16660	180
121	16810		16590		
128	13680	13670	13420	13415	255
128	13660		13410		
132	16910	16915	19170	19360	-2445
132	16920		19550		
MEAN =	**17866**	**17866**	**20159**	**20159**	**-2293**
STDEV =	**6124**	**6181**	**5220**	**5160**	**4681**
VAR =	**37503522**	**38202633**	**27253015**	**26622355**	**21910791**

Table 50 High Temperature TS Data for Paired T-Test

Date Sampled	FEED		MW FEED		Difference TS AvgFeed-AvgMWFeed (mg/L)
	TS (mg/L)	Average TS (mg/L)	TS (mg/L)	Average TS (mg/L)	
210	15840	15755	16920	17510	-1755
210	15670		18100		
213	12670	12745	14340	14780	-2035
213	12820		15220		
217	14560	14495	16640	16615	-2120
217	14430		16590		
220	13680	13550	15810	16045	-2495
220	13420		16280		
223	8650	8655	9260	9265	-610
223	8660		9270		
227	15500	15570	17730	17940	-2370
227	15640		18150		
230	15400	15265	18350	18615	-3350
230	15130		18880		
233	16940	16800	19340	19340	-2540
233	16660				
237	16690	16890	18640	18505	-1615
237	17090		18370		
240	19200	18995	20440	20520	-1525
240	18790		20600		
244	15930	15815	18550	18785	-2970
244	15700		19020		
247	18740	18625	20590	20705	-2080
247	18510		20820		
251	15780	14745	10990	11025	3720
251	13710		11060		
254	7440	7410	6510	6440	970
254	7380		6370		
258	12200	12105	11730	11560	545
258	12010		11390		
261	16880	16630	18350	19600	-2970
261	16380		20850		
266	17200	17215	23270	23565	-6350
266	17230		23860		
268	16630	16510	18140	19330	-2820
268	16390		20520		
273	20400	20125	19630	19795	330
273	19850		19960		
275	20240	20105	23650	24050	-3945
275	19970		24450		
279	17820	17705	23840	24175	-6470
279	17590		24510		
282	21290	21110	24960	25305	-4195
282	20930		25650		
MEAN =	**15765**	**15765**	**17851**	**17885**	**-2120**
STDEV =	**3388**	**3417**	**4893**	**4877**	**2269**
VAR =	**11476286**	**11678914**	**23937582**	**23783250**	**5146128**

Table 51 Total Solids Paired T-Test Information

	Low Temperature Paired t-test	High Temperature Paired t-test
Number of Samples (n)	24	22
Mean TS Difference Between Feed and Microwaved Feed (d)	-2293 mg/L	-2120 mg/L
Standard Deviation of the TS Difference (s_d)	4681 mg^2/L^2	2269 mg^2/L^2
Standard Error of Mean TS Difference (SE_d)	955.5 mg/L	483.6 mg/L
$t_{calculated}$	-2.4	-4.4
$t_{critical}$ (95% C.I.)	2.069	2.080

The results from the paired t-test displayed in the table above show that for both the low temperature operations the TS sample sets for the feed and the microwaved feed were significantly different. The $t_{calculated}$ value for the low temperature TS comparison was determined to be -2.4, which has an absolute value greater than the $t_{critical}$ value of 2.069 indicating that the feed and the microwaved feed were significantly different. For the high temperature operations the $t_{calculated}$ value for the high temperature TS comparison was determined to be -4.4, the absolute value of which is greater than the $t_{critical}$ value of 2.080 indicating that the feed and the microwaved feed were significantly different.

Appendix C.3

Table 52 Low Temperature VS Data for Paired T-Test

Date Sampled	FEED		MW FEED		Difference VS AvgFeed-AvgMWFeed (mg/L)
	VS (mg/L)	Average VS (mg/L)	VS (mg/L)	Average VS (mg/L)	
14	10290	10310	10890	11085	-775
14	10330		11280		
17	13150	12975	15690	16030	-3055
17	12800		16370		
31	4170	4190	10740	11115	-6925
31	4210		11490		
35	10790	10965	15240	16300	-5335
35	11140		17360		
38	7300	7320	17030	17935	-10615
38	7340		18840		
42	10200	10220	12970	12590	-2370
42	10240		12210		
45	6870	6920	13480	14020	-7100
45	6970		14560		
49	12750	12690	13080	13235	-545
49	12630		13390		
52	14310	14410	16590	16500	-2090
52	14510		16410		
56	13330	13170	14160	14210	-1040
56	13010		14260		
63	13450	13590	14620	14660	-1070
63	13730		14700		
66	14190	14175	14270	14405	-230
66	14160		14540		
80	12340	12230	15110	15240	-3010
80	12120		15370		
90	8500	8095	9900	10110	-2015
90	7690		10320		
93	13370	13365	8680	8645	4720
93	13360		8610		
97	16470	16370	17180	17230	-860
97	16270		17280		
100	10450	10495	9370	9320	1175
100	10540		9270		
101	21290	21275	20330	20450	825
101	21260		20570		
104	12990	12985	14450	11860	1125
104	12980		9270		
107	7960	7875	8030	8010	-135
107	7790		7990		
108	21840	20900	18850	18855	2045
108	19960		18860		
121	11380	11345	11260	11220	125
121	11310		11180		
128	9340	9325	9170	9170	155
128	9310		9170		
132	11680	11665	13310	13355	-1690
132	11650		13400		
MEAN =	**11953**	**11953**	**13565**	**13565**	**-1612**
STDEV =	**3926**	**3962**	**3396**	**3371**	**3272**
VAR =	**15415304**	**15698015**	**11529430**	**11366109**	**10703950**

Table 53 High Temperature VS Data for Paired T-Test

Date Sampled	FEED		MW FEED		Difference VS AvgFeed-AvgMWFeed (mg/L)
	VS (mg/L)	Average VS (mg/L)	VS (mg/L)	Average VS (mg/L)	
210	11330	11265	12040	12480	-1215
210	11200		12920		
213	9140	9200	10400	10705	-1505
213	9260		11010		
217	10610	10535	12130	12080	-1545
217	10460		12030		
220	9780	9665	11400	11580	-1915
220	9550		11760		
223	5870	5875	6350	6345	-470
223	5880		6340		
227	11200	11230	12860	13020	-1790
227	11260		13180		
230	11050	10925	13140	13345	-2420
230	10800		13550		
233	12230	12135	14130	14130	-1995
233	12040				
237	11890	12080		13490	-1410
237	12270		13490		
240	14120	13960	15020	15075	-1115
240	13800		15130		
244	11580	11510	13620	13790	-2280
244	11440		13960		
247	13250	13205	14660	14745	-1540
247	13160		14830		
251	10680	9905	7250	7255	2650
251	9130		7260		
254	4520	4520	3980	3910	610
254	4520		3840		
258	7980	7980	7910	7815	165
258	7980		7720		
261	11890	11720	13240	14100	-2380
261	11550		14960		
266	12660	12650	17220	17430	-4780
266	12640		17640		
268	12140	12060	13420	14315	-2255
268	11980		15210		
273	14630	14430	14080	14200	230
273	14230		14320		
275	14450	14360	16990	17290	-2930
275	14270		17590		
279	12380	12290	16310	16510	-4220
279	12200		16710		
282	15030	14900	17760	18025	-3125
282	14770		18290		
MEAN =	**11200**	**11200**	**12754**	**12802**	**-1602**
STDEV =	**2593**	**2616**	**3729**	**3677**	**1613**
VAR =	**6722051**	**6842576**	**13902663**	**13520508**	**2602746**

Table 54 Total Solids Paired T-Test Information

	Low Temperature Paired t-test	High Temperature Paired t-test
Number of Samples (n)	24	22
Mean TS Difference Between Feed and Microwaved Feed (d)	-1612 mg/L	-1602 mg/L
Standard Deviation of the TS Difference (s_d)	3272 mg^2/L^2	1613 mg^2/L^2
Standard Error of Mean TS Difference (SE_d)	667.8 mg/L	344.0 mg/L
$t_{calculated}$	-2.4	-4.7
$t_{critical}$ (95% C.I.)	2.069	2.080

The results from the paired t-test displayed in the table above show that for both the low temperature operations the VS sample sets for the feed and the microwaved feed were significantly different. The $t_{calculated}$ value for the low temperature VS comparison was determined to be -2.4, which has an absolute value greater than the $t_{critical}$ value of 2.069 indicating that the feed and the microwaved feed were significantly different. For the high temperature operations the $t_{calculated}$ value for the high temperature TS comparison was determined to be -4.7, the absolute value of which is greater than the $t_{critical}$ value of 2.080 indicating that the feed and the microwaved feed were significantly different.

Appendix D
Solids Anaerobic Digestion Data

Appendix D.1

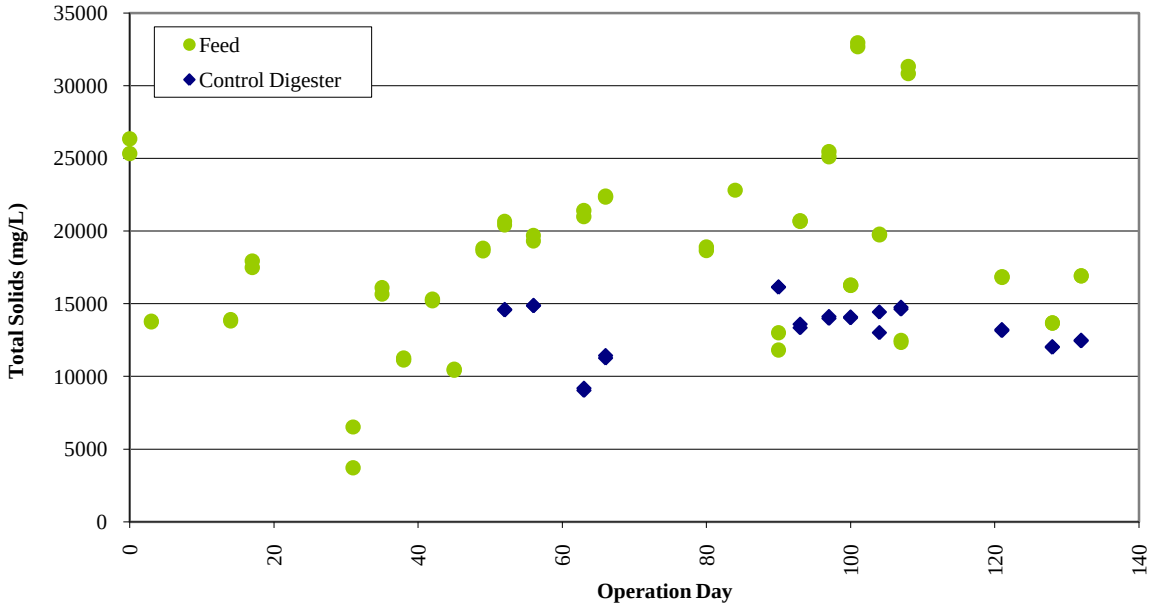

Figure 47 TS Concentration for Feed and Control Digester – Low Temperature

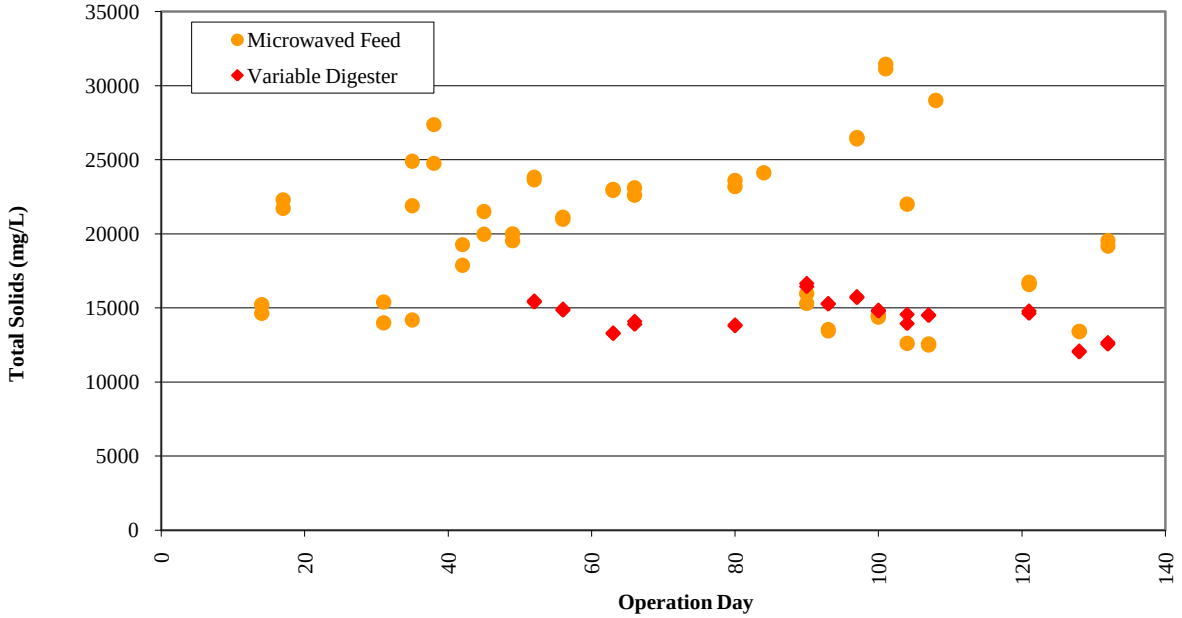

Figure 48 TS Concentration for MWFeed and Test Digester – Low Temperature

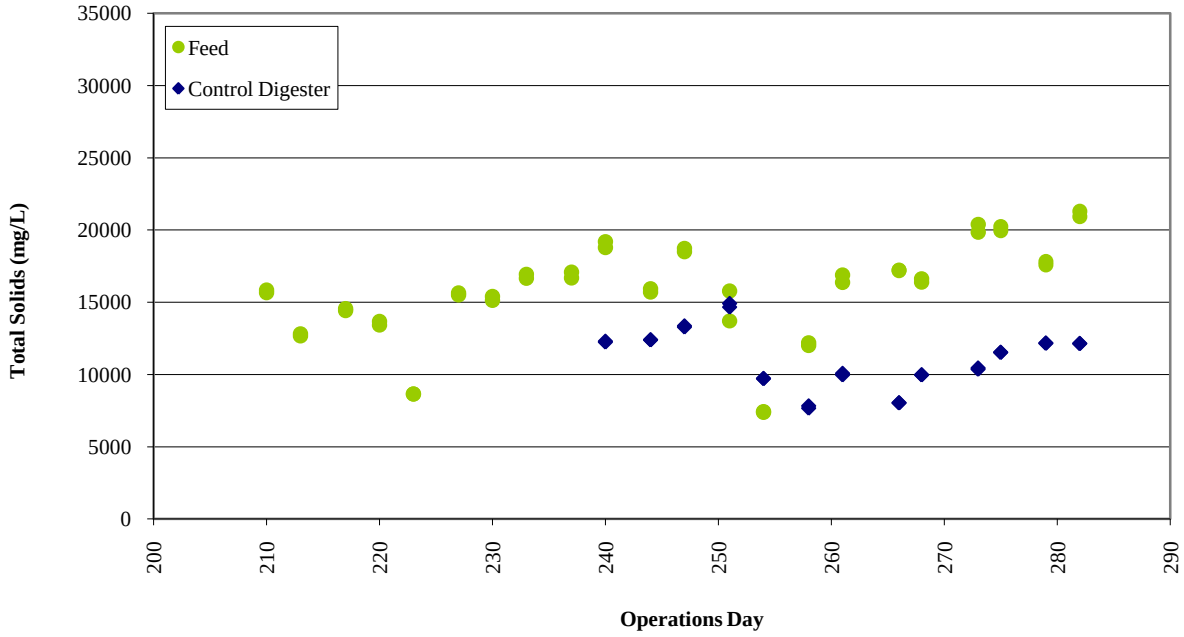

Figure 49 TS Concentration for Feed and Control Digester – High Temperature

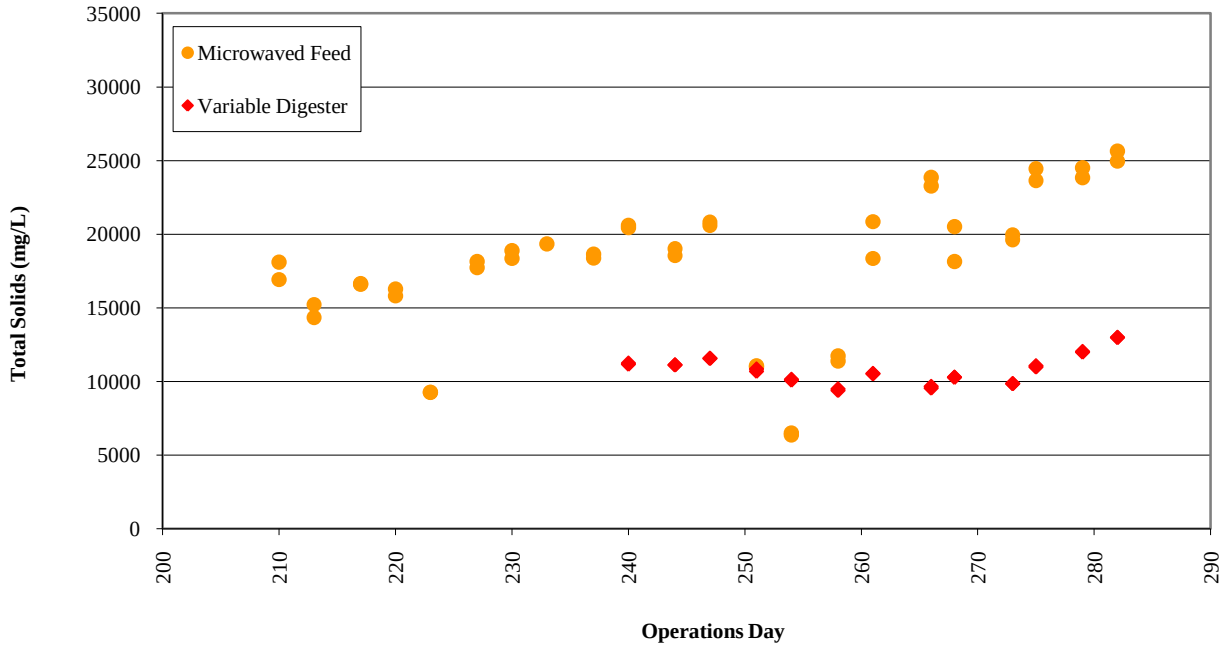

Figure 50 TS Concentration for MWFeed and Test Digester – High Temperature

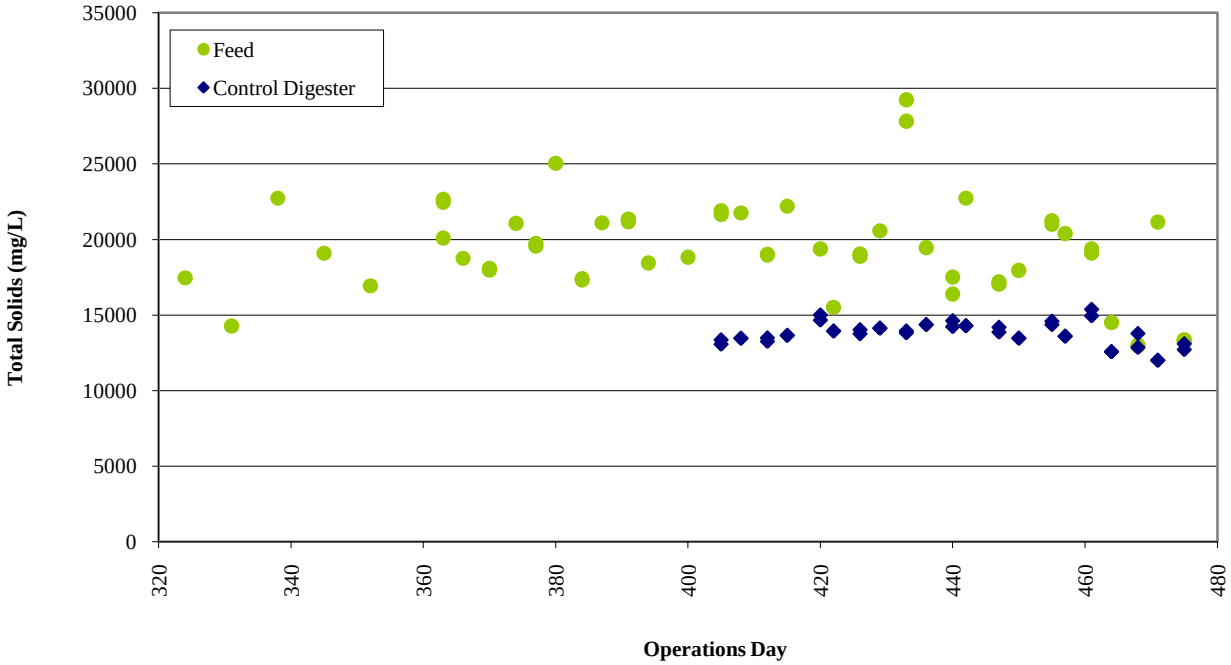

Figure 51 TS Concentration for Feed and Control Digester – Membrane

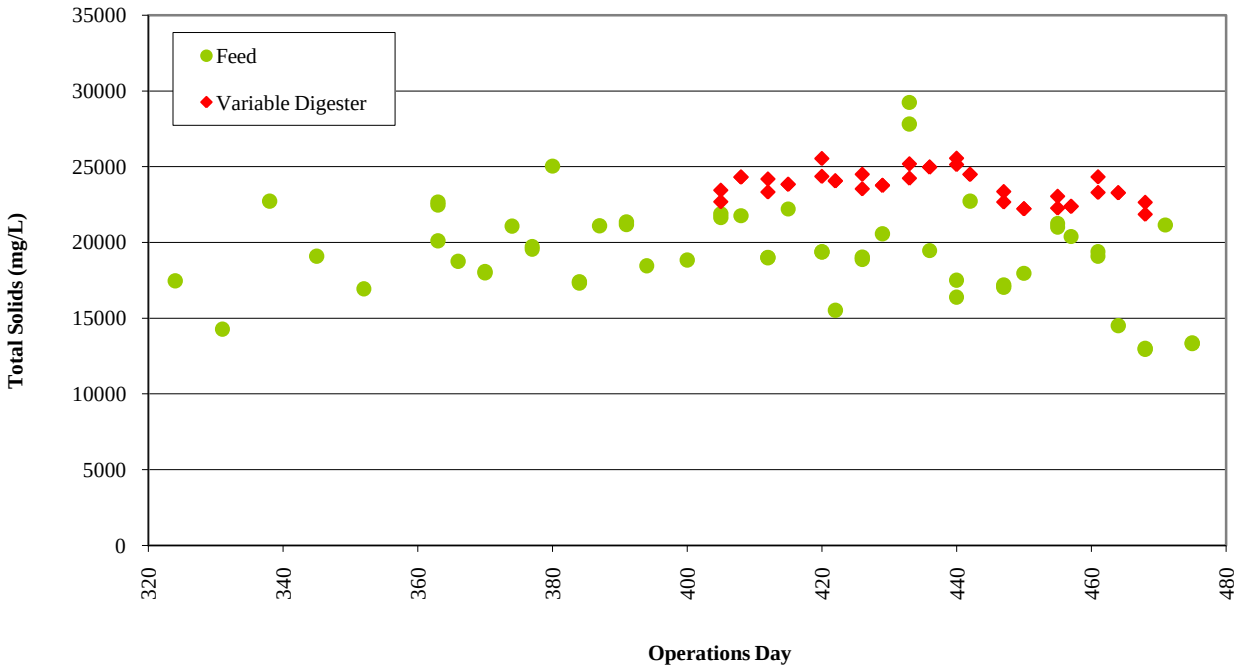

Figure 52 TS Concentration for MWFeed and Test Digester – Membrane

Appendix D.2

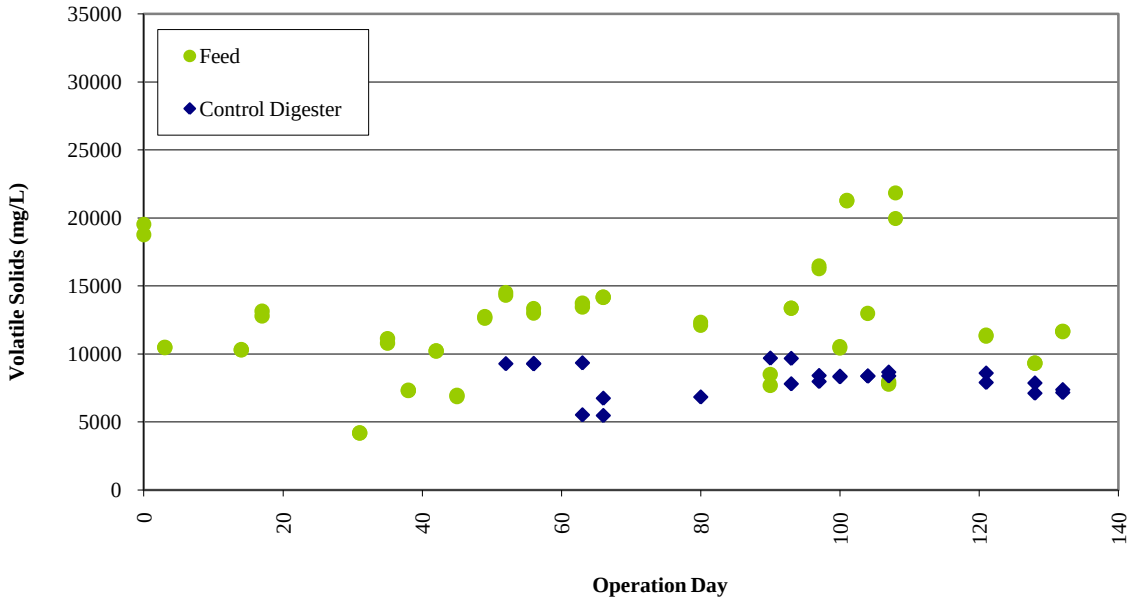

Figure 53 VS Concentration for Feed and Control Digester – Low Temperature

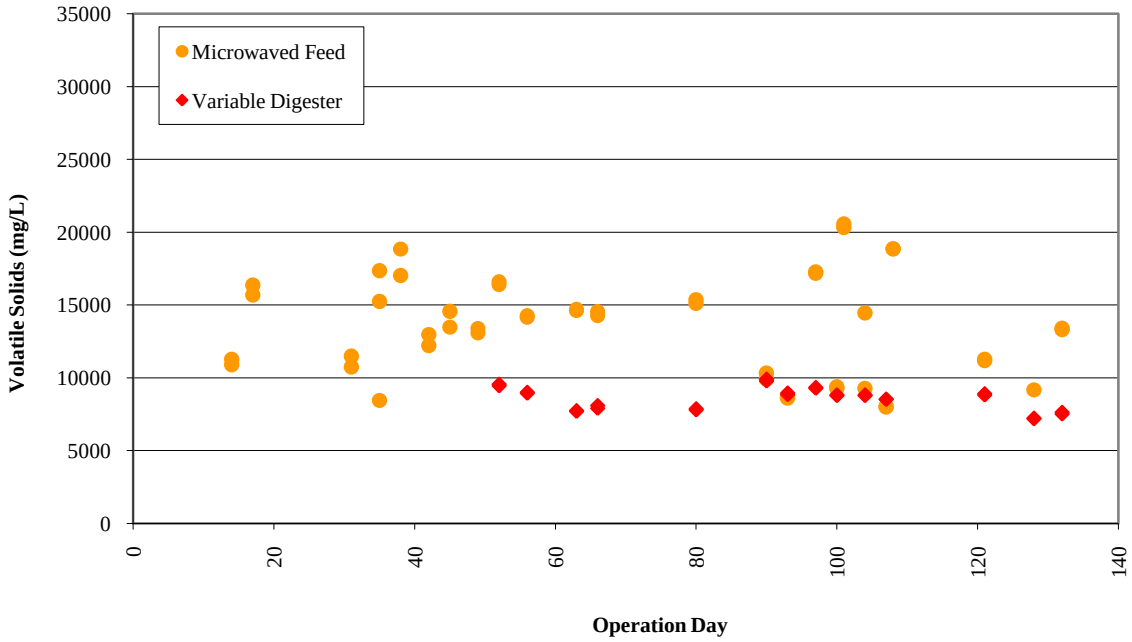

Figure 54 VS Concentration for MWFeed and Test Digester – Low Temperature

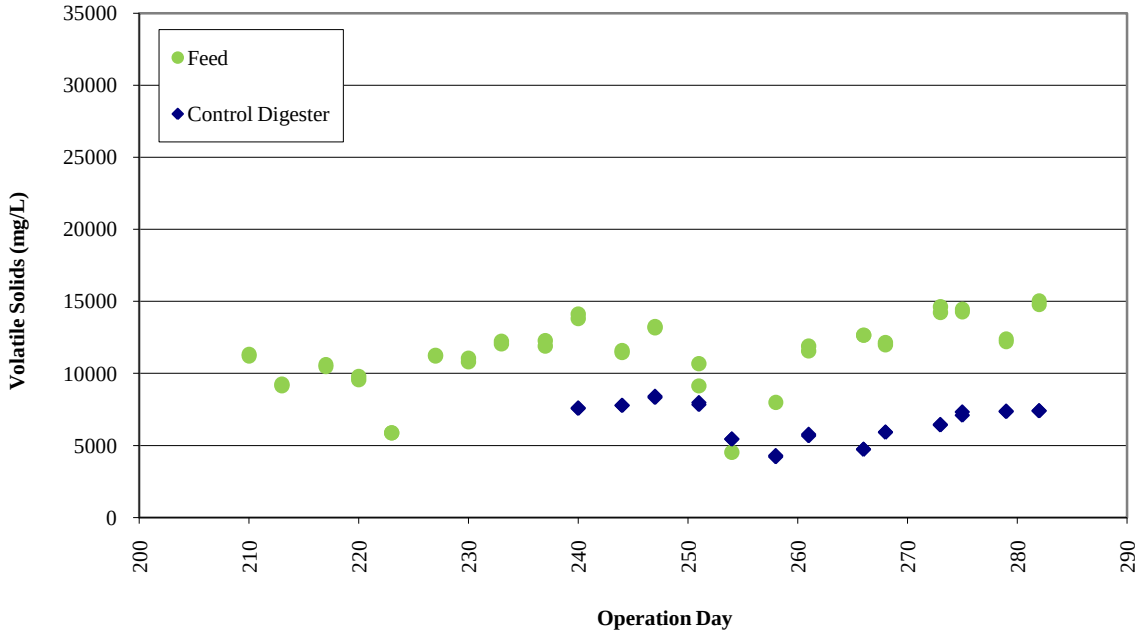

Figure 55 VS Concentration for Feed and Control Digester – High Temperature

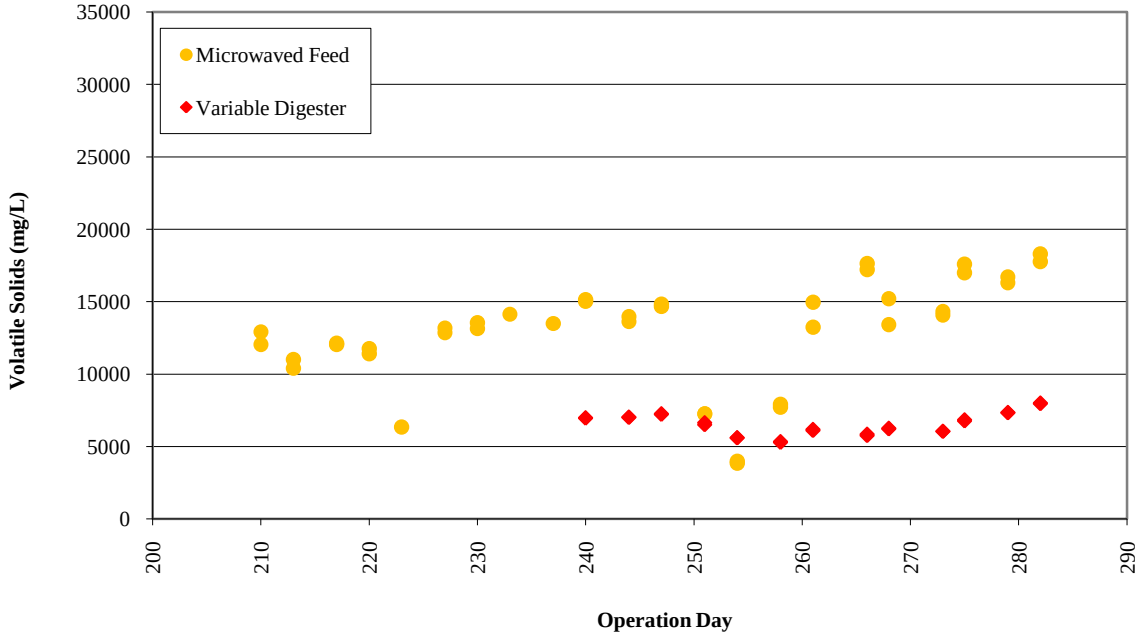

Figure 56 VS Concentration for MWFeed and Test Digester – High Temperature

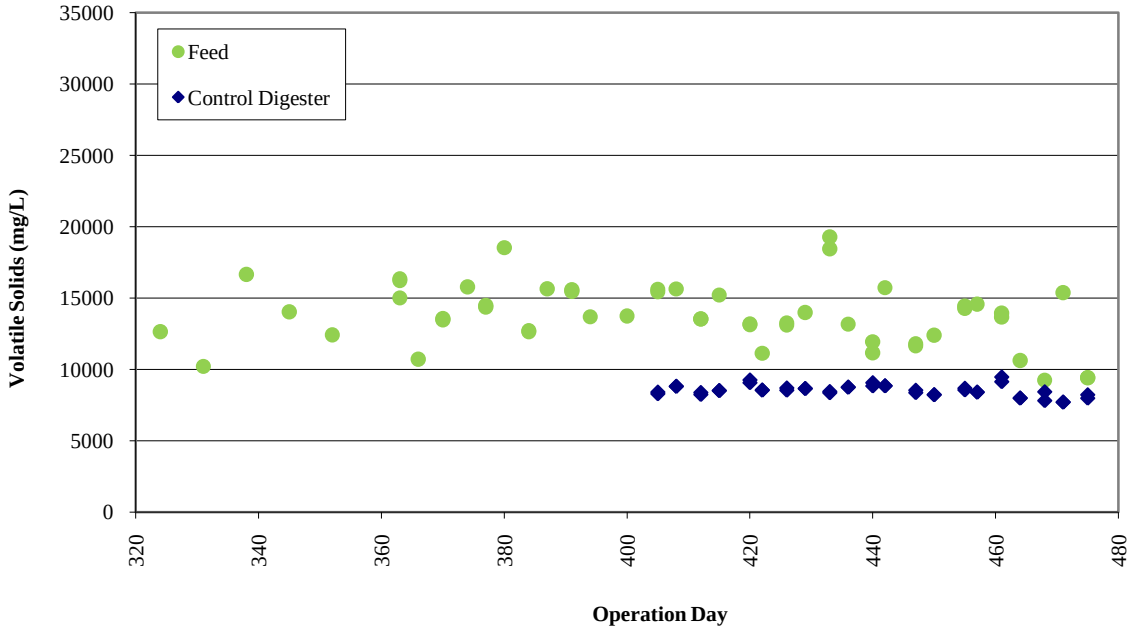

Figure 57 VS Concentration for Feed and Control Digester – Membrane

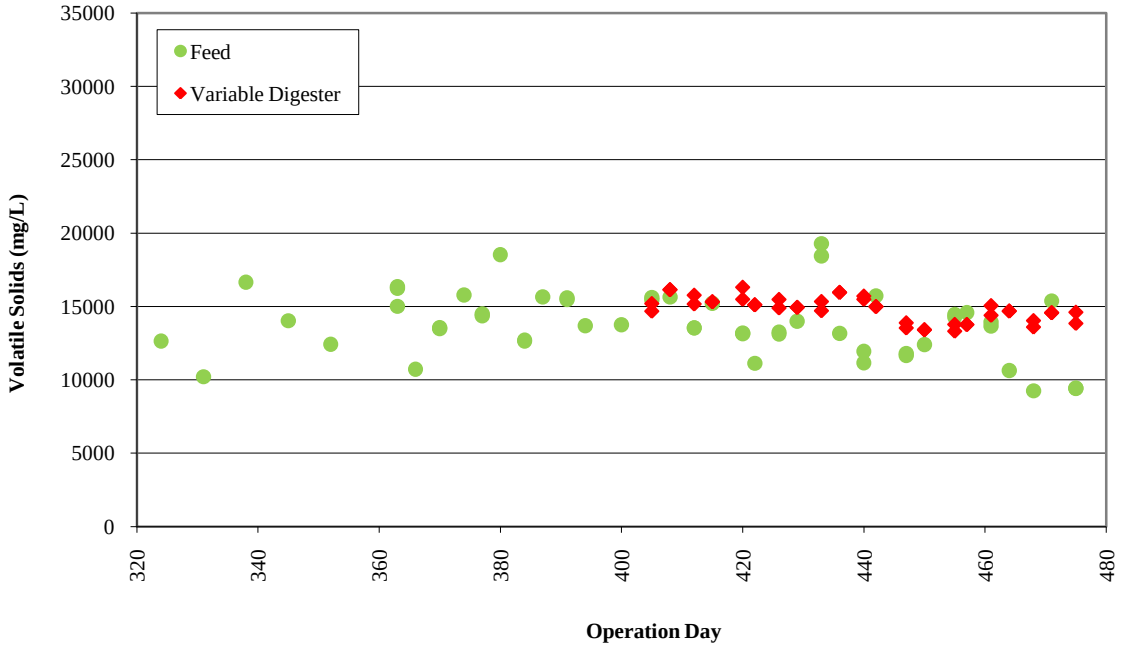

Figure 58 VS Concentration for MWFeed and Test Digester – Membrane

Appendix D.3

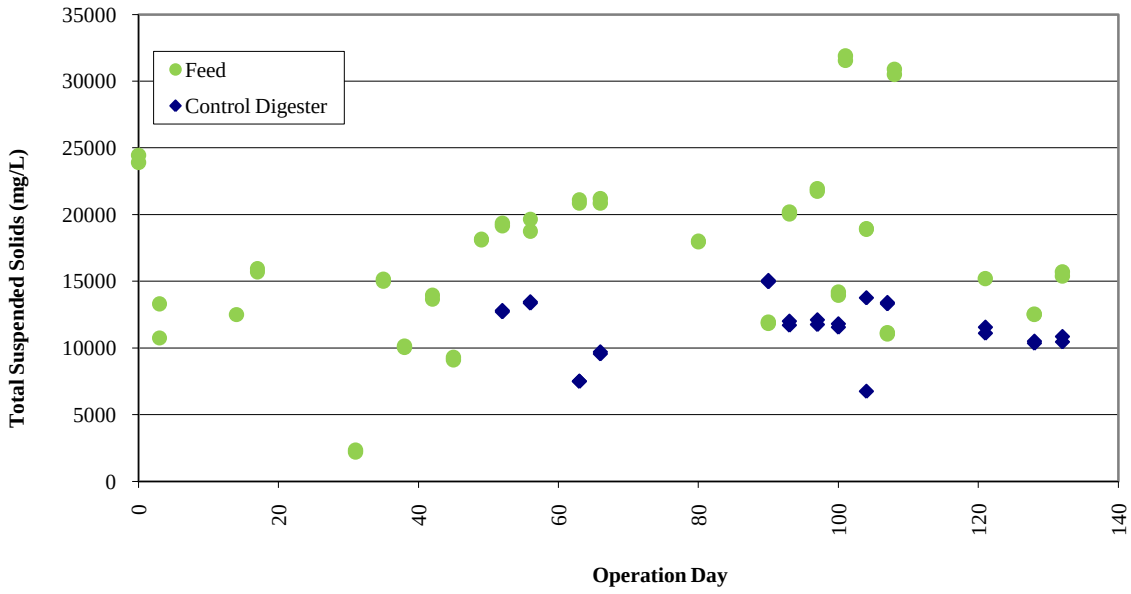

Figure 59 TSS Concentration for Feed and Control Digester – Low Temperature

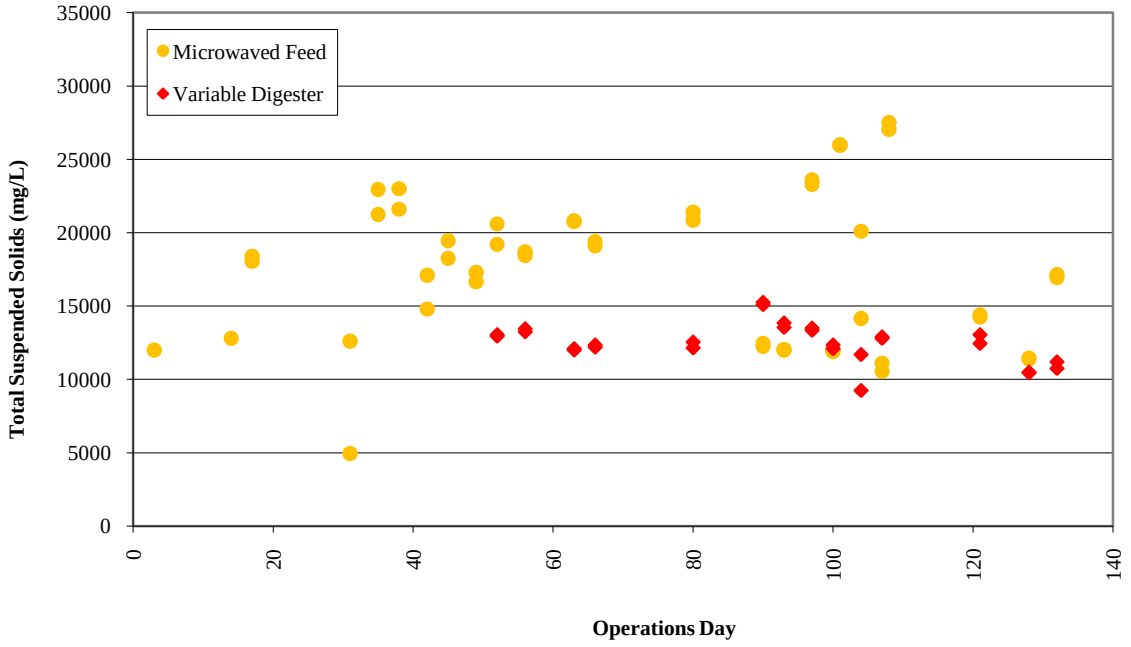

Figure 60 TSS Concentration for MWFeed and Test Digester – Low Temperature

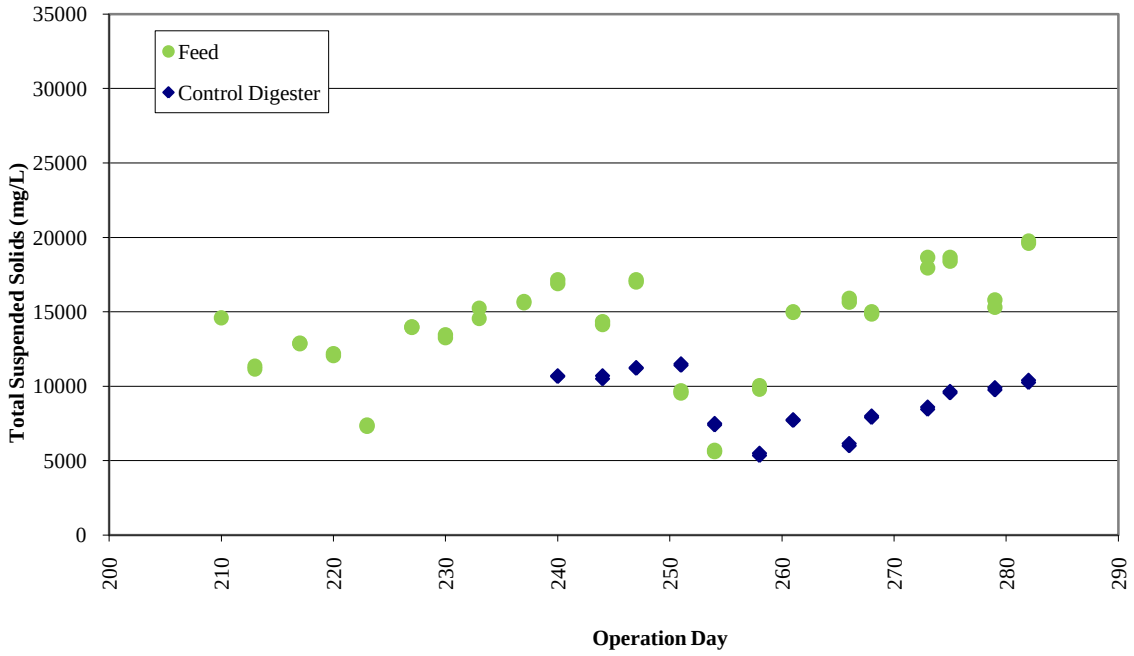

Figure 61 TSS Concentration for Feed and Control Digester – High Temperature

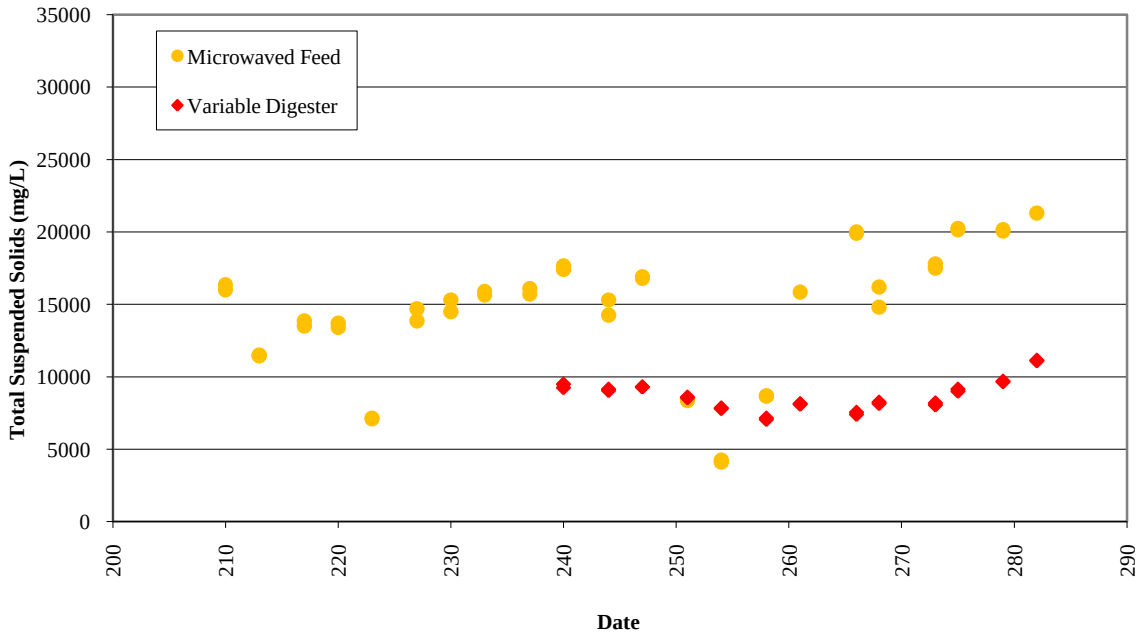

Figure 62 TSS Concentration for MWFeed and Test Digester – High Temperature

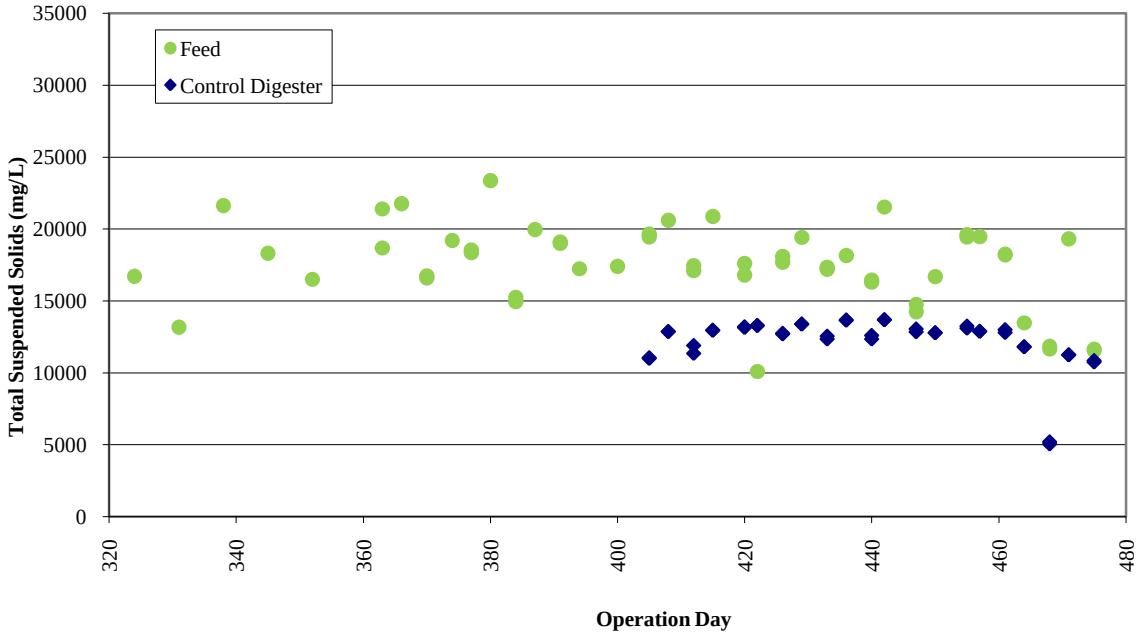

Figure 63 TSS Concentration for Feed and Control Digester – Membrane

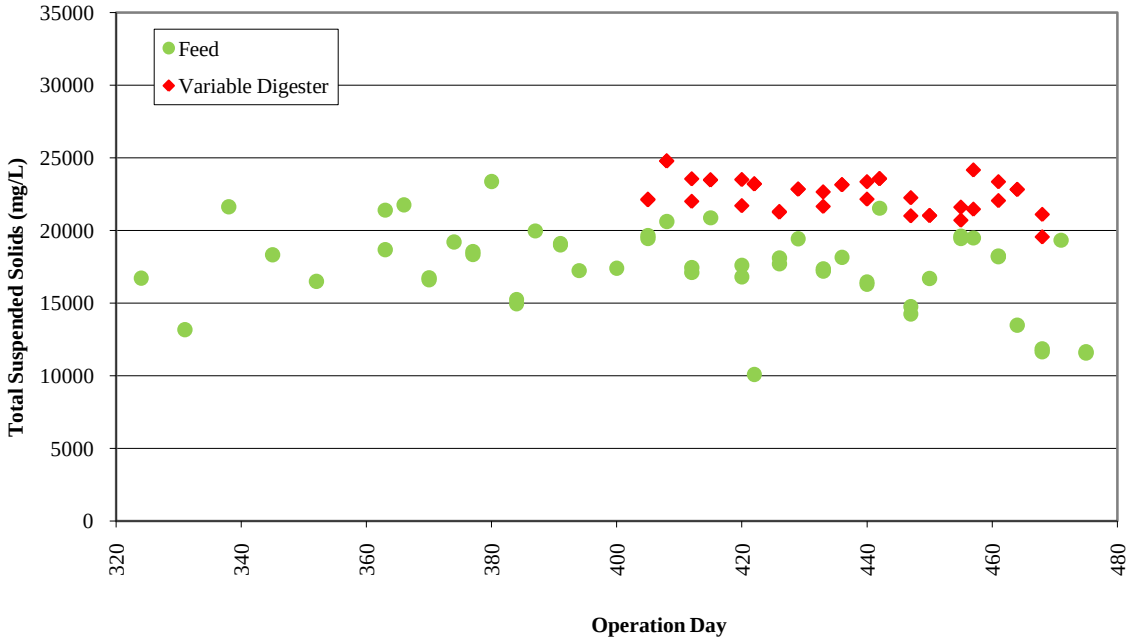

Figure 64 TSS Concentration for MWFeed and Test Digester – Membrane

Appendix D.4

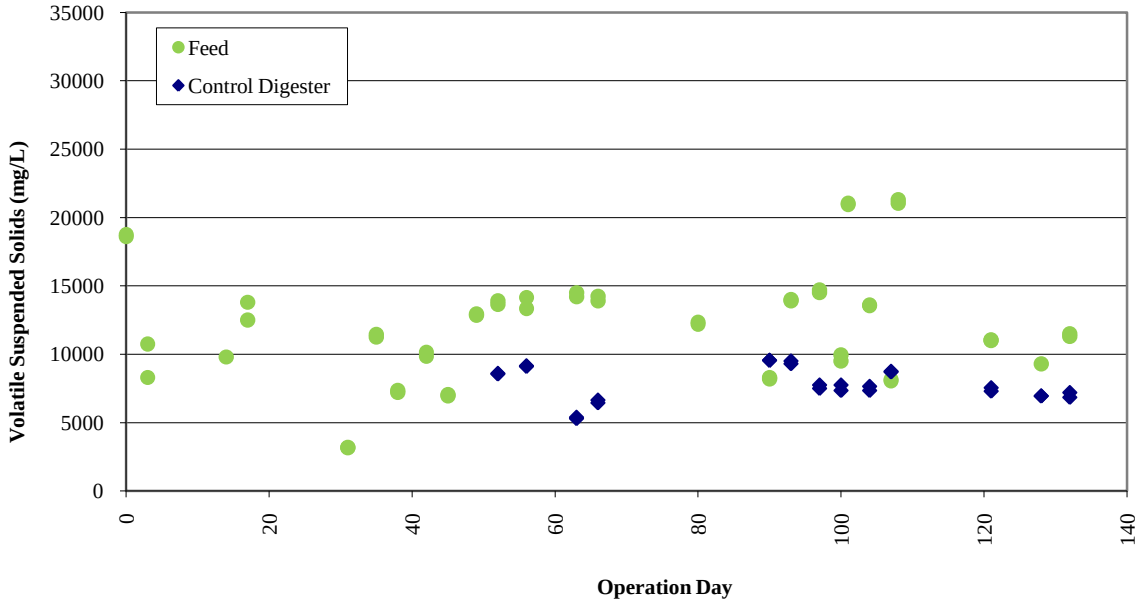

Figure 65 VSS Concentration for Feed and Control Digester – Low Temperature

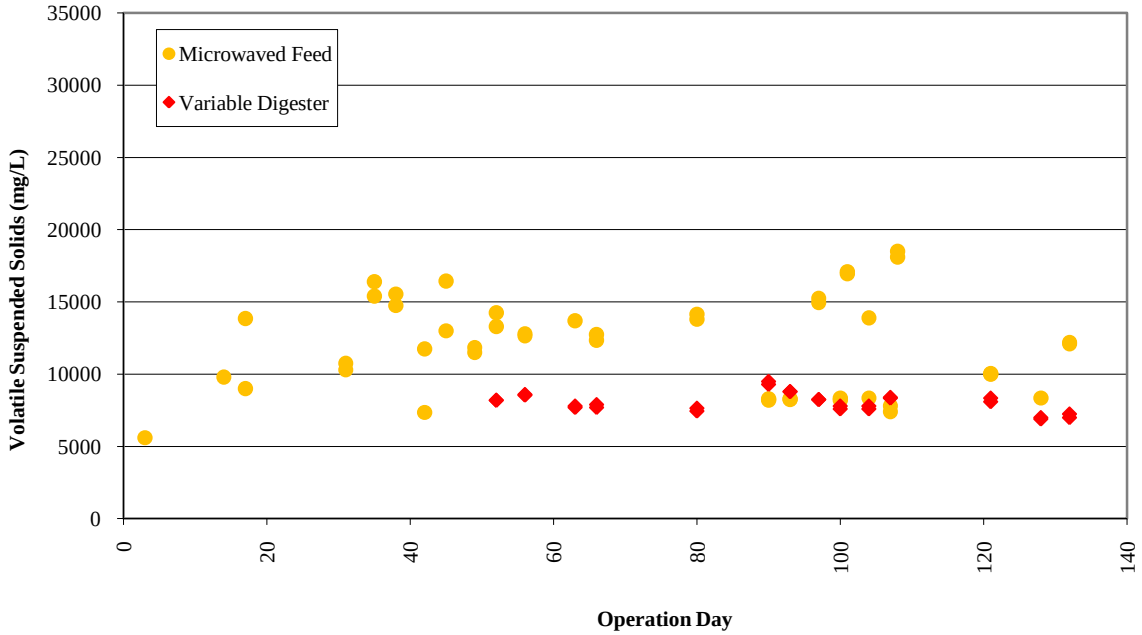

Figure 66 VSS Concentration for MWFeed and Test Digester – Low Temperature

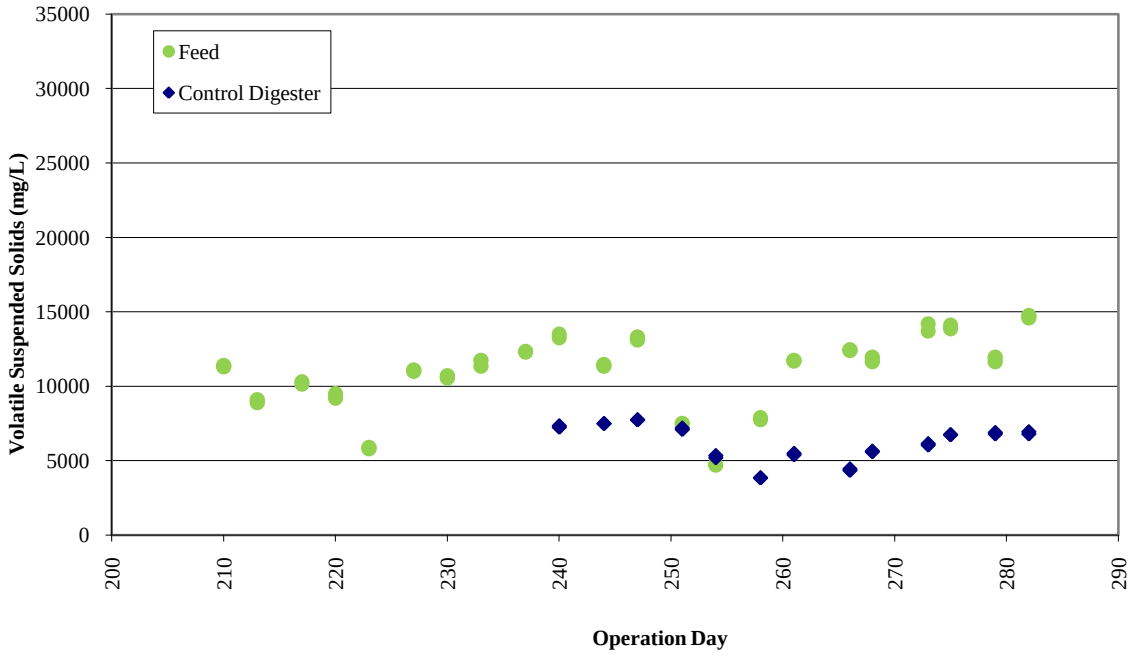

Figure 67 VSS Concentration for Feed and Control Digester – High Temperature

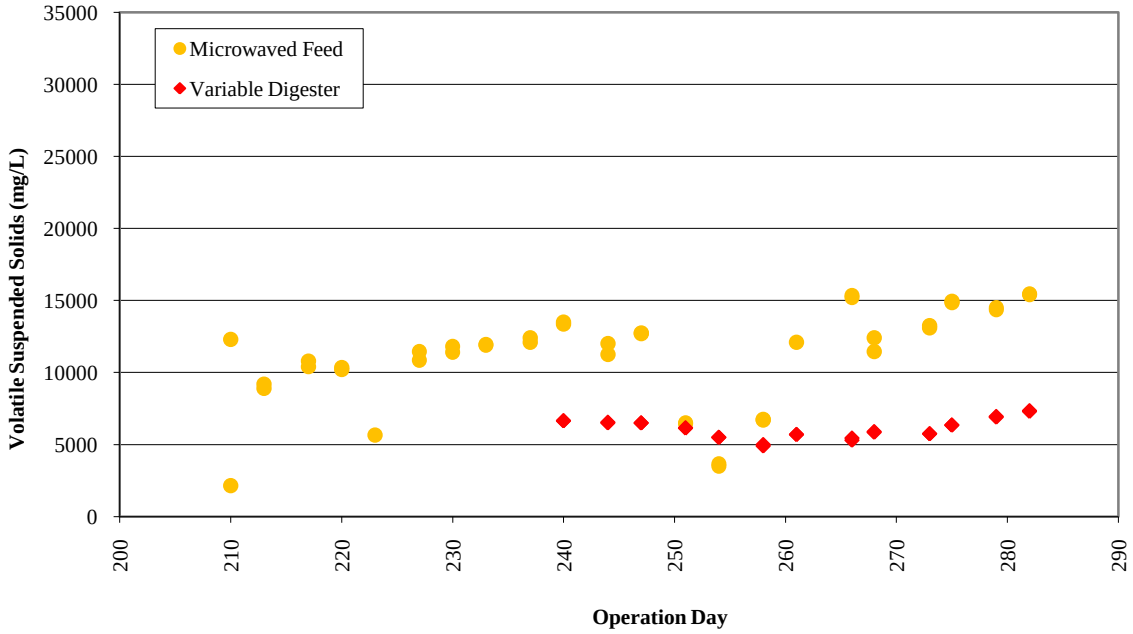

Figure 68 VSS Concentration for MWFeed and Test Digester – High Temperature

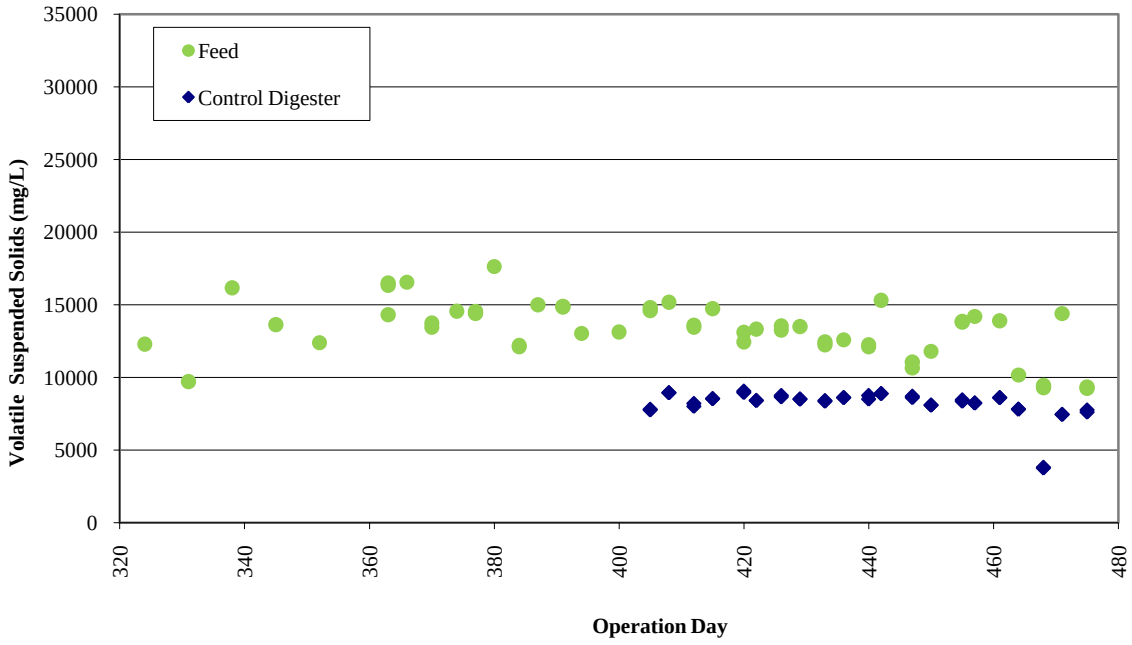

Figure 69 VSS Concentration for Feed and Control Digester – Membrane

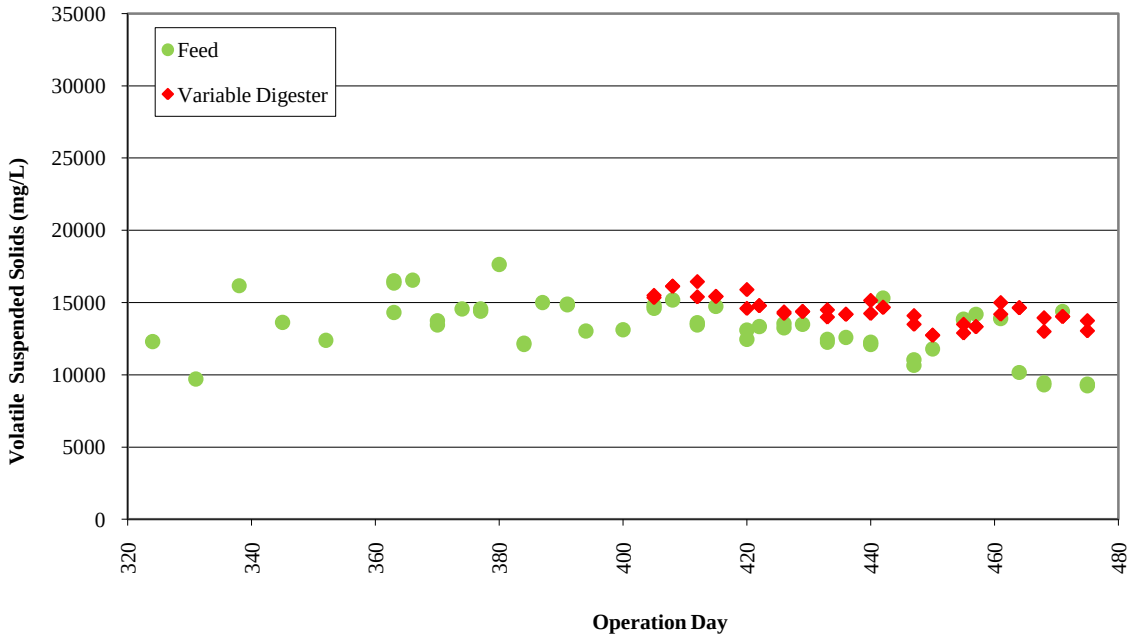

Figure 70 VSS Concentration for MWFeed and Test Digester – Membrane

Appendix E
Biogas Composition Data

Appendix E.1

Table 55 Biogas Characteristics During Membrane Operations

Control					Membrane				
Date	**O_2**	**N_2**	**CH_4**	**CO_2**	**Date**	**O_2**	**N_2**	**CH_4**	**CO_2**
3-Sep-09	0.09539	0.23423	70.50933	25.34941	3-Sep-09	0	0.70332	73.10301	22.51023
	0.09088	0.23326	70.84394	25.34362		0	0.69971	73.13758	22.5138
	0.08852	0.23104	70.86667	25.1793		0.15049	0.39494	73.30406	22.46282
Avg	0.09	0.23	70.74	25.29	Avg	0.05	0.60	73.18	22.50
9-Sep-09	0.21226	0.49614	70.692	25.22677	8-Sep-09	0.1659	0.34481	72.77304	23.51394
	0.12885	0.25962	71.15185	25.12236		0.1385	0.30342	72.49668	23.26114
	0.09348	0.14882	70.77573	24.72738		0.29021	0.73794	72.17357	22.54667
	0.09254	0.14841	71.12947	24.7779		0.18145	0.41041	72.61072	22.64407
Avg	0.13	0.26	70.94	24.96	Avg	0.19	0.45	72.51	22.99
16-Sep-09	0.73342	2.16501	67.14745	25.71614	17-Sep-09	0.23072	0.48088	70.80343	25.50685
	0.37539	1.0812	68.47181	26.03657		0.19247	0.41162	70.60339	25.31507
	0.22107	0.59771	68.79543	26.22222		0.18861	0.41267	70.81879	25.27936
Avg	0.44	1.28	68.14	25.99	Avg	0.20	0.44	70.74	25.37
23-Sep-09	0.17223	0.72215	67.79512	27.14495	25-Sep-09	0.1817	0.59514	71.71505	24.5038
	0.13179	0.59535	67.96468	27.11959		0.18039	0.58439	71.73399	24.40893
	0.12435	0.55779	67.96832	26.98753		0.1782	0.58519	71.49305	24.23825
Avg	0.14	0.63	67.91	27.08	Avg	0.18	0.59	71.65	24.38
30-Sep-09	0.16759	0.52232	68.82255	26.92569	29-Sep-09	0.19426	0.45551	69.32357	25.72421
	0.12957	0.41605	68.64186	26.90512		0.19625	0.45778	68.93559	25.35443
	0.10972	0.36215	68.88425	26.74165		0.19946	0.46021	69.15261	25.25192
Avg	0.14	0.43	68.78	26.86	Avg	0.20	0.46	69.14	25.44
Overall Average	**0.19**	**0.55**	**69.40**	**25.97**		**0.17**	**0.50**	**71.51**	**24.06**

Appendix F

Acids Data

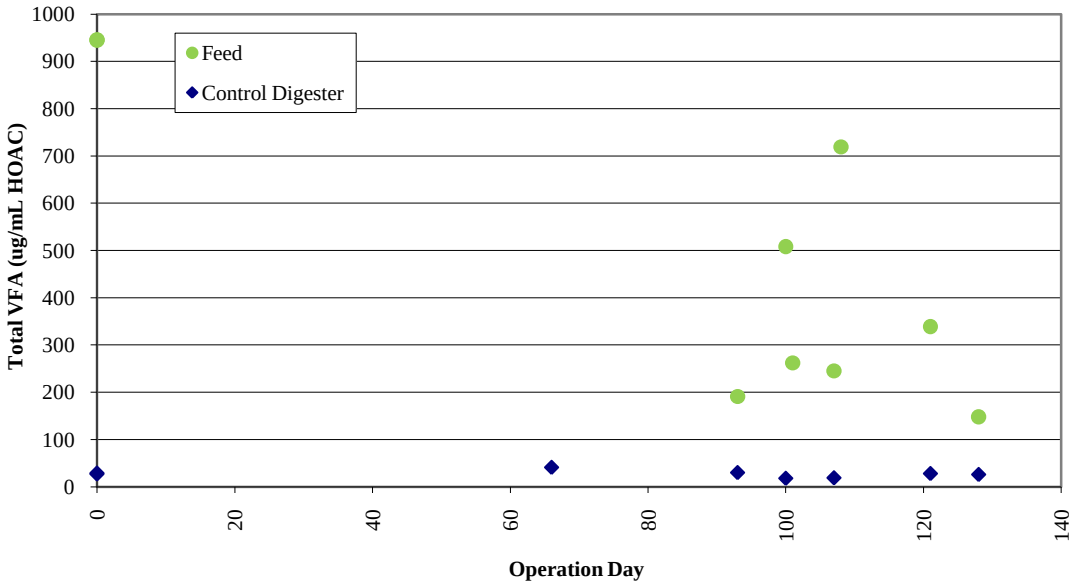

Figure 71 Volatile Fatty Acids for Control Digester – Low Temperature

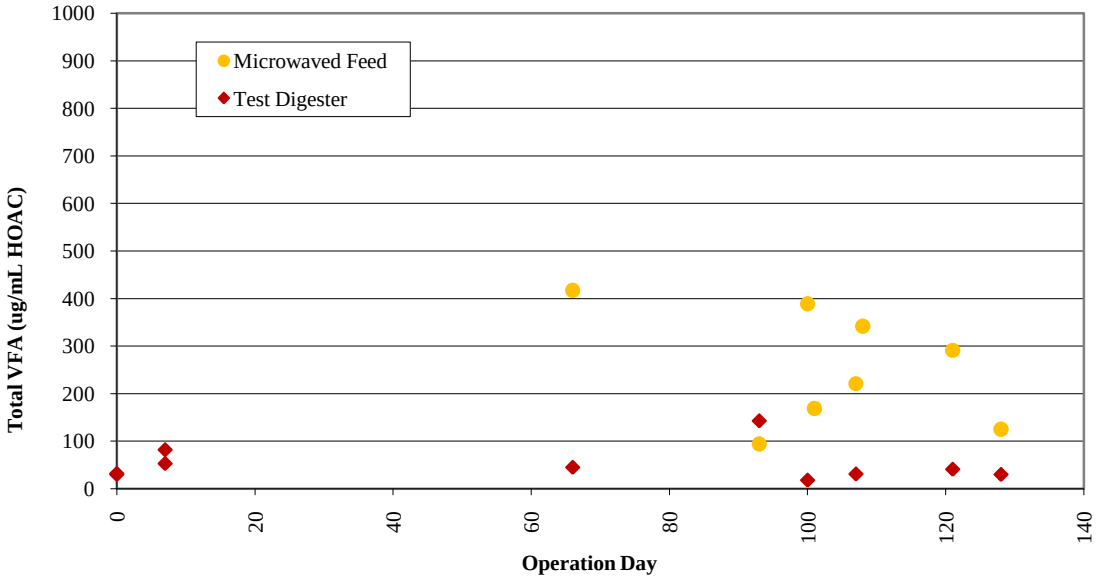

Figure 72 Volatile Fatty Acids for Test Digester – Low Temperature

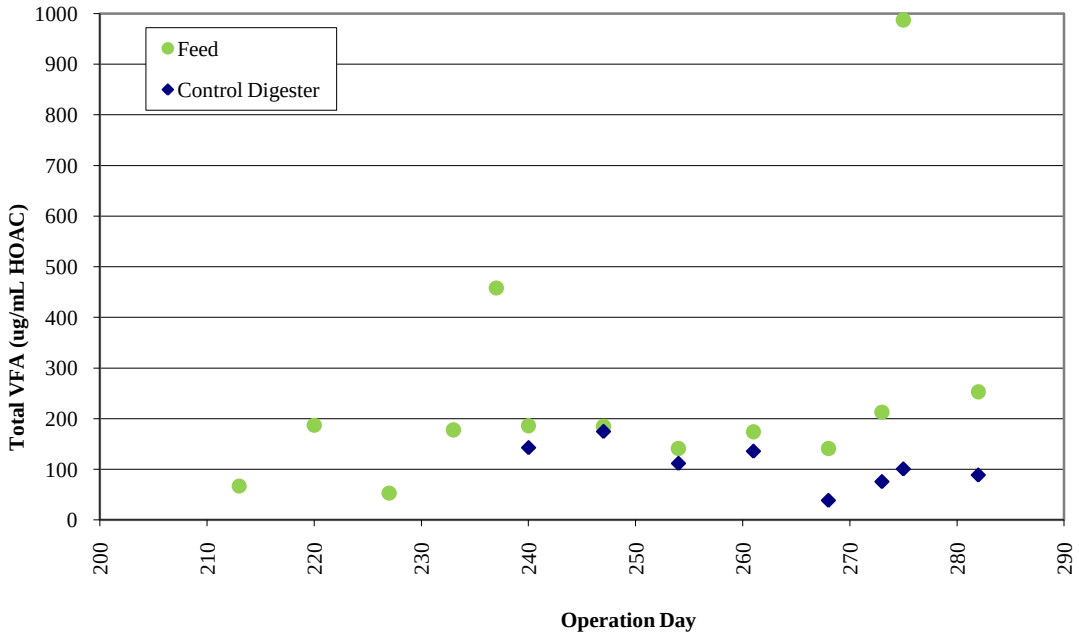

Figure 73 Volatile Fatty Acids for Control Digester – HighTemperature

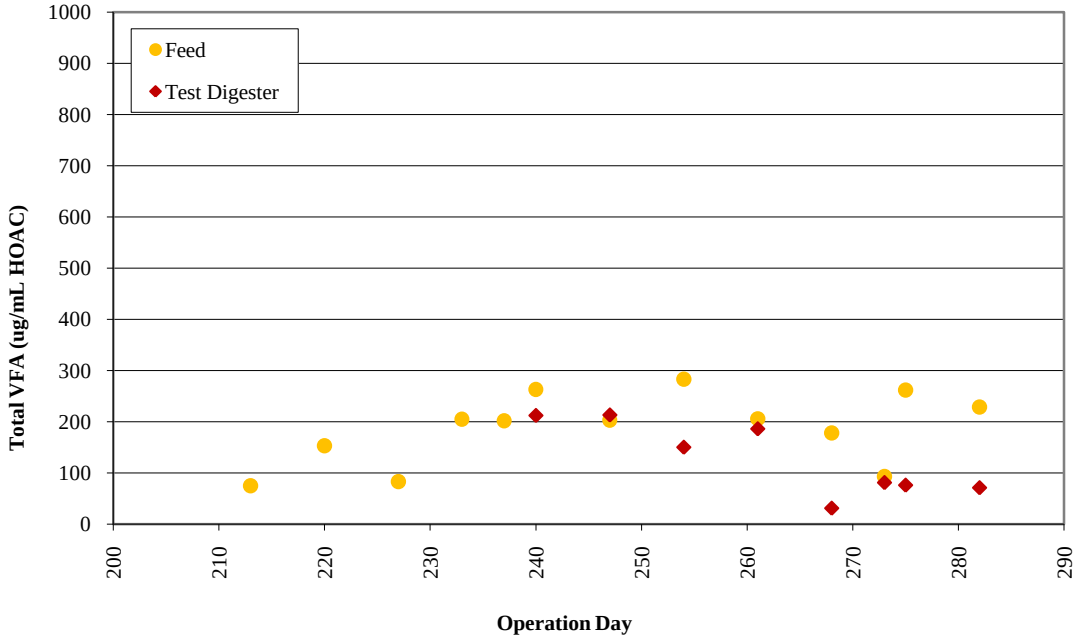

Figure 74 Volatile Fatty Acids for Test Digester – High Temperature

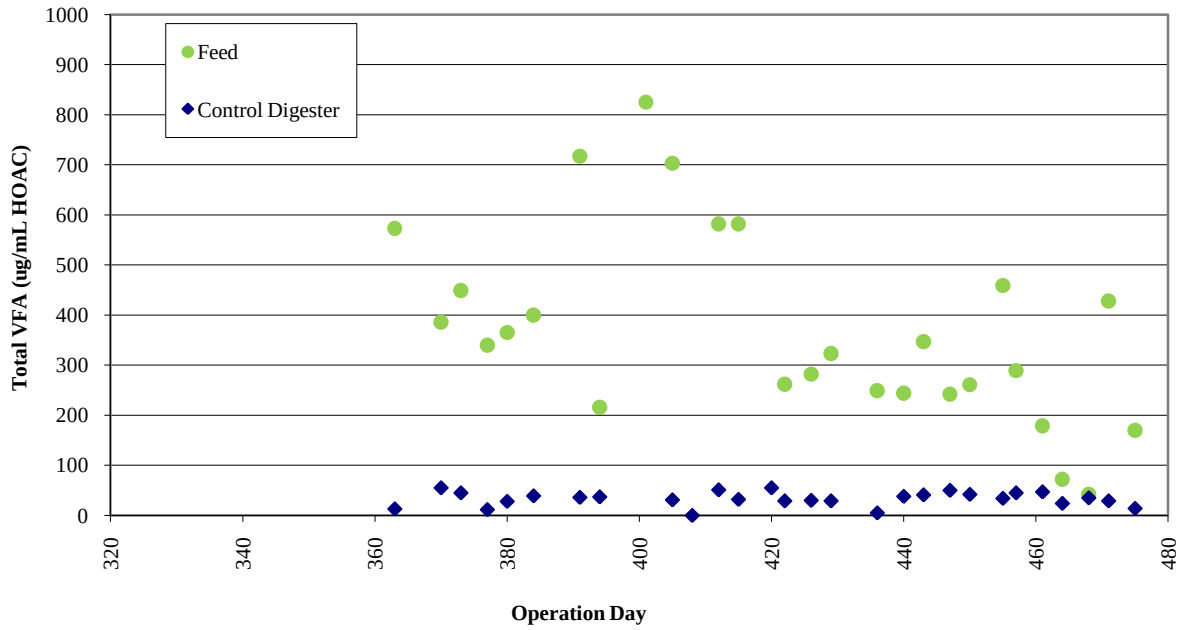

Figure 75 Volatile Fatty Acids for Control Digester – Membrane

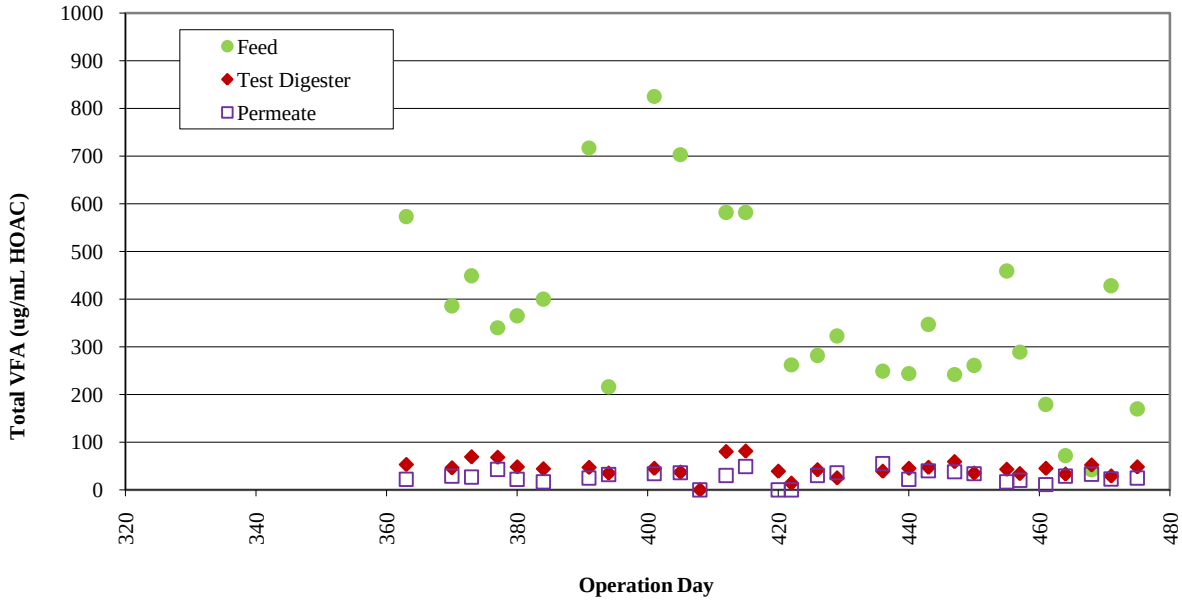

Figure 76 Volatile Fatty Acids for Test Digester – Membrane

Appendix G
Nitrogen Data

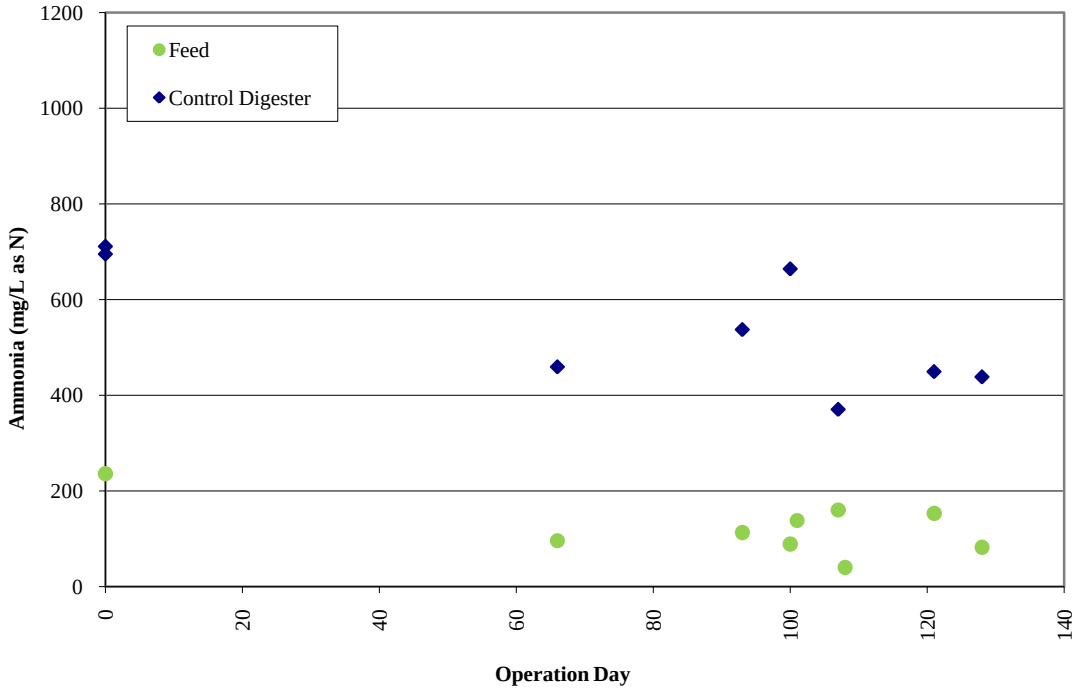

Figure 77 Ammonia for Control Digester – Low Temperature

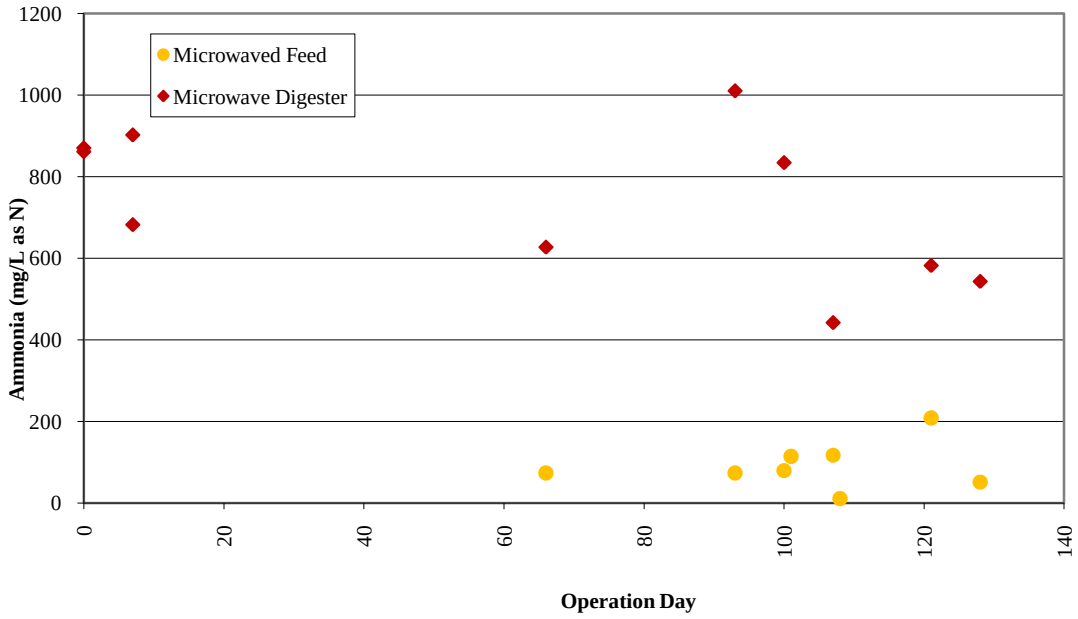

Figure 78 Ammonia for Test Digester – Low Temperature

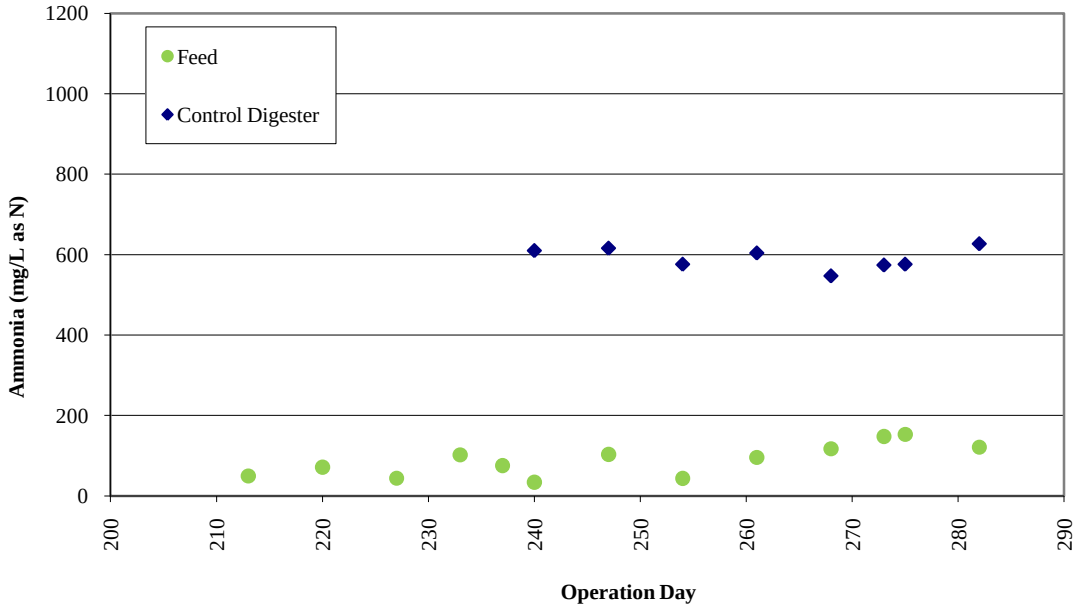

Figure 79 Ammonia for Control Digester – HighTemperature

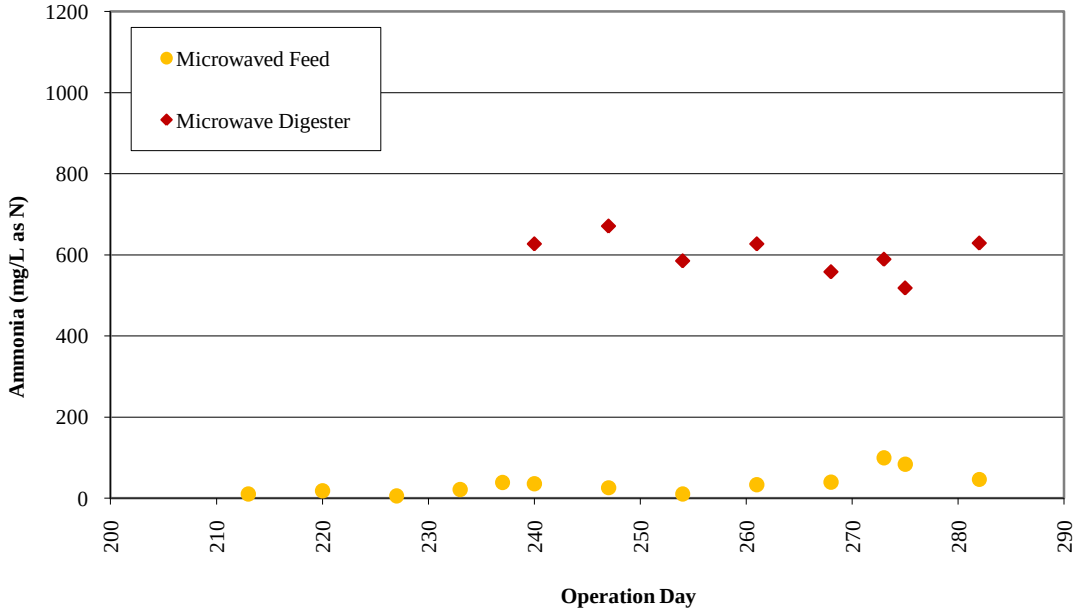

Figure 80 Ammonia for Test Digester – High Temperature

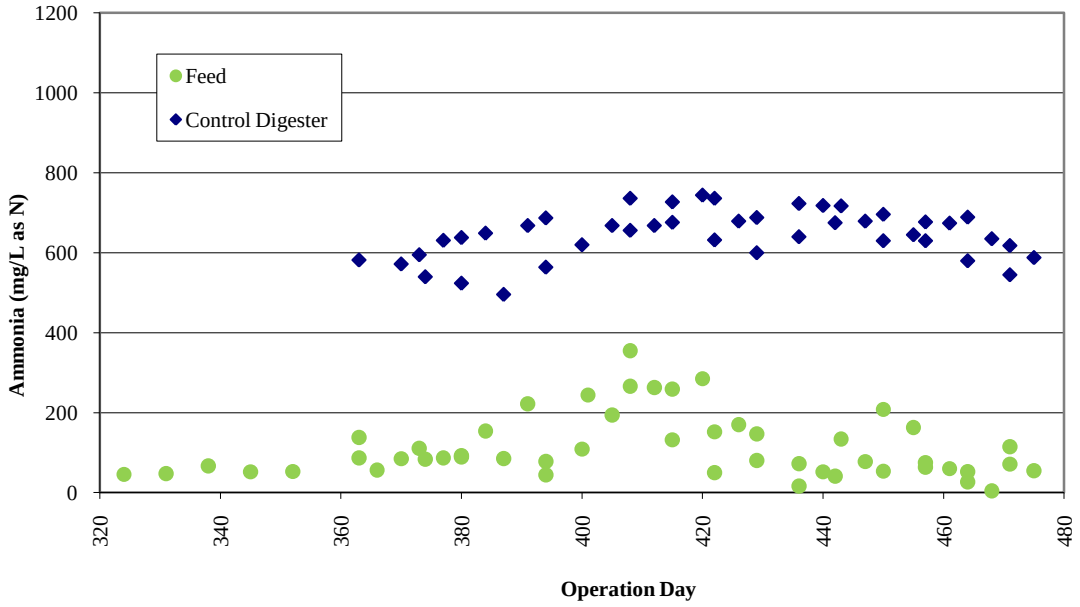

Figure 81 Ammonia for Control Digester – Membrane

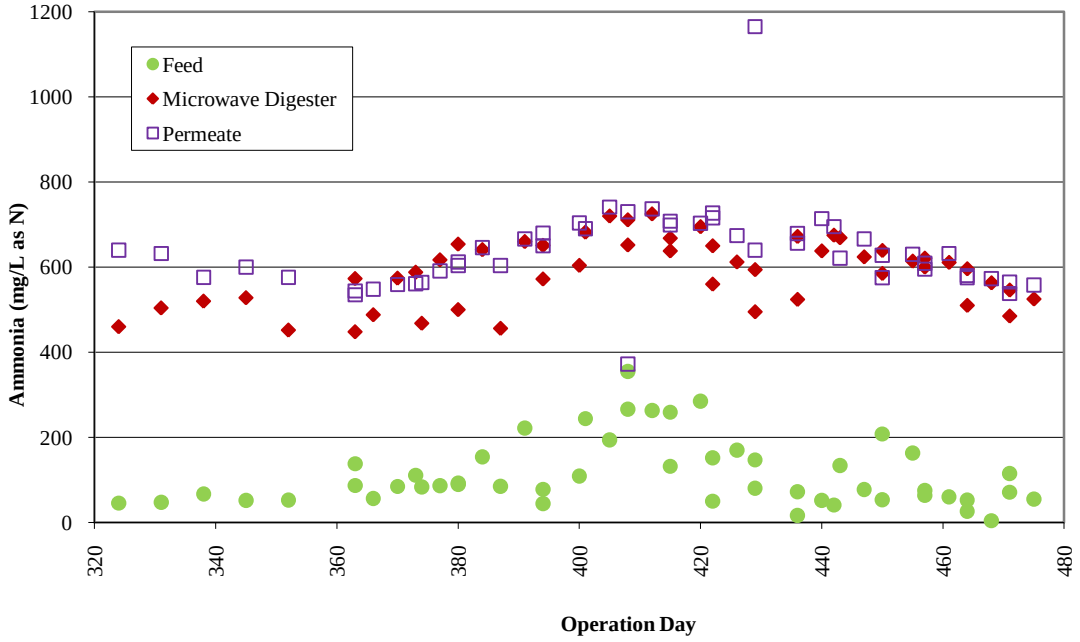

Figure 82 Ammonia for Test Digester – Membrane

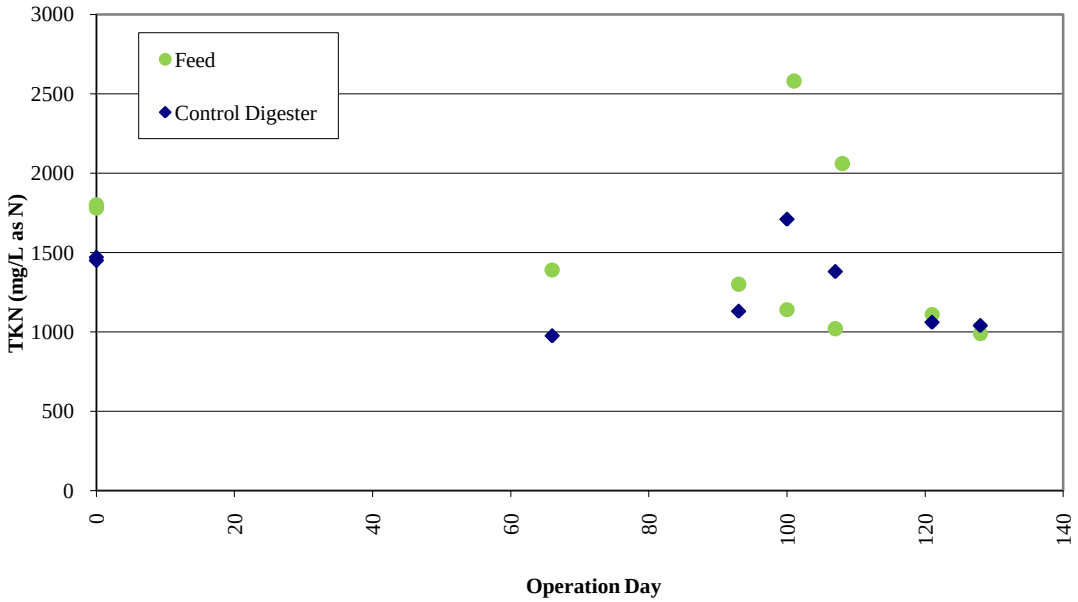

Figure 83 TKN for Control Digester – Low Temperature

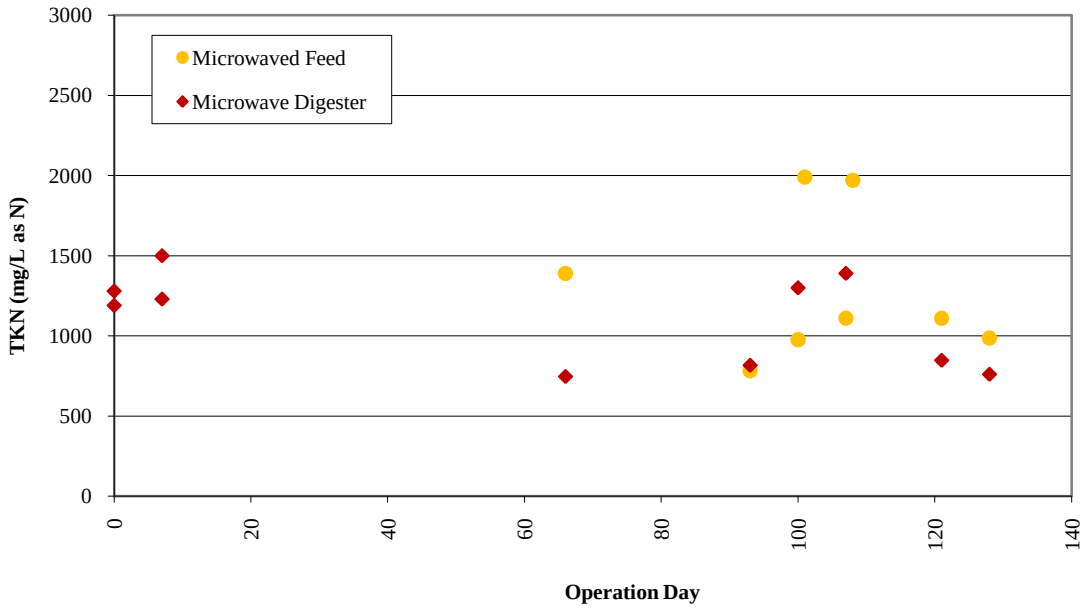

Figure 84 TKN for Test Digester – Low Temperature

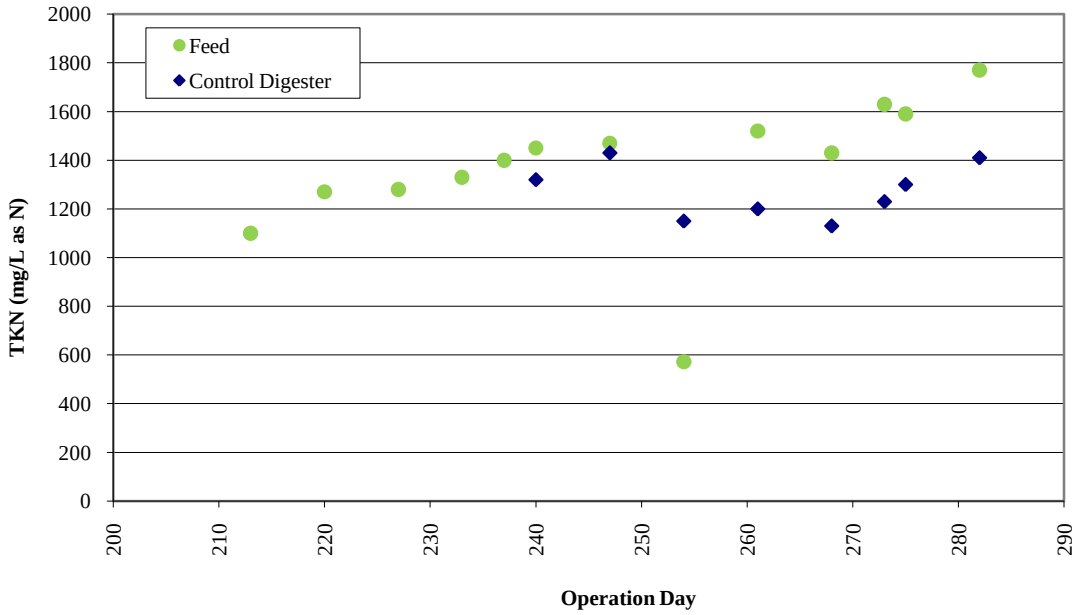

Figure 85 TKN for Control Digester – HighTemperature

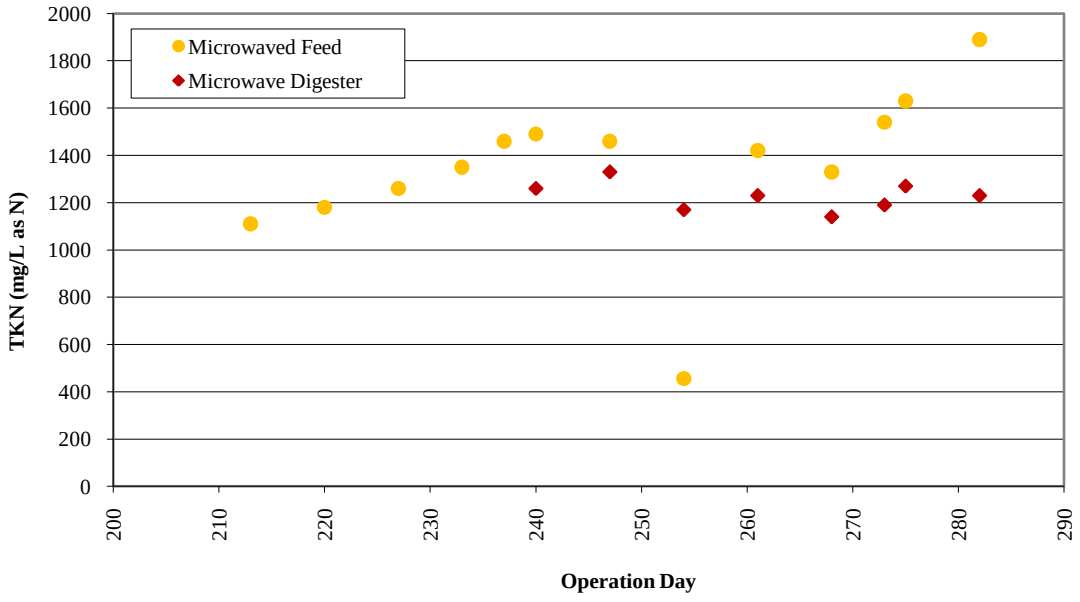

Figure 86 TKN for Test Digester – High Temperature

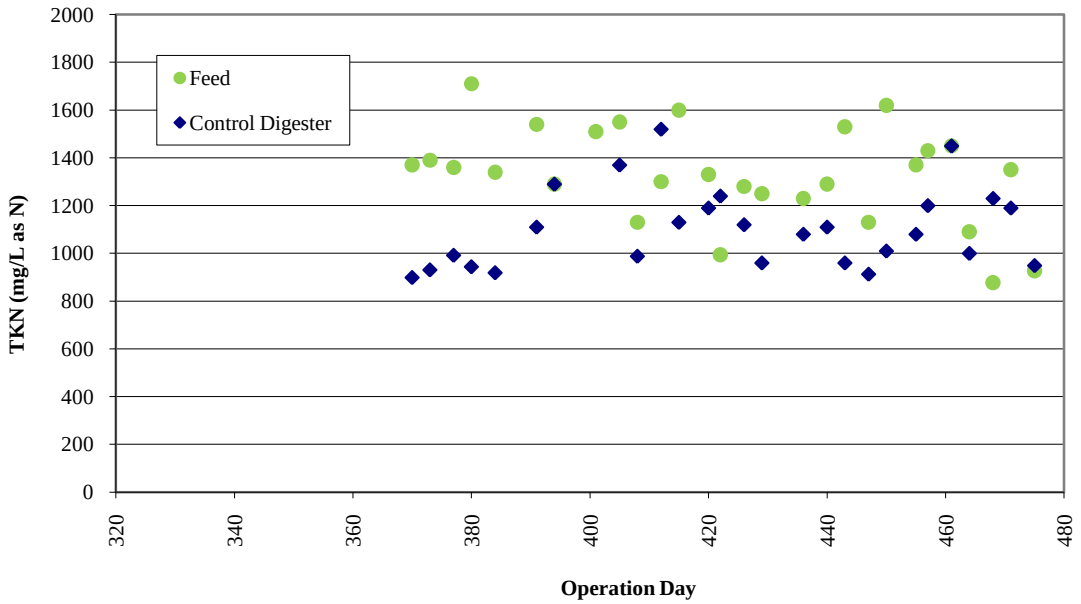

Figure 87 TKN for Control Digester – Membrane

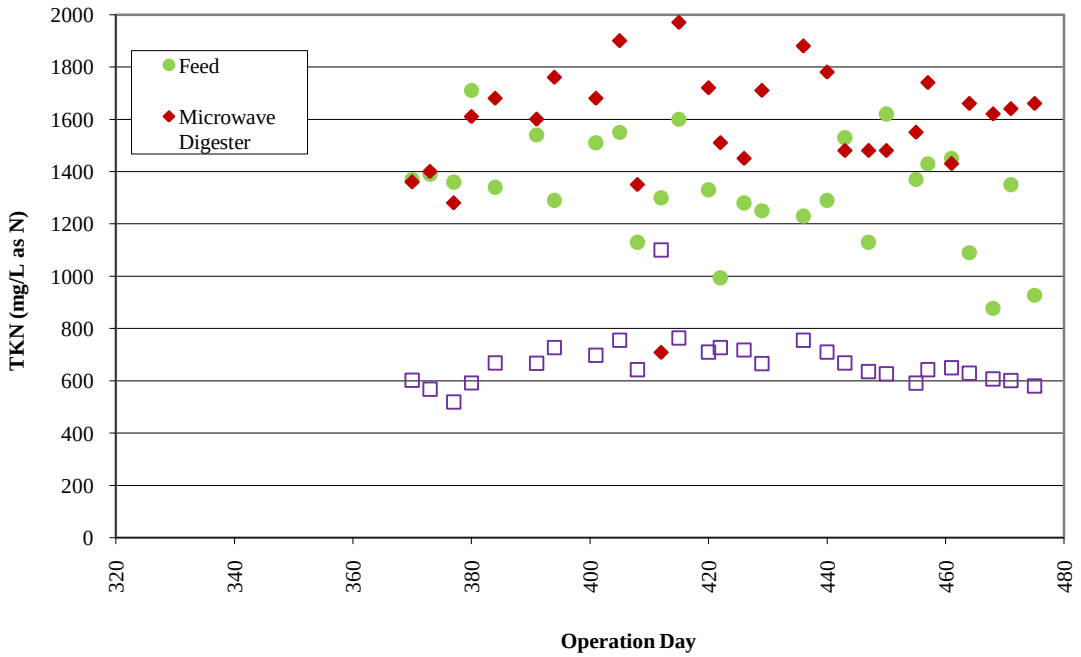

Figure 88 TKN for Test Digester – Membrane