EFFECTS OF THERMAL HYDROLYSIS PRE-TREATMENT ON ANAEROBIC DIGESTION OF SLUDGE

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ABSTRACT

The increased demand for advanced techniques in anaerobic digestion over the last few years has led to the employment of various pre-treatment methods prior to anaerobic digestion to increase gas production. These pre-treatment methods alter the physical and chemical properties of sludge in order to make it more readily degradable by anaerobic digestion. The thermal hydrolysis process has been used in several treatment plants around the world, but none currently operate in the US. Thermal hydrolysis causes cell walls to rupture under the effect of high temperature and high pressure and results in highly solubilized product which is readily biodegradable. The performance of the process was evaluated for a treatment plant located in Dallas, TX. The performance assessment was based on various characteristics including pH, solids removal, COD removal and gas production. The study was conducted in two phases to investigate the effect of change in mesophilic temperature (37^oC and 42^oC) and the effect of solids retention time (SRT) (15 days and 20 days). Thermally hydrolyzed combined (1:1) primary and waste activated sludge was fed to a Thermal Hydrolysis (TH) anaerobic digester and its performance was compared to a conventional mesophilic anaerobic digester receiving non pre-treated sludge. The thermal hydrolysis pre-treatment was found to be more effective as compared to the conventional anaerobic digester. The efficiency of the process varied slightly with increase in temperature but the change in SRT was seen to have a greater impact on the digester's performance. The pre-treatment technique was observed to deliver the best results at a 20 day SRT.

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Chapter 1

Introduction

Over the last century, anaerobic digestion (AD) has emerged as a reliable treatment solution for the stabilization and disintegration of sludge. The process was initially used for the treatment of household waste and sewage sludge in the municipal treatment plants. But over the past 20 years, the true potential of anaerobic digestion has been explored and major advances in reactor design, configuration and operation and in our understanding of the nature of the microbial biochemistry; physiology and ecology have been reported [Craik et al., 1995]. The growing interest of the researchers in this process is a testimony to the viability and applicability of the process.

The growing population and industrial expansion have both led to the production of increasing volumes of wastewater and thus, the demand for a sustainable method for the treatment and disposal of this wastewater has also been amplified. Due to the multiple advantages of anaerobic digestion over aerobic processes [McCarty, 1986], researchers are exploring ways to enhance the process and make it more pertinent for a variety of applications. Also, the market requirements such as energy recovery, reduction of greenhouse emissions, high quality biosolids, and a low odor product and high-rate reactor systems have driven the research on various advanced anaerobic digestion techniques.

Pre-treatment of sludge prior to anaerobic digestion has attracted the attention of many researchers over the past decade. Various pre-treatment techniques such as mechanical grinding, chemical pre-treatment etc. have been utilized to alter the quality and characteristics of the sludge entering the anaerobic digesters. Thermal hydrolysis pre-treatment has been one of the most popular pre-treatment methods for conditioning the sludge prior to anaerobic digestion. It is known for its highly efficient sludge disintegration capability enhancing the biogas production and for destroying pathogens.

Anaerobic digestion with thermal hydrolysis as pre-treatment has shown effective results in VS removal, COD removal and biogas generation [Camacho et al., 2008]. The thermal hydrolysis pre-treatment solubilizes organic particulate matter and causes lysis of cell walls. The main advantage of the process is that the hydrolysis, which is considered to be the rate limiting step in anaerobic digestion, occurs via physical rather than biological means [Wilson et al., 2008].

The problem of biosolids handling and disposal has also been improved by the thermal hydrolysis pre-treatment technology. Certain commercial thermal hydrolysis processes like Cambi claim to produce Class A biosolids with minimal odor issues [Higgins et al., 2006]. The thermal hydrolysis process is often associated with ammonia build-up issues in the anaerobic digesters causing inhibition of methanogenic activity in the system. This depends primarily on the sludge characteristics and the ammonia concentration in the feed. An aerobic digester downstream to anaerobic digester has proved to be effective to avoid the ammonia inhibition issues in the system [Tanneru et al., 2009]

The main objective of this research is to evaluate the performance of a thermal hydrolysis process as a pre-treatment step prior to anaerobic digestion for the Trinity River treatment plant in Dallas, Texas. The efficiency on the thermal hydrolysis process depends on the sludge characteristics and operational parameters such as pH, temperature, SRT, organic loading and nitrogen content. In this study, the effect of two main parameters has been investigated. These are:-

(a) Comparison of two solids retention times (15 days and 20 days) in anaerobic digestion.

(b) Comparison of two mesophilic temperatures (37^{o}C and 42^{o}C) in anaerobic digestion.

The work was conducted in two phases. The SRT was maintained at 20 days during the first phase and 15 days during the second phase. During both the phases, the TH reactors were initially operated at 37^{o}C and performance analysis was conducted and thereafter the temperature was increased to 42^{o}C to study the effect of increase in temperature.

Chapter 2

Literature Review

2.1 Anaerobic Digestion

Anaerobic methods have been used for stabilization of wastewater sludges for over a century. The comparative advantages of AD include: ability to treat high organic loads; low quantities of biomass generation; production of a useable fuel (biogas methane); and production of stabilized sludge, to which post-treatment technologies may be applied to facilitate the recovery of nutrients.

Anaerobic digestion is a biological process that involves decomposition of organic and inorganic matter in the absence of oxygen. Various groups of bacteria and archaea act upon the organic substrate and convert it into more stabilized end products such as methane carbon-dioxide and ammonia. The decomposition occurs in four major steps- hydrolysis, acidogenesis, acetogenesis and methanogenesis (Fig.2-1). These processes involve complex and important interactions between bacteria and archaea which are fundamental to the successful functioning of methanogenic communities [Grady et al., 1999].

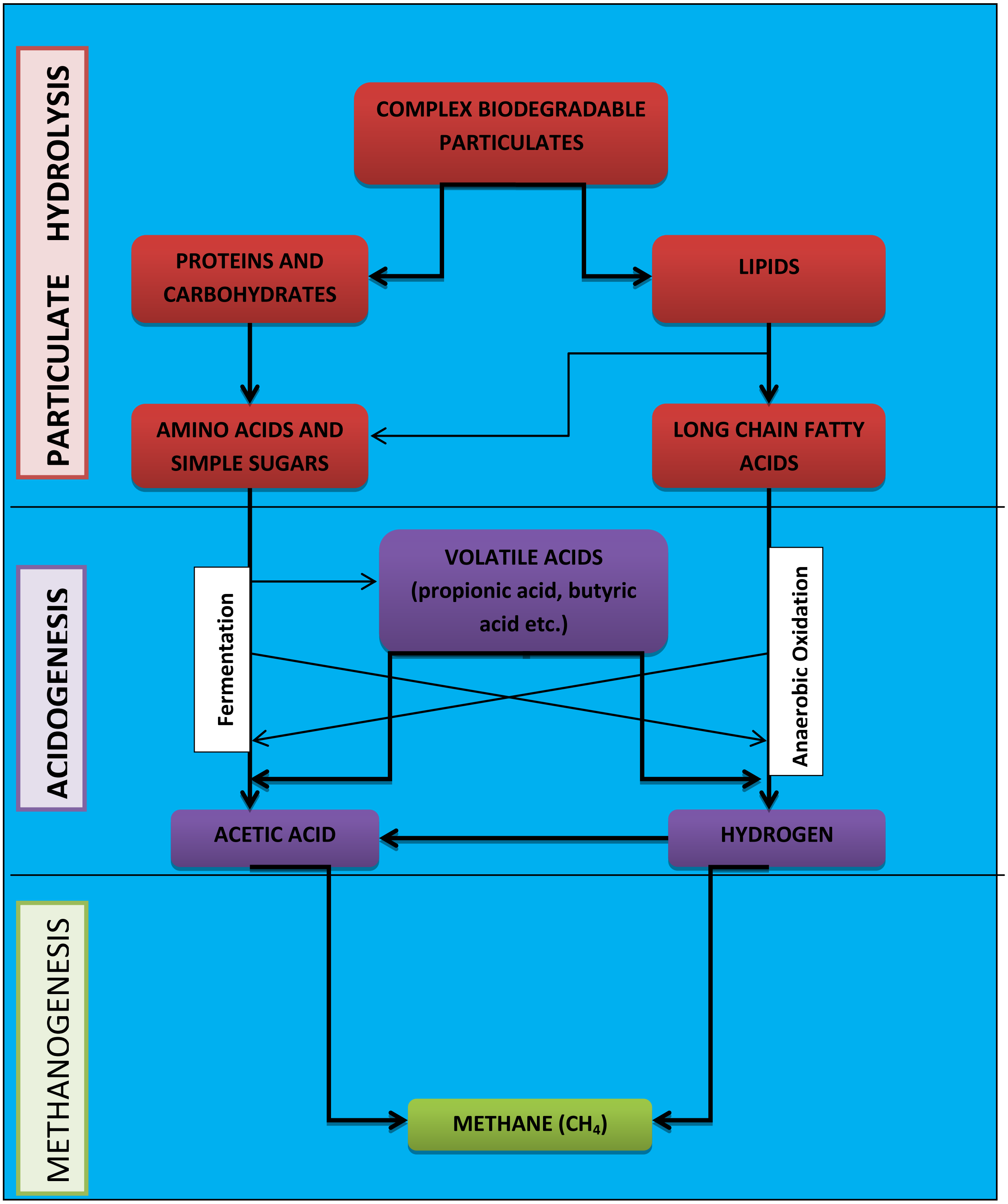

Figure 2-1: Multi-step nature of anaerobic operations

In the first phase both insoluble organic material and high molecular weight compounds such as lipids, polysaccharides, proteins and nucleic acids are solubilized into soluble organic substances (e.g. amino acids, sugars and fatty acids) under the effect of many extracellular enzymes such as cellulases, ammonylases and proteases produced by fermentative bacteria. The components formed during hydrolysis are further disintegrated during the acidogenesis step resulting in the formation of volatile fatty acids (VFA) along with ammonia, CO_2, H_2S and other by-products. The third stage is acetogenesis where these intermediaries VFA are further digested by the acetogenic bacteria to form direct methane precursors acetic acid and H_2. The partial pressure of H_2 in the mixture controls the dynamics of this reaction and thus the production of H_2 by anaerobic oxidation is very important for proper functioning of anaerobic processes [Grady et al., 1999].

During the final stage of methanogenesis, the methanogens convert the previously produced acetic acid and hydrogen into methane gas and carbon dioxide. Two types of methanogenic archaea are involved in this process. Aceticlastic methanogens split acetic acid in to methane and carbon dioxide. Hydrogen oxidizing archaea use hydrogen as an electron donor and carbon dioxide as an electron acceptor to produce methane [Appels et al., 2008]. Methanogens are strict anaerobes and have very slow growth rate. Consequently, their metabolism is usually considered rate-limiting and a long detention time is required for growth [Metcalf and Eddy, 1991].

Within the anaerobic environment, various important parameters affect the rates of the different steps of the digestion process, i.e. pH and alkalinity, temperature, and retention times. For effective performance of an anaerobic digestion system these parameters must be maintained at optimal levels and the production of toxic and inhibitory substances must be repressed.

pH

Anaerobic Digestion is generally operated under neutral pH conditions (pH 6.5-7.6). The observed toxicity under low pH conditions is associated with the presence of un-dissociated VFA [vanLier et al., 2001]. The methanogenic activity will slow considerably at a pH less than 6.3 and higher than 7.8 and this will inhibit biogas production [Leitao et al., 2006]. A lower pH will result in the growth of filamentous bacteria and a high pH results in buildup of ammonia [Grady et al., 1999]. Anaerobic treatment under acidic and alkaline conditions may play an important future role when in line - or side stream - treatment processes in industries call for a higher tolerance for extreme conditions.

Temperature

Another important factor for anaerobic treatment is temperature. Anaerobic reactors are normally operated at mesophilic (30 to 40°C) or at moderate thermophilic (50 to 60°C) temperatures in accordance with the optimal temperature range for the groups of anaerobic microorganisms performing the whole digestion process [Ahring 1994, 1995, van Lier, 1996]. For thermophilic treatment higher temperatures can result in instability of the treatment process [van Lier et al., 1993]. At 50-60°C anaerobic digestion will be stable and will perform as well as or better than mesophilic digestion.

Solids Retention Time (SRT)

The solids retention time is the average time the solids spend in the digester, whereas the hydraulic retention time (HRT) is the average time the liquid sludge is held in the digester. For most conventional anaerobic digesters, the HRT and SRT are equal because no biomass is retained or recycled. The subsequent steps of the digestion process are directly related to the SRT. It is the most important design parameter available for an engineer as it is directly related to specific growth rate of biomass in a continuously stirred tank reactor [Grady et al., 1999]. Detention times for anaerobic digestion of municipal sludges range from 15 to 60 d, depending on temperature and mixing within the digester tank [Hairston et al. 1997].

Taricska et al. [2009] studied the effect of operating temperature and SRT on the volatile solids reduction in anaerobic digestion of sludge and reported that VS reduction increased with increase in SRT to a limit and then remained constant for any further increase. An increase in temperature also showed an increase in VS reduction.

Toxic and Inhibitory compounds

Several materials can cause an inhibitory response: the materials of the greatest concern are light metal cations, ammonia, sulfide, and heavy metals. In addition, sulfate interferes with methane production by providing an alternate electron acceptor. Sulfide exerts an oxygen demand that reduces the amount of COD stabilized. Many organic compounds are also inhibitory, particularly to methanogens [Grady et al., 1999].

Ammonia toxicity is of major concern for anaerobic treatment of wastewaters containing high concentrations of total ammonia nitrogen (TAN). The free ammonia (or unionized ammonia, NH_3) is considered to be toxic for methanogenic bacteria. The toxicity threshold for free ammonia has been reported to be 100 mg/L as N [Grady et al., 1999]. In batch tests, Lay et al. [1998] found a steady inhibition of methanogenic activity as ammonia-nitrogen concentration was increased from 50 to 500 mg/L. McCarty et al. reported an ammonia toxicity concentration range of 1500 to 3000 mg/L as TAN at pH>7.4.

Grady et al. [1999] stated that "for mesophillic operating conditions, total ammonia concentrations of about 2,000 mg/L can result in toxic free ammonia concentrations (100 mg/L as N) as pH approaches 7.5 to 8.0."

Organic loading rates

For solid wastes and organic sludges; loadings most commonly are based on volatile solids (VS), whereas for dilute wastewaters loadings would be expressed in terms of biochemical oxygen demand (BOD) or chemical oxygen demand (COD). The stability of the anaerobic process and the rate of gas production are both dependent upon organic loading rates. At higher loadings, the process often becomes unbalanced because of the excessive production of volatile acids.

2.2 Pre-treatment prior to Anaerobic Digestion

Although anaerobic digestion is one of the best available techniques for the treatment of sludges, its application is often limited by very long retention times (20–30 days) and a low overall degradation of the organic dry solids (30–50%). Space limitation is also one of the restraining factors in the usage of anaerobic digestion at large scale. By reducing the footprint of the process, the versatility and the applicability of the process can be enhanced.

The methanogenic process is generally limited by the rate of hydrolysis of suspended matter and organic solids. During hydrolysis, cell walls are ruptured and extracellular polymeric substances (EPS) are degraded resulting in the release of readily available organic material for the acidogenic micro-organisms [Appels et al., 2008]. By means of efficient pre-treatment the suspended substrate can be made more accessible for the anaerobic bacteria, optimizing the methanogenic potential of the waste to be treated.

Various sludge disintegration methods have hence been studied as a pre-treatment: these include mechanical grinding, ultrasonic disintegration, chemical methods, thermal pre-treatment, enzymatic and microbial pre-treatments.

2.3 Thermal Hydrolysis Pretreatment

Thermal Hydrolysis is a process where high temperature (130^{o}C-180^{o}C) and corresponding pressure (5-8 bar), along with the optimum retention time, destroys the cell walls of the organic substrate. By de-coupling the long-chain polymers and hydrolyzing the proteins, it makes the sludge more readily available for anaerobic digestion. Thermal hydrolysis is able to split and decompose a significant part of the sludge solid fraction into soluble and less complex molecules [McCarty et al., 1976; Haug et al., 1983]. It has been shown that the anaerobic digestion can consistently achieve 55 to 60% volatile solids destruction after thermal hydrolysis [Jolis et al., 2008]. Thermal hydrolysis is also a well proven method to increase the dewaterability of sludge [Kepp et al., 2000].

Various authors have studied the effects of thermal hydrolysis treatment on the sludge structure and properties. Although, all studies reveal that thermal hydrolysis pre-treatment has a positive impact on anaerobic digestion, it has also been observed that the hydrolysis conditions play a vital role in determining the efficiency of the process [Wilson et al., 2011]. It had been reported that Thermal pretreatment in the temperature range from 100^{o}C-180^{o}C destroys cell walls and makes the proteins accessible for biological degradation [Muller, 2001].

Gavala et al. [2003] conducted studies on the parameters that control the effectiveness of the thermal hydrolysis process and concluded that temperature and retention time for the optimum pre-treatment results depend greatly on the nature of the sludge: the greater the difficulty in hydrolyzing the sludge, the higher intensity of pre-treatment required.

Some commercial processes have been developed based on thermal pre-treatments. The Norwegian company Cambi Inc. developed a system based on thermal hydrolysis. Solids solubilization of approximately 30% was reported (dependent on the type of sludge being processes) for a 30 min treatment at 180^{o}C. An associated increase of biogas production by 150% is reported by the company [Cambi.no]. A similar thermal treatment is sold as EXELYS by Kruger Inc., a subsidiary of Veolia Water. As compared to conventional digestion, the treatment offers 30-40% less sludge for disposal, 20-40% more biogas, upto 50% capacity increase for existing digesters and a reduced carbon footprint [Veolia Water].

2.4 The CAMBI Process

The Cambi process serves as an effective pre-treatment technique to the anaerobic digestion of sludge [Pickworth wt al., 2006]. The thermal hydrolysis treatment hydrolyzes the cell walls and releases soluble COD for digestion. Cambi process is better than the conventional thermal hydrolysis process because it operates at comparatively lower temperatures (160^{o}C-180^{o}C) and utilizes steam instead of heat exchangers for primary heating of sludge. The process offers the following advantages over conventional systems:

- High volatile solids destruction (~60-70%)
- Low digester volume
- High biogas yield (~150%)
- Well dewatered cake (~35%)
- Pasteurized product(Class “A” biosoilds) with low transportation cost

In addition, the dewatered cake solids also increase substantially. All these advantages can be explained by the bioavailability of organic material to fermentative organisms after thermal hydrolysis. The process takes place in 4 steps (Fig. 2-2):

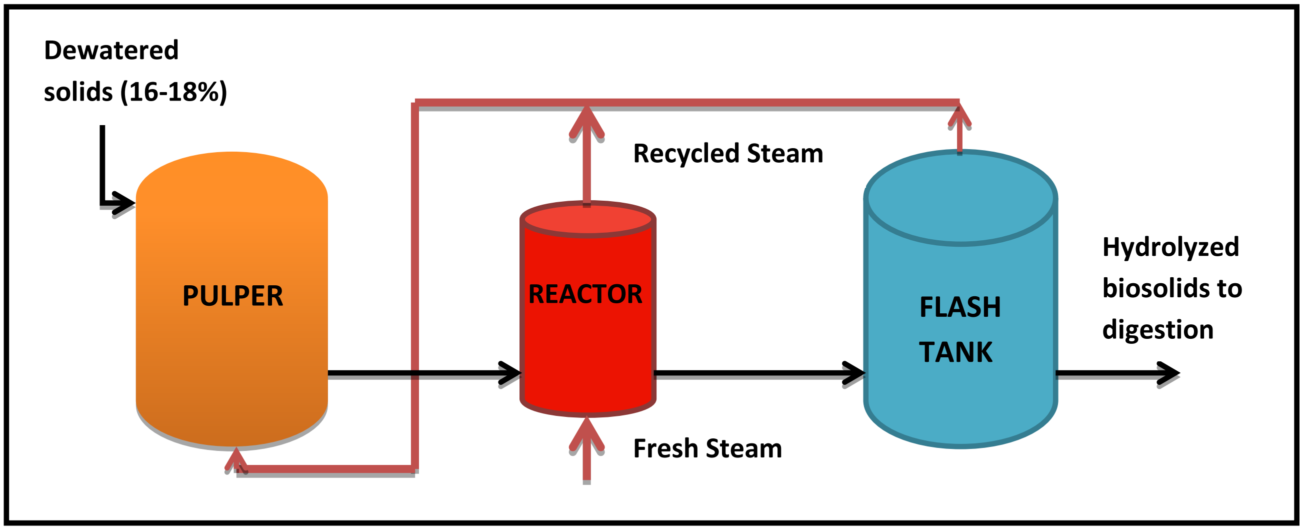

Figure 2-2: Schematic representation of Cambi Process

Thickening: The sludge is dewatered to 16-17% dry solids using a centrifuge, belt and filter press or other dewatering devices.

Pulper: The dewatered sludge is fed into the pulper to be mixed and heated by recycled steam from the reactor(s) and the flash tank. Process gases are compressed and broken down biologically in the digesters (minimal odor).

Reactor: The sludge is batchwise pumped into the reactor. Thermal hydrolysis takes place in reactor(s) at 165ºC for 20-30 minutes. The steam is gradually released and sent back to the pulper. The batch reactor is maintained at these conditions for approximately 30 minutes to produce a product that meets Class A biosolids standards and causes no filamentous bulking problems in the digester. The sludge is homogenized using a recycle loop and a macerator.

Flash Tank: The sterilized sludge is then passed rapidly into the flash tank, resulting in cell destruction from the pressure drop. The sludge temperature is decreased to approximately 102º C by flashing steam back to the pulper.

By relieving the kinetic limitations of biological hydrolysis, higher solids loading rates can be applied such that a significantly smaller digester volume is required. Hydrolysis temperature is more important than the contact time and seems to govern the extent to which sludge disintegration occurs [Li and Noike, 1992]. It was reported that a temperature higher than the optimum range leads to a sharp reduction in biodegradability of sludge hydrolysate [Stuckey and McCarty, 1978].

2.5 Anaerobic Digestion of Thermally Hydrolyzed Sludge

The thermal hydrolysis (TH) process has been used in recent years as a pretreatment method for sewage sludge prior to anaerobic digestion. Thermal hydrolysis makes the sludge readily available for biodegradation. It reduces sludge viscosity and increases soluble COD. Thus, when this thermally hydrolyzed product is used as substrate in anaerobic digesters, the degradation is observed to be higher than that in conventional anaerobic digestion systems. This also results in higher solids loading rates in the digesters which lower the overall cost of the process.

Research has shown that anaerobic digestion of thermally hydrolyzed sludge has net energy production due to high degradability lesser digester heating requirements. The process typically routes sludge through a high pressure (5-8 bar) and temperature (150^oC to 170^oC) reactor for approximately 30 minutes and then anaerobically digests the sludge using temperatures ranging from 35^oC to 42^oC. The higher temperature reduces the need for cooling of the sludge prior to anaerobic digestion. Research [Wilson, 2008] indicates that there is little difference in digester performance between 35^oC and 42^oC.

The solids reduction in a digester has been reported to increase by ~10% as compared to conventional mesophilic anaerobic digester after using the thermally hydrolyzed sludge as the feed for digestion [Wilson et al., 2008]. Not only does the pre-treatment affect the digester operation results but it also enhances the quality of effluent from the digester. The digestion product exhibits high dewaterability characteristics and has very low odor as compared to the conventional digestion products. The product qualifies as Class A biosolids and can readily be used as an effective fertilizer given its low odor content [Novak et al., 2006].

2.6 Ammonia Inhibition

In aqueous phase, ammonium ion ($NH_{4\,(aq)}^+$), free ammonia ($NH_{3\,(aq)}$) in solution, ammonia ($NH_{3\,(g)}$) in the gas phase, hydrogen ion (H^+) and hydroxyl ion (OH^-) species exist in equilibria with one another. The [ammonium]/[ammonia] ratio is pH dependent. At lower pH values, ammonium and hydrogen ions are the major species while ammonia and hydroxyl ions become dominant at higher pH.

Free ammonia has been suggested to be the main cause of inhibition in anaerobic digesters due to its high membrane permeability [Kroeker et al., 1979; de Baere et al., 1984]. A significant difference in inhibiting ammonia concentration has been observed in various studies and this can be attributed to the differences in substrates and inocula, environmental conditions (temperature, pH), and acclimation periods [van Velsen et al., 1979, de Baere et al., 1984]. Grady et al. [1999] stated that "for mesophillic operating conditions, total ammonia concentrations of about 2,000 mg/L can result in toxic free ammonia concentrations (100 mg/L as N) as pH approaches 7.5 to 8.0."

The fraction of un-ionized ammonia has been reported to increase with temperature [Speece et al., 1996, Gallert et al., 1997]. Poggi- Varaldo et al. [1997] reported that thermophilic cultures were more susceptible to ammonia nitrogen than the mesophilic ones. Gallert et al. [1997] reported that free ammonia of 560–568 mg NH_3 N/l caused a 50% inhibition of methanogenesis at pH of 7.6 under thermophilic condition. Ahring et al. [1994] reported that free ammonia above 700 mg NH_3 N/l resulted in poor treatment performance at a pH of 7.4–7.9.

Sung et al. [2003] studied the effect of acclamation (to high TAN concentrations) on the methanogenic activity in anaerobic digesters and reported that although acclimation caused a decrease in overall methanogenic activity, it increased the tolerance of methanogens to TAN and pH variations.

Chapter 3

Research Objectives

Laboratory testing prior to installation of a TH system is desirable for several reasons. First, sludges differ with regard to their contents so the actual performance of the TH system cannot be predicted without some testing. A major advantage of the TH process is that the solids concentration in the feed sludge can be increased to 10 to 12% solids because the TH process alters the physical properties, rendering a10% solids slurry similar in viscosity to 5% solids for non-hydrolyzed sludge.

As a consequence of the high feed solids, the ammonia concentration increases in proportion to the increase in solids in the feed. Total ammonia concentrations can increase to levels that are inhibitory, resulting in high volatile fatty acids in the digester because ammonia inhibits the organisms that convert acetic acid to methane. The inhibitory ammonia species is unionized ammonia (NH_3) and a concentration of 100 mg/L of NH_3 is thought to be the inhibitory threshold. The unionized ammonia concentration is influenced by the temperature, total ammonia concentration and pH. These will differ from one sludge to another and this information will be critical to the design of the digestion system. More inhibition will require a larger anaerobic digester to counter the slower rate of acetate degradation caused by NH_3.

Another aspect of laboratory testing is to determine the gas production. Data from DC WATER showed that the specific gas production (Ft^3/lb VS destroyed) is much higher than that for conventional digestion. We have proposed that the increase in specific gas production results from the thermal hydrolysis of insoluble fats that would otherwise not digest. These fats are high in gas production and also can be an important fraction of volatile solids that degrade as a result of the thermal treatment.

Therefore, in order to properly design a TH system, lab data are needed to assess the increase in solids destruction, gas production and dewatered cake solids. In addition, the odor from the dewatered cake needs to be quantified.

The objectives of this research are:

- To evaluate the performance of a thermal hydrolysis process as a pre-treatment step prior to anaerobic digestion.
- To study the effect of operating temperature ($37^{o}C$ and $42^{o}C$) on the efficiency of TH system.
- To study the effect of SRT (15 days and 20 days) on the efficiency of TH system.

Chapter 4

Methodology

4.1 Digester Setup and Operation

High-density polyethylene batch fermentation reactors supplied by Hobby Beverage Equipment Company (Temecula, California) were used as anaerobic digesters for this study. The conical bottom of these vessels is advantageous in terms of mixing and suspension of grit, similar to the full-scale application of egg-shaped anaerobic digesters. The nominal volume of each vessel was 25 liters (L) and all the digesters were operated with an active volume of 10.0 L. The reactor vessels were modified to accept a threaded stainless steel thermometer, also supplied by Hobby Beverage.

Reactors were continuously mixed by digester gas recirculation from the headspace through the conical bottom of the reactor. A variable speed peristaltic pump (Cole-Parmer 600 rpm, Vernon Hills, Illinois) was utilized for circulating the gas from headspace of each digester to a valve at the bottom of the digester. The degree of mixing was controlled by changing the speed of the pump. Normally, the pumps were set at 50% of their maximum speed. This corresponds to approximately 0.8 L/minute with the Cole-Parmer “L/S-18” tubing used on the pump heads. The biogas produced from the reactors was collected in Tedlar bags of 40 L capacity each connected to the reactors.

Lab-scaled Thermal Hydrolysis mesophillic anaerobic digesters (TH) were initially seeded with reconstituted biosolids obtained from Ringsend Wastewater Treatment Works (Dublin, Ireland). The biosolids had an initial solids concentration on receipt of approximately 30%, and were diluted to the estimated final working solids concentration of the reactors (~ 10%) by addition of anaerobic digested sludge belt filtrate obtained from a local source. All reactors were operated at Virginia Tech Laboratory. Feed sludge for TH reactors was shipped to Virginia Tech as a primary/secondary dewatered mixture from Trinity River Authority (TRA), Texas. The sludge was slug fed daily operated in a batch fashion. Solids were removed first and then replaced.

The experimental setup of digesters was arranged as shown in Figure 4-1.

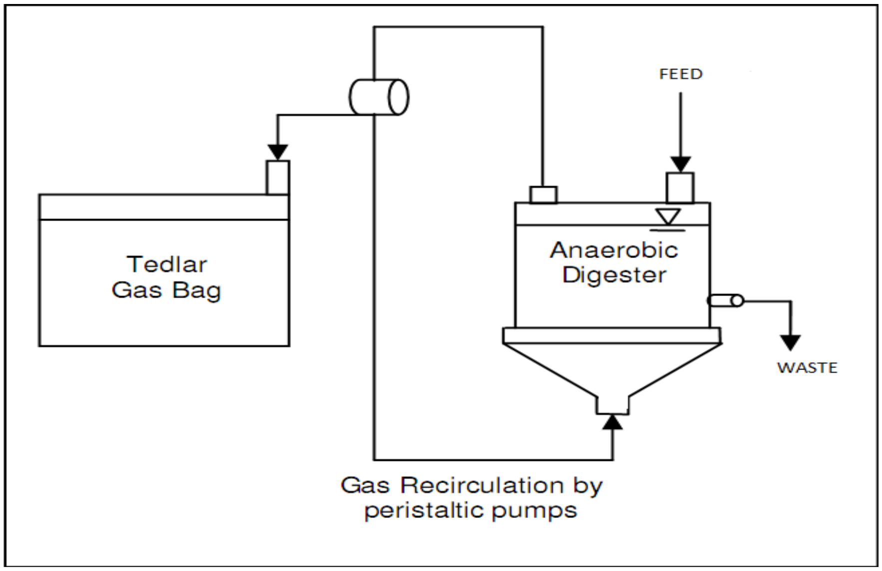

Figure 4-1: A representation of the experimental setup of anaerobic digester design

The laboratory setup of the reactors and the “bomb” employed for thermal hydrolysis of the sludge are show in Figure 4-2. The “bomb” was designed in such a way that it kept the sludge at an elevated pressure. Thus, when the sludge was heated to 170^{o}C, there was an increase in pressure inside the bomb. This pressure aided the cell lysis process during thermal hydrolysis.

Figure 4-2: Laboratory setup of anaerobic digesters at Virginia Tech, The "bomb" used for thermal hydrolysis of sludge

A matrix of experimental studies was carried out to address the questions on the efficiency of the process. Two kinds of variations were studied:

i. Effect of temperature, 37^{o}C and 42^{o}C.
ii. Effect of SRT, 15 days and 20 days.

The work was conducted in two phases. The SRT was maintained at 20 days during the first phase and 15 days during the second phase. During both the phases, the TH reactors were initially operated at 37^{o}C and performance analysis was conducted and thereafter the temperature was increased to 42^{o}C to study the effect of increase in temperature. The digester temperature was raised to 42^{o}C using thermolyte heating tape. Thermolyte is a device used for temperature control. The thermolyte tape is wrapped to the digester and the temperature can be adjusted to 42^{o}C using a heat control knob.

For both the phases, a conventional mesophillic control digester was run parallel at the same SRT as the TH reactor and at a temperature of 37^{o}C so that a TH system could be compared to a non-TH system. The feed for the Control reactor was obtained directly from TRA and consisted of a 50% ratio of primary and secondary solids. This feed material is referred to as Control-Feed.

Table 4-1 shows all the digesters, their operating temperature and SRT during the two phases.

Table 4-1: Anaerobic digester acronyms used during analysis and operational characteristics

Phase	SRT (days)	Digester Name	Temperature (^{o}C)
I	**20**	**Control-20D**	**37**
		TH-20D-37	**37**
		TH-20D-42	**42**
II	**15**	**Control-15D**	**37**
		TH-15D-37	**37**
		TH-15D-42	**42**

The dewatered sludge was diluted to the desired concentration and then placed in a “bomb” (a high temperature, high pressure vessel) using 800mL sample volumes. The sludge was hydrolyzed at 170^{o}C for 3 hours contact time. The bomb was removed from the oven and left at room temperature overnight for cooling. The thermally hydrolyzed sludge was used as feed for the TH digester, herein referred to as TH-Feed. The feed solids concentration was ~10% for TH and ~4% for Control.

4.2 Analytical Methods

The following parameters were monitored in order to evaluate the efficiency of the digesters:

- pH
- Solids Reduction
- COD
- Biogas production and composition
- Volatile Fatty Acids
- Total Ammonia and TKN
- Optimum Polymer Dose and Cake solids concentration
- TVOSC Concentration- Odor analysis

COD, pH, TKN and solids (Method 2540-G) testing were conducted according to Standards Methods for the Examination of Water and Wastewater (APHA, 1999). The pH was measured on the fresh effluent daily by using a pH probe. Total solids (TS) and volatile solids (VS) and COD concentration of the feed and the effluent was measured thrice a week. Volatile solids destruction was calculated by the formula $(VS_{initial}-VS_{final})/VS_{initial}$. For measuring total COD, samples were first diluted (1:100) and then acidified using concentrated H_2SO_4 to lower the pH to less than 2. The closed reflux method (APHA 1999) was used to measure COD concentration in the samples.

The biogas produced from each digester was collected in the 25 L Tedlar bags. The rate of production of biogas was calculated by measuring the volume of gas in the bags using a calibrated peristaltic pump. For determining the gas composition, the samples were analyzed using a Shimadzu model GC-14A gas chromatograph (Shimadzu Scientific Instruments, Columbia, MD) with the thermal conductivity 28 detector (TCD). The column used was made from a 4 meter length of copper tubing with a 0.25 inch inner diameter. The column was coiled to fit in the GC-14A oven and packed with Haysep Q media (Supelco, Bellefonte, PA). Helium was used as the carrier gas, with column flow set at 17 mL/min.

For the ammonium ion, phosphate ion and VFA analysis, samples were collected twice a week and were frozen to avoid any methanogenic activity. In order to analyze the samples, they were first thawed at room temperature and then centrifuged at 13,500 x g for 25 minutes. The supernatant was then filtered through a 0.45 micron syringe filter. From this filtered sample, dilutions were made for the ions and VFA testing. The VFA samples were acidified with 85% phosphoric acid (1:10) before the run. The VFA samples were analyzed on a Hewlett Packard Model 5890 gas chromatograph using a flame ionization detector (FID). The samples were injected in splitless mode into a Nukol capillary column (Supelco) with Helium gas as the carrier gas. The Helium flow rate was set to 17mL/min. Flow rates for the other gases used were as follows: Nitrogen – 13 mL/min, Hydrogen – 45 mL/min, Air – 450 mL/min.

Diluted ammonium ion samples were analyzed on a Dionex ICS-1000 ion chromatograph utilizing a CS-12 column and conductivity detection with self-generating suppression of the eluent (Dionex Corp., Sunnyvale, CA). 25mM methanesulfonic acid was used for eluent at a flow rate of 1 mL/min. Total Phosphate in the sample was analyzed using an ion chromatograph (Model No. DX-120, Dionex) equipped with AS9-HC column (Model No. 051786, IonPac). The eluent was 9.0 mM Na2CO3 and the flow rate was 1.0 ml/min.

The method used for the sludge dewatering for the preparation of dewatered sludge cakes for organic sulfur analysis is described by Muller et al. (2004). The sludge was conditioned using 1% high molecular weight cationic polymer (Clarifloc 3275, Polydyne). The polymer dose was determined for 100ml conditioned samples. The polymer dose that resulted in the lowest capillary suction time was selected as the optimum polymer dose. A mixture of optimum polymer and sludge was then sheared in a blender (Oster 12 speed, 6811-C) at 450 Watts for 45 sec and then centrifuged at 10000 rpm (17700 G) for 15 min at 4^{o}C temperature. The centrate was discarded and the sludge pellet was collected and pressed at 207 kPa for 15 min by a lab press. A dewatered sludge cake was obtained from this which was further used for volatile organic sulfur compounds measurement and determining the cake solids concentration.

Total solids and volatile solids in the cake solids were measured by the method described in Standard Methods for the Examination of Water and Wastewater (APHA 1999). The cake solids were then used to make samples for odor analysis.

For odor analysis, 50 mL glass bottle were used for incubating 5 grams of dewatered cake. Duplicate samples were prepared for each sample for more consistent readings. To maintain anaerobic conditions inside the glass bottles, they were sealed with screw caps and Teflon-lined rubber septa and they were then incubated at a constant temperature (at 25^{o}C).

Using a micro-syringe, 25 microliter headspace gas from each incubation bottle was collected and injected into gas-chromatography/mass spectrometer (Model No. GC 6890, MSD 5970, Hewlett-Packard) with a cryo-trapping system. Liquid nitrogen was used to form the cryo-trap. The cryo-trap was employed to accumulate gas samples and to generate narrow chromatographic peaks. A Supelco column (Model No. 20751-01A), 30 m long and 0.25 mm internal diameter was used for injection. Helium was used as a carrier gas at a flow rate of 2 ml/min. The oven temperature was increased from 50^{o}C to 265^{o}C at a rate of 35C/min. Each sample was analyzed for a total time of around 6 minutes. The volatile organic sulfur compounds (VOSC) namely methanethiol (MT), dimethyl sulfide (DMS) and dimethyl disulfide (DMDS) were measured during this analysis. Peak areas of each organic sulfur compound were integrated by the data analysis program, G1034C version C.03.00 (Hewlett-Packard). The amount of organic sulfur in each sample was quantified by comparing the sample peak area with the area of a standard gas mixture of known amounts of H2S, MT and DMS (Scott Specialty Gases Inc., PA). The concentration of odor generating compounds was expressed in the form of total VOSC (TVOSC) which is the sum of MT, DMS and DMDS concentrations.

Chapter 5

Results and Discussion

5.1 Research approach

The research was conducted to study the applicability of thermal hydrolysis pre-treatment on the TRA sludge. Also, the performance of the process at two different temperatures (35^oC and 42^oC) and two different SRTs (15d and 20d) was compared in order to optimize the conditions for the best results.

It has been shown that the efficiency of this process depends greatly on the sludge characteristics such as organic content, COD, ammonia concentration etc. [Gavala et al., 2003]. Hence all the relevant sludge characteristics (pH, TS, VS, and COD etc.) were regularly measured and analyzed. All the analyses were performed at steady state. Steady state was determined by daily monitoring of pH, biogas production, solids removal and COD reduction. When little or no variation was observed in the values of the parameters over 2 weeks, it was considered as an indication of steady state in the digester.

As mentioned earlier, the study was conducted in two phases: 15d SRT and 20d SRT. For both the phases the effects of variation of temperature (35^oC and 42^oC) were tested.

5.2 Experimental results

The results from the testing and a discussion of the results are presented in the following sections:

5.2.1 pH

Anaerobic digestion is generally operated under at near neutral pH conditions (pH 6.5-7.6). The observed toxicity under low pH conditions is associated with the presence of un-dissociated VFA [van Lier et al. 2001]. The methanogenic activity will slow considerably with pH less than 6.3 and higher than 7.8 and this will inhibit the biogas production [Leitao et al., 2006]. Lower pH will result in the growth of filamentous bacteria and a high pH results in buildup of unionized ammonia [Grady et al., 1999].

During phase I, the pH of the Control digester was one of the initial problems observed during this research. The initial pH for the Control digester was measured as 6.2 which was less than optimum for the methanogenic population inside the digester. It was necessary to raise the pH to >6.5, thus the Control digester was dosed with alkalinity (sodium bicarbonate) for a period of 30 days. Sodium bicarbonate was weighed on a scale and mixed with the Control feed. The amount of sodium bicarbonate added to the digester was gradually decreased from 4g/l to 1g/l according to the changes observed in the pH over time. When the pH reached a steady state value between 7.03-7.12, the dosing was terminated. As shown in Figure 6, steady state required about 30 days to achieve. The reason for initial low pH is not known.

The pH values were phase II were higher than those for phase I. This can be attributed to high ammonia concentrations associated with the digesters at the lower SRT. The variation of pH in all the digesters over time is shown in Figures 5-1 and 5-2.

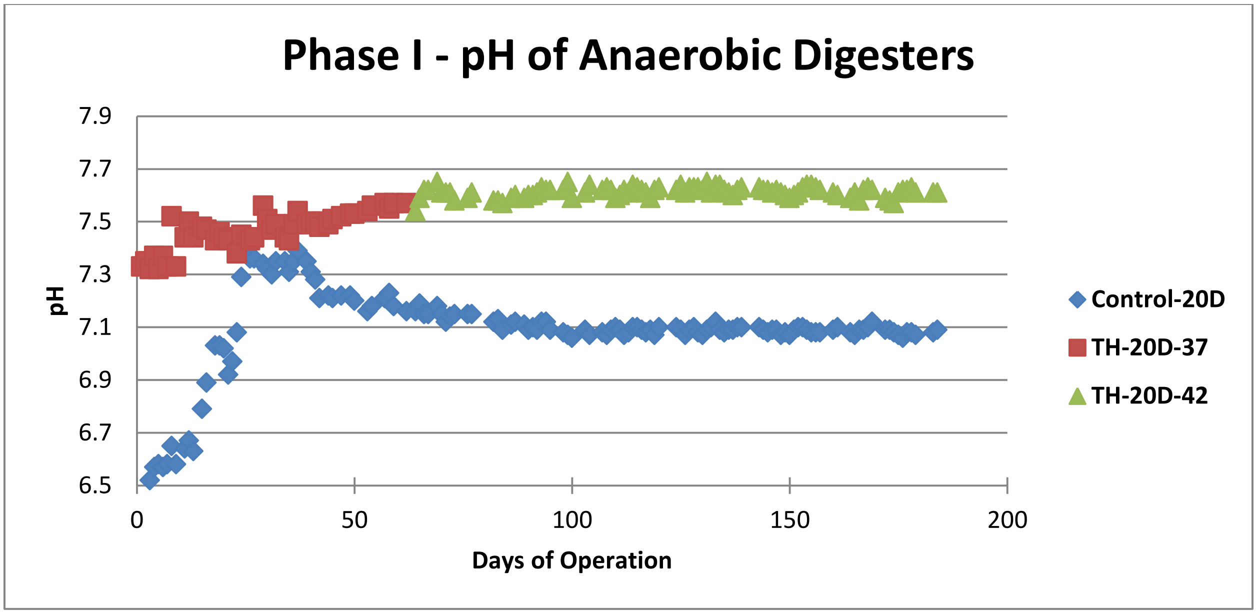

Figure 5-1: pH of anaerobic digesters during phase I (20 day SRT)

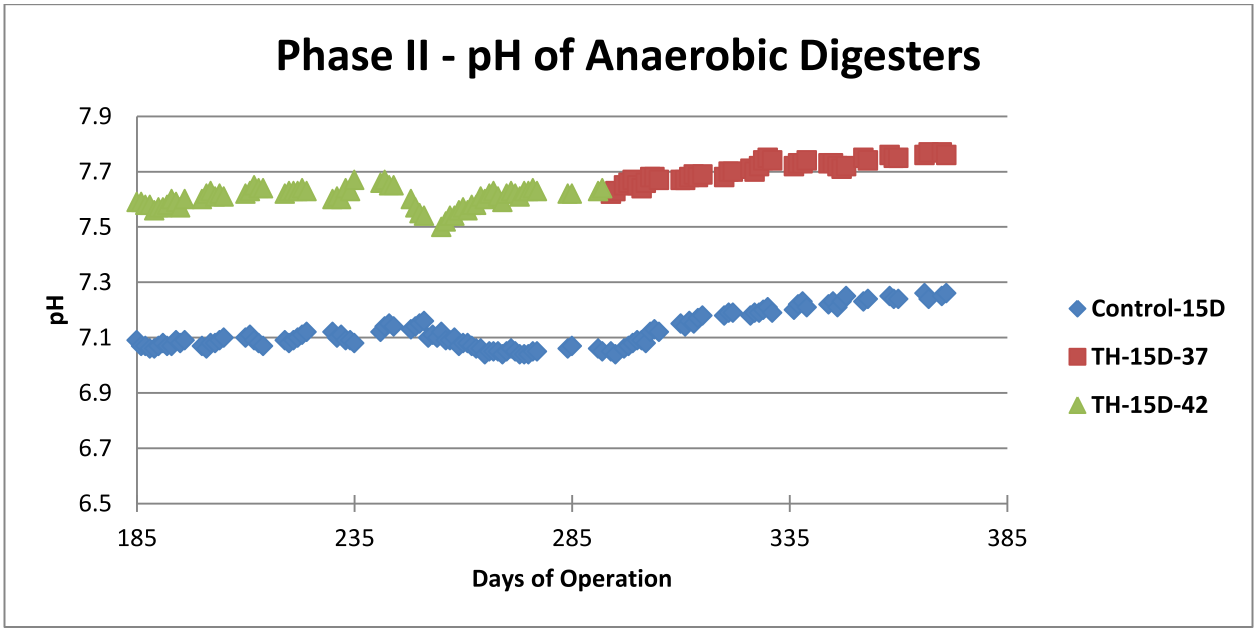

Figure 5-2: pH of anaerobic digesters during phase II (15 day SRT)

The pH for the TH digester was always observed to be >7.3. This indicated healthy conditions inside the digester and a good amount of methanogenic activity was expected. Although, due to the high concentration of feed solids (~10%) variations in the pH values were observed during startup.

The overall pH of the digesters varied as shown in Figure 5-3. The variation in temperature also affected the pH of the digesters. The pH increased with an increase in temperature for phase I but due to the ammonia build up issues in phase II the pH was seen to decrease with an increase in temperature. During phase II, the digester was initially operated at 42°C and then the temperature was dropped to 37°C in the later stage. An increase in pH was observed after this temperature shift. This could be attributed to the ammonia that was building up in the digester during phase II. The ammonia concentration was seen to increase with the time period of operation as shown in Figure 5-13.

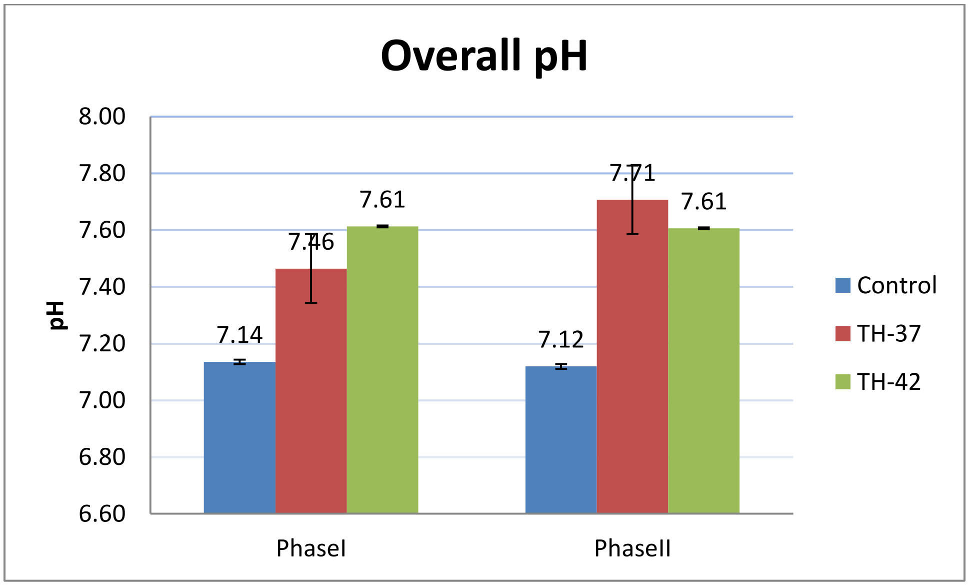

Figure 5-3: Overall pH for the digesters during phase I and II

5.2.2 Solids Reduction

TS and VS were measured for all the digester influent and effluent sludges, three times per week. The reduction in solids content in the effluent was then plotted as shown in Figure 5-4 and 5-5.

As expected, both the TS and VS reduction for the TH system was approximately 10% greater than that for the control. This indicated the better performance of the TH digested sludge as compared to the conventionally digested sludge. An increase of 8-10% was expected based on prior studies for DC WATER [Wilson et al., 2008]. The increase in VS reduction was 11% for the TH system operated at 37^oC. When the temperature of the TH digester increased to 42^oC, a slight reduction in the TS and VS reduction was observed which could be due to changes in the sludge characteristics. However, the increased VS reduction at 42^oC was 9% for the TH system as compared to the control system, indicating that the TH digester preformed about as well at 42^oC as at 37^oC. This was a very promising result because it is economical to operate at 42^oC because less cooling is required.

During phase II, the TS and VS reductions were statistically the same for the control and the two TH digesters. For the 15 days SRT operation, there was no benefit to thermal hydrolysis. This can be attributed to the increased loading of the digesters with organic substrate by reducing the SRT to 15 days and also the increase in ammonia associated with phase II. The ammonia data will be presented and discussed later. This also suggests that the 20 day SRT conditions were optimum for the TRA sludge.

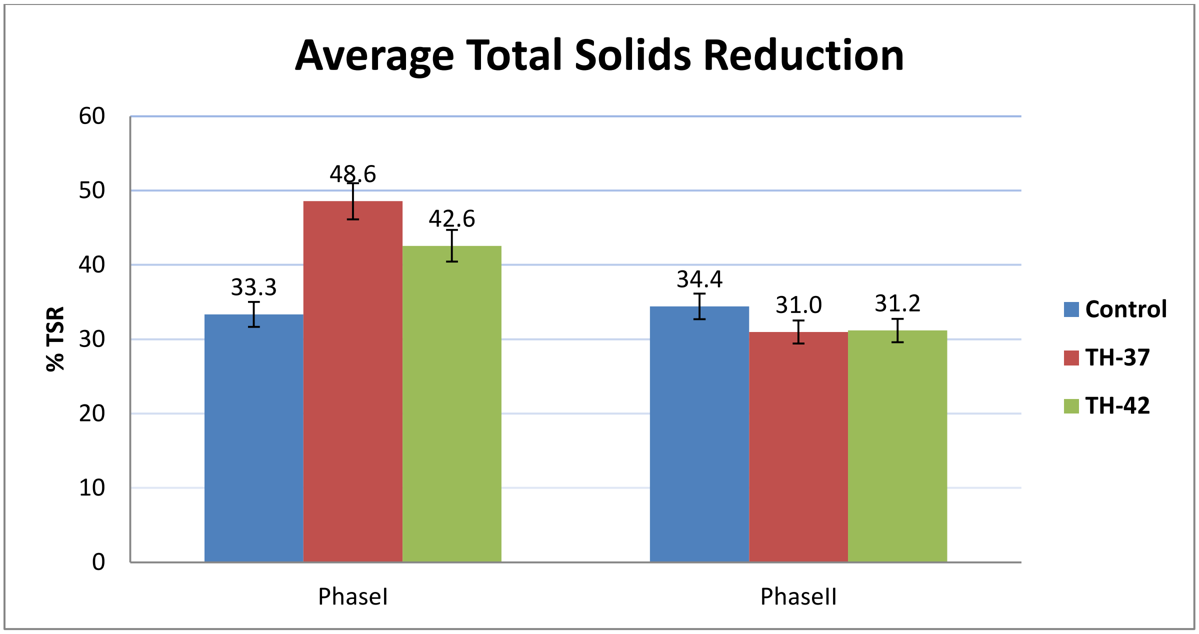

Figure 5-4: Average total solids reduction in anaerobic digesters during phase I and II

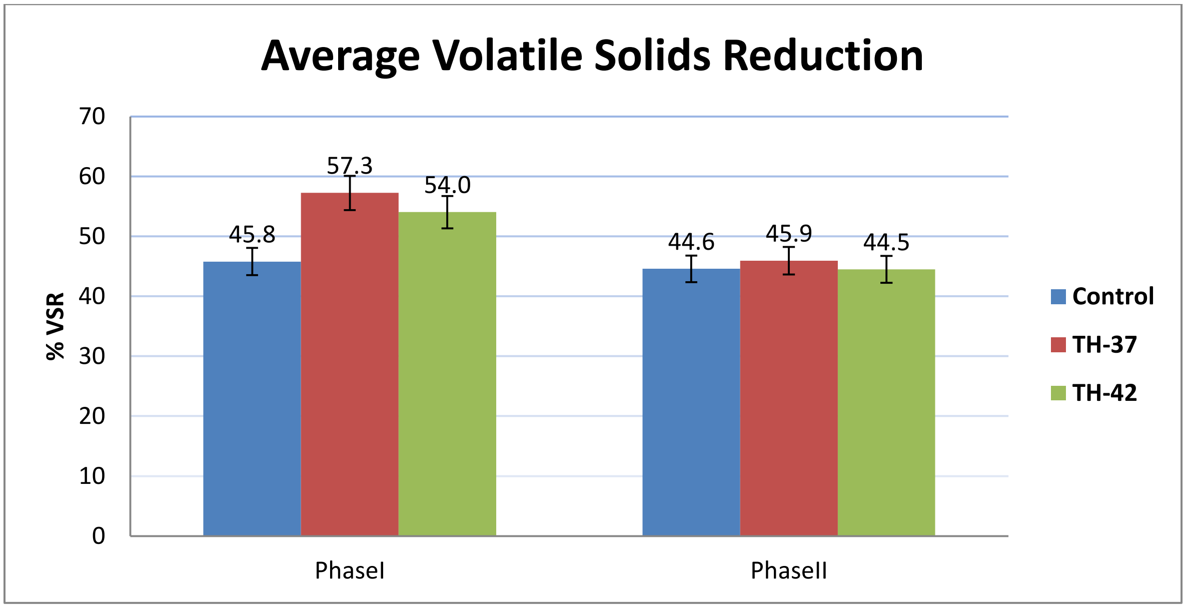

Figure 5-5: Average volatile solids reduction in anaerobic digesters during phase I and II

Due to the limitations on the usage of the bomb, the pressure build-up and the pressure release during this process cannot be compared to the steam explosion and the flash tank in the Cambi process. The sludge could possibly show better digestibility and higher solids reduction after the steam explosion as compared to the thermal hydrolysis technique used in this study.

5.2.3 COD reduction

The reduction in COD is a measure of biodegradation occurring in the digester. The COD in the digester was measured three times per week and the average COD reduction obtained at steady state has been shown in Figure 5-6.

COD reduction is correlated to VS destruction [Spinosa et al., 2001]. Rittman & McCarty [2001] have reported the theoretical COD/VS ratio to be 1.42. The ratio depends on the process employed and the feed characteristics. Gonzalez-Fernandez et al., 2009 studied the effect of COD/VS ratio on the digestion of swine slurry and concluded that COD/VS =1 was the optimum ratio for that process.

During phase I, the average COD reduction was ~48% for control, ~43% for TH-37 and ~59% for TH-42 digesters. The variation in data was observed primarily due to changes in feed characteristics and difficulty in measuring the COD accurately. The TH feed, being ~10% solids was difficult to analyze for accurate COD concentration. An increase in COD reduction was expected in the TH digesters as compared to control based on the VS destruction trend. The COD reduction during the beginning of phase I at 37^{o}C was observed to be lower than expected; this could be due to the fact that methanogens were not yet acclimated in the digester. A higher COD reduction was observed during the later segment of phase I at 42^{o}C due to longer period of operation.

During phase II, the COD reduction profile was similar to the VS reduction profile shown in Figure 5-5. The COD reduction for the digesters in phase II was lower than that during phase I. The COD removal in control and TH digesters was comparable, indicating that the TH pre-treatment was more successful at 20 day SRT than at 15 day SRT.

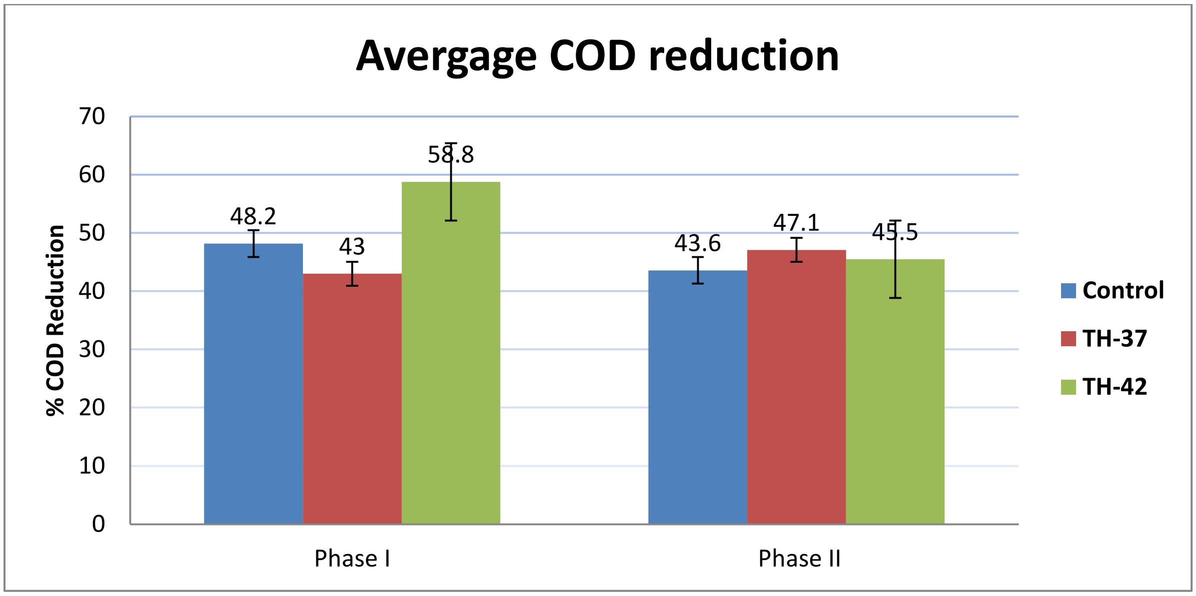

Figure 5-6: Average COD reduction in the digesters during phase I and II

Figure 5-7 shows interaction between VSR and CODR for all the digesters. The red point on the first plot represents the TH-20D-37 digester and it can be seen that the CODR value for this digester was probably incorrect. The second plot in the figure shows the data for all the digesters except TH-20D-37 and it can be observed that VSR and CODR showed a positive proportionality for all the other digesters.

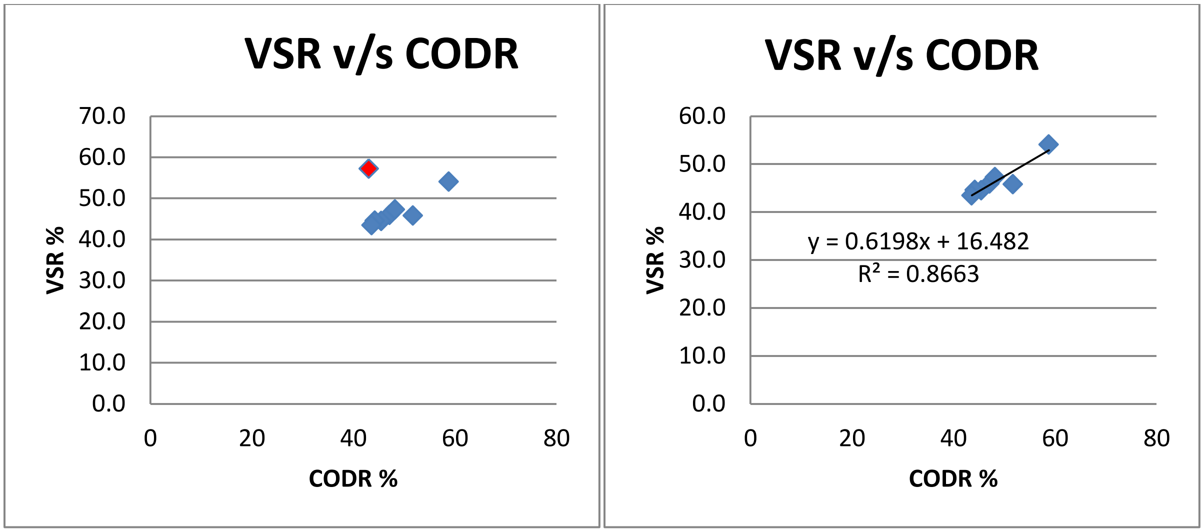

Figure 5-7: Correlation between VSR and CODR for all digesters

5.2.4 Biogas Production and Composition

Biogas production and its use for energy recovery from wastewater treatment is one of the most important attributes of anaerobic digestion and is the motivation for employment of pre-treatment methods. As mentioned earlier, thermal hydrolysis pre-treatment is a promising technology in terms of production of biogas from the digestion of sludge. The methane content in the biogas corresponds to the stability and the performance of the anaerobic digester as it reflects the amount and type of organic matter degraded [Grady et al., 1999].

Gas production from all the digesters was measured regularly and the specific gas production was calculated as L/g of VS destroyed. The specific gas production for all the digesters is shown in Figure 5-8.

This is an important set of data because the normal specific gas production is expected to be 14 to 15 Ft^3/lb of VS destroyed [Rittman, McCarty 2001]. The control values are slightly below that value. However, the thermal hydrolysis values, especially in Phase I, are higher than that, indicating that thermal hydrolysis not only generates more solids destruction but the solids destroyed are different from those destroyed in the control. It is suspected that these additional solids contain a high proportion of grease that otherwise would not degrade. It is important that this extra specific gas production be considered in the design of the thermal hydrolysis digesters.

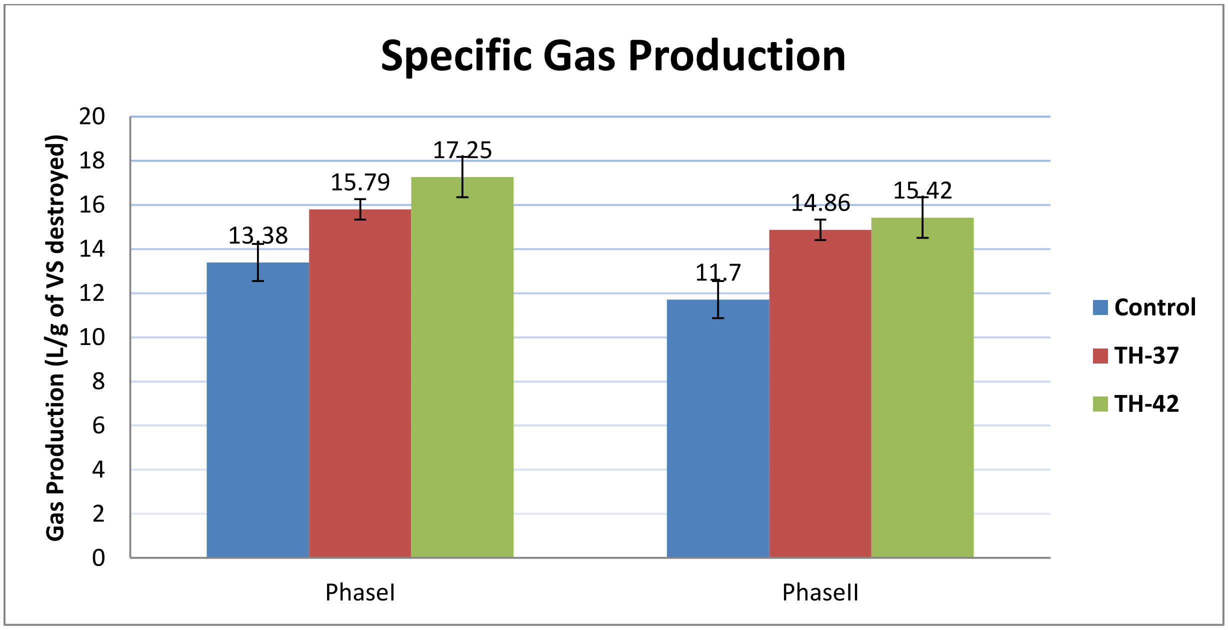

Figure 5-8: Specific gas production values for anaerobic digesters for phase I and II at steady state

In the initial study period, gas production for the control was low due to the low alkalinity and the average values for Control in phase I include the duration when alkalinity was being added which led to high CO_2 exiting the digester. This is consistent with the VFA data that indicated that the system may not have been entirely acclimated. Gas production for TH-42 was much steadier and this reflects the acclimation of the organisms and the improved measurement technique.

As the solids destruction was lowered in Phase II, consequently the gas production was also seen to be lower than that in Phase I. The gas collected from the digesters was analyzed for methane and CO_2. The methane concentration for different digesters is tabulated below.

Table 5-1: %Methane data for the biogas collected from anaerobic digesters

Sample	% Methane (by Volume)
Cotrol-20D	62
TH-20D-37	56.6
TH-20D-42	66.5
Control-15D	56.3
TH-15D-37	59.1
TH-15D-42	58.6

The data shows that the methane production is similar for the TH system and the control are in the expected range of methane production for sewage sludge digesters.

5.2.5 Volatile Fatty Acids

Volatile fatty acids are weak acids that are released as intermediates during the breakdown of organic matter in anaerobic digestion of sludge [Coskuner et al., 2002]. Accumulation of VFA can result from overloading due to increased ammonia and the predicted ammonia inhibition of microbes involved in methanogenesis [Leitao et al., 2006]. The presence of high VFA has been reported to have damaging effects on methanogenic activity.

Volatile fatty acid data is shown in Table 5-2 and Figure 5-9. The VFA were higher in Phase I, although they were within the range considered normal. In Phase 1, the TH system had somewhat higher VFA than did the control digester. At the end of the operational period, the VFA in the Phase I digesters were trending down and that suggests that the methanogenic organisms had not yet adjusted to the digester conditions. Methanogens are very slow growing so it is possible that they were not yet at steady state condition when the experimental phase was terminated. The VFA were the lowest for the TH digester at 42^oC. One sign of ammonia toxicity is an increase in the VFA. Since these were low, this is another indication that no ammonia inhibition was taking place. These data also indicate that acclimation was likely occurring throughout Phase I.

The same sludge was used in Phase II so it was better acclimated as this is reflected in the lower VFA. Higher values of digester ammonia were observed in Phase II which could be the reason for comparatively poorer performance of digesters in this phase. Generally, VFA concentration is reported to increase in the cases of ammonia toxicity [Tanneru et al., 2009]. The total VFA concentration has been shown in Table 5-2. The feed concentration was not measured for phase II. It was assumed to be the same as phase I.

Table 5-2: Total VFA Concentration for feed and effluent in different phases

Sample	**Total VFA Concentration (mg/L)**	
	Phase I	**Phase II**
Control Feed	1650	N/A
TH Feed	3597	N/A
Control	1491	175
TH-37	1613	309
TH-42	776	329

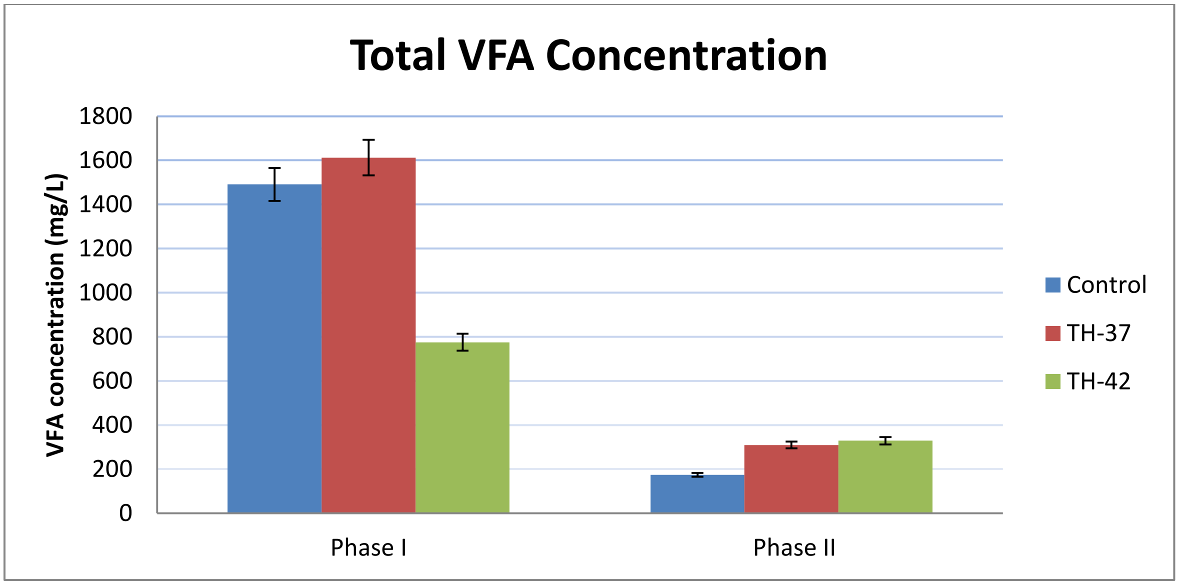

Figure 5-9: Total VFA concentration in anaerobic digesters for phase I and II

The concentrations of the individual VFA for Phase I and II are plotted Figures 5-10 and 5-11.

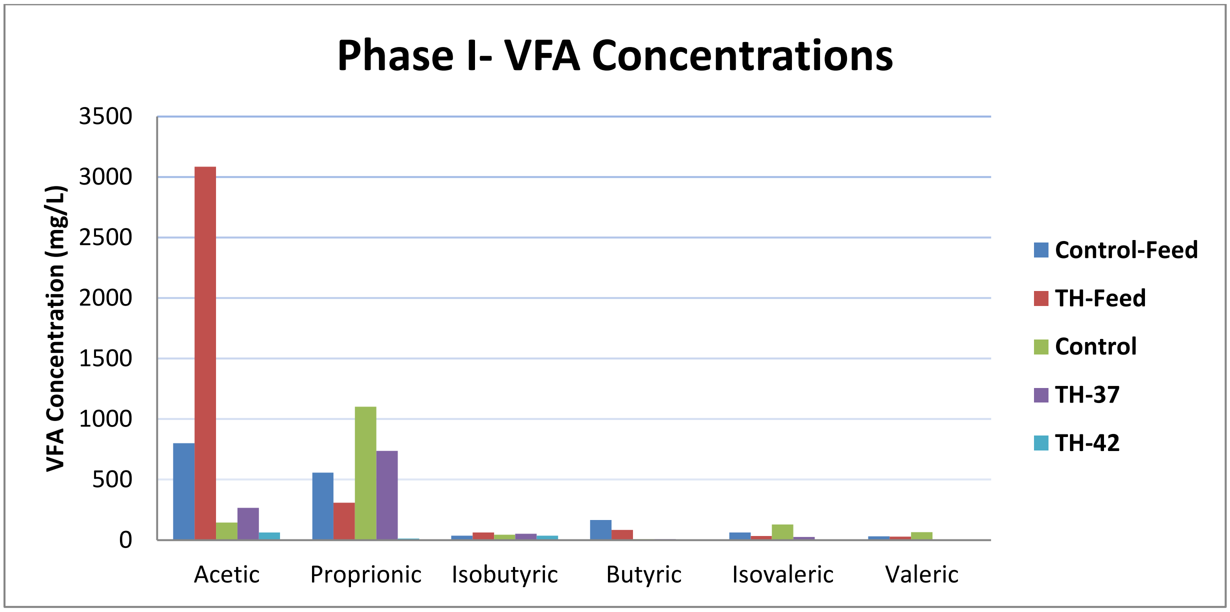

Figure 5-10: VFA concentration in feed and effluents from anaerobic digesters during phase I

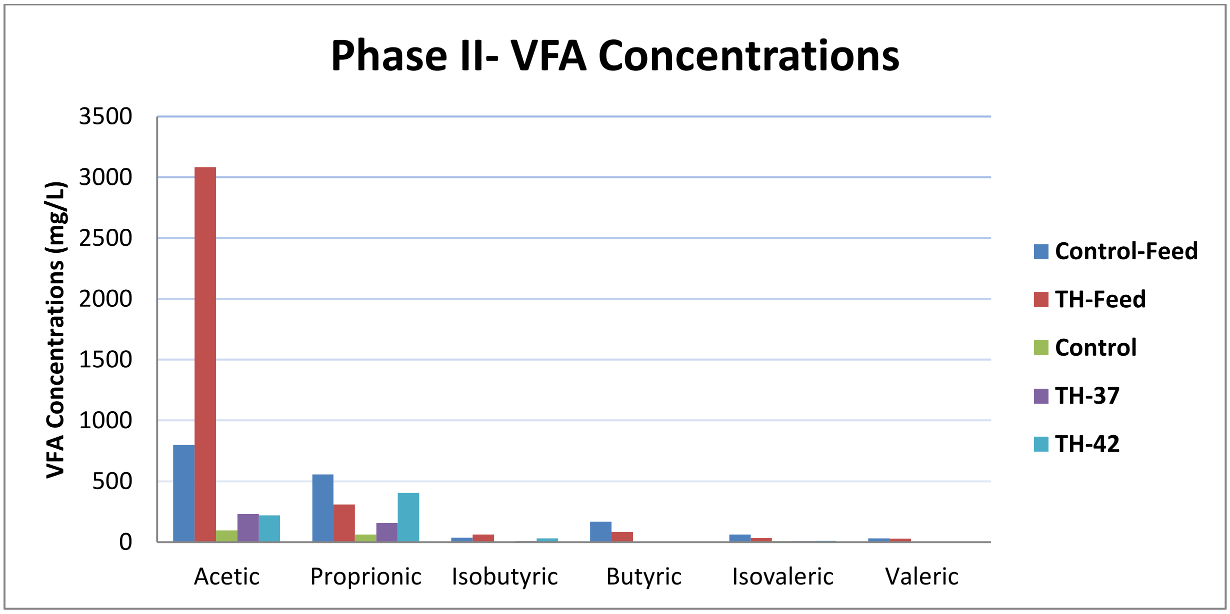

Figure 5-11: VFA concentration in feed and effluents from anaerobic digesters during phase II

5.2.6 Ammonia and TKN

Ammonia is produced in the digesters due to the hydrolysis of protein present in the organic matter. TH treatment stimulates this hydrolysis process and thus can result in high levels of ammonia in the digesters. The high pH conditions and the high solids concentration in the feed can also contribute to the increased levels of Total Ammonia Nitrogen (TAN) [McCarty et al., 1964]. The recycle flows from dewatering of sludge containing high TAN concentrations can increase wastewater influent load by 15 to 20 percent [Kayhanian, 1994].The ammonia data are important because a high ammonia concentration can result in toxicity. It has been reported that free ammonia concentration of >100 mg/L as N is toxic to the system and can be detrimental for methanogenic activity [Grady et al., 1999]. However, data from Wilson, (2006) indicates that the inhibitory concentration may be less than 100 mg/L free ammonia.

Ammonia data are shown in Figure 5-12 for the two phases. The ammonia data are also summarized in Table 5-3. It can be seen that for the TH systems, the ammonia concentration was about 1340 mg/L for TH-20D-37 digester and 1290mg/L as N for the TH-20D-42 digester during phase I. The 37^{o}C digester showed higher concentration of ammonia as compared to the 42^{o}C digester, this could be attributed to comparatively higher solids destruction in the digester at 37^{o}C.

For phase II, the values were slightly higher in all the digesters. The control digester showed an increase from approximately 300mg/L in phase I to 600mg/L in phase II. The TAN concentrations for TH-37 and TH-42 digesters increased from 1340 mg/L and 1291 mg/L in phase I to 1570mg/L and 1740mg/L in phase II, respectively.

These data are very important because they suggest the possibility of ammonia toxicity in the digesters during phase II. The higher ammonia levels during phase II couple with higher pH may be the reason for the relatively poor performance of the digesters during this phase.

Table 5-3: Total ammonia concentration in feed and effluents for different phases

Sample	Total Ammonia Concentration (mg/L as N)	
	Phase I	Phase II
Control Feed	200	N/A
TH Feed	364	N/A
Control	294	600
TH-37	1340	1573
TH-42	1291	1738

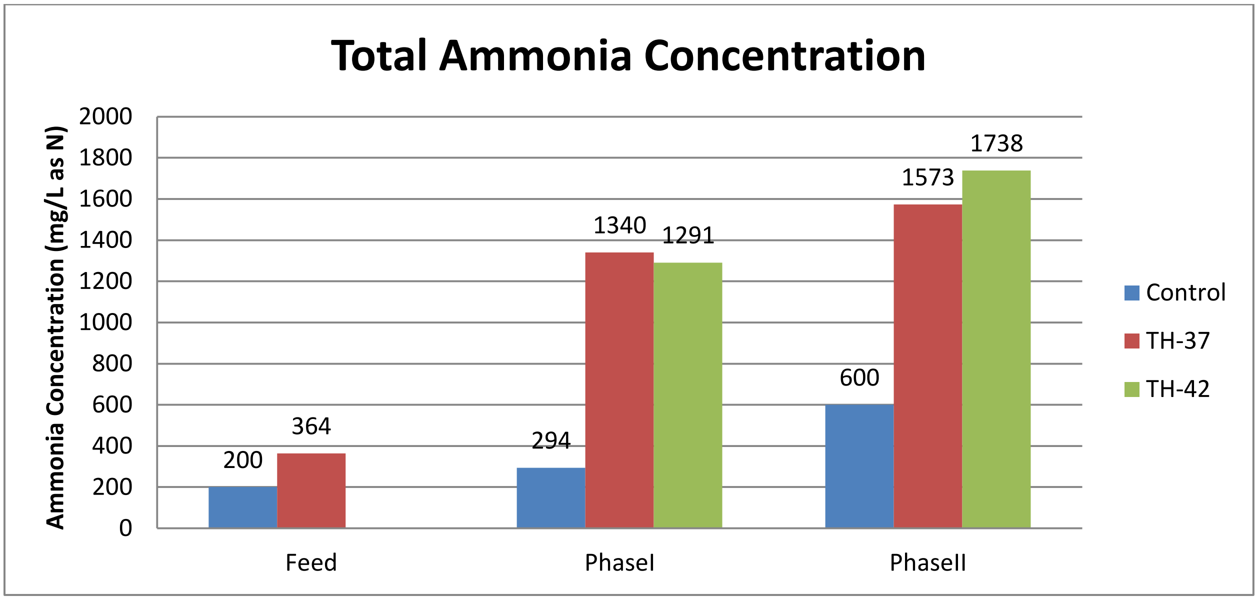

Figure 5-12: Total ammonia concentration in feed and effluents from the anaerobic digesters during phase I and II

Figure 5-13 shows the trend of ammonia accumulation in the digesters during the course of this study. It can be seen that the sludge had a higher ammonia concentration during phase II. This is clearly an issue of importance because the second phase digester performance was poorer. The higher organic loading and the higher pH conditions during phase II could have been the reason for the higher ammonia level during this phase.

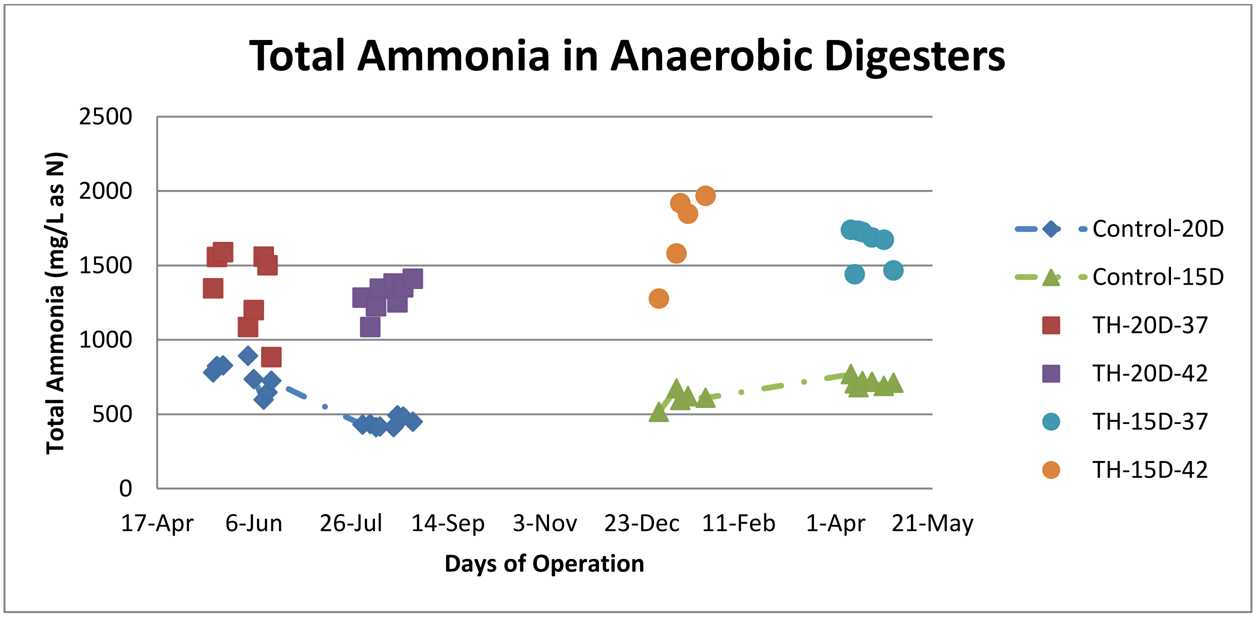

Figure 5-13: Total ammonia concentration in anaerobic digesters varying with time

Total Kjeldahl Nitrogen (TKN) is the sum of organic nitrogen, ammonia (NH3) and ammonium (NH4+). The average TKN values for the feed and effluents for both the phases are summarized in table 5-4. The TKN values for TH feed were much higher than the values for Control feed. This was consistent with the solids concentrations data for both the feeds, the TH feed being ~10% solids and while the Control feed being ~4% solids. The TKN values for all the digesters in both the phases were similar, as expected. The TKN values for TH-42 were noted to be slightly higher than TH-37 digesters; this is consistent with the ammonia data.

Table 5-4: Average values for TKN in feed and effluents from anaerobic digesters

Sample	**TKN (mg/L as N)**	
	Phase I	**Phase II**
Control Feed	1540	1610
TH Feed	3500	3580
Control	1580	1570
TH-37	3170	3250
TH-42	3870	3640

5.2.7 Phosphate

During the degradation of organic substrate, phosphorus is also released in the system along with nitrogen. At higher concentrations, phosphorus can combine with the Magnesium or Iron present in the sludge and form struvite ($MgNH_4PO_4.6H_2O$) and vivianite ($Fe_3(PO_4)_2 \bullet 8(H_2O)$). Struvite is well known for plugging pipes and fouling pumps, aerators, screens, and other equipment. As the TH pre-treatment stimulates hydrolysis, it also promotes the release of phosphorus in the sludge. Therefore, it was necessary to check the phosphorus concentration in the digesters and the feed. Phosphorus was measured and reported as mg/L phosphate in the sludge samples.

The phosphate concentration is presented in Figure 5-14 for the control and Phase I TH-42 system. The phosphate concentrations were similar at about 8 mg/L. Struvite and vivianite formation should be responsible for low phosphate levels in TH systems.

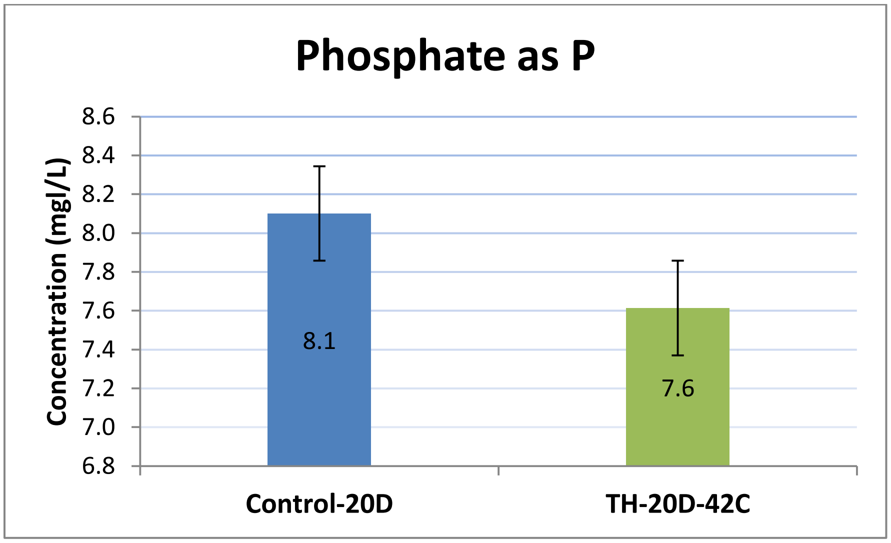

Figure 5-14: Phosphate concentration in control and TH-20D-42 digesters

The concentration of phosphorus in the digesters over time is shown in Figure 5-15.

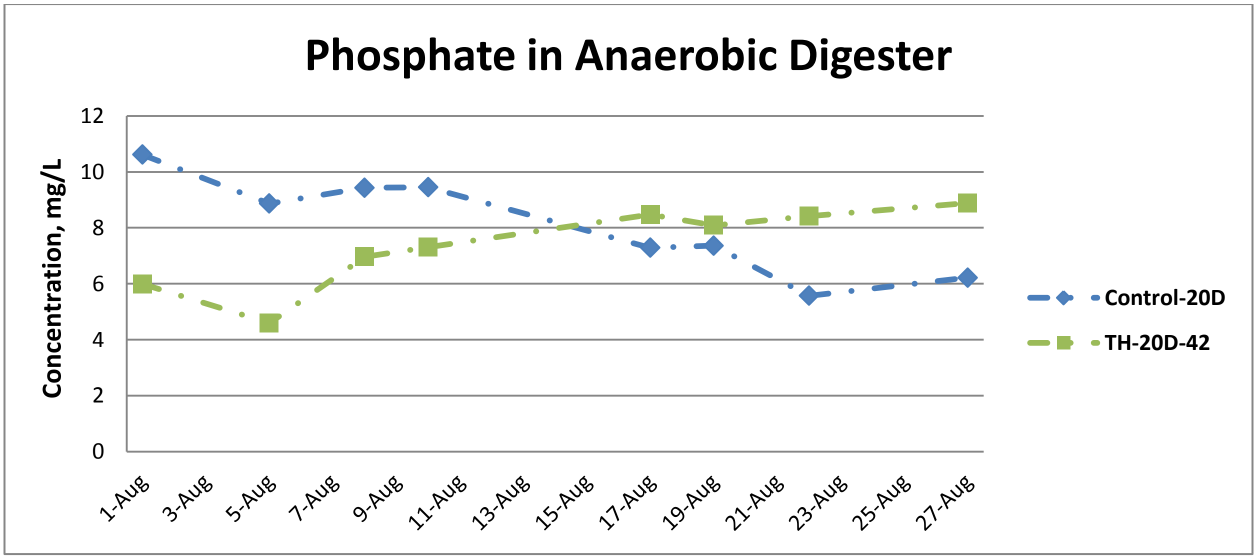

Figure 5-15: Variation of phosphate concentration in control and TH-20D-42 digesters over time

Struvite precipitation occurs when the combined concentrations of Mg^{2+}, NH_4^+ and PO_4^{3-} exceed the struvite solubility product (Ksp between 10^{-10} and $10^{-13.3}$) [Burns eat al., 1982; Taylor et al., 1963]. Marti et al.,[2008] conducted studies to assess the formation of struvite in anaerobic digesters and reported that the sludge with high ammonia concentration was more prone to struvite precipitation.

During phase II, the increase in organics loading led to an increase in ammonia concentration in the digesters which could have resulted in struvite formation in the digester during this phase.

5.2.8 Optimum Polymer Dose and Cake Solids Concentration

The experiments for finding the Optimum Polymer Dose (OPD) for the sludge samples posed one of the major challenges during this study. The pre-calibrated blender which was being used for these experiments was found broken and it was difficult to replace it. Hence a new blender was installed and recalibrated. It took about 50 days to do this task. Various samples were prepared at shearing times of 30s, 45s and 55s. The capillary suction time for each sample was plotted against the optimum polymer dose used for that run. Three curves were obtained for the shearing times of 30s, 45s and 55s.

Figure 5-16 shows the plot obtained from these experiments.

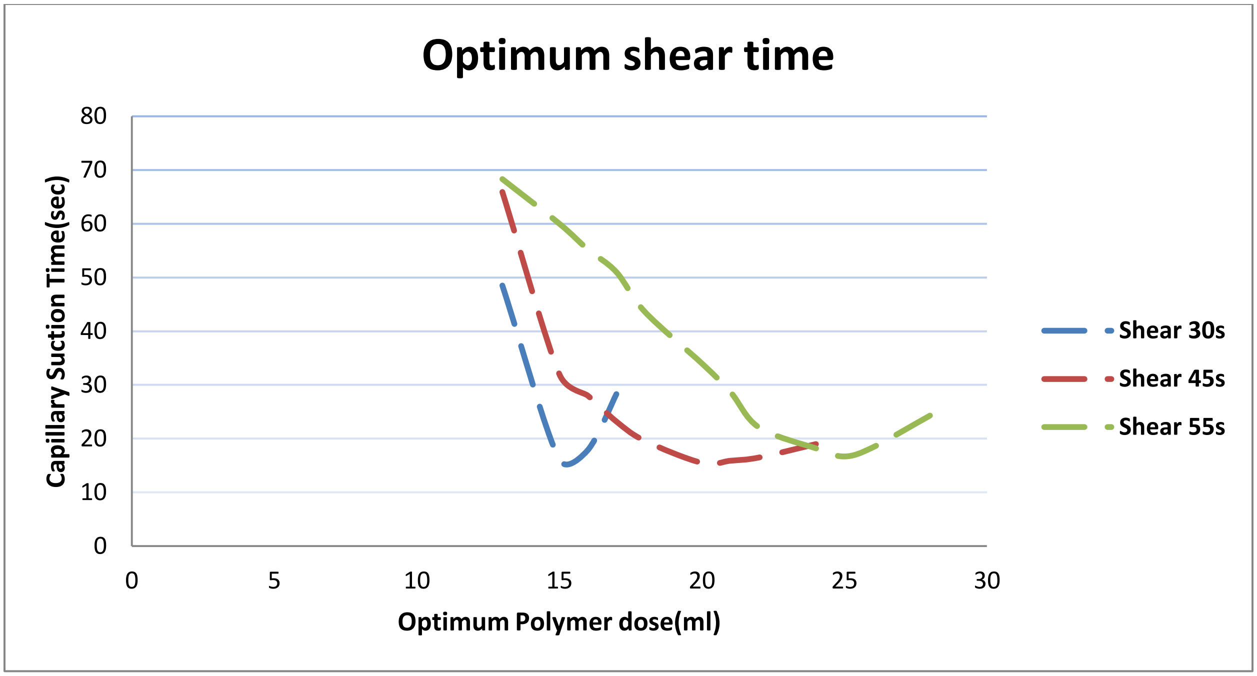

Figure 5-16: Experimental data for determining the optimum shearing time

All these samples were then transformed into cake solids as described in the Methods chapter earlier and a GC analysis was performed on these. The plot obtained from the GC analysis of the TVOSCs present in all the samples is shown in Figure 5-17. Based on this analysis, the optimum shearing time was decided to be 45s. All the samples prepared for the TVOSC analyses in this study have been sheared in a blender (Oster 12 speed, 6811-C) at 450 Watts for 45s.

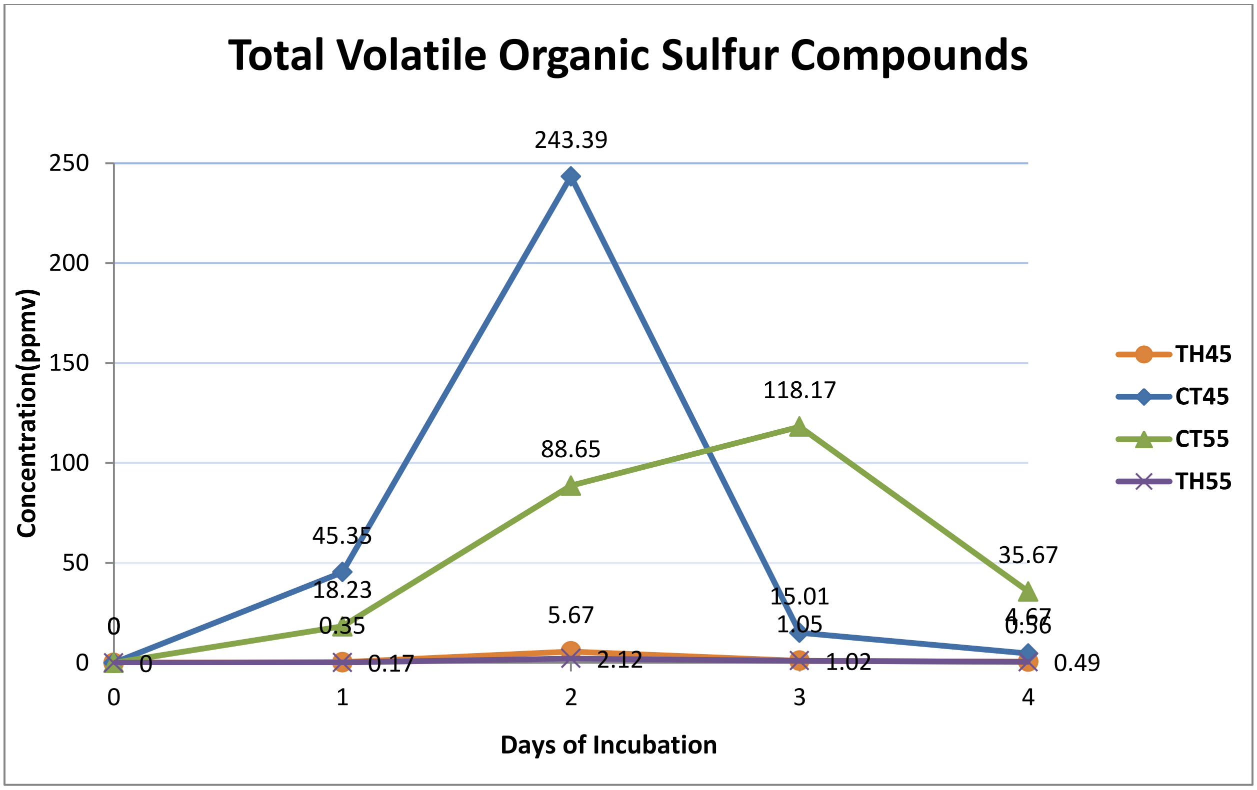

Figure 5-17: TVOSC generation pattern of sludge cakes sheared at 45s and 55s.

The OPD and the cake solids concentration for all the digester samples have been reported in Table 5-5. The OPD for the Control digesters was 17ml and 19ml for Phase I and Phase II respectively. The OPD for TH-20D-37 was not calculated due to technical issues and has been marked as N/A in Table 5-5. The dose for TH-20D-42 was 60ml, TH-15D-37C was 61ml and TH-15D-42 was 63 ml. the OPD for all the TH systems were similar, they were minutely higher for the phase II digesters.

One of the main advantages of TH pre-treatment is the higher concentration of cake solids. This was supported by the results from this research as well. While the cake solids concentration for

Control digesters were about 25% for both the phases, the cake solids concentration for TH-20D-42 was the highest 37%, TH-25D-37 was 35% and TH-15D-42 was 33.6%.

These results showed higher cake solids concentrations which were similar to the work performed by Pickworth et al. [2006] and Kepp et al. [2000]. Anaerobic digestion with Cambi pre-treatment resulted in higher cake solids concentration compared to conventional anaerobic digestion.

Table 5-5: Optimum polymer dose and cake solids concentration for effluents from different digesters

Sample	OPD lb/ton TS	Cake Solids %
Control-20D	3.59	25
TH-20D-37	NA	NA
TH-20D-42	4.99	37
Control-15D	4.26	24.3
TH-15D-37	4.82	35
TH-15D-42	4.83	33.6

5.2.9 Odor generation

Odors are typically a problem for dewatered biosolids since digested but undewatered sludge usually shows no or low odor [Adams et al., 2004]. Odor emissions are a nuisance for the population in the vicinity of the sewage treatment works and near land application sites [Frechen, 1988; Wilson et al., 1980]. Odor emissions have been reported to affect quality of life [Brennan, 1993] leading to psychological stress and symptoms such as insomnia, loss of appetite and irrational behavior [Wilson et al., 1980].

The main odor causing compounds present in the dewatered sludge are volatile inorganic sulfur compounds and volatile organic sulfur compounds (VOSC) which include carbon disulfide, hydrogen-sulfide (H2S), methanethiol (MT), and dimethyl sulfide (DMS), dimethyl disulfide (DMDS) and trimethyl amine. Methanogens play a key role in the degradation of VOSC's [Novak et al., 2006; Higgins et al., 2006].

In Figure 5-18, the odor data are shown for the two phases. No odor data were collected for TH-20D-37C. These data are very important and show that the TH system produced a very low odor final product. A total volatile organic sulfur concentration below 100 ppm is considered to be a low odor product and the peak TVOSC for the TH biosolids reached a maximum of 20 ppm, indicating its very low odor potential.

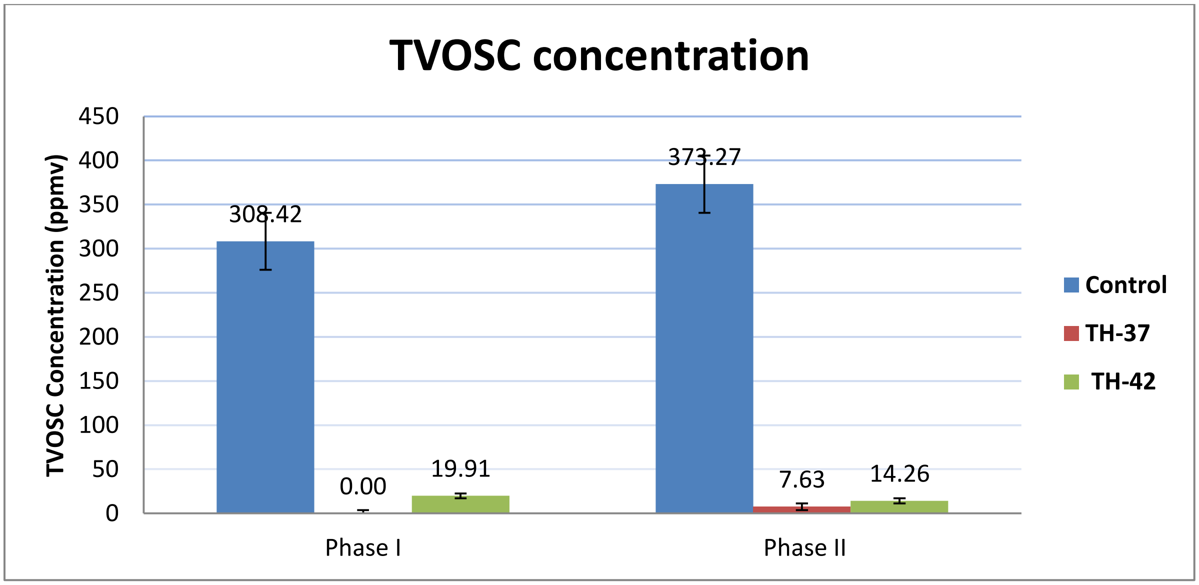

Figure 5-18: Total volatile organic sulfur compounds concentration in the samples from anaerobic digesters during phase I and II

The TVOSC concentration in TH systems was seen not to vary much with temperature and SRT. In all the digesters for both the phases, the odor potential of TH samples was much lower as compared to the conventional mesophilic control digester which resulted in a TVOSC concentration of 308ppmv for phase I and 373ppmv for phase II.

The odor generation pattern of biosolids is very important for their land use aspect. Novak et al., 2004 have reported that the degradation of biosolids show a very predictable pattern of VOSC production reaching a peak concentration between days 1 to 7 and then declining gradually. Kacker et al. [2011] studied the odor generation patterns for various digestion techniques and have reported that Cambi treatment reduces the odor significantly.

The odor generation pattern for the TRA sludge is shown in Figure 5-19. It can be seen that most of the samples reached their peak concentrations on the 2nd day of incubation and the odors died out by Day 4. All the TH systems showed a similar TVOSC generation pattern with comparable concentrations.

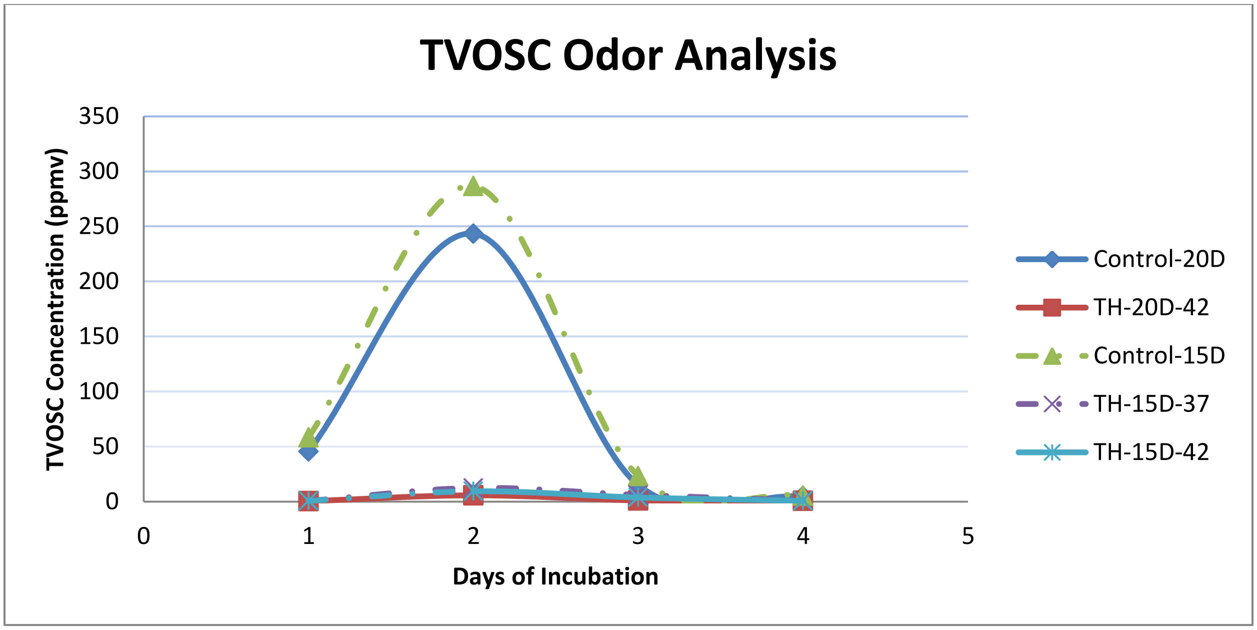

Figure 5-19: TVOSC generation pattern for samples from different anaerobic digesters

5.3 Summary

The results obtained from this study have revealed that the thermal hydrolysis pre-treatment has several advantages over the conventional anaerobic digestion systems. The analysis of these results show that the thermal hydrolysis process worked well during the first phase, i.e. for 20 days SRT. These data show that thermal hydrolysis (Cambi process) will increase VS reduction by 9-10%. Also, not much difference was observed in the performance of digesters at 37^{o} C and 42^{o} C which indicates that the operation of the digesters at 42^{o}C is acceptable. This will help reduce the cooling costs in the digesters. The reason for the good performance at 42^{o}C is that the sludge is a low ammonia product so no ammonia inhibition occurs. However, a significant depreciation in the performance of all the digesters was observed in phase II which can be attributed to the ammonia build-up issues in the digester. The odor potential of the thermally hydrolyzed sludge was observed to be low and the digested sludge demonstrated enhanced dewatering properties. The TH-20D-42 digester delivered a dewatered cake with 37% solids concentration. Thus, a product with low odor and superior dewatering characteristics can be expected.

Chapter 6

Conclusions

This research was conducted in order to evaluate the performance of thermal hydrolysis pre-treatment prior to anaerobic digestion of sludge and to compare its operation to a conventional mesophilic anaerobic digestion system. Various characteristics were measured to assess the advantages and disadvantages of TH systems over conventional anaerobic digestion. Several parameters such as VS destruction, COD removal, biogas production and odor generation were researched and analyzed to compare the efficiency of the two processes. The following conclusions were drawn from the results of these analyses.

- **The thermal hydrolysis pre-treatment system exhibited better performance as compared to the conventional anaerobic digestion system.**

The TH digesters showed ~10% higher solids destruction during phase I. A higher biogas production rate and a lower odor product were obtained from the TH system as compared to the control digester. The effluent sludge from the TH digesters also demonstrated higher dewatering characteristics with cake solids concentrations up to 37% as compared to ~25% obtained from the Control digesters.

- **There was little difference in the performance of TH digesters at 37^{o} C and 42^{o} C**

For both the phases, the digesters were operated at two different temperatures, 37^{o} C and 42^{o} C. This experiment was conducted in order to investigate the effect of temperature on the digester operation. It was observed that during both the phases the performance of the digesters at these two temperatures remained similar. This showed that the TH digesters can be operated at 42^{o} C and the cooling cost can be cut down.

- **The TH-20D (phase I) digesters performed better than TH-15D (phase II) digesters**

The study was conducted in two phases; the SRT was maintained at 20 days for phase I and 15 days for phase II. Degradation in the performance of the digesters was observed during phase II. There was little difference between the solids reduction in the Control and the TH digesters during this phase. This can be attributed to the ammonia build-up in the digesters during lower SRT conditions. The results obtained from this experiment revealed that the TH digesters worked efficiently at 20 days SRT and showed inferior performance at 15 days SRT.

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APPENDIX

Sludge Characterization

Sludge characterization is performed in order to better understand the sludge characteristics, to predict the sludge behavior during various conditions and to assess its treatability. As mentioned earlier, the efficiency of the TH pre-treatment is majorly dependent on the type of sludge and its physical and chemical properties. Therefore, it was necessary to run sludge characterization tests and analyze the sludge properties.

The TH feed sludge was thermally hydrolyzed in the “bomb” for a time duration of 1hr, 3hrs and 6hrs at 2 different temperatures of 170C and 190C. The thermally hydrolyzed sludge obtained after each experimental setup was collected and refrigerated for further testing. Two tests were performed for characterizing the TRA sludge: Solids tests and the Soluble COD experiments.

Solids

Solids (Method 2540-G) testing was conducted according to Standards Methods for the Examination of Water and Wastewater (APHA, 1999). The TS and VS concentrations comparison for the experiment have been summarized in figures below.

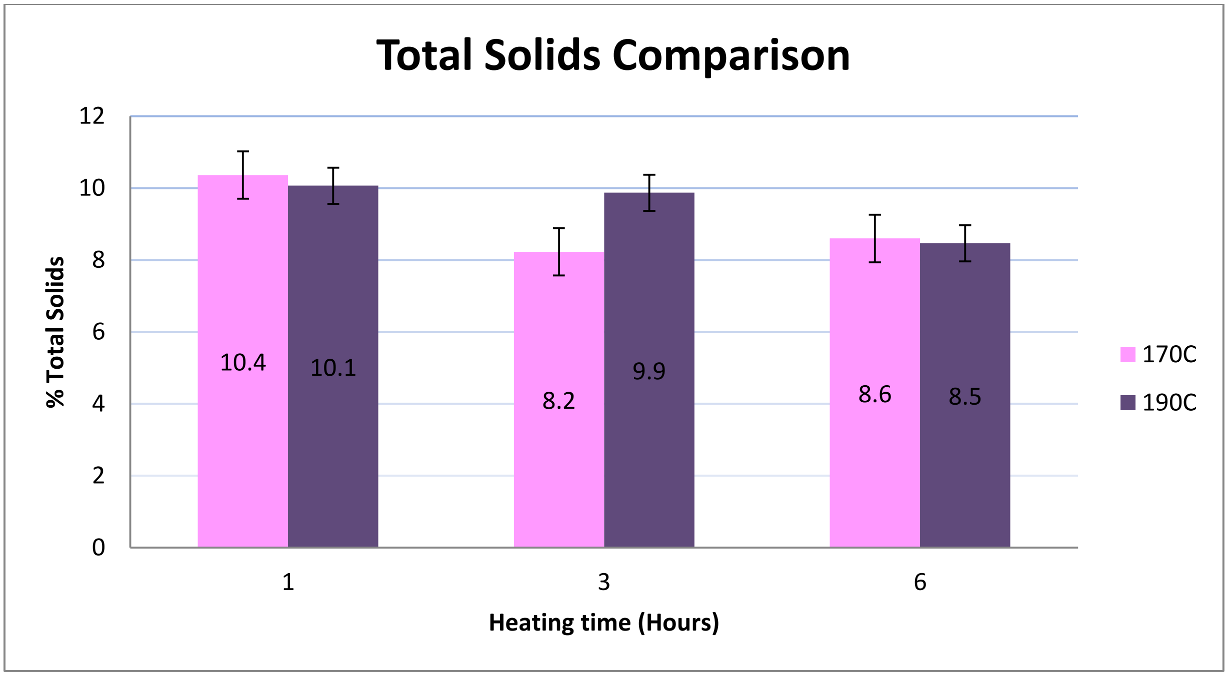

Figure 1: A comparison of total solids concentration in different samples

It can be seen that the TS concentration in the hydrolyzed sludge samples was found to be the least in the sample which was hydrolyzed at 170C for 3hrs duration. Therefore, the Total Solids reduction was the highest when the sample was hydrolyzed at 170C for 3hrs. A considerable difference was observed in the solids concentration of the 1hr and 6hr duration samples. The samples which were hydrolyzed for a longer time (6hr) had ~8.5% solids while the samples which were hydrolyzed for lesser time duration (1hr) had ~10% solids. It can thus be deduced that the solids destruction increased with increase in the time duration of hydrolysis. The hydrolysis temperature on the other hand was seen to have little effect on the solids content of the sample. The samples hydrolyzed at 170C and at 190C both showed similar solids concentrations.

The figure below shows the VS comparison for all the hydrolyzed samples.

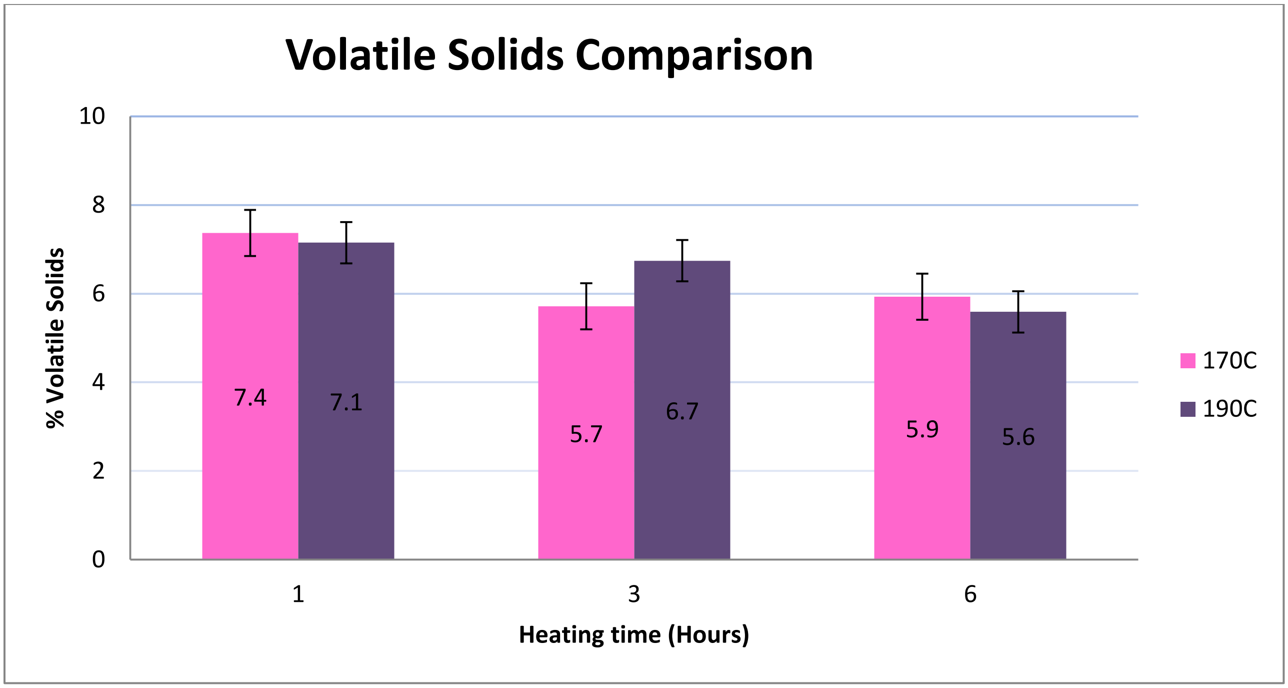

Figure 2: A comparison of volatile solids concentration in different samples

The VS data was consistent with the TS data. Similar VS concentration trends were observed in the hydrolyzed samples. VS destruction was the highest in the samples which were hydrolyzed at 170C for 3hrs.

Soluble COD

The total COD of wastewater is usually made up of biodegradable and non-biodegradable components. The biodegradable fraction consists of soluble, readily biodegradable organics and particulate, slowly biodegradable organics. The non-biodegradable fraction comprises of soluble and particulate organics. Soluble COD concentration is defined as the sum of inert soluble COD and readily biodegradable COD.

In order to prepare the samples for measuring their soluble COD content, the hydrolyzed samples were centrifuged for 10 min in order to separate the centrate. The centrate was collected from the samples and diluted (1:200) then acidified using concentrated H_2SO_4 to lower the pH to less than 2 before measuring COD. Acidifying the sample fixes the carbon. COD measurements were conducted by the closed reflux method (APHA 1999).

The Soluble COD concentrations for all the hydrolyzed samples have been summarized int the figure below.

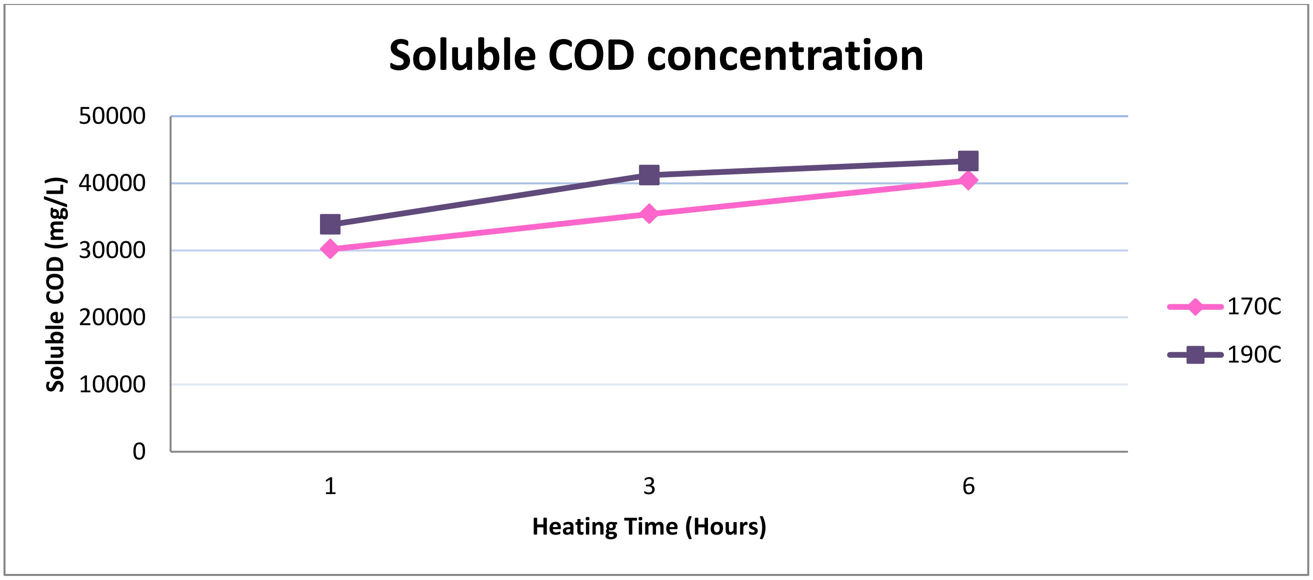

Figure 3: Soluble COD concentration in the samples

As shown in the plot, the soluble COD content of the sludge was seen to increase with temperature and the time duration of hydrolysis. The soluble COD concentration was found to be the least for the samples that were hydrolyzed for 1 hour at 170C and the concentration was the highest for the samples that were hydrolyzed for 6hrs at 190C.