Applicability of ultrasound conditioning of waste activated sludge to reduce foaming potential in the anaerobic digesters

by

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ABSTRACT

This study has primarily established ultrasound disintegration as one of the many methods for the pre-treatment of thickened waste activated solids (TWAS) from Denver, CO and Marshalltown, IA WWTPs ranged from 4% to 4.8% of total solid concentration to control foaming in mesophilic anaerobic reactors. A secondary objective of the study was to evaluate the performance of ultrasound-conditioned (sonicated) TWAS for minimizing the retention time and enhancing the destruction of volatile solids. The sonication duration was adjusted to achieve energy inputs equivalent to 1.25 and 1.95 kWs/g of TWAS. Three reactors; control, full-stream reactor that received 100% sonicated TWAS and part-stream reactor that received 30% sonicated TWAS, were operated at various solids retention times (SRTs) of 15, 10, and 6 days.

The effect of ultrasound disintegration on TWAS was demonstrated in terms of the increase in COD solubilization, the reduction of solids particle size as well as the largest increase in turbidity, and microscopic photographs shown the disintegration of the bacterial cells.

During the lower energy input of 1.25 kWs/g TWAS, the 100% TWAS sonicated unit was able to control foam for both Denver and Marshalltown feed solids effectively. While the reactor that received 100% sonicated Marshalltown TWAS showed the lowest tendency to foam, the reactor that received 100% sonicated Denver TWAS showed the greatest foaming potential during the higher energy input of 1.95 kWs/g TWAS.

Contrary to other reported studies, there was no significant difference in the performance of the three reactors with regard to volatile solids reduction (VSR). All three reactors achieved 47-53% VSR at 15 day HRT, 42-48% VSR at 10 day HRT, and 41-47% VSR at 6 day HRT. Sonication of TWAS increased the rate of substrate degradation, but did not improve the ultimate degree of degradation.

CHAPTER 1. GENERAL INTRODUCTION

Background

Anaerobic digestion is the most applied solids stabilization technology used to degrade complex organic substances in the absence of free or dissolved oxygen. The final products of anaerobic digestion are methane, carbon dioxide, trace gases and stabilized biosolids. However, anaerobic degradation is a slow process and large fermenter volumes are required due to the rate limiting step of biological sludge hydrolysis (Eastman and Ferguson, 1981).

Anaerobic degradation process occurs in three stages. These stages are hydrolysis, acid forming (acidogenesis and acetogenesis) and methanogenesis. Process proceeds efficiently if the reaction rates of all three stages are equal. If the first stage is inhibited, the second and third stages are limited and methane production decreases due to substrate deficiency from the first stage (Gerardi, 2003).

Although there are advantages associated with anaerobic digestion, such as volatile sludge reduction and biogas production, the production and accumulation of foam is one of common problems in many anaerobic digesters causing several operational issues. Foaming occurs as a result of gas bubbles entrapped in the liquid. Excessive foaming might cause operational and maintenance problems and inversion of digester solids profile, fouling of gas collection compressors and recirculating pipes, reducing effective digester volumes.

Foaming can be attributed to one or several of the following factors:

- Alkalinity increase

- Filamentous bulking in waste activated sludge (WAS)
- Carbon dioxide increase
- High WAS/Primary sludge ratios
- Fatty acids increase
- Heterogeneous nature of WAS and primary sludge (PS) combined streams being fed to digesters
- Excessive mixing
- Temperature fluctuations

Ultrasound disintegration, one of the new technologies for sludge treatment, promotes digestion of difficult-to-digest materials by accelerating the rate limiting hydrolysis step (Barber, 2003). This technology is used to accelerate the anaerobic digestion process, increase destruction of volatile solids and digester gas production, and reduce the volume of sludge for disposal (Eimco, 2004; Ultrasonus, 2000).

The primary objective of this research was to evaluate the effects of ultrasound conditioning of thickened TWAS to control foaming in a subsequent anaerobic digestion stage processing a mixture of primary and thickened secondary sludge produced from the treatment of municipal wastewater. A secondary objective was to compare the performance of ultrasound-conditioned WAS in terms of volatile solids reduction and biogas generation against unsonicated WAS in conventional mesophilic anaerobic digestion of combined sludge at different solids retention times. Pilot-scale studies were sponsored by Black & Veatch in collaboration with Metro Wastewater Reclamation District of Denver (MWRD) to evaluate the applicability of this technology for controlling foaming of mesophilic anaerobic digesters at Metro's Central Treatment Plant.

Thesis Organization

The thesis is organized into a total of five chapters. Chapter 1 is a general introduction including a brief description of the research. Chapter 2 is the literature review encompassing information of the previous studies providing a fundamental basis for the study presented. Chapter 3 is a paper entitled "Applicability of Ultrasound Conditioning of Waste Activated Sludge to Reduce Foaming Potential in the Anaerobic Digestion of Combined Sludges. This paper evaluates the effects of ultrasound conditioning of thickened waste activated sludge (TWAS) in controlling foam in mesophilic anaerobic digesters. Chapter 4 is a manuscript titled "The Effects of Ultrasound Disintegration on Anaerobic Digestion at Different Solids Retention Times". This paper compares the performance of ultrasound-conditioned TWAS against unsonicated TWAS in terms of volatile solids reduction and biogas production maintained at different retention times in conventional mesophilic digesters. Chapter 5 provides the engineering significance of the study including some recommendations for future study. The references for each chapter are listed at the end of the thesis.

CHAPTER 2. LITERATURE REVIEW

Anaerobic Digestion

Anaerobic digestion which is a biological decomposition of organic matter in the absence of molecular oxygen can be examined as one of the standard technologies for stabilizing wastes. The products of anaerobic digestion are gases principally composed of methane (CH_4) and carbon dioxide (CO_2) and the stabilized biosolids.

Anaerobic degradation may either occur in nature spontaneously or in a controlled environment such as a biogas plant. Depending on the waste feedstock and the system design, biogas typically consists of 55 to 80% methane; the remaining composition is primarily carbon dioxide, trace gases such as hydrogen sulfide, nitrogen and water.

The process of anaerobic digestion occurs in three steps. Hydrolysis, the first step, is the solubilization of complex organic matter. Complex organics such as carbohydrates, proteins, and lipids are broken down to usable-sized molecules such as sugars, amino acids and peptides (McCarty and Smith, 1986). Hydrolysis has been identified by many researchers to be a rate limiting step of the anaerobic digestion process (Chan and Pearson, 1970; Chyi and Dague, 1997; Eastman and Ferguson, 1981; Ghosh *et al*., 1975; Pheffer, 1974; Pavlostathis and Giraldo-Gomez, 1991).

The second step, acidogenesis and acetogenesis, is the conversion of solubilized matter to organic acids. Acidogenic bacteria degrade the simpler soluble organics to long chain fatty acids such as propionate, butyrate. This step is known as acidogenesis. The long chain fatty acids are then converted to acetate, hydrogen, and carbon dioxide. This process is named acetogenesis.

And finally, acetate and hydrogen are converted to methane and carbon dioxide by methanogenic bacteria. The removal of organics is completed in this final step known as methanogenesis. The steps during anaerobic digestion process and their particular products are shown in Figure 2.1.

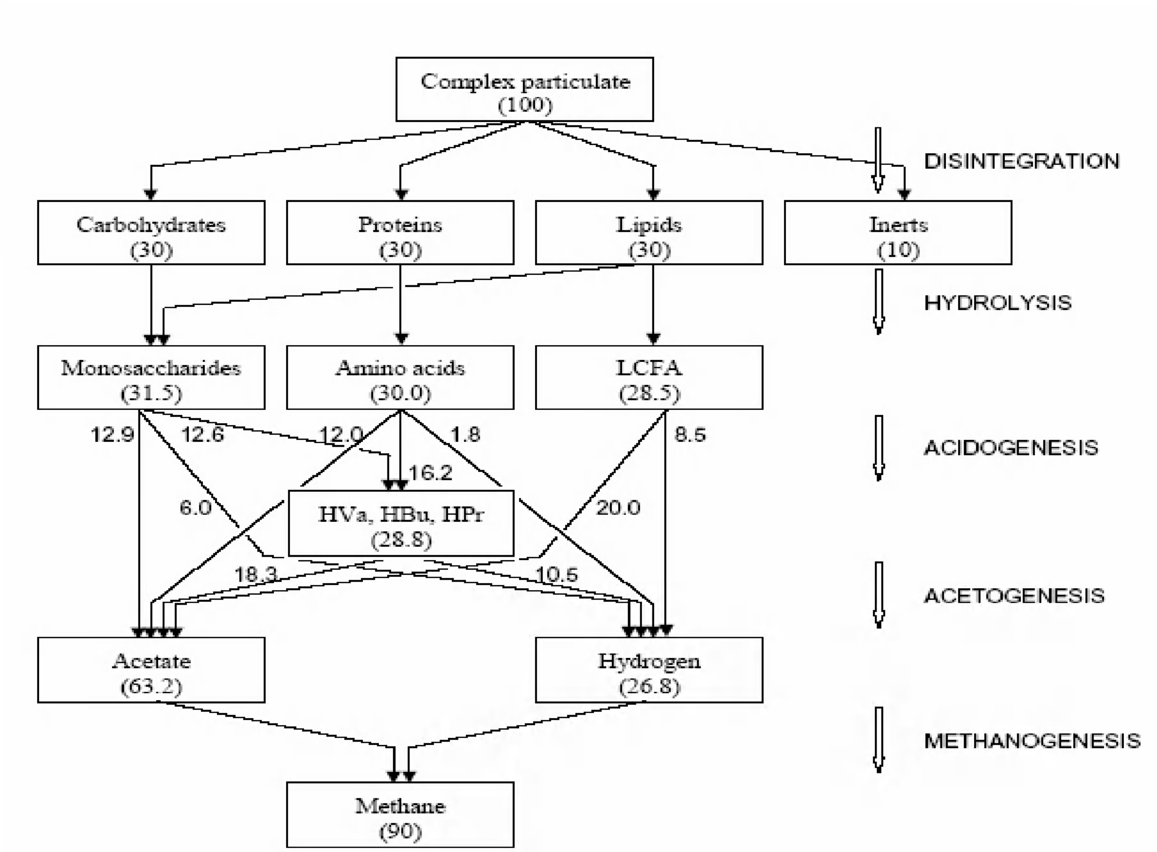

Figure 2.1: Flow-Diagram for the Anaerobic Degradation of a Composite Particulate Material, as Implemented in Anaerobic Digestion Model #1 (ADM1) (Batstone *et al.*, 2002). Valerate (HVa), Butyrate (HBu), and Propionate (HPr) are grouped for simplicity. Numbers in parenthesis indicate COD fractions.

The rate of digestion is affected by process temperature and is usually maintained in the mesophillic range of 30 to 38 °C (95 to 105 °F). It is also possible to operate in the thermophillic range of 50 to 57 °C (122to 136 °F) (Poulsen, 2003). The thermophilic temperatures are preferred due to the two reasons. First, a higher loading rate of organic materials can be processed. Second, higher temperatures increase the destruction rate of pathogens in raw wastes (Adhikari, 2004).

Operational Conditions

Process control of anaerobic digesters is not usually easy due to numerous operational conditions, and changes in one condition may directly affect others. Therefore, optimum operational conditions should be maintained and sustained for satisfactory rates of solids destruction and methane production.

<u>Start- Up</u>

Effective digester startup is dependent upon the quality and quantity of inoculum (Chynoweth & Pullammanappallil, 1996). To seed an anaerobic digester, the inoculum-to-feed ratio (VS basis) is typically greater than 10 in conventional CSTR digesters. During start-up, loading to digesters should maintain slowly, and the control of pH and alkalinity should be essential because more rapid growth of acid formers than methane formers may lead to accumulation of organic acids and consequent pH reduction. The digester start-up pH should be sustained within the optimum range of 6.8-7.2.

Solids Retention Time and Hydraulic Retention Time

There are two significant retention times in an anaerobic digester:

- Solids retention times (SRT)
- Hydraulic retention time (HRT)

The solids retention time (SRT) is the average time that solids (microorganisms) are in the anaerobic digester. A minimum SRT for an anaerobic digestion system is essential to ensure the least time required for necessary microorganisms to regenerate at the same rate as they are wasted to prevent washout. Critical SRT is different for various constituent bacteria groups. While the lipid-metabolizing bacteria are the slowest growing group and, therefore, need a longer SRT, a shorter SRT is required for the cellulose-metabolizing bacteria (WEF, 1995). Typical SRTs for high-rate mesophilic digesters are longer than 10 days. Retention times less than 10 days are not recommended for anaerobic digesters due to the washout of methane-forming bacteria which is the slowest-growing bacteria consortia in the anaerobic digestion process (Gerardi, 2004).

The hydraulic retention time (HRT) is defined as the average time that the substrate (wastewater or solids) remains in the anaerobic digester. It is calculated as the digester working volume divide by the mean volume flowrate. The proper HRT for an anaerobic digestion system is dependent on the degradability of the composition of substrate treated. The lower degradation rate the substrate is required the longer HRT. The rate of degradation of the main organic compounds increase in the following order:

- Cellulose

- Hemicellulose
- Proteins
- Fat
- Carbohydrates (Speece, 1996).

The SRT and the HRT are the same in completely mixed digesters with no solids recycle.

Loading Rate

The loading rate could be defined as the amount of organic matter fed into the digester per unit of working volume per unit of time. It can be calculated from the concentration in the feed divided by the hydraulic retention time (Chynoweth & Pullammanappallil, 1996). Loading rate is usually expressed in terms of volatile solids (VS) or chemical oxygen demand (COD) (kg VS (COD)/m^3/day or lb VS (COD)/ft^3/day).

Typical designed and recommended VS loading rates for high-rate mesophilic anaerobic digesters with mixing and heating are 1.6-4.8 kg VS/m^3/day (Tchobanoglous and Burton, 1991). Excessively low volatile solids loading rates can cause some difficult to operate because the average solid value in the digesters is diluted. Diluted feed may result in the following adverse effects in digester operation:

- Reduced HRT
- Reduced VS destruction
- Reduced methane generation
- Reduced alkalinity

- Increased volumes of digested biosolids and supernatant
- Increased heating requirements

Temperature

Biological activity is directly affected with changing of temperature due to the impact of temperature on enzymatic activity or reactions. Variations in temperature of even a few degrees impact all biological activity including the inhibition of some anaerobic bacteria, especially methane forming bacteria. Therefore, increases in temperature result in more enzymatic activity. Most applications of fermentation process have been performed under either ambient (15 to 25^{o}C), mesophilic (30 to 40^{o}C), or thermophilic (50 to 60^{o}C) temperatures (Chynoweth *et al.*, 1998).

Typically, most anaerobic digesters treating wastewater solids are operated at mesophilic temperatures ranging from 34 to 38 ^{o}C. Thermophilic anaerobic digestion, a faster degradation at higher temperature, is applied at thermophilic temperatures of 55 to 60 ^{o}C. Thermophilic anaerobic digestion is mostly practiced in circumstances when the reduced reactor sizes and the effective pathogen kill justify higher energy requirements and extra effort to ensure stable performance.

pH, Volatile Acids, and Alkalinity

pH, organic acids, and alkalinity are related parameters that influence digester performance (McCarty, 1964). pH extensively influences to digester performance or enzymatic activity. The methane-forming bacteria are the most sensitive consortia to pH compared with other bacterial consortia in the anaerobic digestion process. Therefore, the optimum pH range is determined based on the suitable pH values for the growth of methanogenic bacteria. Optimum pH in a properly operating anaerobic digester is usually within a pH range of 6.8 to 7.2.

Sufficient alkalinity which serves as a buffer is necessary to prevent rapid change in pH. A decrease in alkalinity below the normal operating level can be caused by an accumulation of organic acids due to the failure of the activities of methanogenic bacteria, depletion of buffer and depression of pH. The major alkalis contributing to alkalinity are ammonia and bicarbonate.

Volatile acids are intermediate products of the anaerobic digestion process. The accumulation of volatile acids is an indicator of process instability. Therefore, the volatile acid consumption rate must be equivalent to the production rate to avoid the accumulation of volatile acids. The bicarbonate alkalinity in the digester neutralizes the excess volatile acids to sustain the pH in the optimum range.

The volatile acids/alkalinity ratio is a good indicator for process condition. It is generally accepted that the normal volatile acid-to-alkalinity ratio is 0.1. Increases to ratios of 0.5

indicate the onset of failure and a ratio of 1.0 or greater is associated with total failure (Chynoweth *et al.*, 1998).

Nutrients

The major macronutrients, nitrogen and phosphorus, are required for anaerobic digestion. These elements are directly related to microbial growth requirements in anaerobic digesters. It is generally assumed that the nitrogen and phosphorus requirements for cell growth are 12% and 2%, respectively. These ratios are based on the common empirical formula for cellular material, $C_5H_7O_2NP_{0.06}$ (Speece, 1997).

The need for several micronutrients is essential to ensure both proper degradation of substrate and efficient operation of the digester. Requirements for several micronutrients have been identified, including iron, copper, manganese, zinc, molybdenum, cobalt, nickel, selenium, and vanadium (Speece, 1997). Other nutrients such as sodium, potassium, calcium, magnesium, chlorine, and sulfur are needed in intermediate concentrations (Speece, 1997; Wilkie *et al.*, 1986). Limitations of these micronutrients have been demonstrated in reactors where the analytical procedures failed to distinguish between available and sequestered forms (Jewell *et al.*, 1993).

Mixing and Feeding Mode

Adequate mixing is necessary to enhance the digestion process by disturbing bacteria, substrate, and nutrients throughout the digester. Mixing is also applied:

- To prevent temperature variations
- To minimize the settling of grit
- To minimize any toxic materials entering the digester with rapid dispersion
- To break up floating scum and foam buildup

Proper mixing can be accomplished by mechanical stirring, liquid recycle, or gas recycle. It is not recommended rapid and continuous mixing due to washing out methane-forming bacteria in the effluent.

It is generally preferred as continuous or nearly continuous feeding for the operation of anaerobic digestion systems. Feeding intermittently may cause to shock loading to the digesters because the methanogenic bacteria are very sensitive to the variation of the substrate concentration.

Inhibition (Toxicity)

Anaerobic digestion process is sensitive to a variety of organic and inorganic wastes which may cause toxicity that disturbs a metabolic process or kills the bacteria. The toxic effect of an inhibitory compound is dependent upon its concentration and the ability of the bacteria to acclimate to its effects (Chynoweth *et al.*, 1998). The inhibitory concentration depends upon different variables, including pH, HRT, temperature, and the ratio of the toxic substance concentration to the bacterial mass concentration.

Inhibition in anaerobic digesters is reflected by:

- The accumulation of volatile acids

- The disappearance of methane
- Decreases in alkalinity and pH
- High ammonia levels.

There are several groups of inhibitors creating toxicity in the following:

- Alternate electron acceptors (oxygen, nitrate, and sulfate)
- Ammonia
- Hydrogen sulfide
- Heavy metals (cobalt, copper, iron, nickel, zinc etc.)
- Halogenated hydrocarbons
- Volatile organic acids
- Alkaline cations (calcium, magnesium, potassium, and sodium)
- Cyanide
- Formaldehyde and phenolic wastes
- Recalcitrant compounds (Aliphatic hydrocarbons, chlorinated biphenyls)

Methanogenic populations, the most sensitive consortia to toxicity, can tolerate higher concentrations by acclimating (Speece, 1996).

Ultrasound Technology

The Mechanism of Ultrasound

The term ultrasound is used to define sound energy in a frequency range of 20-100 kHz which is above human hearing. It is approximately divided into three main regions: power ultrasound (20-100 kHz), high frequency ultrasound (100 kHz-1 MHz), and diagnostic ultrasound (1-10 MHz) (Clark and Nujjoo, 2000).

High-power ultrasound is usually generated by a transducer, converting electrical energy to mechanical energy with high frequency vibrations, and is delivered into a liquid by a horn or probe. In high-power ultrasound applications, ultrasound waves on a liquid cause compression (negative) and rarefaction (positive) pressures. These alternating cycles of compression and rarefaction can produce a phenomenon known as 'cavitation'.
Cavitation is the formation, growth and collapse through implosion of microbubbles. It occurs due to the stresses induced in the liquid by the passing of a sound wave through the liquid (Mason and Lorimer, 1988).

The minimum amount of energy, cavitation threshold, is required to initiate cavitation, and only the energy above the threshold contributes to the formation of cavitation gas bubble. These small gas bubbles created above a certain intensity threshold absorb energy from sound waves, and grow before violently collapsing. The violent collapse produces very powerful hydromechanical shear force in the liquid surrounding the bubble. The temperature and pressure inside the collapsing cavitation bubbles may rise up to about 5,000 ^{o}K and several hundred atmospheres (Tiehm *et al.*, 2001). Finally, the cavity no longer absorbs energy as efficiently, and leads to implosion of the cavity.

The cavitation is influenced by a number of the following factors:

- Frequency of ultrasonic vibration
- Ultrasonic intensity
- External temperature and pressure
- Surface tension
- Duration of cavitation
- Temperature of the liquid
- Viscosity (Mason and Lorimer, (1988); Clark and Nujjoo, (2000).

Ultrasound Disintegration

Cell disintegration or lysis is usually the limiting factor in the rate of solids digestion in conventional anaerobic digestion process. This is the case for secondary solids in particular, such as thickened waste activated solids (TWAS). TWAS is mainly formed the substances of bacteria cells and difficult to digest anaerobically. Thus, only a limited portion of TWAS without reducing the efficiency of the digester might be fed to an anaerobic digester.

In order to improve anaerobic digestion process, mechanical disintegration was investigated as an innovative process step in solids treatment. Ultrasound pretreatment, one of the mechanical disintegration methods, has been well applied for many years in solids stabilization. The basic principal is based on the destruction of microbial cells to extract intracellular material, (Harrison, 1991). Therefore, hydrolysis the rate limiting step of solids treatment is promoted.

The digestion of raw solids with ultrasound positively affects the anaerobic digestion as a result of solids dispersion and disruption of cellular material. The component structure of the solids changes, and allows the water to be separated more easily. Ultrasound attacks the cell walls of bacteria as well, and the breakdown of organic material is accelerated.

Ultrasound treatment of raw solids to improve anaerobic digestion process can be applied by two main hypotheses: ‘Full-stream’, and ‘Part-stream’ hypotheses. Full-stream treatment is based on that the entire volume of solids is subjected to ultrasound treatment to maximize the release of cell lysis. Part-stream treatment is conducted to evaluate the concept that full-stream treatment may not be required to realize the benefits of ultrasound pre-treatment, and applied to only a fraction of solids stream which provides maximum benefits.

Ultrasound treatment of raw solids may achieve the following effects in anaerobic digestion process:

- Increased digestion rate
- Increased volatile solids reduction
- Increased gas production
- Decreased solids volume for disposal and handling
- Improvement of dewatering
- Destruction of filamentous bacteria
- Reduced polymer requirements for dewatering of the biosolids
- Allowing shorter retention time and smaller digester volume

- Improvement of C/N ratio in raw solids (Tiehm *et al,* 2001; Barber, 2003; Eimco Sonolyzer, 2004; Ultrasonus, 2003).

Tiehm *et al.* (2001) studied ultrasonic pretreatment of raw solids as an anaerobic digestion enhancement. Cell lysis by ultrasonic disintegration was yielded better anaerobic digestion. >6,000 mg/L soluble COD increase with 96 seconds of ultrasonic treatment was found. However, they never discuss the raw supernatant COD, so the percentage increase may be minimal. Ultrasonic pretreatment improved VS destruction from 45.8% to 50.3% at the 22 day HRT while other VS destructions were: 16 day – 49.3%; 12 day – 47.3%; and 8 day – 44.3%.

Barber (2003) reported that analysis shown that hydrolysis rates on several full-scale solids treatment works increased by 25-50% after the installation of ultrasound. He also reported that HRT may be reduced by approximately 30% for similar performance if organic loading rates are kept constant.

Eimco Sonolyzer (2004) reported that ultrasound pretreatment achieved volatile solids destruction from 42% to 54% with 30% biogas production increase at the 18 day HRT at the municipal full-scale treatment plant of Bamberg, Germany.

CHAPTER 3. APPLICABILITY OF ULTRASOUND CONDITIONING OF WASTE ACTIVATED SOLIDS TO REDUCE FOAMING POTENTIAL IN MESOPHILIC ANAEROBIC DIGESTERS

A paper to be submitted to *The Journal of Water Resources*

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Abstract

Many anaerobic digestion facilities processing waste activated solids (WAS) or combined primary and secondary solids with a high fraction of WAS have reported operational problems associated with excessive foaming of the digester contents. The pre-treatment of thickened waste activated solids (TWAS) ranged from 4% to 4.8% of total solids concentration by ultrasonic disintegration was primarily studied for controlling foam and secondarily improving performance of mesophilic anaerobic reactors treating combined primary and secondary solids. The sonication duration was adjusted to achieve energy inputs equivalent to 1.25 and 1.95 kWs/g of TWAS. During the lower energy input of 1.25 kWs/g TWAS, the 100% TWAS sonicated unit was able to control foam for both Denver and Marshalltown raw solids effectively. While the reactor that received 100% sonicated TWAS showed the lowest tendency to foam during the study with Marshalltown raw solids, the reactor that received 100% sonicated TWAS showed the greatest foaming potential during the higher energy input of 1.95 kWs/g TWAS with Denver raw solids. Contrary to other

reported studies, there was no significant difference in the performance of the three reactors as full stream reactor that received 100% sonicated TWAS, part stream reactor that received 30% sonicated TWAS and control reactor with regard to volatile solids reduction (VSR). Sonication of TWAS increased the rate of substrate degradation, but did not improve the ultimate degree of degradation.

Keywords: Ultrasound, pre-treatment, disintegration, full-stream, part-stream, mesophilic, digestion, foaming.

Introduction

Anaerobic digestion is the most widely adopted technology for stabilization of wastewater solids in municipal wastewater treatment plants. The production of biogas, reduction of volatile solids, and improvement of dewatering of the digested solids are important features of anaerobic digestion. However, one of the disadvantages of anaerobic digestion is that it is a slow process. The hydrolysis step of anaerobic digestion has been identified as the rate limiting step (Eastmen and Ferguson, 1981; Shimuzu *et al.*, 1993). Therefore, long retention times and large digester volumes are required for anaerobic digestion due to the rate limiting step of hydrolysis (Tiehm *et al.*, 2000).

In addition, many anaerobic digestion facilities processing waste activated solids (WAS) or combined primary and secondary solids with a high fraction of WAS have reported operational problems associated with excessive foaming of their contents. Excessive foaming might cause a decrease in effective digester volumes, negatively impacting biogas handling systems, and damaging digester covers (Moen *et al.,* 2002). Foaming incidents have been related to filamentous bulking of WAS, low PS/WAS ratios, heterogeneous nature of WAS and PS combined streams fed to digesters.

The anaerobic digestion process has been studied in the past years and various methods have been explored for process improvement. The biodegradability of wastewater solids can be improved by mechanical disintegration (Muller, 1998), thermal pretreatment (Stuckey and McCarty, 1984; Li and Noike, 1992), addition of enzymes (Knapp and Howell, 1978), ozonation (Yasui and Shibata, 1994), chemical solubilization by acidification (Gaudy *et al.*, 1971; Woodard, 1994) or alkaline hydrolysis (Mukherjee and Levine, 1992).

One of the mechanical solids disintegration methods, ultrasound treatment involves the application of high intensity sound waves into the solids stream to promote the hydrolysis step during digestion. Ultrasound treatment is based on the disintegration of bacterial cells, thereby extracting the intracellular materials and making them amenable for digestion (Harrison, 1991). The difficult-to-degrade organic substances are disintegrated into smaller readily biodegradable fractions. The increased concentration of biodegradable material improves both volatile solids reduction and biogas production, decreasing quantities of stabilized biosolids for disposal. Many of these installations have also reported observing a decrease in occurrences of foaming incidents as a result of ultrasound conditioning.

While studies on ultrasound as a pretreatment disintegration method have focused primarily on the improvements in anaerobic digester performance (Brown *et al.,* 2003), controlling digester foaming has not yet been fully studied. Therefore, the purpose of this study was to evaluate the effects of ultrasound conditioning of thickened waste activated solids (TWAS) to control foaming in mesophilic anaerobic digesters. A secondary objective of the study was to compare the performance of ultrasound conditioned (sonicated) TWAS in terms of volatile solids reduction and biogas production against unsonicated TWAS during conventional mesophilic digestion of combined solids.

Material and Methods

The pilot-scale studies were performed in three 30-liter laboratory-scale reactors, each with an operating volume of 18 Liters. The reactors were maintained in a constant temperature room at 36-37 °C. The temperature of the biosolids was monitored at regular intervals to ensure operation at 36-37 °C. A schematic of the reactor setup is shown in Figure 3.1.

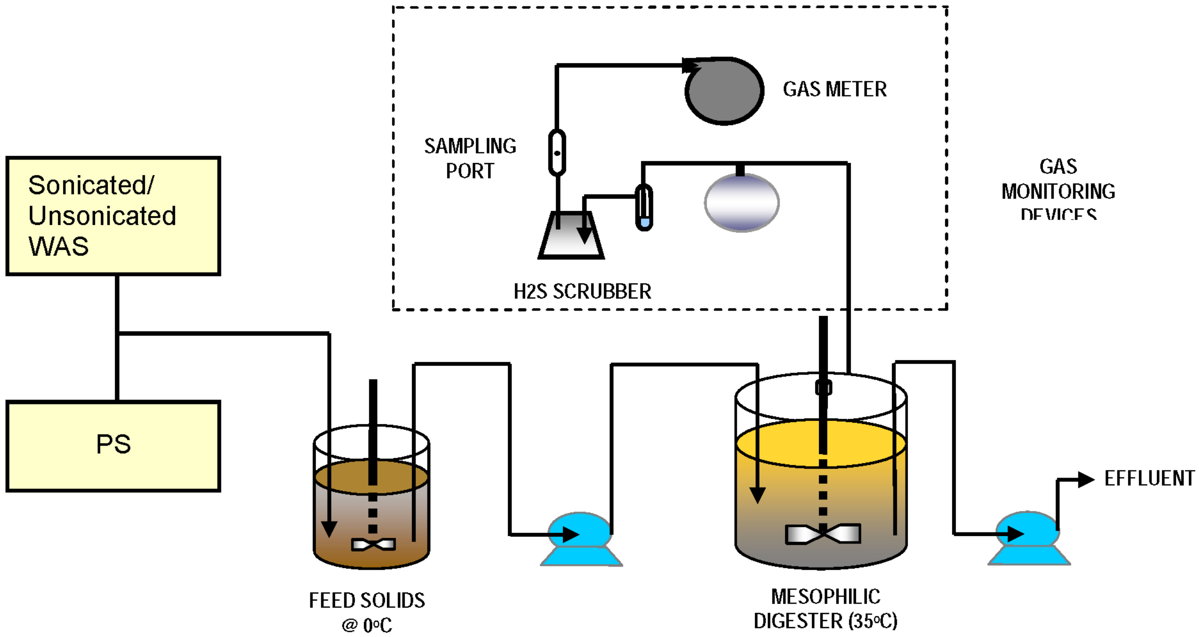

Figure 3.1. Schematic of the Reactor Setup

Two of three pilot reactors were operated as test units and were fed sonicated TWAS combined with PS. One of these test reactors was operated based the sonication of 100% of the TWAS (full-stream treatment), while the other was operated with 30% by volume of sonicated TWAS (part-stream treatment). The feed to the second test reactor consisted of 30% sonicated TWAS and 70% unsonicated TWAS, which were then combined with PS. Part stream sonication tests were conducted to evaluate the hypothesis that full stream treatment may not be required to accomplish the benefits of ultrasound pre-treatment (Friedrich, 2002). The control reactor was operated with a mixture of PS and unsonicated TWAS. All three reactors were fed a 40:60 blend of PS:TWAS on a dry weight basis.

Solids from two different sources were examined in order to evaluate the effect of ultrasound pre-treatment during the study. The feed solids for the experimental study were obtained from Denver Metropolitan Wastewater Reclamation District (MWRD), Colorado and Marshalltown Wastewater Treatment Plant (WWTP), Iowa on a weekly basis. The feed solids from Denver MWRD were shipped overnight in ice-packed coolers to minimize degradation. The solids were kept refrigerated at 0 °C prior to feeding to maintain consistent feed characteristics. The average raw PS and TWAS characteristics of both Denver and Marshalltown solids for the two runs are summarized in Table 3.1A and 3.1B, respectively.

Table 3.1A. Primary Solids Characteristics

Parameter	**Run 1**		**Run 2**	
	Denver	Marshalltown	Denver	Marshalltown
Total Solids (%)	5.01	6.08	5.27	5.92
Volatile Solids (%)	4.20	4.59	4.39	4.38
VFA (mg/L as Ac)	1,190	1,000	1,870	1,200
Alkalinity (mg/L as CaCO3)	1,000	1,300	1,050	1,450
Average pH	5.60	5.75	5.44	5.85
Soluble COD (mg/L)	--	2,850	2,340	1,790

Number of Samples- Run 1: n=12, Run 2: n=8

Table 3.1B. Thickened Waste Activated Solids Characteristics

Parameter	Run 1		Run 2	
	Denver	Marshalltown	Denver	Marshalltown
Total Solids (%)	4.04	4.25	4.21	4.81
Volatile Solids (%)	3.36	3.09	3.51	3.65
VFA (mg/L as Ac)	790	850	1,310	880
Alkalinity (mg/L as CaCO3)	1,475	1,600	1,470	1,670
Average pH	6.35	6.48	6.37	6.53
Soluble COD (mg/L)	1,635	230	1,850	270

Number of Samples- Run 1: n=12, Run 2: n=8

The reactors were operated at a 15-day solids retention time. All three reactors were operated in a semi-continuous mode, feeding and decanting at 6 hour intervals. The contents of the reactors were mixed intermittently during digestion.

TWAS sonication was performed on a daily basis, and accomplished with the ultrasound equipment provided by Etrema Products Inc., Ames, IA. It consisted of one 3 kW, 20 kHz transducer, with an air-cooled refrigeration system. Solids circulation was accomplished by continuous through the flow cell using a solids pump to mix of solids during sonication. The frequency of ultrasound and the sonication power were maintained at 20 kHz and 1.5 kW, respectively.

Typically, the soluble COD concentration in the solid increases up to a certain critical value during sonication. Beyond this critical value, soluble COD concentration shows no further increase even at higher energy inputs. Therefore, the key during the study was to adjust the residence time of the solids in the sonication chamber so as to maintain the soluble COD concentrations in the treated solids below the critical value to avoid energy wastage. Therefore, the energy input required to achieve an appreciable solubilization of COD without exceeding the maximum value was determined by trial and error. During this study, the sonication duration was adjusted to achieve energy inputs equivalent to 1.25 and 1.95 kWs/g of TWAS. For Run 1, the sonication duration was adjusted to achieve an energy input equivalent to 1.25 kWs/ g TWAS. The results of Run 1 did not show significant difference in the secondary performance parameters-volatile solids reduction and biogas production-among the three digesters. It was speculated that COD release achieved during Run 1 might not have been sufficient to cause a noticeable difference in performance among the digesters. Therefore, the energy input for Run 2 was increased to 1.95 kWs/g TWAS. The sonication time was varied to achieve the desired energy input (kWs/g), depending upon the TWAS concentration. The system setup is shown in Figure 3.2.

The pilot-scale studies were conducted for fourteen months. In the daily operation of the pilot reactors, foam accumulation in the reactors, pH, digestion temperature, and biogas production were monitored. Other parameters like total solids (TS) and volatile solids (VS), biogas composition, volatile fatty acids (VFA), alkalinity, total chemical oxygen demand (TCOD), and soluble chemical oxygen demand (SCOD) were monitored weekly. The turbidity of solid samples was measured after centrifugation with a nephelometer. Aqueous phase supernatants were obtained by centrifugation (30 minute at 40,000 rcf) followed by filtration through a 0.5 μ membrane filter to measure soluble COD (SCOD) and. Microscopic photography and turbidity tests were conducted for TWAS samples before and after sonication. More intensive testing was performed over a week after reaching each steady-state condition. Steady state condition was assumed after three retention times (3×15 days).

The degree of solids disintegration was defined as increase in SCOD in the supernatant due to ultrasound treatment. For reference, the maximum possible solids disintegration was determined by chemical disintegration, by incubating a fixed volume of solids with 0.5 N sodium hydroxide for 22 h at 20 °C (Tiehm *et al.*, 2001). The degree of disintegration of TWAS was calculated as the ratio of SCOD-increase by sonication to the SCOD-increase by the chemical disintegration.

Figure 3.2. Ultrasound System Setup

Results and Discussion

<u>Solids Disintegration</u>

The effect of ultrasonic disintegration was measured in terms of the increase in COD solubilization. Figure 3.3 shows the increase in soluble COD concentration after sonication for solids from the two sources.

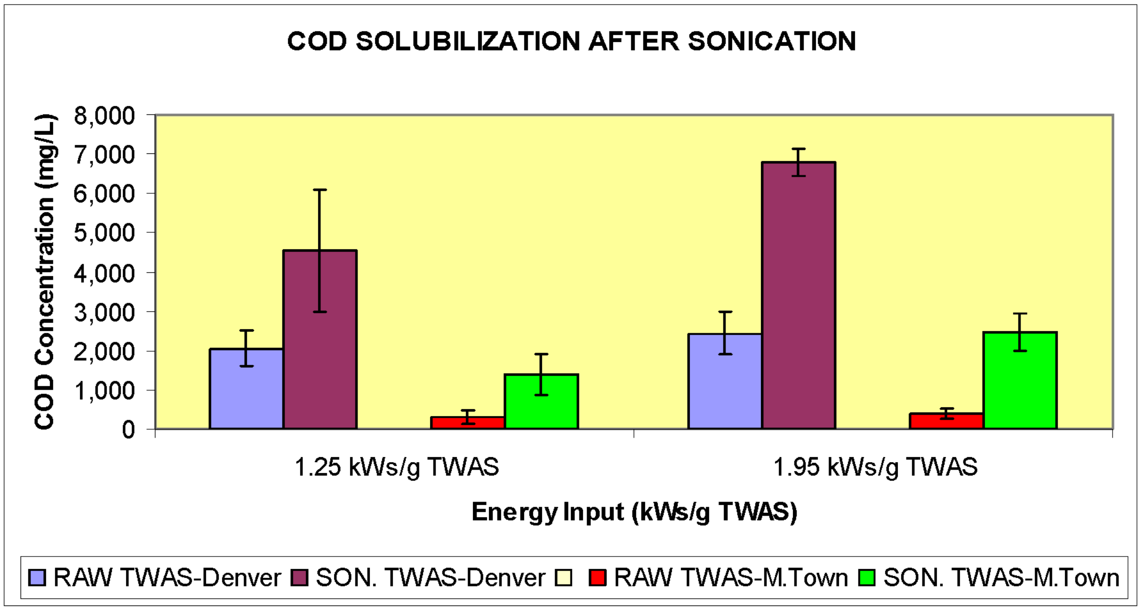

Figure 3.3. Variation in SCOD After Sonication

The average increase in SCOD concentration after sonication for the solids from Denver and Marshalltown was approximately 120% and 440% at the energy input of 1.25 kWs/g TWAS, and 190% and 550% at the energy input of 1.95 kWs/g TWAS, respectively. The significant increase in SCOD concentration of Marshalltown solids was probably due to lower COD of raw solids compared to Denver solids.

Solids disintegration was studied to compare the increase of COD in the solids supernatant due to ultrasound treatment. The maximum possible COD solubilization was determined by chemical disintegration of TWAS, a treating volume of raw solid with 0.5 N sodium hydroxide.

Figure 3.4 presents the degree of raw solids disintegration as a function of the specific energy input. The degree of disintegration was calculated as the ratio of COD-increase by sonication of the COD-increase by the chemical disintegration.

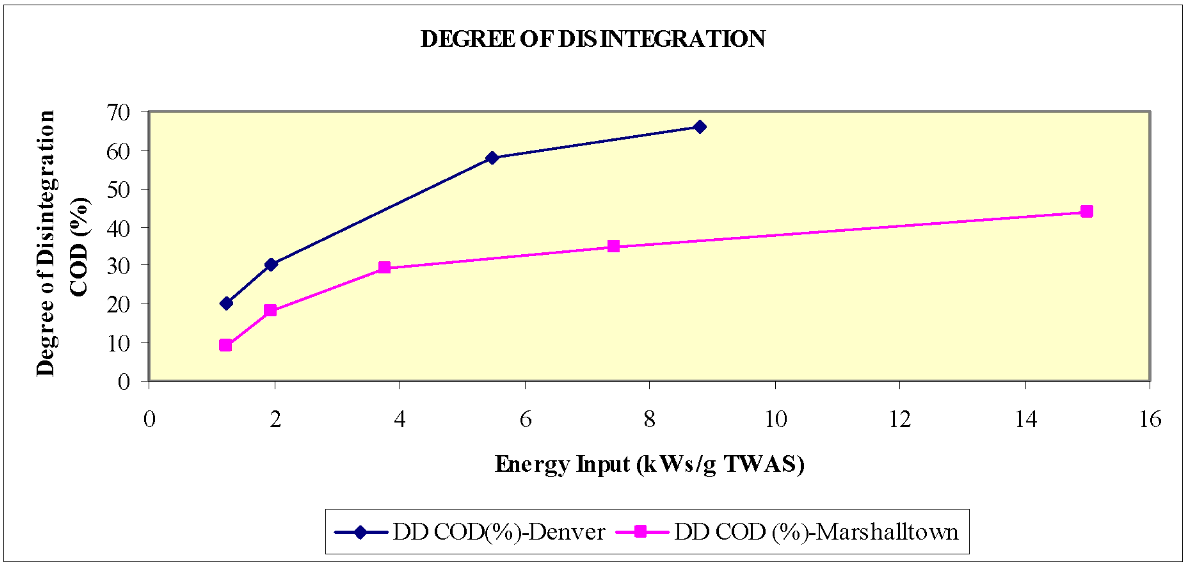

Figure 3.4. Degree of Raw Solids Disintegration at Different Sonication Durations

Degrees of disintegration of 20% and 9% for the solids from Denver and Marshalltown were achieved at energy inputs of 1.25 kWs/g TWAS and 30% and 18% at energy inputs of 1.95 kWs/g TWAS, respectively. It was found that maximum degree of disintegration achievable by sonication of TWAS with Denver MWRD was approximately 65-70%, and 40-45% with Marshalltown WWTP, irrespective of the sonication duration. The maximum degree of disintegration achieved by chemical treatment was 18,350 mg/L for Denver solids, and 11,290 mg/L for Marshalltown solids, respectively. The lower degree of disintegration for

Marshalltown solids could be explained due to lower volatile portion of TWAS (73% VS of TS) in comparison of Denver solids (83% VS of TS).

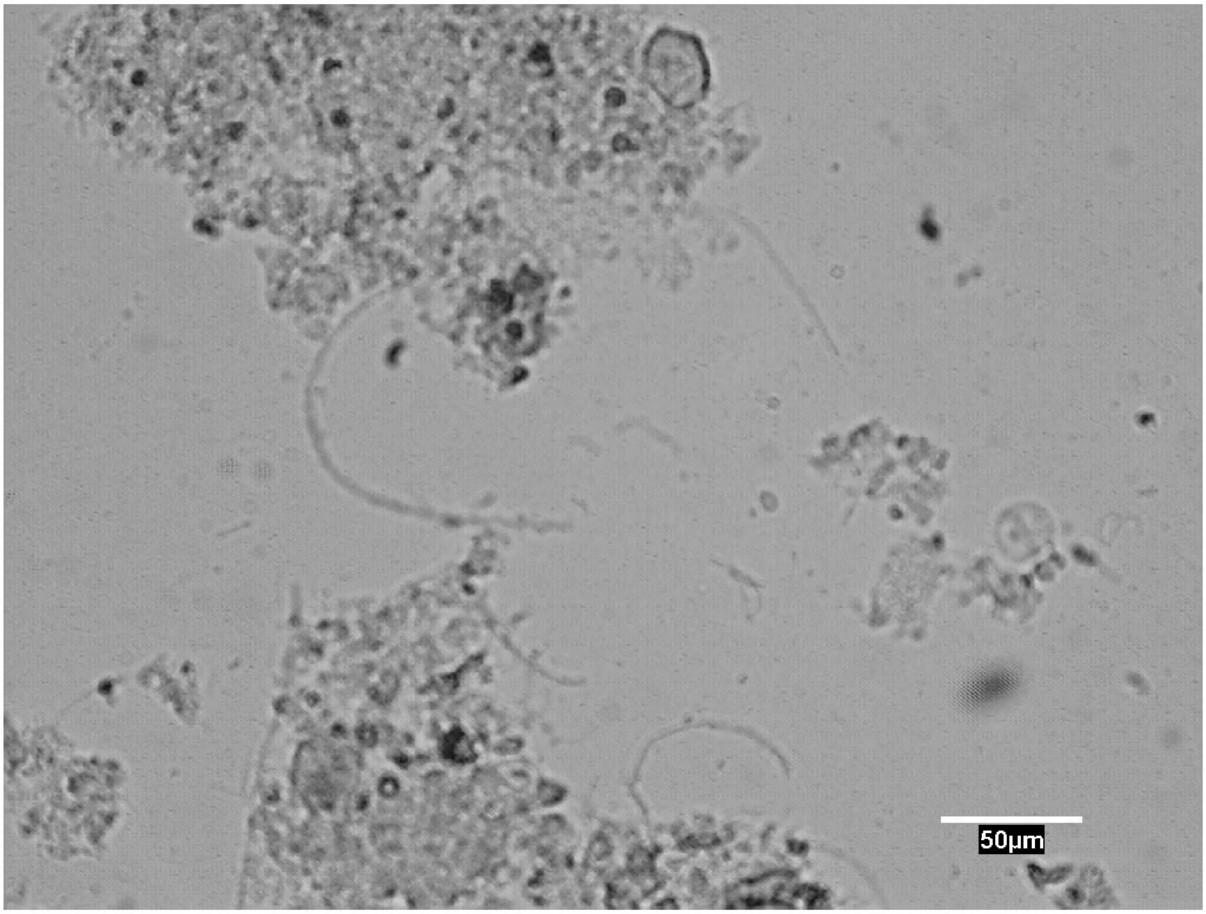

Figure 3.5. TWAS Untreated

By ultrasound pre-treatment was effective in disintegrating the bacterial cells as was evident from the microscopic photographs. Figures 3.5 and 3.6 are the microscopic snapshots of TWAS. Figure 3.5 shows microscopic view before ultrasound treatment and Figure 3.6 shows the view after ultrasound treatment.

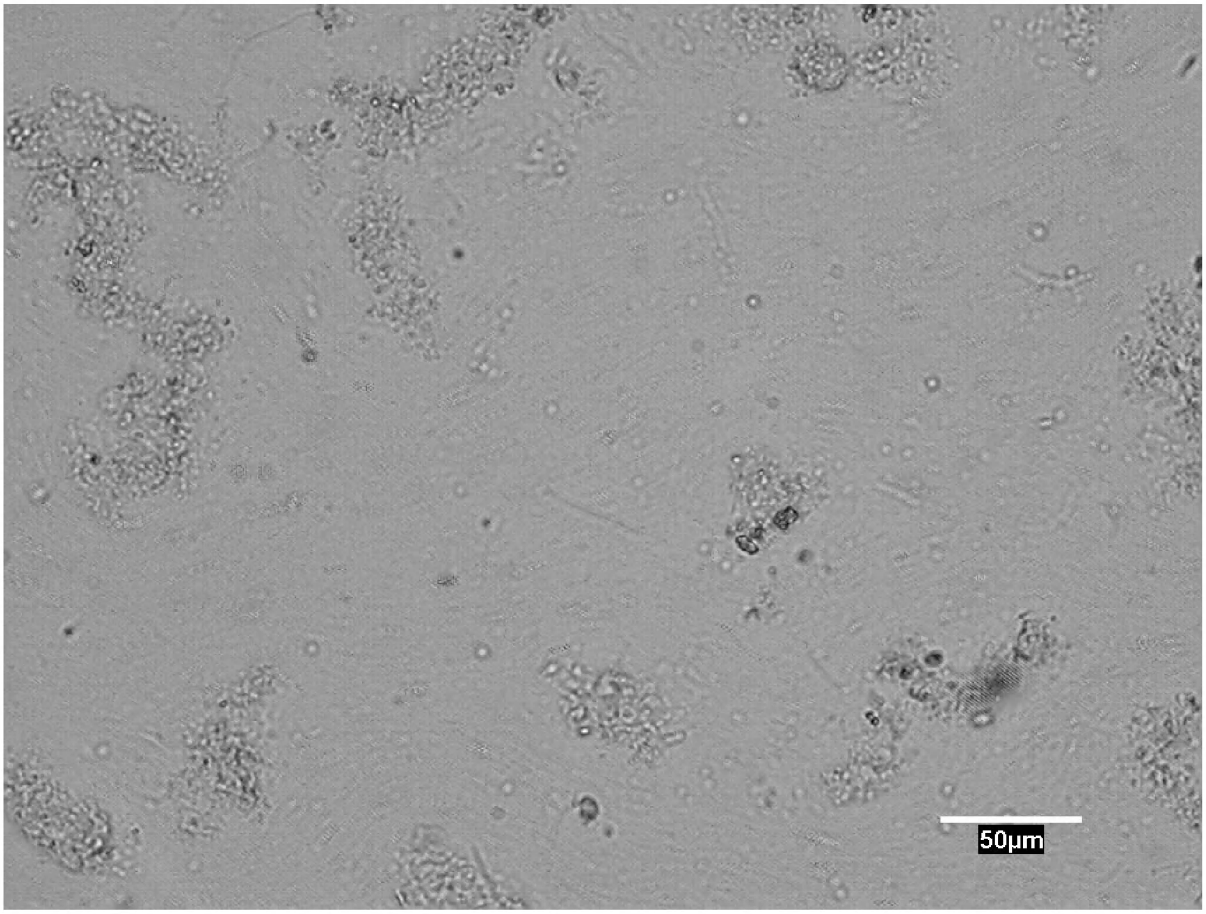

Figure 3.6. TWAS Treated with Ultrasound

The turbidity of the TWAS samples was tested before and after sonication. Raw TWAS, prior to sonication had a turbidity reading of 60-70 NTU. The turbidity readings after sonication at 1.25 kWs/g TWAS and 1.95 kWs/g TWAS were approximately 150 NTU and 200 NTU, respectively. The increase in turbidity noted at the higher energy input of 1.95 kWs/g TWAS was possibly due to the greater disintegration of the microbial cells contained in TWAS.

Foaming Potential

The tendency to foam was determined in terms of daily foam accumulation in the reactors. Initially, the potential to foam was measured as the height of foam layer (over the liquid) in the reactors. However, by this approach, it was not easy to separate freshly formed foam from any foam that was previously present. Hence, a new modified approach was adapted to determine foaming potential. All the reactors were mixed at maximum intensity (100 rpm) once daily until the foam layer was completely combined into the liquid phase (approximately 3-5 minutes). The liquid volume and the volume of freshly formed foam in the reactors were measured every 24 hours. In addition to breaking up the foam layer, the increased mixing intensity also helped in distributing the feed substrate within the reactors, giving more consistent reactor effluent solids concentrations.

During the energy input of 1.25 kWs/g TWAS, while the control reactor and the reactor that received 30% sonicated TWAS 'foamed over', the test reactor that received 100% sonicated TWAS showed the lowest tendency to foam for both Denver and Marshalltown solids (Run 1).

At the higher energy input of 1.95 kWs/g TWAS (Run 2), Marshalltown solids showed similar results to Run 1. The test reactor that received 100% sonicated TWAS showed the lowest tendency to foam. On the other hand, limited COD solubilization after sonication achieved between the two energy inputs values of 1.25 and 1.95 kWs/g TWAS (Runs 1 and 2). Therefore, it was speculated that the COD release achieved during the energy input of 1.95 kWs/ g TWAS might not have been sufficient to show a noticeable difference in performance with respect to foaming potential. These results indicated the need to study higher energy inputs than 1.95 kWs/g TWAS to increase more COD solubilization to evaluate in performance of foaming potential for Marshalltown solids.

In contrast to our observation with Marshalltown solids at the energy input of 1.95 kWs/g TWAS, the reactor that received 100% sonicated TWAS showed the greatest foaming potential with Denver solids (Run 2). The test reactor that received 30% TWAS showed the lowest propensity to foam. It was speculated that since the reactors were not mixed at maximum intensity during effluent discharge, the foam accumulation from the previous phase was not substantially washed out off the reactors even after three volume turnovers during the energy input of 1.95 kWs/g TWAS. The residual foam from Run 1 coupled with larger quantities of solubilized substrate in the reactor feed at the higher sonication energy input was making the reactor that received 100% sonicated TWAS more prone to foaming. To confirm this thought, all three reactors were reseeded with fresh solids from Newton Wastewater Treatment Facility, IA (no foaming problems in the full-scale reactors at Newton) for the next run. This assured that the reactors were free of any foam at the start of new run for Denver solids (Run 3). The reactors were also mixed at the maximum intensity at least once during effluent discharge to eliminate any foam accumulating in the reactors with the effluent.

Even with the operational changes, the reactor that received 100% sonicated TWAS showed the greatest tendency to foam, while the test reactor that received 30% sonicated TWAS showed the lowest foaming potential. However, foam accumulation did not hinder operations in any of the reactors during this run. The cause for increased foaming in the reactor that received 100% sonicated TWAS was established by monitoring the gas production rates from the three reactors over a 6-hour period soon after the feeding cycle. Figures 3.7A and 3.7B show the gas production rates from the three reactors during the first 30 minutes and over the six hour period, respectively. The gas production rate from the test reactor that received 100% sonicated TWAS was considerably higher than the other two reactors during the first 30 minutes. The higher energy input used for sonication during Run 3 solubilized more COD in the feed, thereby enhancing the rate of substrate degradation. The initial higher rate of gas production was accentuating foam accumulation in this reactor.

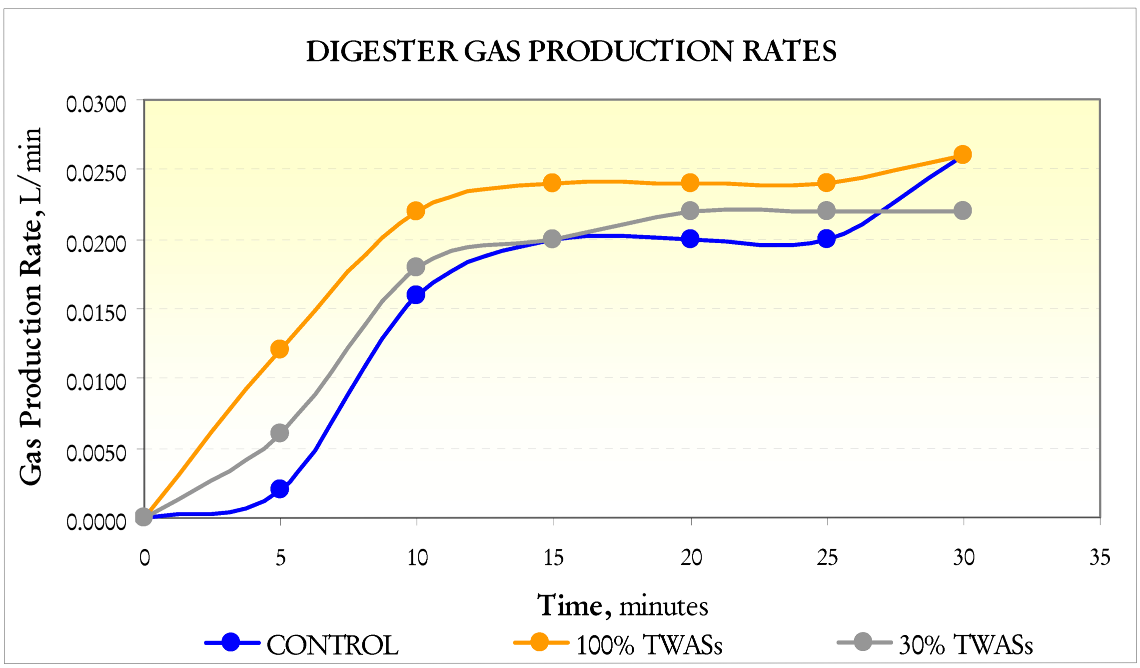

Figure 3.7A. Digester Gas Production Rate – First 30 Minutes after Feeding

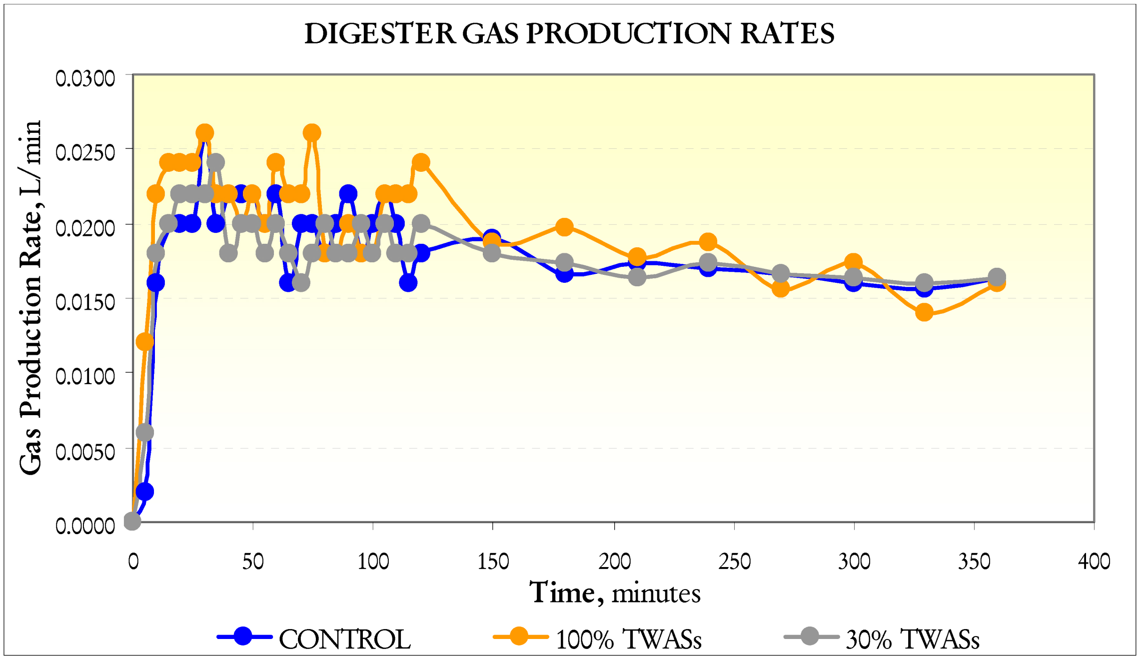

Figure 3.7B. Digester Gas Production Rates – 6 Hour Cycle

Although the three Runs were conducted with the same 40:60 blend of PS and TWAS, the control reactor showed less foam accumulation during Run 3, following reseeding with fresh solids than during the lower energy input of 1.25 kWs/g TWAS. The only noticeable difference between the two runs was digester loading. While there were wide fluctuations in reactor loads during the energy input of 1.25 kWs/g TWAS, the loads were relatively stable during the energy input of 1.95 kWs/g TWAS. This implied that a 40:60 blend of PS and unsonicated TWAS is susceptible to foaming if the digester loading is variable, but may not be a problem if loading is constant.

Under variable load conditions, part-stream sonication at an energy input of 1.25 kWs/g TWAS was not effective in controlling foam, but seemed effective at the higher energy input of 1.95 kWs/g TWAS. On the other hand, full-stream sonication of TWAS was effective in controlling reactor foaming at the lower energy input used for Run 1 (1.25 kWs/g TWAS) even with variable loads, but increased substrate solubilization at the higher energy input used for Run 2 (1.95 kWs/g TWAS) intensified foaming. This indicated the need to optimize energy inputs during sonication if the primary objective is foam control.

Volatile Solids Reduction and Biogas Production

There was no significant difference in the performance of the three reactors in terms of volatile solids reduction (VSR) during this study. The reactor that received 100% sonicated TWAS achieved marginally higher VSR (approximately 2-3% for Denver solids and approximately 5-6% for Marshalltown solids) than the control and the reactor that received 30% sonicated TWAS. All three reactors achieved 50-55% VSR with Denver solids, and 47-53% VSR with Marshalltown solids. The reason for lower VSR achieved with Marshalltown solids could be explained with lower volatile portion of the total solids portion (73% of TS) compared to Denver solids (83% of TS). Figure 3.8 shows the VSR achieved in the reactors for the two different solids at energy inputs of 1.25 and 1.95 kWs/g TWAS.

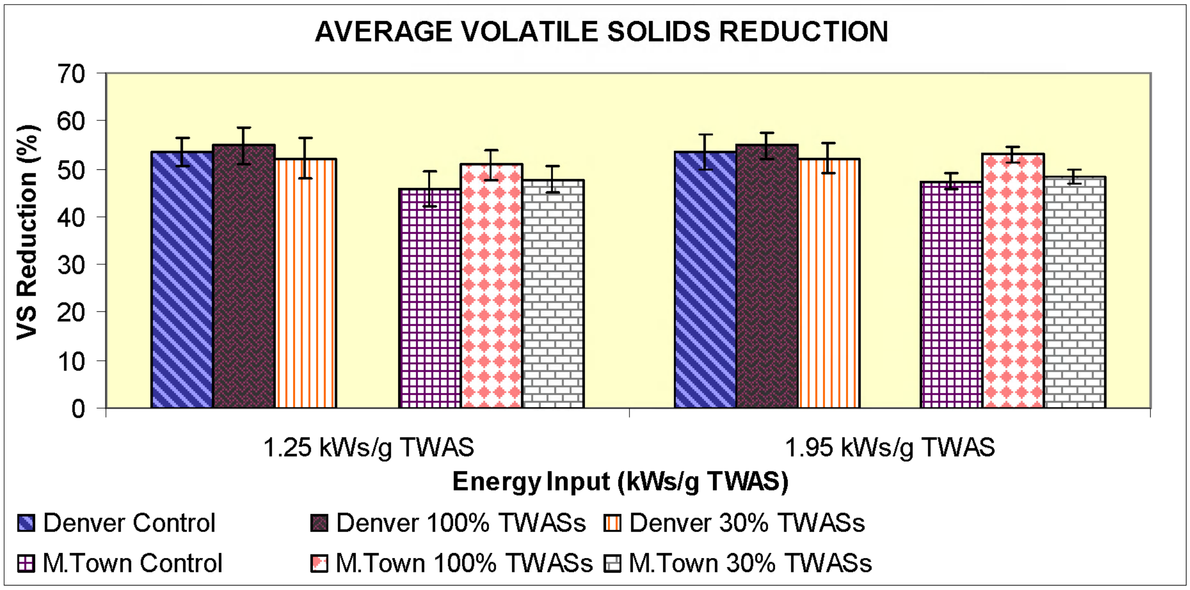

Figure 3.8. Volatile Solids Reduction in the Reactors

By running Marshalltown solids, the reactor that 100% sonicated TWAS achieved higher VSR in comparison of Denver solids. At the energy input of 1.25 kWs/g TWAS, VSR achieved in the reactors was 46% of the control reactor, 51% of the reactor that received 100% sonicated TWAS, 48% of the reactor that 30% sonicated TWAS, respectively. This higher VSR could be explained by increase in SCOD concentration after sonication, (440%) at the energy input of 1.25 kWs/g TWAS. However, there was no significant improvement at the energy input of 1.95 kWs/g TWAS compared to the energy input of 1.25 kWs/g TWAS. The reactor that received 100% sonicated TWAS only achieved 1% higher VSR than the other reactors. The energy input of 1.95 kWs/g TWAS did not show a significant increase in COD solubilization on VSR (Figure 3.9).

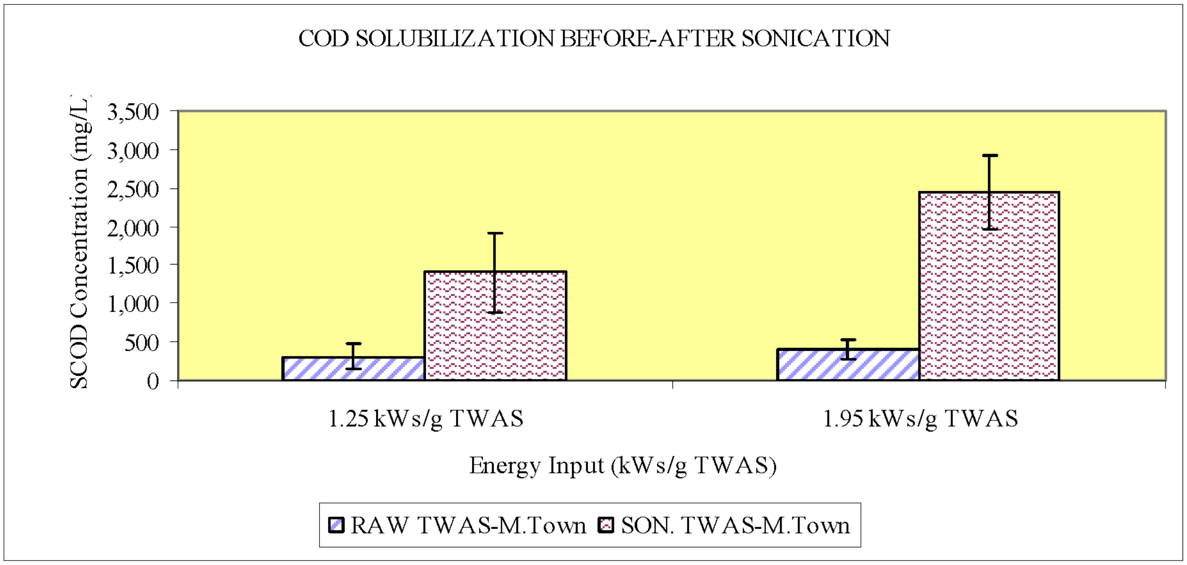

Figure 3.9. Variation in SCOD Before-After Sonication for Marshalltown Solids

The daily biogas production at standard temperature and pressure from the three reactors is shown in Figure 3.10. The corresponding biogas yields from the digesters averaged 0.70-1.00 L/g VSR (12-15 cf/lb VSR) for the two solids. Biogas production, consistent with the higher volatile solids reduction, was slightly higher from the reactor that received 100% sonicated TWAS compared to the control and the reactor that received 30% sonicated TWAS. The biogas from all the three reactors for both solids contained approximately 60-65% methane.

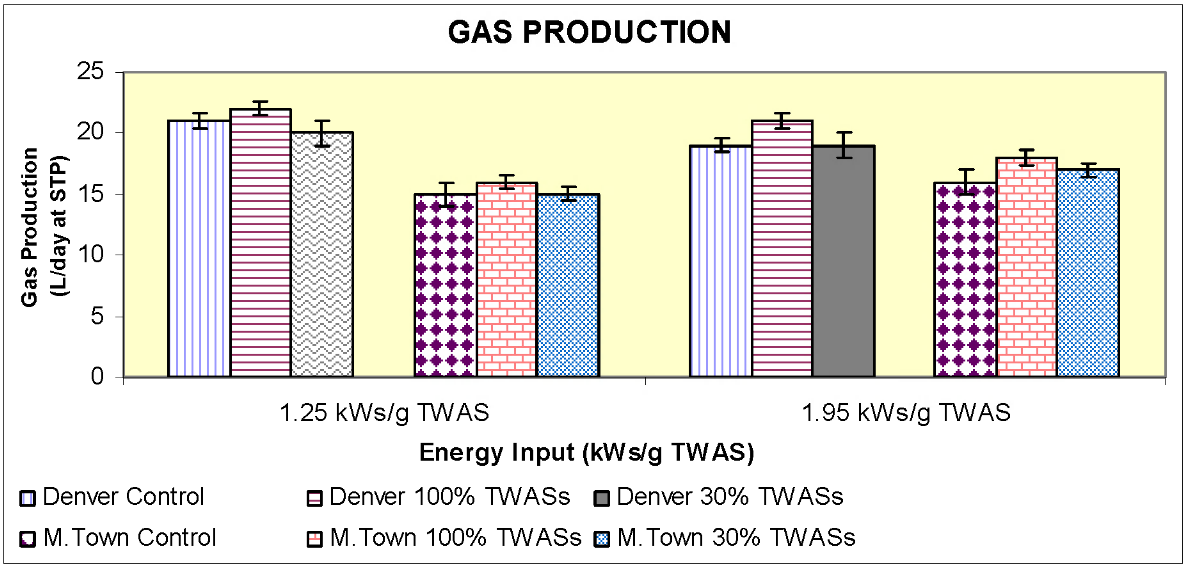

Figure 3.10. Biogas Production from the Reactors

Contradictory to other studies (Neis *et al.*, 2000; Tiehm *et al.*, 2001; Onyeche *et al.*, 2002), the test reactors that received sonicated TWAS did not achieve higher VSR with either solids. During the run with Denver and Marshalltown solids, it was speculated that this was possibly due to low COD solubilization achieved at the energy input of 1.25 kWs/g TWAS. Therefore, a higher energy input was used for next run to increase COD solubilization and to evaluate the impact of higher soluble COD concentration on VSR. However, in spite of solubilizing more COD, the reactors achieved only minimal variation in performance at the energy input of 1.95 kWs/g TWAS. Subsequently, it was decided to monitor the soluble COD concentrations in the reactor feed and effluent to determine the removal efficiencies achieved in the three reactors. The reactor feed/effluent soluble COD concentrations for the Denver and Marshalltown solids are plotted in Figures 3.11A and 3.11B.

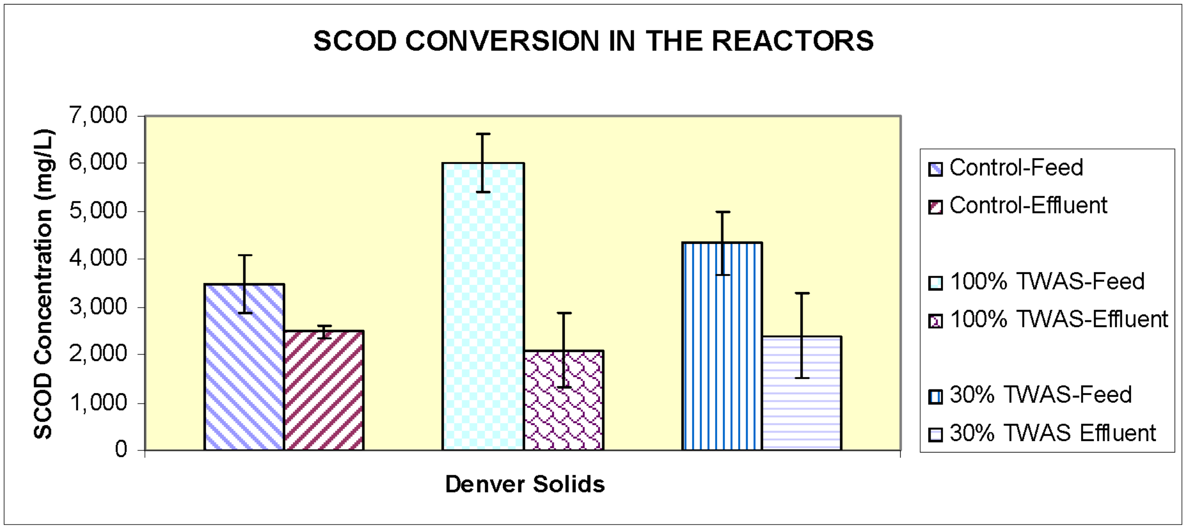

Figure 3.11A. Soluble COD Conversion in the Reactors for Denver Solids

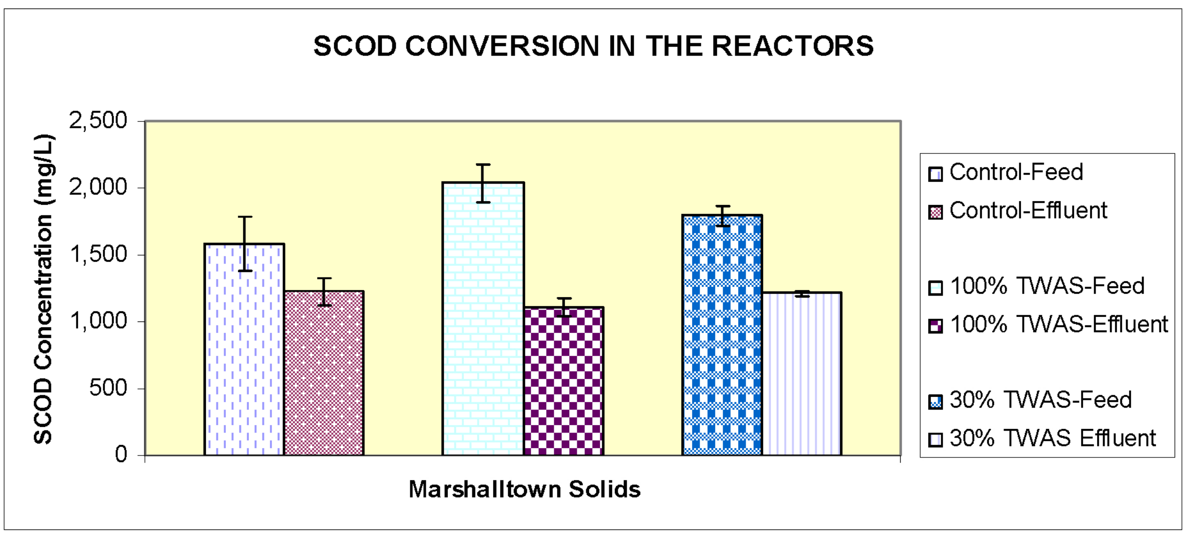

Figure 3.11B. Soluble COD Conversion in the Reactors for Marshalltown Solids

It was found that the control reactor operating at 15 day retention time was able to reduce soluble COD concentrations in the feed to levels comparable to those achieved in the reactors receiving sonicated TWAS. Therefore, it can be hypothesized that greater COD solubilization achieved via sonication improved the rate, but not necessarily the final degree of degradation. Solubilization of substrate by ultrasonic pre-treatment may be beneficial for plants operating digesters at shorter retention times (overloaded digesters).

Volatile Fatty Acids & Alkalinity

It is speculated that ultrasonic disintegration disrupts the microbial cells in the TWAS, extracting intracellular organics and making them amenable for digestion. Release of intracellular organics is measured as the increase in soluble COD of the samples. The volatile fatty acids concentrations do not generally change after sonication since a change in VFA levels would result only from biological activity. The volatile fatty acids concentrations (VFA) in reactor feed and effluent are plotted in Figure 3.12 for both Denver and Marshalltown solids.

As shown in Figure 3.12, there was no significant difference in VFA concentrations in the feed to the three reactors during the energy input of 1.25 and 1.95 kWs/g TWAS. During the study, the variation in VFA feed concentrations for both solids was resulting from the partial degradation of feed solids at higher temperatures, typical of the summer months. The reactor that received 100% sonicated TWAS had the lowest effluent VFA concentration. This could be explained that the solids sonicated by ultrasonic pre-treatment were amenable to better degradation. Average effluent VFA concentrations were always below 500 mg/L in the reactors. These VFA concentrations were in the typical range for mesophilic digesters and showed an uninhibited digestion process.

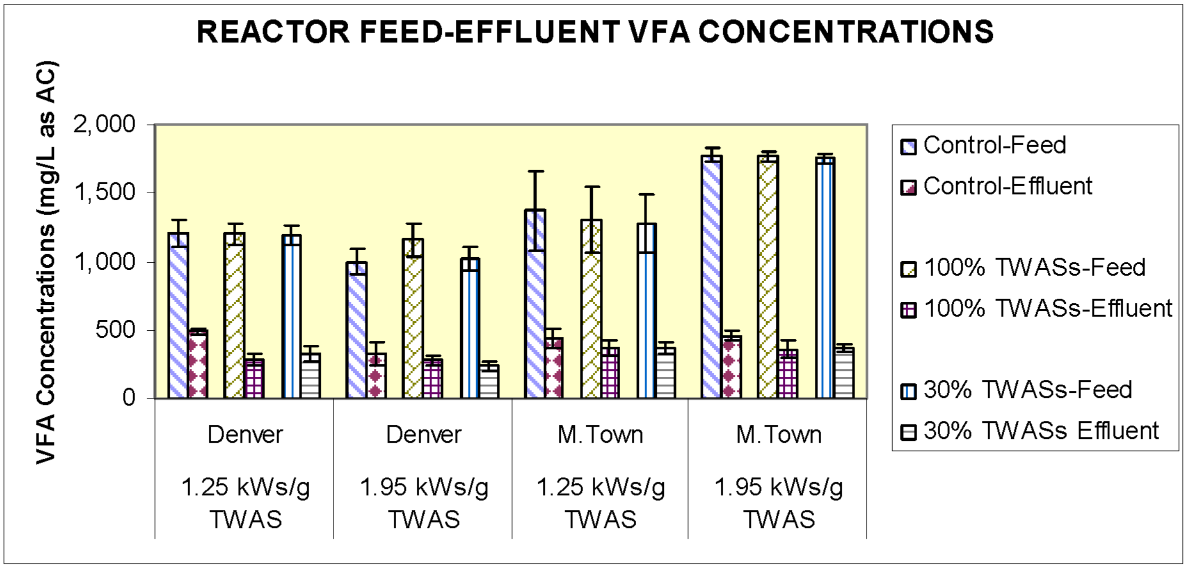

Figure 3.12. VFA Concentrations

As clearly shown from Figure 3.13, the alkalinity levels in the reactors increased with solubilization. The reactor that received 100% sonicated TWAS had the highest effluent alkalinity followed by the reactor that received 30% sonicated TWAS and the control. Sonication increased the ammonia nitrogen concentration in the feed due to greater release of cellular proteins. This resulted in increased conversion of organic nitrogen to the inorganic form, thereby increasing ammonia bicarbonate alkalinity.

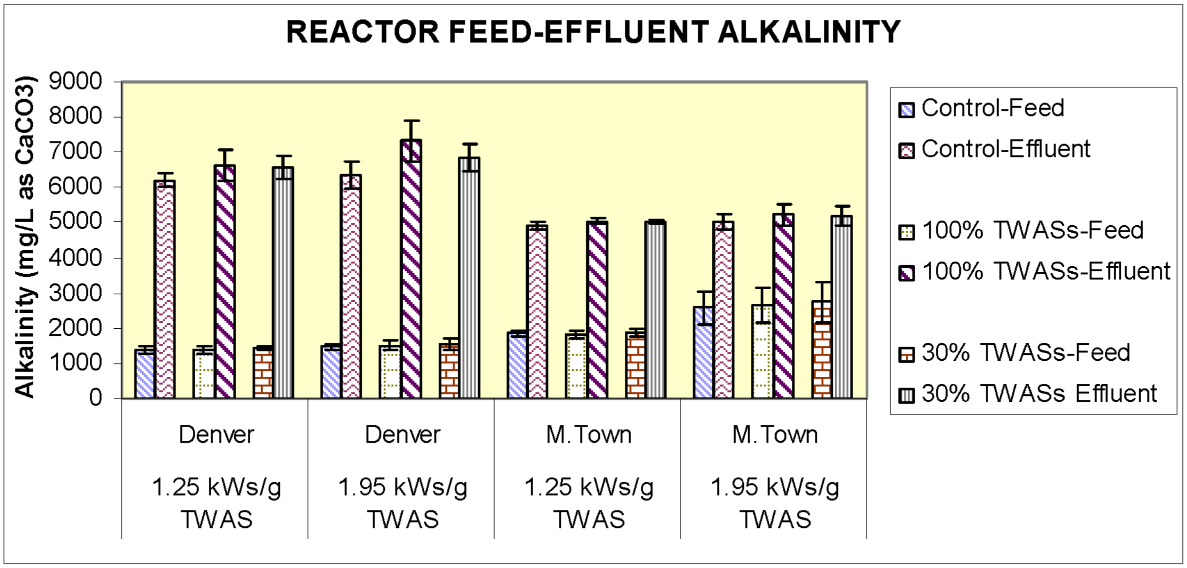

Figure 3.13. Reactor Alkalinity

Conclusions

In this study, ultrasonic disintegration of TWAS was primarily studied as a foam control measure. A secondary objective of the study was to compare the performance of ultrasound-conditioned (sonicated) TWAS in terms of volatile solids reduction and biogas production against unsonicated TWAS. The most important results drawn from the study are:

- With a 40:60 blend of PS:TWAS, mesophilic digester did not have foaming problem when the digester loading was relatively constant. This shows that PS and TWAS fractions in the digester feed have to be controlled for exercising better foam control.

- At the lower energy input of 1.25 kWs/g TWAS, the 100% TWAS sonicated unit was able to control foam effectively while the control unit and the 30% sonicated test unit 'foamed over' under variable loading conditions with 40:60 blend of PS and TWAS.

- The energy inputs during sonication have to be optimized to control foam effectively. Full-stream sonication of TWAS was able to be effective in foam control at the lower energy input while increased solubilization of feed substrate at the higher energy input used for Run 2 exacerbated foaming problems due to higher rates of substrate degradation.

- Sonication of TWAS by increasing SCOD values in 1,500-2,500 mg/L range did not result in significant volatile solids reduction in the reactors contrary to other reported studies. However, this does not necessarily imply that an increase in VSR and corresponding increase biogas production would not be achieved at higher energy inputs.

- Sonication of TWAS only increased the rate of substrate degradation, but did not improve the ultimate degree of degradation. Hence, in case of sufficient long retention times are ensured in the reactors, the control reactor could achieve comparable results as the reactors that receive sonicated TWAS. As seen for the experimental study, the control reactor operating at 15-day retention time was able to achieve comparable SCOD removal as the reactors that received sonicated TWAS.

Acknowledgements

This project was supported by the Black & Veatch in collaboration with Metro Wastewater Reclamation District of Denver (MWRD). The authors would like to express thanks for the support of Etrema Products Inc., Ames, IA for the ultrasound equipment and Marshalltown WWTP, Marshalltown, IA for raw primary and secondary solids.

CHAPTER 4. THE EFFECTS OF ULTRASOUND DISINTEGRATION ON ANAEROBIC DIGESTION AT DIFFERENT SOLIDS RETENTION TIMES

A paper to be submitted to *WEFTEC'2007*

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Abstract

Thickened waste activated solid (TWAS) is usually considered difficult to digest due to its complex structure and low degradability. Therefore, the low rate of TWAS degradation is usually depicted to be the rate limiting stage in anaerobic digestion process. The effect of disintegration on thickened waste activated solids by ultrasound treatment was studied to minimize both the retention time and the destruction of volatile solids in a mesophilic anaerobic reactor processing a mixture of primary and thickened secondary solids. The sonication duration was adjusted to achieve energy inputs equivalent to 1.95 kWs/g TWAS. The reactors were operated at various solids retention times (SRT) of 15, 10, and 6 days. Ultrasound treatment resulted in disintegration on TWAS was demonstrated in terms of the increase in COD solubilization, the reduction of solids particle size as well as the increase in turbidity, and microscopic photographs shown the disintegration of the bacterial cells. Contrary to other reported studies, there was no significant difference in the performance of the three reactors with regard to volatile solids reduction (VSR). All three reactors achieved 47-53% VSR at 15 day HRT, 42-48% VSR at 10 day HRT, and 41-47% VSR at 6 day HRT.

Sonication of TWAS increased the rate of substrate degradation, but did not improve the ultimate degree of degradation.

Keywords: Anaerobic digestion, solids destruction, thickened waste activated solids, ultrasound, retention time, disintegration.

Introduction

Anaerobic digestion is traditionally a biological process in which raw solids are destructed and then converted to biogas which is mostly of methane and carbon dioxide. The long hydraulic retention time is the principal disadvantage of the anaerobic digestion process due to the rate limiting stage of hydrolysis (Tiehm *et al.*, 2001; Neis *et al.*, 1997; Barber, 2003; Clark and Nujjoo, 2000; Wang *et al.*, 1999; Nickel, 2002).

Contrary to primary solids, thickened waste activated solid (TWAS) is considerably difficult to digest due to its complex structure and low degradability. Therefore, the low rate of TWAS degradation is usually depicted to be the rate limiting stage in anaerobic digestion process (Li and Noike, 1992). Previous studies showed that the digestibility of TWAS was limited to about 25-30% to achieve efficient and stable operation (Mormede, 2003).

Several pretreatment methods have been studied recently to improve hydrolysis rate of raw solids (Wang *et al.*, 1995). These methods led to disrupt of the cell walls and membrane of bacteria in raw solids resulting in the organic substances within cells released by destroying the cell walls. These organic substances increase of biodegradable material can be easily hydrolyzed, and improve the anaerobic digestion process.

The potential of ultrasound disintegration has been investigated for many years to enhance anaerobic digestion. Ultrasound disintegration, which includes the high intensity sound waves into a solid medium, is based on the destruction of both bacterial cells and difficult to degrade organic substances (Barber, 2003). The bacterial cells available for consumption by other species then release while the organics are broken down into smaller readily biodegradable fractions. The subsequent enhancement of biodegradable material improves anaerobic digestion process. Improved performance is demonstrated by increased volatile solids destruction, higher gas production, elimination of foaming, and improved dewaterability.

The primary objective of this study was to evaluate the performance of ultrasound-conditioned (sonicated) TWAS to minimize the retention time and to enhance the destruction of volatile solids in a mesophilic anaerobic reactor processing a mixture of primary and thickened secondary solids. A secondary objective of the study was to determine the effect of disintegration on thickened waste activated solids by ultrasound treatment.

Material and Methods

Three laboratory-scale mesophilic anaerobic reactors, each with a total volume of 30 liter, were used with a working volume of 18 liters. Anaerobic reactors used in this study were made from cylindrical Plexiglas^{TM} in the Chemistry Machine Shop at Iowa State University.

Feeding and decanting at 6 hour intervals were performed on a semi-continuous basis. The reactor contents were mixed mechanically at 40 rpm for 5 minutes every 15 minutes.

Masterflex I/P 7591-00 peristaltic pumps were used to transfer raw solids from feed tank to each reactor, and to discharge biosolids from each reactor. Each reactor was equipped with an EMI mixer (Eastern Mixer Brand) to mix the reactors intermittently. ChronTrol XT timers were used to control all pumps and mixers, and automate the feeding, decanting, and mixing cycles. The gas collection system included airtight plastic tubing, a steel wool in a glass vessel to remove H_2S from the digester gas followed by a glass flowrate observation bubbler and a glass sampling port with a rubber septum. Wet-tip gas meter was used to record volumetric biogas production. A schematic of one full reactor configuration is shown in Figure 4.1.

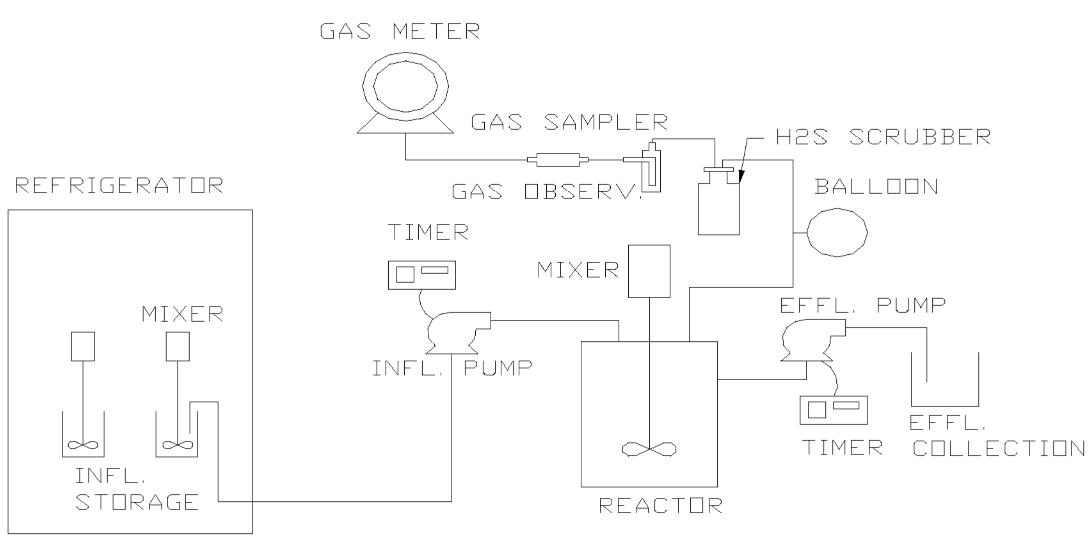

Figure 4.1. Mesophilic Anaerobic Reactor Schematic

The feed solid (PS and TWAS) for the experimental study was obtained from Marshalltown WWTP, Iowa, and kept in a refrigerator maintained at 0 ^{0}C prior to feeding to minimize the decomposition of the substrate. The reactors were operated in a constant temperature room maintained at 35 ^{0}C. The temperature in the mesophilic anaerobic reactors was maintained at 35 ±1 ^{0}C. The reactors were operated at various solids retention times (SRT) of 15, 10, and 6 days. Two of three pilot reactors were operated as test units and were fed sonicated TWAS combined with PS. One of these test reactors was operated based the sonication of 100% of the TWAS (full-stream treatment), while the other was operated with 30% by volume of sonicated TWAS (part-stream treatment). The feed to the second test reactor consisted of 30% sonicated TWAS and 70% unsonicated TWAS, which were then combined with PS. Part stream sonication tests were conducted to evaluate the hypothesis that full stream treatment may not be required to accomplish the benefits of ultrasound pre-treatment (Friedrich, 2002). The control reactor was operated with a mixture of PS and unsonicated TWAS. All three reactors were fed a 40:60 blend of PS:TWAS on a dry weight basis. During the study, the sonication duration was adjusted to achieve energy inputs equivalent to 1.25 and 1.95 kWs/g of TWAS. The sonication time was varied to achieve the desired energy input (kWs/g), depending upon the TWAS concentration.

The raw TWAS for the study was pretreated by a Maxonics ultrasonic unit provided by Etrema Products, Inc., Ames, IA. The sonication vessel was made of stainless steel with an external jacket with a direct connection to a cold water tap, and could sonicate 1.4 L of raw solids in batch operation. TWAS was circulated continuously during sonication using an external peristaltic pump. The ultrasound unit was operated at a frequency of 20 kHz and 1.5

kW of sonication power, respectively. The sonication time was varied to adjust the energy input (kWs/g) to the solids.

On a daily basis, volumetric biogas production, digestion temperature, and pH were recorded. Biogas composition was measured approximately once per week with a gas chromatograph. Measurements of total solids (TS), volatile solids (VS), volatile fatty acids (VFA), and alkalinity were made on a weekly basis. Soluble chemical oxygen demand (SCOD) analysis and microscopic photography were conducted for TWAS samples before and after sonication.

More intensive testing was conducted after steady state was reached. Standard Methods (1995) was followed for TS, VS, VFA, SCOD, and alkalinity analysis procedures. Fisher Scientific G4 filter papers (1.2 micron retention, 90 mm diameter), and Osmonics MAGNA LIFT nylon transfer membranes (0.45 micron retention, 82 mm diameter) were used for the soluble COD determination.

Results and Discussion

Effect of Ultrasound Disintegration on Solubilization of TWAS

The effect of ultrasound disintegration on TWAS was demonstrated in terms of the increase in COD solubilization, the reduction of solids particle size as well as the largest increase in turbidity, and microscopic photographs shown the disintegration of the bacterial cells.

A break-up of microbial cell walls is achieved by ultrasound disintegration, and intracellular components are released resulting in an increase of COD on the supernatant fraction of the raw solids (Neis *et al.*, 2000). The results increasing in SCOD before and after sonication tests are presented in Figure 4.2. Significant difference in COD solubilization was noted. The average increase in SCOD concentration after sonication was approximately 440% at the energy input of 1.25 kWs/g TWAS, and 550% at the energy input of 1.95 kWs/g TWAS, respectively. The efficiency of solids disintegration was dependent on specific energy input, and increased with increasing specific energy input. The results showed that COD solubilization indicated a good indicator to determine the effect of ultrasound treatment on solids disintegration.

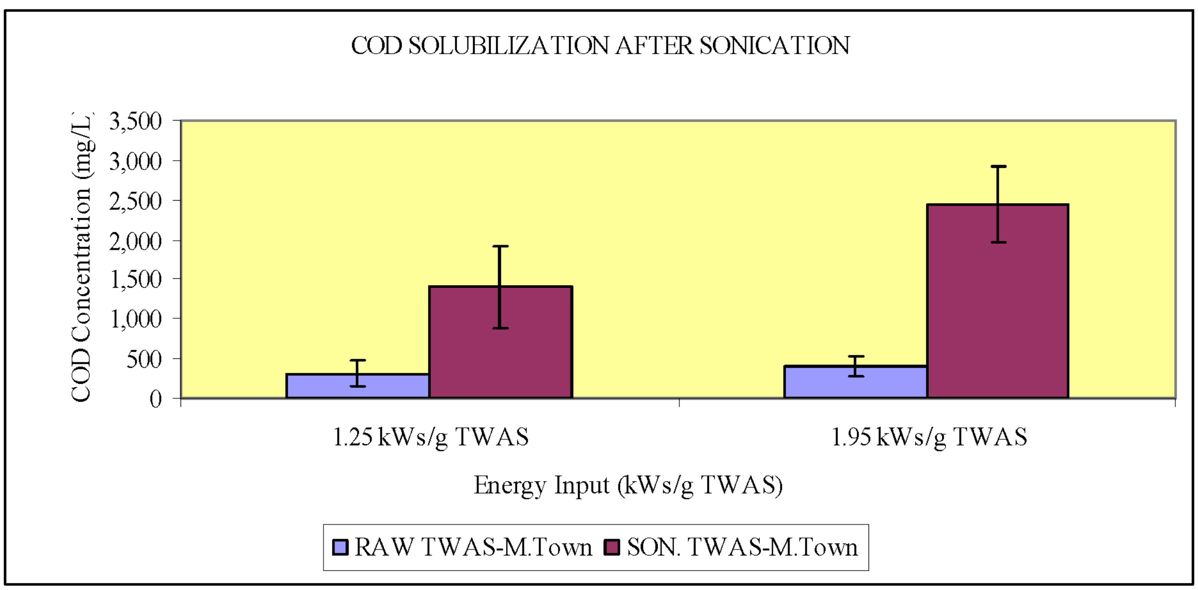

Figure 4.2. COD Solubilization Before-After Sonication at Different Energy Input Values

The effect of ultrasound treatment on TWAS disintegration was also determined by the measurement of the turbidity. The turbidity of the TWAS samples was tested before and after sonication. The turbidity readings after sonication were 150 NTU at the energy input of 1.25 kWs/g TWAS and 200 NTU at the energy input of 1.95 kWs/g TWAS, respectively while raw TWAS had a turbidity reading of 60-70 NTU. A significant increase in turbidity after sonication could be explained with the reduction of the particle size due to the disintegration of the microbial cells contained in TWAS.

Furthermore, microscopic photograph of the disintegration of the bacterial cells was an evidence of ultrasound treatment. Figure 4.3, the microscopic snapshots of TWAS solids, shows microscopic view before and after ultrasound treatment. The structure of bacterial cells was significantly disrupted by the effect of ultrasound treatment.

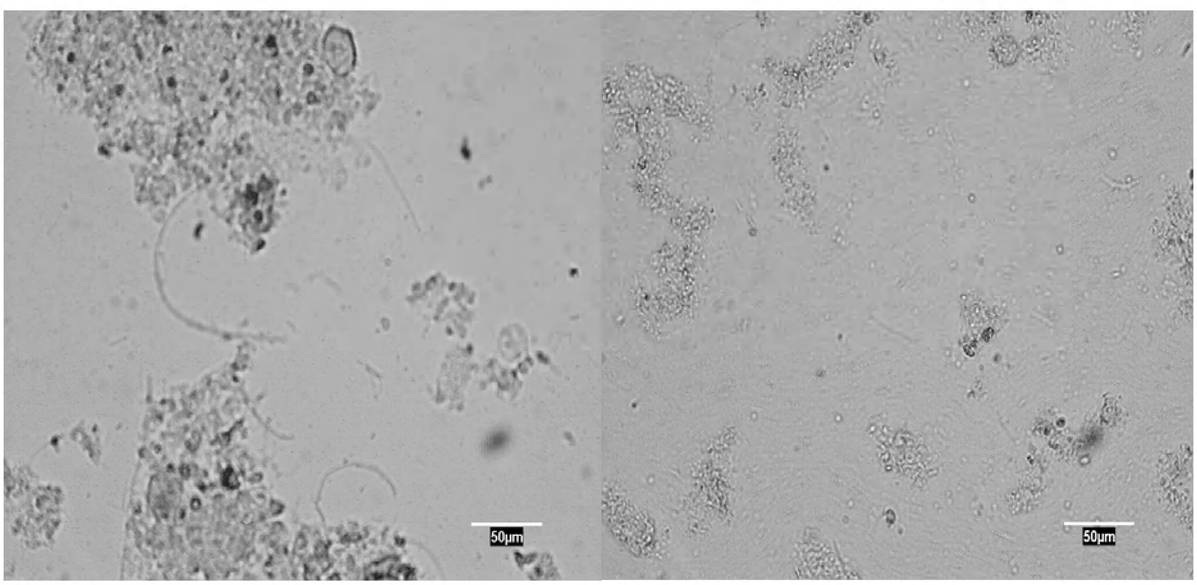

Figure 4.3. TWAS-Untreated and Treated with Ultrasound

Anaerobic Solids Digestion Following Ultrasound Treatment

Anaerobic digestion was not significantly improved by ultrasound treatment as expected. Many studies have shown that ultrasonic pretreatment improves anaerobic digestion, but there was no noticeable benefit for enhancing anaerobic digestion during this study. The reactor that received 100% sonicated TWAS showed marginally higher (~4-6%) volatile solids reduction (VSR) than the reactor that received 30% sonicated TWAS and the control at different retention times of 15, 10, and 6 days. All three reactors achieved 47-53% VSR at 15 day HRT, 42-48% VSR at 10 day HRT, and 41-47% VSR at 6 day HRT. Figure 4.4 shows the VSR achieved in the reactors at the different retention times at energy input of 1.95 kWs/g TWAS. VSR progressively declined with decreasing HRT. There was no significant VSR difference in the performance of the three reactors at the retention times between 10 and 6 days. The reactors at 10 day HRT only achieved 1% higher VSR than the reactors at 6 day HRT.

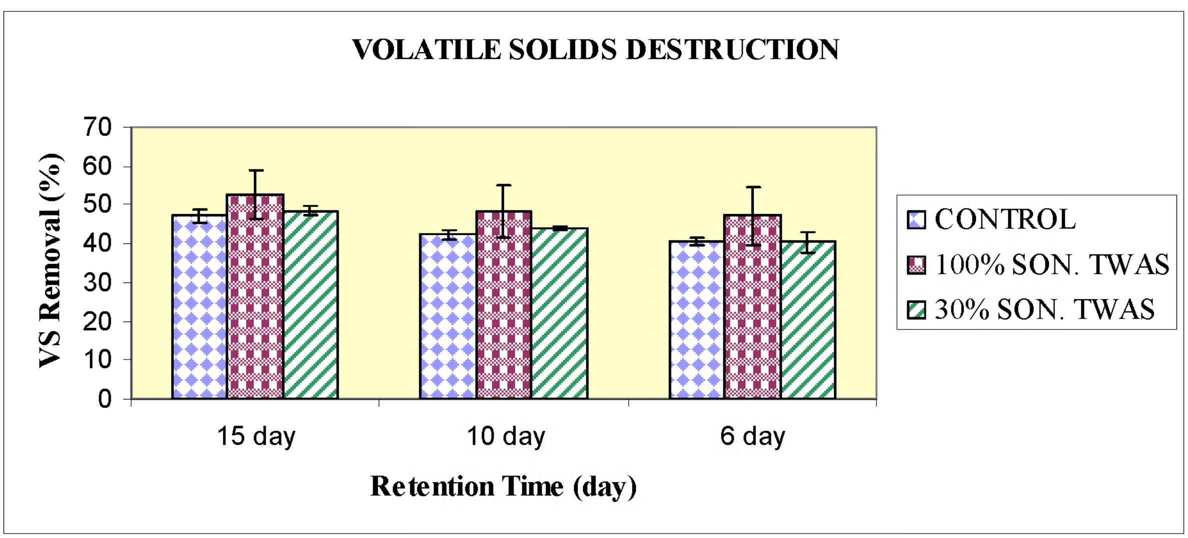

Figure 4.4. Volatile Solids Reduction in the Reactors at Different Retention Times

The results in this study did not achieve higher VSR compared to other reported studies (Neis *et al.*, 2000; Tiehm *et al.*, 2001; Onyeche *et al.*, 2002). It was speculated that this was probably due to low COD solubilization obtained at the energy input used during the study. Therefore, it was decided to determine the degree of disintegration of TWAS. For this reason, the maximum possible COD solubilization was determined by chemical disintegration of TWAS, a treating volume of raw solid with 0.5 N sodium hydroxide. The degree of disintegration of TWAS was approximately 18% at energy input of 1.95 kWs/g TWAS. This was slightly lower in the comparison of other studies. Neis *et al.*, (2000) studied with the average degree of disintegration of 20% while Tiehm *et al.*, (2001) carried out with higher than 70% of the degree of disintegration. Low COD solubilization could probably be explained one reason for lower VSR determined in this study.

Furthermore, the previous studies with regard to ultrasound disintegration of solids have involved either only thickened waste activated solids (TWAS) or a mixture of primary and thickened secondary solids. Most of these studies accomplished with both solids have been achieved at a lower fraction of TWAS feed such a ratio of 70:30 PS:TWAS, 60:40 PS:TWAS, or 53:47 PS:TWAS (Barber, 2003; Nickel, 2002; Tiehm *et al.*, 1997). Contrary to other studies, the reactors used in this study was fed with a higher fraction of TWAS, 40:60 PS:TWAS. A lower VSR achieved during this study might probably be explained due to a higher fraction of TWAS which is inherently difficult to digest and has a low biodegradability.

Although there was no significant VSR difference in the reactor that received 100% sonicated TWAS compared to the other two reactors, greater COD solubilization in the reactor that received 100% sonicated TWAS achieved via sonication improved the rate. To validate this thought, the removal efficiency of soluble COD concentrations in the three reactors was determined. Soluble COD conversion in the reactors based on the calculation of the reactor feed and effluent SCOD concentrations is plotted in Figure 4.5.

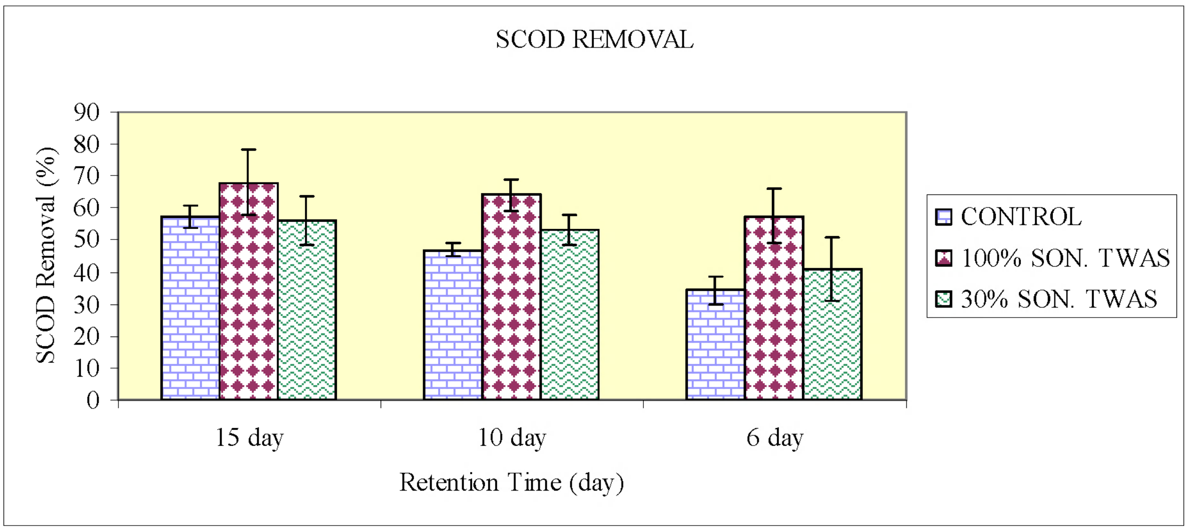

Figure 4.5. SCOD Conversion in the Reactors

The results showed that the greatest soluble COD conversion with 58% achieved in the reactor that received 100% sonicated TWAS in comparison of 34 and 41% of soluble COD conversion determined in the control reactor and the reactor that received 30% sonicated TWAS at the shortest retention time of 6 days, respectively. It may be concluded that solubilization of substrate by ultrasonic pre-treatment may be effective and beneficial for plants operating digesters at shorter retention times (overloaded digesters).

Contrary to VSR results, biogas production in the reactors significantly increased with reduced retention times. The corresponding biogas yields from the digesters averaged 0.70-1.00 L/g VSR (12-15 cf/lb VSR). The biogas composition in all the three reactors contained approximately 60-65% methane content during the study. Figure 4.6 presents biogas production from the three reactors at different retention times.

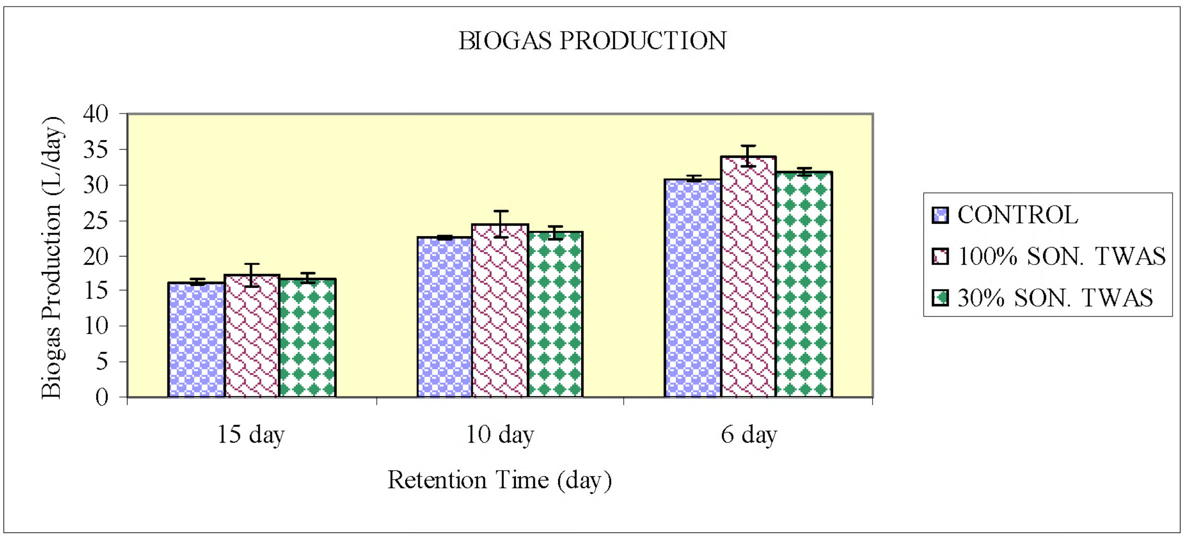

Figure 4.6. Biogas Production from the reactors at Different Retention Times

Gas production approximately increased 29% and 49% more biogas at the retention times of 10 and 6 days compared to the 15 day HRT, respectively. Higher biogas production obtained with increasing loading rates. Organic loading rates the reactors varied from 2.50 to 5.55 g VS/L/day during the study. Figure 4.7 illustrates the organic loading rates to the reactors during the three runs. Biogas production and the percent methane in the reactor fed with sonicated TWAS were always slightly higher than in the other two reactors. The reactor that received 100% sonicated TWAS showed 6%, 7%, and 9% higher biogas than the control reactor at the 15, 10, and 6 day HRT, respectively. This could be explained that the effect of sonication in TWAS increased the rate of substrate of degradation.

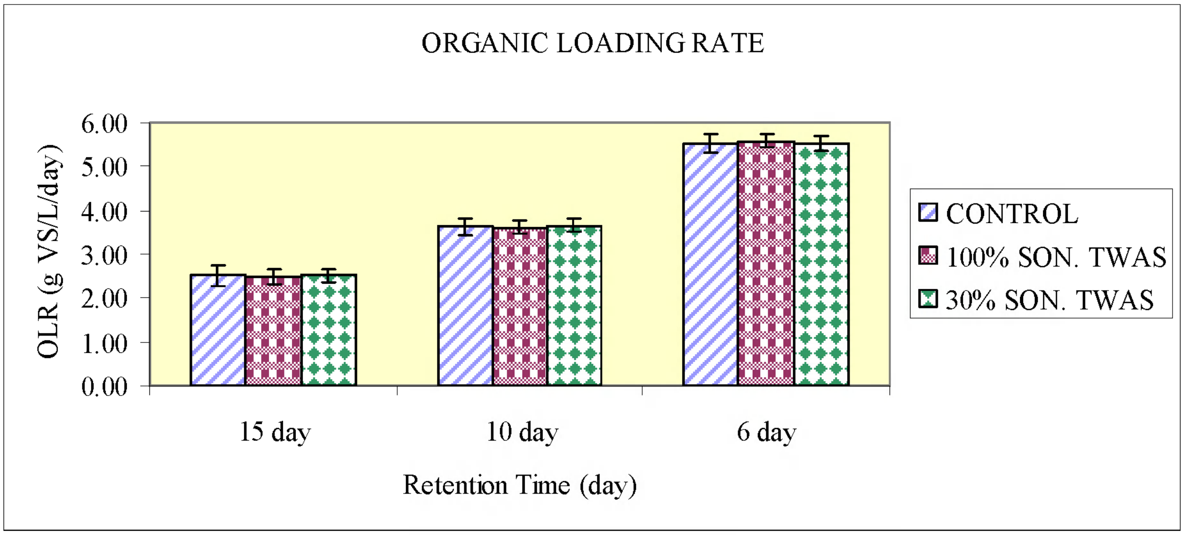

Figure 4.7. Organic Loading Rates to the Reactors

VFA and alkalinity are good indicators for the stability of an anaerobic digestion process. Low VFA:Alkalinity ratios indicate stable operation. A low VFA indicates that the methanogens are capable to utilize the VFA substrate, and a relatively high alkalinity indicates that the pH is stable enough for the pH sensitive microbes. Figure 4.8 and 4.9 illustrate the effluent VFA and Alkalinity concentrations with increasing instability of the reactors by decreasing HRT.

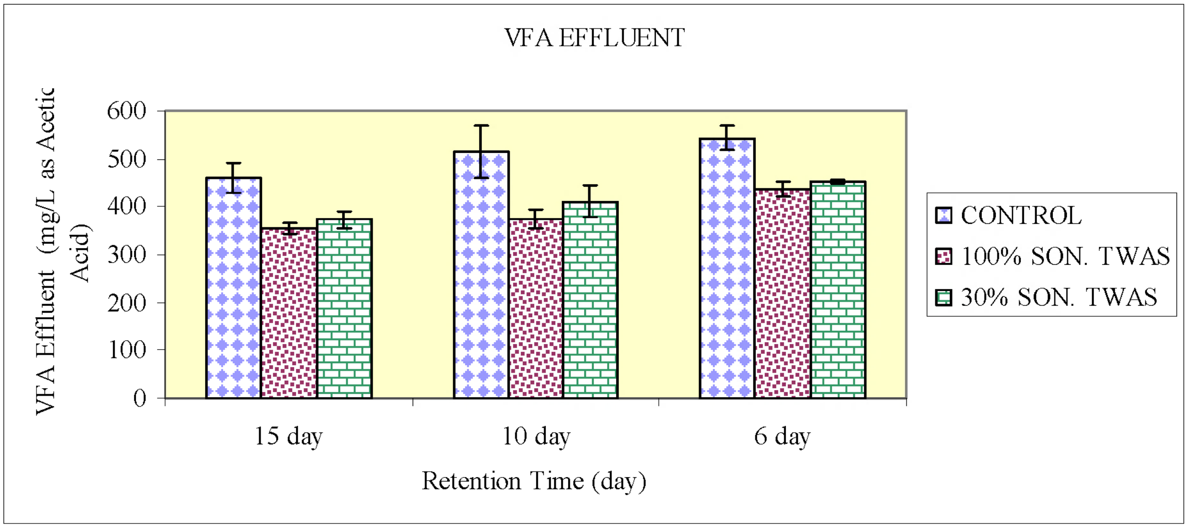

Figure 4.8. VFA Effluent Concentrations in the Reactors at Different Retention Times

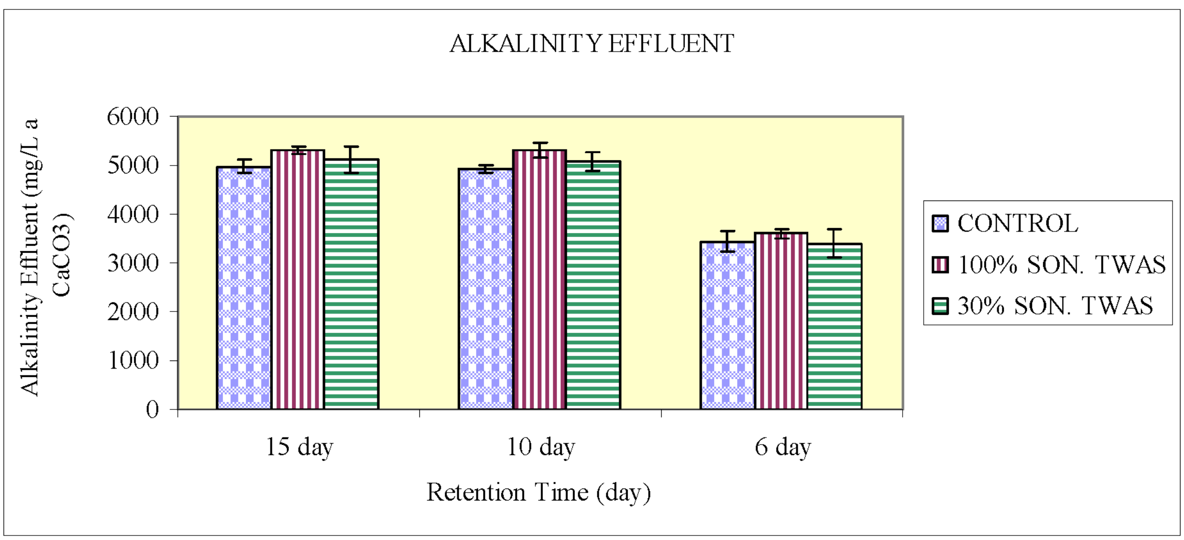

Figure 4.9. Alkalinity Effluent Concentrations in the Reactors at Different Retention Times

As expected, due to the better biodegradation of disintegrated TWAS, the reactor that received 100% sonicated TWAS showed the lowest VFA and the highest alkalinity concentrations followed by the reactor that received 30% sonicated TWAS and the control reactor at each retention time throughout the study. The 15 and 10 day HRTs exhibited low

VFA:Alkalinity ratio with 0.08. The 6 day HRT, with 0.14 VFA/Alkalinity ratio, began to show signs of instability with increased levels of effluent VFA and decreased effluent alkalinity in three reactors indicating that the methanogens were not able to utilize the VFA substrate efficiently due to the shorter retention time which was insufficient time required for necessary microorganisms to regenerate at the same rate as they were wasted to prevent washout. pH was also a good indicator to show for the instability of the reactors at the 6 day HRT. While the mean pH values at the 15 and 10 day HRT ranged from 7.34 to 7.55, it was gradually declined in the range of 6.99. Figure 4.10 presents pH changes in the reactors.

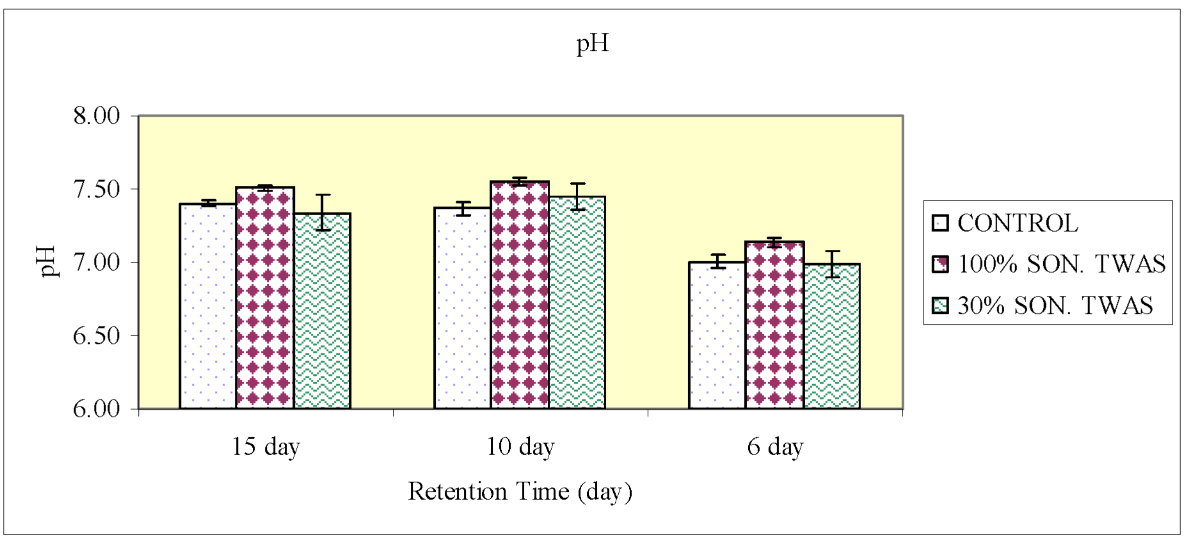

Figure 4.10. pH in the Reactors at Different Retention Times

Conclusions

This study has investigated the effects of ultrasound treatment on anaerobic digestion to evaluate the performance of ultrasound-conditioned (sonicated) TWAS to minimize both the retention time and the destruction of volatile solids. Based on the results of the experiments conducted in this study, the following conclusions are evident:

- Ultrasound treatment is an appropriate method to disintegrate of thickened waste activated solids. The effect of disintegration of TWAS can be increased with increasing specific energy input.

- Hydrolysis, the rate limiting stage of anaerobic digestion process, was improved with a break-up of microbial cell walls resulting in solubilization and enhancing biodegradability of solids by ultrasound treatment.

- Sonication of TWAS did not show any significant increase in volatile solids reduction and biogas production contrary to other reported studies. The reactor fed with 100% sonicated TWAS only achieved 5-6% VSR and 6-9% more biogas than the other reactors.

- The reactor fed with 100% sonicated TWAS always showed greater soluble COD conversion in comparison of the other two reactors. Solubilization of substrate by ultrasonic pre-treatment could be effective and beneficial for plants at shorter retention times (overloaded digesters).

- Although the reactor fed with 100% sonicated TWAS shows better results, anaerobic digestion may successfully be operated at shorter retention times without ultrasound treatment.

Acknowledgements

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CHAPTER 5. GENERAL CONCLUSIONS

Engineering Significance

Anaerobic digestion, one of the most important processes for solids reduction and stabilization, is limited due to the rate limiting step of solids hydrolysis. In an effort to improve solids hydrolysis, many studies have been investigated utilizing different methods of solids pre-treatment. References may be made to pre-treatment methods that are thermal, chemical, mechanical disintegration, enzymatic.

Using ultrasound, one of the mechanical disintegration methods, for solids treatment has been a well known method for many years. The use of ultrasound disintegration in order to enhance the anaerobic digestion process is the best-researched full-scale application today. However, the specific conditions of each treatment plant may supply further arguments for disintegration. Especially some operational problems are important factors such as difficult digestion properties of the solids, overload of digesters, foaming problems due to filamentous micro-organisms in excess solids.

There have been several investigations of many anaerobic digestion facilities processing waste activated solids (WAS) or combined primary and secondary solids that have identified digester foaming as a series operational problem. A survey by the American Society of Civil Engineers indicated that plant operators cited foaming as the most persistent operating problem associated with anaerobic solids digestion, with half of all anaerobic digester installations surveyed having experienced foaming problems at one time or another (Filbert, 1985). Foaming has often shown significant problems such as reducing the operating volumes of digesters, causing to biogas handling systems and structural damage to digester covers.

Metro Wastewater Reclamation District (MWRD) of Denver, sponsored for this pilot-scale study in collaboration with Black & Veatch, operates mesophilic anaerobic digesters for stabilization of PS and a combination of conventional WAS and pure oxygen WAS produced at the District's wastewater treatment plants. The digestion facilities at the plant have periodically experienced operational problems associated with excessive foaming in the winter months and the District has been exploring options to alleviate digester foaming. The foaming episodes have been attributed mainly to low seasonal PS:TWAS ratios, the way these two solids streams are fed to the digesters, and to the heterogeneous nature of the WAS component in the feed solids since the District is treating WAS fractions originating from different sources. Therefore, pre-treatment of WAS prior to digestion seemed a promising option for the District to help address the foaming issue.

Therefore, this study has primarily established ultrasound disintegration as one of the many methods for the pretreatment of thickened waste activated solids (TWAS) to control foaming in mesophilic anaerobic reactors. A secondary objective of the study has been to evaluate the performance of ultrasound-conditioned (sonicated) TWAS for minimizing the retention time and enhancing the destruction of volatile solids.

In spite of being an effective treatment technology for achieving more complete digestion and enhancing volatile solids reduction with observing a decrease in occurrences of foaming incidents, the ultrasound disintegration has not found recognition as a pre-treatment method for TWAS. The mesophilic reactor operated with conditioned TWAS could neither satisfy for controlling foaming problem nor improve for enhancing the destruction of volatile solids.

Recommendations for Future Study

The issues stated below are recommended for future research:

1. Effect of loading rates at constant retention time: It would be interesting to study the effect of organic loading rates if hydraulic retention time remains constant. It is also feasible to keep the organic loading rate constant by reducing the retention time. Reports have shown that organic loading rates can be increased by 20-50% while keeping hydraulic retention times constant with ultrasound. If organic loading rates are kept constant, then hydraulic retention times can be decreased by approximately 30% for similar performance.

2. How does influent dry solids content determine the energy balance due to ultrasound? Reports have showed that energy increase due to ultrasound is greatly improved as the dry solids content of the digester influent is increased. An evaluative comparison with this study would provide valuable information on the economics of operation of the system.

3. The effect of ultrasound pre-treatment on raw solids combined primary and secondary solids with fractions of PS:WAS ratios at different retention times: Ultrasound pre-treatment may be beneficial with an appropriate fraction of PS:WAS ratio at shorter retention times.

Conclusions

The Ultrasound pre-treatment method is a viable alternative for promoting the rate limiting stage of solids hydrolysis. If operated in an appropriate condition, the process can also satisfy to enhance anaerobic digestion process.

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