# ANAEROBIC DIGESTION OF WASTE ACTIVATED SLUDGE WITH ULTRASONIC PRETREATMENT

by

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#### Abstract

Ultrasonication of waste activated sludge is usually applied in the enhancement of sludge solubilization and sludge disintegration. This research examines the effectiveness of ultrasound pretreatment on waste activated sludge (WAS) disintegration at different specific energies and sonication durations in the anaerobic digestibility of control, full stream and part stream (50% sonicated + 50% nonsonicated sludge) sonicated WAS. A specific energy of 12 kWs/gTS was the maximum specific energy for the effective disintegration of WAS. The mesophilic digester was operated with SRTs of 20 and 15 days. The ultrasonic pretreatment enhanced the subsequent anaerobic digestibility resulting better removal of TS and VS. The biogas production rate in the full stream and part stream digesters was increased by 102% and 91% respectively, for a SRT of 20 days in comparison to the control digester, whereas at an SRT of 15 days, the biogas production rate was increased in the full stream and part stream digesters by 81.4% and 57.1% respectively. Although, the specific biogas production was the same in full stream and part stream digesters for both SRTs, which is 0.500 L/g VS removed in a SRT of 20 days and 0.64 L/g VS removed in a SRT of 15 days respectively. The dewaterability of the sludge was ranked in the ascending order as full stream, part stream, control digester sludge and raw sludge. Based on the energy balance, energy gained from biogas in the full stream is around two folds compared to energy gained from biogas from control digester. In part stream digester, around 88% of sonication energy input can be replenished in the form of biogas energy. The ultrasonic pretreatment improved the VS hydrolysis coefficient in the full and part stream digesters for both SRTs.

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# **List of Abbreviations**

APHA	American Public Health Association
BMP	Biochemical Methane Potential
BOD	Biochemical Oxygen Demand (mg O <sub>2</sub> /L)
CFR	Code of Federal Regulations
COD	Chemical Oxygen Demand (mg O <sub>2</sub> /L)
CST	Capillary Suction Time
$d_{50}$	Cut diameter (µm)
DD	Degree of Disintegration
DD <sub>COD</sub>	Degree of Disintegration in term of COD
DD <sub>OUR</sub>	Degree of Disintegration in term OUR
DS	Dry Solids
ECD	Electron Capture Detector
EPS	Extracellular Polymetric Substance
FFAP	Free Fatty Acid Phase
FID	Flame Ionization Detector
MPN	Most Probable Number
NTU	Nephelometric Turbidity Unit
OCSD	Orange Country Sanitation District
PN	Protein
PP	Peak to Peak
RF	Radio Frequency
RMS	Root Mean Square
SCOD	Soluble Chemical Oxygen Demand (mg O <sub>2</sub> /L)
SE	Specific Energy (kJ/kg TS)
SEM	Scanning Electron Micrographs
SOUR	Soluble Oxygen Uptake Rate (mg DO/gVSSh)
SRT	Solids Retention Time
S.T.P	Standard Temperature and Pressure
TKN	Total Kjeldhal Nitrogen
TS	Total Solids
TWAS	Thickened Waste Activated Sludge
VFA	Volatile Fatty Acid
VS	Volatile Solids
WAS	Waste Activated Sludge

### Chapter 1

#### Introduction

### 1.1 Background

The activated sludge process is the most widely used biological process for domestic wastewater treatment. During aerobic biological treatment, organic pollutants are mineralized into carbon dioxide and water with the generation of excess bacterial biomass commonly known as waste activated sludge (WAS). The activated sludge process generates a significantly high amount of sludge because biomass yield in aerobic biological treatment is 0.4 gVSS/gCOD. As a result of the quantitative and qualitative expansion of wastewater treatment plants over time, the production of WAS has also increased. In the same time, disposal routes are subject to more legal and social constraints, and incineration is quite expensive. The treatment, handling and disposal of this excess sludge amounts to up to 60% of the total wastewater treatment plant operating costs (Weemaes and Verstraete, 1999). Although sludge is rich in nutrients, it is not vet generally accepted for use as a fertilizer for agricultural purposes. The resistances from the farming industry concerns mostly fear of heavy metals and other presumably toxic compounds. As long as no definitive solution to the sludge problem exists, it is necessary to reduce sludge production to the source that is to say in the wastewater treatment plant. Means of minimizing the amount of sludge are highly interesting. Therefore, minimization of the amount of sludge produced coupled with the generation of value-added products from the sludge is the best strategy for sustainable sludge management.

Biological methods such as aerobic and anaerobic digestion are widely used for sludge stabilization, which not only reduces the quantity of sludge to be disposed off, but also produces valuable methane gas (during anaerobic digestion), high quality biosolids for land application, and as a carbon source for denitrification. Aerobic digestion is not economically viable to treat large amount of sludge, because large capacity aerator consumes a lot of power resulting in high operating cost. Therefore anaerobic digestion is the most often applied in medium and large wastewater treatment plant and is generally applied to mixture of primary and secondary (waste-activated) sludge, but waste-activated sludge (WAS) is known to be more difficult to digest than primary sludge (Lafitte-Trouque and Forster, 2002). Therefore, the organic fraction of excess activated sludge is only about 30-45 % digestible in conventional anaerobic digester (Tasuo et al., 1993; Harrison et al., 1991) and the anaerobic stabilization is a slow process. Therefore, long residence times in the digester and large digester volumes are required. Anaerobic degradation of particulate organic matter and macromolecules follow series of four steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. In the case of sewage digestion, the biological hydrolysis has been identified as the rate limit step. Therefore, the pretreatment of sewage sludge by mechanical, chemical, or thermal disintegration can improve the subsequent anaerobic digestion. When sludge disintegration is employed, the hydrolysis reaction is accelerated. Hence, the sludge retention time in the anaerobic digestion reactor can be reduced from the standard of 15-30 days. As a consequence, the digester volume can be reduced, which leads to savings in investment and operating costs, which allows more compact plants. Finally, due to the destruction of filamentous bacteria sludge flocs are deagglomerated during disintegration

Thus, the biological sludge needs to be pretreated to enhance the digestibility. The aim of such pretreatment is to rupture the cell wall and membrane to the release the intra and extra cellular matter into the aqueous phase for subsequent degradation, and this process is called sludge disintegration. These pretreatments include physical (ball milling, ultrasonic, etc.), chemical (ozone, hydrogen peroxide, acid and base), thermal and biological (enzymatic hydrolysis).

Important parameters such as duration of sonication, power input, TS content and operating frequency need to be optimized to maximize sludge disintegration in ultrasonic systems and the use SOUR could be a new tool to evaluate the progress with bacterial cell disintegration (Khanal et al., 2006c). Efficiency of ultrasonication is also dependent on the specific energy of sludge. For ultrasonic specific energy between 0 and 7000 kJ/kg TS, biogas production increased with energy supplied, but for energy supplied of 7000 and 15,000 kJ/kg TS, biogas production was almost the same. The optimum ultrasonic energy was thus about 7000 kJ/kgTS (Bougrier et al., 2005). Nevertheless, Benabdallah El-Hadj et al. (2006) had found that Optimum specific energy of 11,000kJ/kgTS promoted the best solubilization yield and enhanced biogas production in the subsequent anaerobic stabilization under both mesophilic and thermophilic conditions. Therefore, no specific conclusion can be arrived at regarding the value of optimum specific energy.

Although some technical installations have already been introduced, taking advantage of active reaction of ultrasonic field for the preparation of sludge prior to anaerobic stabilization, keeping in mind the mechanisms of the process and methods to control the run of the process, the technology in question is still in the experimental phase. Efficiency of ultrasonication depends on several parameters such as specific energy input and ultrasonic density; sonication period, TS contents of sludge, temperature needs to be controlled during ultrasound disintegration, and pH of sludge to maximize sludge disintegration prior to testing the sludge for anaerobic digestibility. Many researchers found the correlation between Efficiency of ultrasonication and theses parameters, so war there is a lack of information as to how different degrees of sludge disintegration impact on the digestion process (Yoon et al., 2004; Tiehm et al., 2001). However, no conclusion can be arrived at regarding the predominant parameter. At present, no mathematical model that gives relationship between efficiency of ultrasonication and each parameter has been found. Therefore Statistical analysis has to be applied to elucidate the relative significance of each factor.

Many of researchers had observed that ultrasonic disintegration of activated sludge could improve the dewaterability of sludge, but Wang et al. (2006) had found that the dewaterability is increasingly reduced with increasing ultrasonic density and time. Therefore, dewaterability after sonication and digestion has to be determined more in this study to find out influence of ultrasonication on dewaterability of waste activated sludge. On account of digestion for sonicated sludge cause the end product that shows a substantially better biological stability than the digested sludge without sonication. Therefore, land-fill disposal and limited agricultural use as a fertilizer are possible. Ultrasonication controls pathogen in the digested sludge in term of *E. coli* levels and density of *Salmonella sp.* Effects of sonication on biosolids quality have to be determined using optimum sonication condition. A cost-benefit analysis of ultrasonic integrated systems needs to be conducted to justify the economics of the process in full-scale applications.

### **1.2** Objectives of the Study

The main goals of this study was to investigate ultrasonic disintegration of thickened WAS and to evaluate anaerobic digestibility of ultrasonic pretreated WAS. The specific objectives include the following:

- 1. To optimize ultrasonic pretreatment to maximize WAS disintegration.
- 2. To examine the anaerobic digestibility of full-stream (100% sonicated) and part stream (50% sonicated and 50% non-sonicated) WAS at different solids retention time.
- 3. To evaluate the quality of ultrasonic pretreated WAS following digestion with respect to dewaterability and pathogen count.
- 4. To determine the hydrolysis coefficient for both sonicated and non-sonicated sludge during anaerobic digestion.

### 1.3 Scope of the Study

This study was based on laboratory scale experimental research that includes:

- 1. The sewage waste activated sludge used in this research was sampled from the Thammasat domestic wastewater treatment plant located in Pathumthani.
- 2. Semi-continuous feeding will apply to anaerobic reactor two times per day in equal time intervals. Solids retention time of the anaerobic reactors was selected as 15, and 20 days.
- 3. TS content of sample sludge was increased to 3% by centrifugation.

# Chapter 2

### Literature review

### 2.1 Introduction

The domestic waste water is mostly treated by biological process such as activated sludge process, aerobic pond, and anaerobic treatment. Waste activated sludge process is more efficient technology to meet stringent standard. It results in the generation of a considerable amount of activated sludge that has to be wasted (Weemaes et al., 2000). The expense for excess sludge treatment has been estimated to be up to 60% of the total operating cost of a wastewater treatment plant (Egemen et al., 2001). Moreover the conventional disposal method of landfilling causes secondary pollution problems. Therefore, an interest in methods to reduce the volume and mass of excess sludge has been growing rapidly. For the purpose of reducing the volume of sludge, anaerobic digestion has been widely used. Before study anaerobic treatment, Characteristics and types of sludge produced in biological treatment plant should be known.

## 2.2 Types and Characteristics of Sludge

### 2.2.1 Primary sludge

Primary sludge is essentially raw waste which comes from the bottom of the primary clarifier. It is putrescible and must be stabilized before being disposed off (Liu and Liptak, 1999). In comparison with activated sludge, primary sludge generally contains more fat and protein and less carbohydrates (Sykes, 2003). Because of this, the gas yield is higher, but the methane content of the gas is lower. Primary sludge is easily digestible compared to activated sludge.

### 2.2.2 Activated sludge

Activated sludge comes from the secondary treatment. The excess sludge is called waste activated sludge and is a result of overproduction of microorganisms in the active sludge process. It is light and fluffy and composed of microorganisms flocculated organic matter (Liu and Liptak, 1999). The organisms are primarily bacteria and protozoa, but also rotifers and filamentous bacteria. Activated sludge is more difficult to digest than primary sludge. Filamentous bacteria are a normal part of the activated sludge microflora (Bitton, 1999). If the process is run suboptimally the filamentous bacteria can increase in numbers and cause foaming of the active sludge process. A high number of filamentous bacteria in the waste activated sludge can also cause foaming of anaerobic digesters. Common species are *Nocardia* spp and *Microthrix parvicella*.

### 2.2.3 Digested sludge

After anaerobic digestion of primary and activated sludge, the residual product is digested sludge. The digested sludge is reduced in mass, less odorous, and safer in the aspect of pathogens (Bitton, 1999) and more easily dewatered than the primary and activated sludges (Liu and Liptak, 1999).

### 2.3 Anaerobic Sludge Digestion

Anaerobic digestion is the biochemical process by which organic matter is degraded by microorganisms in the absence of oxygen. In sewage treatment it has primary served the purpose of sludge stabilization and sludge volume reduction.

### 2.3.1 History of anaerobic digestion

In the nineteenth century, the microorganisms responsible for the anaerobic process were first described by Pasteur (Hughes, 1980). It was also concluded that methane forms from the biological breakdown of cellulose (Klass, 1984). For the last hundred years anaerobic digestion systems much like the ones seen today have been used for waste disposal and stabilization (Klass, 1984). As of today anaerobic digestion is the most commonly applied method of treatment for sewage sludge (Tiehm et al., 1997; Gronroos et al., 2005).

### 2.3.2 Microbiology of anaerobic digestion

Anaerobic digestion is a multi-step process carried out by a mixed culture of different groups of microorganisms. The process consists of four main steps which are carried out by at least three groups of microorganisms: acidogenic bacteria, acetogenic bacteria and methanogenic bacteria (Ecke and Lagerkvist, 2000; De Mes et al., 2003). Figure 2.1 summarizes the process. Organic matter consists of particulate, water-insoluble polymers such as carbohydrates, lipids and proteins. Insoluble polymers cannot penetrate cellular membranes and are therefore not directly available to the microorganisms. In the first step, hydrolysis, acidogens excrete hydrolytic enzymes which break up the insoluble polymers to soluble mono- and oligomers. Carbohydrates are converted to sugars, lipids are broken down to long-chain fatty acids and proteins are split into amino acids. These soluble molecules are, through the acidogenesis, converted by acidogens to acetic acid and other longer volatile fatty acids, alcohols, carbon dioxide and hydrogen. During the acetogenesis the longer volatile fatty acids and alcohols are oxidised by proton-reducing acetogens to acetic acid and hydrogen. In the last step, methanogenesis, methanogenesis use acetic acid or carbon dioxide and hydrogen to produce methane and carbon dioxide.



Figure 2.1 Summary of the anaerobic digestion chain

For mesophilic bacteria, the optimal methane production rate is mostly reached at 35-37°C. The thermophilic methanogens differ from the mesophilic one and their maximum methanogenic activity is reached at about 55°C. A thermophilic digestion process can sustain a higher organic loading compared to a mesophilic one. But the thermophilic process produces a gas with a lower methane concentration (Ecke and Lagerkvist, 2000) and is more sensitive to toxicants (Bitton, 1999). Methanogens are more sensitive toward changes in temperature than the other species, because of their slower growth rate in the reactor environment. In all four digestion steps only the methanogenesis is critically pH dependent (De Mes et al., 2003) in the digestion of sewage sludge. Methanogenesis occurs at neutral pH; in the range of 6.5–7.5, although optimum lies at pH 7.0–7.2 (Bitton, 1999). If, for example, a temperature shift affects the methanogens negatively there can be a build up of VFAs. This lowers the pH which further affects the methanogens in a negative way which leads to a vicious circle of negative feedback.

The digestion efficiency and its stability can vary significantly depending upon the mode of operation, waste type, digestion temperature, digestion design, and other factors. The longer a substrate is kept under proper reaction conditions the more complete its degradation will become. But the reaction rate will decrease with increasing residence time. The disadvantage of a longer retention time is the increasing reactor size needed for a given amount of substrate to be treated. A shorter retention time will lead to a higher production rate per reactor volume unit, but a lower overall degradation. These two effects have to be balanced in the design of the full scale reactor.

### 2.3.3 Criteria of Digestion

**Loading rate:** The system's design will dictate loading rates and contents, but experience indicates that uniform loading, on a daily basis, of WAS with 2-6% solids generally works best. The load's retention time in the digester will typically range from 15 to 30 days.

**Mixing:** The loaded sludge needs to be mixed regularly to prevent settling and to maintain contact between the bacteria and the sludge. The mixing action also prevents the formation of scum and facilitates release of the biogas.

**Nutrients:** The best digestion occurs with a carbon / nitrogen ratio between 15:1 and 30:1 (optimally 20:1).

**Management:** Anaerobic digesters require regular and frequent monitoring, primarily to maintain a constant desired temperature and to ensure that the system flow is not clogged. Failure to properly manage the digester's sensitivity to its environment can result in a significant decline in gas production and require months to correct.

**Safety:** Working with anaerobic digester biogas, and especially with methane, the major component of the gas warrants extreme caution. Methane, when mixed with air, is highly explosive. In addition, because digester gas is heavier than air, it displaces oxygen near the ground, and if hydrogen sulfide is still present, the gas can act as a deadly poison. It is critical that digester systems be designed with adequate venting to avoid these dangerous situations.

**Storage:** Because of the high pressure and low temperature required, it is impractical to liquefy methane for use as a liquid fuel. Instead, the gas can be collected and stored for a

period of time until it can be used. The most common means of collecting and storing the gas produced by a digester is with a floating cover-a weighted pontoon that floats on the liquid surface of a collection/storage basin. Skirt plates on the sides of the pontoon extend down into the liquid, thereby creating a seal and preventing the gas from collection basin coming into contact with the open atmosphere. High-pressure storage is also possible, but is both more expensive and more dangerous and should be pursued only with the help of a qualified engineer.

## 2.3.4 Biogas production and Composition

Pafaranaa	Gas production (mL/g VS)					
Reference	Primary sludge	Activated sludge				
Sato et al. (2001)	612	380				
Speece (2001)	362	281				
Rittmann and McCarty (2000)	375	275				

Table 2.1 Gas production from primary sludge and activated sludge

Source: Cited in Brown et al. (2003)

### 2.3.5 Utilization of biogas

Biogas can be used for heat production, co-generation of electricity and heat or be upgraded to motor vehicle fuel. Generation of heat and/or electricity in a gas boiler, gas engine, gas turbine or fuel cell system can be accomplished with the methane content normally reached in a digester (55–75 % (de Mes et al., 2003)). For use as motor vehicle fuel the methane content has to be increased to at least 96–97 %. Biogas of vehicle fuel quality has the same methane concentration as natural gas and can be co-distributed in a natural gas network.

### 2.3.6 Enhancement of Anaerobic Digestibility

Anaerobic digestion is the most applied technique for sewage sludge stabilization resulting in the reduction of sludge volatile solids and the production of biogas. The anaerobic stabilization is a slow process. Therefore, long residence times in the digester and large digester volumes are required. Anaerobic degradation of particulate material and macromolecules is considered to follow a sequence of four steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. In the case of sewage sludge digestion, the biological hydrolysis has been identified as the rate-limiting step (Eastman and Ferguson, 1981; Shimizu et al., 1993). Therefore, pre-treatment of waste activated sludge has been developed to improve anaerobic digestion by mechanical, chemical, or thermal disintegration can improve the subsequent anaerobic digestion (Chiu et al., 1997; Dohanyos et al., 1997; Hiraoka et al., 1985; Mueller et al., 1998).

### 2.4 Pretreatments

The rate-limiting step of anaerobic digestion of waste activated sludge is hydrolysis (Tiehm et al., 1997), e.g. break up of cell walls and disintegration of sludge flocs. A pretreatment step would render hydrolysis less difficult, thus giving a more efficient process. Several pretreatments processes are developed such as ultrasound, enzyme

addition, ozonation, chemical solubilization by acid or base addition According to (Tiehm et al., 1997), microwave irradiation (Eskicioglu et al., 2006), thermal pretreatment (Camacho et al., 2002), mechanical disintegration (Nah et al., 2000). Although the methods were different, the aim of them was the release of the organic substances inside and outside the cells in the sludge solids into liquid phase, and this process was called sludge disintegration. After the sludge was disintegrated, the soluble chemical oxygen demand (SCOD) increased. The product can be utilized both as a substrate in aerobic and anaerobic biological processes. Pretreatments are classified into four categories given below;

- 1. Mechanical: Ultrasound, Homogenizer, stirred ball mills;
- 2. Thermal: Thermal hydrolysis (autoclave or steam heating), Wet oxidation;
- 3. Chemical: Use of enzymes, Alkaline/Acid hydrolysis;
- 4. Biological: Thermophilic Aerobe/Anaerobe pretreatment.

The anaerobic digestion rate and the biodegradability of sludge solids can be improved by pretreatment resulting in solids solubilization. Positive effects were shown for thermal pretreatment, addition of enzymes, ozonation, chemical solubilization by acidification (Woodard & Wukasch, 1994), alkaline hydrolysis, and mechanical sludge disintegration (Muller et al., 1998; Kopp et al., 1997).

### 2.4.1 Mechanism of ultrasonication



Ultrasound is above the human audible range, part of the sonic spectrum that ranges from 20 kHz to 10 MHz, is generated by a transducer in the Ultrasonic device that converts mechanical or electrical energy into high frequency acoustical (sound) energy. The sound energy is then fed to a horn that transmits the energy as high frequency vibrations to the liquid being processed. A wave propagates in a liquid through alternating cycles of compression and rarefaction.

Ultrasonic disintegration is a well-known method for the break-up of microbial cells to extract intracellular material (Harrison, 1991). The impact of ultrasound waves on a liquid causes the periodical compression (Positive pressures) and rarefaction (negative pressures) of the medium. Micro bubbles occur above a certain intensity threshold in rarefaction due to the negative pressure. These micro bubbles also known as cavitation bubbles, essentially containing vaporized liquid and gas that was previously dissolved in the liquid. As the wave fronts propagate, microbubbles oscillate under the influence of positive pressure. Cavitation bubbles first grow in size until reach resonant bubble size within a few microseconds and then violently collapse when they reach its critical size is shown in the figure 2.2 and Cavitation bubble is shown in the figure 2.3.



Figure 2.2 The illustration shows how a cavity builds up successively until it implodes

The violent collapse produces very powerful hydromechanical shear forces in the bulk liquid surrounding the bubble. It has been shown that macromolecules with a molar mass above 40,000 Dolton are disrupted by the hydromechanical shear forces produced by ultrasonic cavitation. The mechanical forces are most effective at frequencies below 100 kHz (Portenlanger, 1999). In implosion phase, the temperature increases to 5 000 K and the pressure increases to 500 bar within the cavity. When the cavity implodes near a wall, a jet beam is sent out against the wall with a speed up to 100 m/s.



Figure 2.3 Cavitation Bubble

These extreme conditions can lead to the thermal destruction of compounds present in the cavitation bubbles and to the generation of very reactive radicals (H°, HO<sub>2</sub>° and OH°) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) (Mason, 1991; Young, 1989). In this way sonochemical reactions can degrade volatile pollutants by pyrolytic processes inside the cavitation bubbles and non-volatile pollutants by hydroxyl radical reactions in the bulk liquid (Petrier and Francony, 1997; Tiehm, 1999). While sonochemical degradation processes can occur in a broad ultrasound frequency range from 20 kHz up to about 1 MHz the highest efficiency of sonochemical reactions was observed at more than 100 kHz (Hua and Hoffmann, 1997; Petrier and Francony, 1997). Both the hydromechanical shear forces and the sonochemical effects can contribute to the ultrasonic disintegration of sewage sludge. These extreme temperatures and pressures, which last only microseconds, do not exist long enough to heat the liquids being processed. However, the localized temperature and pressure increases are sufficient to increase chemical reactivity, polymer degradation, and chemical free-radical production.

### 2.4.2 Generating ultrasound



Figure 2.4 Details of typical 20 kHz piezo-electric ultrasonic system

Ultra Sonix uses piezoelectric ceramics to generate ultrasound. When a piezoelectric ceramic is affected by an electrical field the dimension of the ceramic is altered. The repetition of the increase respective decrease of the ceramics dimension is created by the electrical fields changing polarity. The changes in the ceramics dimensions create ultrasound of a specific frequency. Three major components of an ultrasound system are the converter (transducer), booster and horn (sonotrode). A converter basically converts electrical energy into ultrasound energy (vibration). The booster is a mechanical amplifier that helps to increase the amplitude generated by the converter. The horn is a specially designed tool that delivers the ultrasonic energy to the sludge. Figure 4 shows the arrangement of converter, booster and horn in a typical ultrasound system. The booster is designed and tuned to operate at a desired frequency. The booster often acts as a mounting component. For example, in order to hold or affix the stack assembly, the stack is clamped at nodal points. The two most common places to clamp the stack assembly are either at the converter or booster nodal ring. Similar to the booster, the horn, which delivers the motion to the sludge, often amplifies the motion even further. In addition, the horn is usually half a wavelength long, but full wavelength designs are also common depending on the application.

Thus, both booster and horn are designed to magnify the amplitude of the ultrasonic motion (built-in gain). For example, a large contact area is usually desired in sludge processing. However, this limits the possibility of a built-in gain, and thus a booster (mechanical gain device) is often placed between the converter and the horn. Such arrangement is required because the typical displacement of the ultrasonic energy is 20  $\mu$ mpp, and this is not sufficient to efficiently process the sludge where amplitudes greater than 50  $\mu$ mpp are often employed. The gain of the booster (and horn) can be approximated

by evaluating the mass above and below the nodal plan (area with no motion). Because of equilibrium and compatibility, the forces (F) above and below the nodal plan must be equal. However, when the mass  $(M_1)$  above the nodal plan is different from the mass below the nodal plan  $(M_2)$ , the accelerations  $(a_1, and a_2)$  must also be different, resulting in different amplitudes of motion.

$$F_1 = F_2 \Longrightarrow m_1 a_1 = m_2 a_2 \Longrightarrow \frac{a_1}{a_2} = \frac{m_1}{m_2}$$
 (Eq. 2.1)

One of the major design criteria of the horn is uniformity of amplitude. For horns with relatively small faces this is usually not an issue, but with large block horns (see Figure 4), uniformity can be difficult to obtain. Often masses are added to the back drive of these horns or undercuts are made near the face of the horn where uniformity drops off. In addition, with larger horns, the so- called "Poisson" effect can produce undesired modes of vibration. In this case, the material expands outward because of the materials' Poisson ratio. There are many possible designs for horns; but some typical designs are shown in Figure 2.5.



Figure 2.5 Examples of common horns used for liquid processing

### 2.4.3 Quantification of energy or power input to sludge

The economy of an ultrasound system is primarily governed by the power (W or kW) or energy (J or kJ) input needed to achieve effective sludge disintegration. Thus, quantification of energy/power input to obtain a desired degree of disintegration is critical to evaluate the relative efficiency of ultrasound systems. This will be a key factor in selecting the ultrasound system for field application. The power or energy input needed to obtain a desired degree of sludge disintegration depends on both sludge characteristics (e.g., TS content, sludge viscosity, organic fraction, nature of sludge, i.e., fraction of primary and secondary sludge to be sonicated, etc.) and design of the ultrasound system (horn, booster and converter). Currently no rational model is available that accounts for all of these factors. Therefore, all ultrasound systems need to be laboratory and field-tested to examine their energy efficiency before full-scale installation. The power or energy supplied for sludge disintegration can be expressed in a number of ways as elucidated below:

#### **Specific energy input (SE):**

It is defined as the energy supplied per unit of mass of sludge solid (as TS) to achieve a certain degree of disintegration. The specific energy input is a function of ultrasonic power, ultrasonic duration, volume of sonicated sludge and TS concentration, and can be calculated using the following equation (Bougrier et al., 2005):

$$SE = \frac{P \times T}{TS \times V}$$
 (Eq. 2.2)

Where,

SE = Specific energy input in kJ/kg TS

P = Ultrasonic power in kW

t = Ultrasonic duration in second (s)

V = Volume of sonicated sludge in liter (L)

TS = Total solids concentration in kg/L

#### **Ultrasonic dose:**

*Ultrasonic dose* = 
$$\frac{P \times t}{V}$$
 (Eq. 2.3)

It relates to the amount of energy supplied per unit volume of sludge and is expressed as J/L or kJ/L. However, it does not depend on total solid concentration. The ultrasonic dose can't use to compare the sludge with different TS content. As long as the TS content remains fairly constant, the ultrasound density is a practical method of expressing the energy input for the disintegration of sludge on a volume basis.

#### Ultrasonic density:

*Ultrasonic density* = 
$$\frac{P}{V}$$
 (Eq. 2.4)

It relates to the power supplied per unit volume of sludge and has a unit of W/ml. Ultrasound density also relates power input to the volume of sludge, similar to ultrasound dose. However, ultrasound density does not take into account the sonication duration.

#### **Ultrasonic Intensity:**

*Ultrasonici Intensity* = 
$$\frac{P \times t}{A}$$
 (Eq. 2.5)

Where A = Converter surface area (cm<sup>2</sup>)

It relates to power supplied to sludge per unit of converter surface area and is expressed as  $W/cm^2$ . Ultrasonic intensity therefore reflects the power generating capacity of the converter. When amplitude of the ultrasound emitted by horn increases, power supplied to the sludge will increase.

### 2.5 Ultrasonication

Ultrasonication is a novel pretreatment to treat the sludge. In real application, Ultrasonication is used in the two locations as shown in the figure 2.6. First location is on excess sludge before entering to anaerobic digestion. When sludge is subjected to ultrasonication, temperature of sludge is increased. If the excess sludge is thermally treated prior to further dewatering, these thermal effects generally have a positive impact in dewaterability and biosolids quality (Neyens and Baeyens, 2003). Second location is especially to reduce the number of filamentous organisms in recycled sludge (in case of bulking sludge). In this location, Excess temperature due to ultrasonication should be avoided to prevent all organisms to be destructed. Because when WAS is recycled to the biological treatment, Active bacteria should be maintained in Aeration tank.



Figure 2.6 Location of ultrasonication in the sludge treatment

The mechanism of cavitation plays an important role when applying ultrasounds to WAS. Effects of cavitation generated in sludge can be summarized as follows:

- High mechanical shear stress
- ▶ Radical reactions: creation of OH° and H° radicals;
- Chemical transformation of substances
- Thermal breakdown of volatile substances

These effects yield disintegration of sludge resulting as minimum sludge production, as a carbon source for denitrification, and the improvement of subsequent anaerobic digestion and dewaterabilty.

### The primary benefits are:

- Enhanced sludge destruction rates
- Increased biogas production
- Reduced solids production
- Increased dewaterability of final sludge cake
- Increased sludge loading rates (due to increased reaction times)
- Improved digester operation and stability

#### 2.6 **Evaluation of Ultrasound Disintegration Efficiency**

Ultrasound pretreatment is destroyed the cell wall of microbes and release the intracellular materials to the aqueous phase. In addition, ultrasound also helps to deagglomerate the biological flocs and disrupt large organic particles into smaller-size particles. It gives the changes in physical, chemical and biological properties of WAS. Therefore it is necessary to Quantify degree of disintegration of sludge. So far, data obtained from researches is not sufficient to fit the model to predict sludge disintegration. Because, Degree of Disintegration depends many variables associated with ultrasound pretreatment. Some of the variables are operating frequency, horn, booster and converter designs, types of sludge, TS content, organic fraction, operating temperature, ultrasonic density (or power density), etc. Horn design is considered to be one of the most important factors affecting the degree of sludge disintegration and its design is often a proprietary. This makes it often harder to quantify many important operating conditions of ultrasound systems.

The quantitative data provides much valuable information, such as:

- Efficiency of a selected ultrasound system, (converter, booster and horn design);
- Assessment of minimum energy input needed for cell rupture;
- Various optimal operating data (TS content, sonication duration, ultrasonic density, frequency, and amplitude, etc.) to maximize sludge disintegration;
- Overall operating cost of ultrasound system for sludge disintegration.

Different parameters have been employed to evaluate sludge disintegration efficiency. They can be collectively classified into three categories namely, physical (such as change in particle size distribution and microscopic examination), chemical (such as increase in soluble COD concentration and ammonia concentration, and release of protein) and biological (oxygen uptake rate and heterotrophic count).

### 2.6.1 Physical evaluation

Particle size analysis, microscopic image, turbidity, and sludge dewaterability are some of the techniques used to judge the Degree of ultrasonic disintegration. Physical evaluation, especially particle size distribution and microscopic image analysis have been widely employed for simplicity as qualitative measures of sludge disintegration.

### Particle size analysis

Tiehm et al. (2001) evaluated sludge disintegration in terms of change in medium particle size and turbidity at different ultrasound frequencies. The authors found the lowest medium particle size of about 20 µm and the highest turbidity of 120 NTU at a frequency of 41 kHz. There was no mention of medium particle size and turbidity without sonic treatment. Chu et al. (2001) also examined the effect of different sonication densities and times on floc size at frequency of 20 kHz and maximum power input of 110W. During 120 min of sonication, the floc size did not decrease at sonication density of 0.11 W/mL. When the sonication density increased to 0.22 W/mL, the floc size started to decrease. At higher sonication densities of 0.33 and 0.44 W/mL, the floc size decreased from 99 µm to about 22 and 3  $\mu$ m, respectively during 20 min of sonication. The authors observed no decrease in floc size beyond 3 µm with further sonication up to 120 min. Thus, the ultrasonic density is more important than the sonication time for effective sludge disintegration. Bougrier et al. (2005) examined the particle size distribution at different specific energy

inputs (kJ/kgTS) using ultrasonics at 20 kHz frequency and supplied power input of 225 W. The cut diameter,  $d_{50}$  (that is 50% of the particles (by volume) with diameter equal or lower than  $d_{50}$ ) of sludge particles showed a decreasing trend with increase in specific energy input as shown in the table 2.2. In another study, Tiehm et al. (1997) also showed that the  $d_{50}$  of sludge particles decreased from 165 µm (unsonicated) to 135 and 85µm during 29.5 and 96 sec of sonic treatment at a frequency of 31 kHz and power input of 3.6W.

Specific energy input (kJ/kgTS)	Cut Diameter, $d_{50}$ (µm)
0	32
660	19.6
1350	18.5
6950	17.6
14550	12.7

Table 2.2 Particle size variation with specific energy input

### Microscopic image evaluation

The sludge disintegration has widely been examined based on visual observation using light and electron microscopes. A light-based micrograph depicts qualitative information, such as structural changes in flocs, disappearance of filaments, etc., during ultrasonic treatment (Khanal et al., 2006b). However, the light microscopic image does not provide information at the cellular level. When WAS was observed under a light microscope, floc-like structures entangled within a large numbers of filaments were seen prior to sonication. Within two minutes of sonication, the filaments and flocs were almost completely disintegrated and a more or less homogeneous texture was observed.

Scanning electron micrographs (SEM) provide more thorough information on sludge disintegration particularly at the cellular level as depicted in (Khanal et al., 2006c). Prior to sonication, flocs entangled within large numbers of filaments were observed (Figure 2.7A). These filaments-like structures are essentially organic debris (with diameter less than one-fourth of a micron) attached to the flocs. During 2 min of sonication, the structural integrity of flocs as well as filaments was significantly disrupted without appreciable destruction of bacterial cells as seen in Figure 2.7 B.



Figure 2.7 Microscopic observation of WAS; (A) before sonication; (B) after 2 min of sonication at constant power input of 1.5 kW and frequency of 20 kHz, (1000 x)

At a longer sonication duration of 10 min, nearly complete disintegration of flocs and filament-like structures with a very few scattered bacterial cells was observed (Figure 2.8C). When the sludge was sonicated for 30 min, more or less complete break-up of cell walls was observed with several punctured cells (Figure 2.8D).



Figure 2.8 SEM images of undigested WAS at different sonication duration with constant power input of 1.5 kW and frequency of 20 kHz: (A) 0 min (control); (B) 2 min; (C) 10 min; and (D) 30 min

### 2.6.2 Chemical evaluation

Chemical evaluation is far more quantitative for measuring sludge disintegration than physical. It primarily measures the solubilization of WAS in the aqueous phase. In environmental engineering, all released organic matter is lumped together and measured as an increase in soluble chemical oxygen demand (SCOD). However, it is important to note that ultrasonic pre-treatment also disintegrates extracellular matter including organic debris and Extracellular Polymeric substances (EPS), which also become part of SCOD. Thus, SCOD is a gross parameter to quantify the sludge disintegration.

### a) SCOD assessment

Many of research studies presented the SCOD increase with respect to sonication duration, which makes it harder to compare one study to the other. This is because ultrasonic disintegration depends on several factors such as TS content, frequency, sludge type, ultrasonic density, temperature, duration of sonication, etc., and such information is not well described in the literature. For better comparison, the SCOD release needs to be correlated with the specific energy input (see equation 2). Figure 2.9 shows a typical SCOD increase pattern at different specific energy inputs (Khanal et al., 2006a). As

evidence from the figure, better disintegration of biological and non-biological solids was achieved (as evident from continuous increase in SCOD) at a longer sonication time, and thus at a higher specific energy input. This is because at longer sonication time or at higher specific energy input, there was ample opportunity for cells and debris to come under perpetual attack of large numbers of collapsing cavitation bubbles. However, the release in SCOD slowed down at an energy input of over 35 kJ / g TS. This is most likely due to exhaustion of readily disintegrable biological and non-biological organic particles in the vicinity of the collapsing cavitation bubbles or exhaustion of dissolved gases that aid cavitation bubble formation. Based on this finding, an energy input of 35 kJ/gTS was found to be optimal for maximal sludge disintegration at 3% TS content. It is important to point out here that optimization of energy requirements for efficient sludge disintegration is extremely important for cost effective digestion of sonicated sludge.



Figure 2.9 Effect of specific energy input on SCOD increase (Frequency: 20 kHz; Maximum power: 1.5kW; TS content: 3%; and ultrasound density: 1.07 W/mL)

Muller proposed an equation to calculate  $DD_{COD}$  as given by Schmitz et al. (2000):

$$DD_{M} = \left[\frac{COD_{ultrasound} - COD_{original}}{COD_{NaOH_{22hr}} - COD_{original}}\right] \times 100(\%)$$
(Eq. 2.6)

In the above equation,  $COD_{ultrasound}$  is the COD in the supernatant of the sonicated sample (mg/L);  $COD_{original}$  is the COD of supernatant original (untreated sample) (mg/L) and  $COD_{NaOH}$  is the maximum COD release in the supernatant after NaOH digestion at 22 hours. The NaOH digestion is carried out by treating the sludge samples with 1 M NaOH in the ratio of 1:2 for 10 min at 90°C. The supernatant is obtained by centrifugation for 10 min at 30,000g and temperature of 4°C.

Rai et al. (2004) adopted Müller's method to determine the  $DD_{COD}$  in their ultrasonic study. The authors also filtered the supernatant sample after centrifugation at 10,000 g through 0.45 µm pore size membrane filter. Tiehm et al. (2001) also employed Müller's method in calculating the  $DD_{COD}$ . However, the chemical disintegration of sludge was determined by adding 1M NaOH in the ratio of 1:2 (0.5 mol/L) after 22 h at 20°C. In another study, Bougrier et al. (2005) employed Müller's method for  $DD_{COD}$  determination.

The authors, however, mixed the sludge with 1 mol/L of NaOH for 24 h at room temperature for determining the chemical disintegration. Although the literature shows a significant variation in the conditions for chemical disintegration, the overall goal of chemical disintegration is to obtain the maximum release of soluble organics from the sludge. This data is taken as a baseline to elucidate the efficacy of ultrasonic disintegration. It is important to note that the conditions for testing chemical disintegration could vary depending on sludge type and TS content. Thus, researchers need to conduct a series of exploratory studies with respect to concentration and amount of NaOH to be used, reaction time and temperature to develop a standardized protocol for chemical disintegration that ideally suits their sludge samples.

### b) Protein assessment

Since the degree of disintegration is primarily based on COD determination, researchers argue that DD determination is rather slow in the range of a day and is also expensive due to the need of large numbers of COD sample analyses (Schmitz et al., 2000). The authors therefore proposed protein measurement as an alternative to DD<sub>COD</sub> determination. In their study, correlation coefficients (R<sub>2</sub>) for increase in protein ( $\Delta$ Protien), DD<sub>KW</sub>, and DD<sub>M</sub> with respect to increase in biogas yield ( $\Delta$ biogas) due to sonication were compared to evaluate the reliability of the new sludge disintegration assessment technique. When the sludge samples were collected twice from one plant and once from another plant and sonicated, the authors found that the combined coefficients for  $\Delta$ Protien/ $\Delta$ biogas (R2=0.83) suggesting that irrespective of sources of sludge and times of collection, protein determination was a more reliable technique of assessing the ultrasonic disintegration of sludge.

Wang et al. (2005b) examined the release of protein, polysaccharide and deoxyribonucleic acid (DNA) in aqueous phase during ultrasonic disintegration of WAS at different specific energy inputs. Protein was predominant in the aqueous phase of sonicated sludge and its concentration increased almost exponentially to as high as 2,500 mg/L up to a specific energy input of 50 kJ/gTS. Thereafter, the increase in protein concentration slowed down with further increase in specific energy input. The increase in DNA and polysaccharide level was marginal.

The release of soluble protein (shown in table 2.3) and carbohydrate  $COD_{Cr}$  in the aqueous phase during different sonication durations was also investigated by Wang et al. (1999). The authors found that the release of soluble protein was significantly higher than the other two parameters during sonication.

Sonication duration (min)	Soluble protein concentration (mg/L)
0	50
10	1,200
20	3,000
30	5,200
40	6,000

Table 2.3 The soluble protein in the aqueous phase with sonication duration

Protein is an important building block of all microbial cells. The microbial extracellular polymeric substances (EPS), which provide the structural matrix for all microbial aggregates such as flocs in activated sludge, also contain protein. Wang et al. (2005b) reported protein contents of 698 and 11,338 mg/L, respectively in EPS and inside the microbial cells with 3% TS content. Therefore, quantification of sludge disintegration particularly WAS by protein measurement could be used reliably. However, for field application, protein measurement is still not common as none of the published studies employed protein measurement to assess the efficacy of ultrasonic sludge disintegration. The COD measurement will continue to be the method of choice for daily operation due to its simplicity.

### c) NH<sub>3</sub> assessment

Khanal et al. (2006c) studied the release of ammonia-N concentration at different TS contents and specific energy inputs (kJ/gTS) during ultrasonic disintegration of WAS. The results showed that the release of ammonia-N concentration increased with increase in specific energy inputs and TS contents (shown in Figure 2.10). The ammonia-N concentration reached a fairly constant level at specific energy inputs of 20 kJ/gTS for 2.0, 2.5 and 3% TS contents, and 10 kJ/gTS for 1.5% TS content. During sonication, bacterial cells are disintegrated releasing intracellular organic nitrogen into the aqueous phase, which is subsequently hydrolyzed to ammonia. This results in an increase in ammonia nitrogen in the aqueous phase. It is important to point out that the disintegration of organic nitrogen from non-biological debris could also contribute to the release of ammonia nitrogen.



Figure 2.10 Release NH<sub>3</sub>-N during sludge disintegration at different specific energy inputs and TS contents, (Khanal et al., 2006c)

Bougrier et al. (2005) monitored nitrogen release (soluble organic and ammonia nitrogen) during sonication of thickened WAS at different specific energy inputs. The total Kjeldhal nitrogen (TKN) in the whole sludge did not change the specific energy inputs at all. This apparently suggests that ultrasound does not contribute to nitrogen mineralization or volatilization. However, organic nitrogen and ammonia nitrogen increased in the aqueous phase with increase in the specific energy input during sonication with concomitant decrease of organic nitrogen in the solid phase. The maximum solubilization of organic nitrogen was achieved at a specific energy input of 10 kJ/g TS.

#### 2.6.3 Biological evaluation

#### a) Heterotrophic plate counts

WAS mainly consists of heterotrophic bacteria, the measure of their survival during ultrasonic treatment could also furnish data on efficacy of ultrasonic disintegration. Chu et al. (2001) reported a survival ratio (ratio of viable bacteria after sonication to the original sample) of 44% for heterotrophic bacteria at a sonication density of 0.33W/mL during 120 min of sonication. However, heterotrophic plate count is not a pragmatic method for judging the sludge disintegration efficiency in field applications and is not discussed here.

#### b) Specific oxygen uptake rate (SOUR)

The WAS mainly consists of aerobic and facultative bacteria. Therefore, measurement of oxygen uptake rate is a good indicator of bioactivity of WAS. Since ultrasonic treatment disrupts the bacterial cells, the measurement of SOUR of sonicated WAS could be used to assess the effectiveness of sludge disintegration. Based on this premise, Khanal et al. (2006c) examined the SOUR of WAS samples at different sonication durations. The SOUR test was conducted using 20 mL of sonicated sludge with a TS content of 1.5%, and synthetic substrate with SCOD of 500 mg/L containing all essential macro- and micro-nutrients was used as the sole carbon source. The SOUR results are shown in Figure 2.11.



Figure 2.11 SOUR of WAS at different sonication durations, (Khanal et al., 2006c)

As seen from the figure, the biological activity of sonicated sludge decreased almost exponentially during the first 16 min of sonication; after that it decreased at a lower rate. The activity decreased by as much as 55% when the WAS was sonicated for 16 min compared to a control (without sonication). This finding suggests that sonication was effective in disintegrating the bacterial cells. It is important to point out that the release of SCOD may not be a true measure of effectiveness of sonication. The rupturing of the bacterial cells does not necessarily release the intracellular matter. However, it exposes the cell content to exo-enzymes thereby enhancing efficient digestion. Thus, the use of oxygen-uptake rate could be a useful and practical tool to evaluate the cell disintegration.

Rai et al. (2004) coined the term degree of inactivation ( $DD_{OUR}$ ) based on oxygen uptake rate (OUR) data, which is somewhat similar to degree of disintegration ( $DD_{COD}$ ) as discussed earlier. The  $DD_{OUR}$  can be calculated using the following expression:

$$DD_{OUR}(\%) = \left[1 - \frac{OUR_{sonicated}}{OUR_{original}}\right] \times 100$$
 (Eq. 2.7)

Where, OURsonocated is the oxygen uptake rate of sonicated sludge. OURoriginal is the oxygen uptake rate of the original sample (without sonication)

$$OUR = -\frac{d[O_2]}{dt}$$
(Eq. 2.8)

The  $DD_{OUR}$  increased rapidly with increase in specific energy input up to 40 kJ/gTS, after that the increase slowed down as shown in the figure 2.12 (Rai et al., 2004). At a specific energy input of 8 kJ/gTS, the  $DD_{OUR}$  was found to be negative. This means that the OUR of sonicated sludge was higher than that of the unsonicated. This was mainly because at low energy input, the microbial cells were not disrupted and the flocs were simply deagglomerated into individual microbial cells, which eventually participated in the biological activity. The measurement of oxygen uptake rate is relatively simple and takes only 20 min or less. Besides, it measures the true biological activity. Thus,  $DD_{OUR}$ determination based on OUR measurement could be a very useful tool for field application to assess the ultrasonic disintegration of sludge, particularly WAS. However, the versatility of this method needs to be tested under different conditions and correlated with sludge digestibility both under aerobic or anaerobic conditions.



Figure 2.12 Degree of inactivation at different specific energy inputs, (Khanal et al., 2006c)

### 2.7 Factors Affecting Efficacy of Ultrasonic Disintegration

The efficiency of ultrasonic disintegration is depended by several factors. These factors can be broadly classified into three categories,

- (a) Sludge (solid) characteristics;
- (b) Sonication conditions; and
- (c) Design of ultrasonic components.

#### 2.7.1 Sludge (Solid) characteristics

The sludge characteristics such as type of sludge (primary solids, waste activated sludge or animal manure, etc.), TS content, and particle size play an important role during ultrasic treatment. An increased effectiveness of ultrasonic disintegration is observed at increasing concentrations of DS in the WAS (Tiehm et al., 2001; Onyeche et al., 2002; Neyens et al., 2004). This finding is explained by the fact that (1) more DS creates more sites for cavitation and (2) more particles are exposed to the resulting shear force. Grönroos et al. (2005) reported the maximum SCOD concentration at the highest dry solid (DS) content. However, the authors did not present the data in terms of mg SCOD/gDS, which made it difficult to understand whether sludge disintegration was efficient at higher solids content. Dewil et al. (2006) conducted a thorough study to evaluate the effect of TS contents on SCOD release at different specific energy inputs. The results are presented in Figure 2.13.



Figure 2.13 SCOD release at different energy inputs and TS contents, (Khanal et al., 2006c)

As evidence from the figure, the efficiency of sludge disintegration increases clearly with the dry solids content; a higher  $\Delta$ COD is observed for an equal SE. This higher efficiency is in agreement with previous literature findings (Onyeche et al., 2002; Nickel, 1999). Two factors are responsible for this phenomenon: (1) the presence of more enhances cavitation by DS-particles that act as nuclei and (2) due to the higher concentration; particles are more affected by the cavitation that is taking place. This trend reaches a maximum at about 15g.L<sup>-1</sup>. Higher concentrations were also used, but the  $\Delta$ COD-effect was seen to decrease dramatically. This is caused by the increasing viscosity of the sludge, too high a viscosity reduces cavitation since the ultrasonic waves are scattered by the DS-particles and absorbed by the fluid to generate heat rather than creating bubbles that are needed for cavitation. Wang et al. (2005a) also reported a significant effect of TS content on SCOD release. The SCOD release increased from 3,966 to 9,019 mg/L when the TS content was increased from 0.5 to 1% during 30 min of sonication at an ultrasonic density of 1.44 W/mL. These findings apparently show that a higher TS content is more energy efficient for ultrasonic disintegration than the lower TS content.

Nevertheless, Akin et al. (2007) conducted the experiment to evaluate ultrasonication pretreatment efficiency with different TS contents. The cut-off diameter and SCOD increment per specific energy used to evaluate ultrasonic pretreatment efficiency. Results

show that the cut-off diameter for WAS with 2% Ts content declined by nearly 6.5 fold at ultrasonic density of 0.67 W/ml. For higher TS contents of 4 and 6 %, higher densities of 1.03 and 0.86 W/ml, respectively, were needed to achieve the same degree of particle size reduction. SCOD release of about 320 mg SCOD/gTS was obtained at TS content of 2% and specific energy input of 5 kWs/gTS. The SCOD release, however, decreased to 160 and 90 mg SCOD/gTS at 4% and 6% TS contents respectively. Author concluded that low TS contents give high effectiveness of ultrasonic pretreatment. However, the effect of the number of particles (TS contents) on the formation of cavitation bubbles in the sludge matrix is still unknown.

The ease of ultrasonic disintegration is also governed by the composition of the sludge matrix. It is believed that non-biological solids, e.g., primary sludge and animal manure, are relatively easy to disintegrate compared to biological sludge such as WAS. However, no study evaluated the effects of different sludge types and particle size on ultrasonic disintegration. Such a study is important if ultrasonic technology is to be applied for primary solids or animal manure disintegration. The data obtained from biological sludge disintegration may not be directly extrapolated due to the different degree of disintegration.

### 2.7.2 Sonication conditions

The oscillation frequency, Ultrasonic energy input, sonication time, temperature, pH, and amplitude are some of the important parameters that affect the ultrasonic disintegration.

### a) Frequency

As we have outlined before in section 2.4.2, two cavitation phenomena might be responsible for the destruction of solid cell matter: powerful hydromechanical shear forces and sonochemical reactions. Both the hydromechanical shear forces and the sonochemical effects can contribute to the ultrasonic disintegration of sewage sludge.

Tiehm et al. (2001) found the  $DD_{COD}$  to be 13.9, 3.6, 3.1 and 1.0%, respectively at frequencies of 41, 207, 360 and 1,068 kHz and concluded that a frequency lower than 41 kHz would yield better sludge disintegration. This is demonstrated by the most pronounced reduction of the median sludge particle size as well as the largest increase in turbidity of the sludge samples at low frequency. Obviously particulate sludge material was broken down into smaller pieces. We also measured the highest degree of disintegration ( $DD_{COD}$ ) at 41 kHz. The efficiency of sludge disintegration decreased with increasing frequency. Hence we would expect the best disintegration results with the lowest ultrasound frequency of 20 kHz. However such a frequency could not be set with the device available. Sonochemical reactions are particularly predominant at a higher ultrasonic frequency 200 to 1000 kHz (Mark et al., 1998). Thus, nearly all sludge disintegration tests are conducted at the lower frequency range of 20 kHz (Wang et al., 2005a, b; Bougrier et al., 2005; Khanal et al., 2006a, b).

Theoretical considerations are useful to understand the decrease in disintegration efficacy with increasing ultrasound frequency. Cavitation bubble collapse occurs when the expanding bubbles have reached their resonant radius. The resonant cavitation bubble radius is a function of the ultrasound frequency. In the case of air bubbles in water at atmospheric pressure, the ultrasonic cavitation bubble radius can be approximated as

 $R_r \approx 3.28 f_r^{-1}$  (Eq. 2.9)

Where the resonant bubble radius  $R_r$  is expressed in millimeters and  $f_r$  is the resonance frequency in kHz (Young, 1989). The bubble radius is inversely proportional to the ultrasound frequency. The application of low frequencies creates larger cavitation bubbles. Upon bubble collapse, hard mechanical jet streams are produced that are responsible for many cavitation effects observed on solid surfaces. A valid assumption might be that the energy released by a jet stream is a function of the bubble size at the moment of collapse. The number and size of cavitation bubbles in a sludge media may was depended to number of solids, density of sludge and the presence of dissolved gases. However, the degree of sludge disintegration could be related to the theoretical bubble size calculated by using equation (3). The theoretical approach gives evidence that the hydromechanical shear forces produced by ultrasonic cavitation are more important for sewage sludge disintegration than sonochemical processes. As review above, High efficiency of sludge disintegration is obtained at low frequency as 20 kHz.

#### b) Ultrasonic energy input

The SCOD release must also be correlated with ultrasonic energy input (expressed as ultrasonic density, ultrasonic intensity or specific energy input). Such correlations will help to optimize the energy needs to achieve maximum sludge disintegration. A numbers of studies evaluated SCOD release at different specific energy inputs and ultrasonic densities as shown in Table 2.4.

Released SCOD and disintegration rate can also directly be expresses as a function of specific energy (SE) that is applied to the sludge (Dewil et al., 2006). In addition, the authors are obtained; there is a minimum SE required before destruction starts. There, this minimum lies at about 1500 kJ/KgTS



Figure 2.14 SCOD release at different energy inputs and TS contents, (Khanal et al., 2006c)

As evident from the figure 13 (Khanal, et al., 2006c), SCOD release showed an increasing trend with increase in both TS content and energy input. However, the release in SCOD slowed down at an energy input of over 35 kWs/gTS for all TS contents. Based on linear regression analysis (R2 > 0.90), SCOD releases were 1.6, 2.2, 2.5 and 3.2 mg/kWs at TS contents of 1.5, 2.0, 2.5 and 3.0%, respectively. This corresponds to 38, 59 and 98% increase in SCOD release at TS contents of 2.0, 2.5 and 3.0%, respectively as compared to 1.5%.

#### c) Duration of Ultrasonication

As evident from equation (2), the specific energy input is proportional to sonication time. The longer sonication time means a higher specific energy input; thus resulting in higher SCOD release. Wang et al. (2005b) examined the release in SCOD concentration at three different sonication times of 5, 15 and 20 min at TS content of 3%, frequency of 20 KHz and ultrasonic density of 0.768 W/mL. The authors observed an increase in SCOD release from 2,581 to 7,509 mg/L, when the sonication time was increased from 5 to 15 min. However, when the disintegration was continued for 20 min, the SCOD release slowed down significantly with final SCOD concentration of 8,912 mg/L. Several studies confirmed this trend (Wang et al., 2005a; Khanal et al., 2006; Zhang et al., 2006). The highest SCOD release is the major goal of ultrasonic pretreatment. Although the degree of solubilization improved with increase in specific energy input, the improvement was not in direct proportion to the energy input. For example, Khanal et al. (2006c) obtained SCOD/COD of 16.2% at an energy input of 66,800 kJ/kgTS; whereas Bougrier et al. (2005) achieved as much as twice that at an energy input of only 6,951 kJ/kg TS. In another study, DD<sub>COD</sub> of 40% was obtained at a specific energy input of 60,000 kJ/kg TS (Tiehm et al. 2001); whereas Rai et al. (2004) reported DD<sub>COD</sub> of 25% at energy input of 64,000 kJ/kg TS. Such variations are most likely attributed to energy transfer efficiencies of ultrasonic units. Many of the sludge disintegration studies reported in Table 2.4 were conducted at frequencies of 20 to 40 kHz with 20 kHz being optimal for cavitation.

Interestingly, for the same ultrasonic energy input of 3W-min/mL, the sludge disintegration at an ultrasonic density of 0.5W/mL (sonicated for 6 min) yielded DD<sub>COD</sub> of 9.2%, whereas an ultrasonic density of 0.1W/mL (sonicated for 30 min) yielded DD<sub>COD</sub> of 7.3% (Zhang et al., 2006). Along the same line, the authors reported DD<sub>COD</sub> of 15.8% at an ultrasonic density of 0.5W/mL (sonicated for 10 min) and 11.3% at an ultrasonic density of 0.2W/mL (sonicated for 30 min) with energy inputs of 5 and 6W-min/mL, respectively. Grönroos et al. (2005) also observed a better sludge disintegration at the same specific energy input, when the sludge was sonicated at higher ultrasonic density for a short duration than a lower sonication density for a longer duration. These findings show that for efficient sludge disintegration, ultrasonic density is apparently more important than the sonication time.

Table 2.4 Organic solids solubilization at different sonication condition	Table	e 2.4	Organic	solids	solubilization	at	different	sonication	conditions
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<b>T C L L</b>	Power input (W)		Specific energy	Ultrasonic density	Deg	Reference				
Type of sludge	and Frequency (kHz)		input (kJ/kg TS)	or intensity	SCOD release	DD <sub>COD</sub> (%)	SCOD/COD (%)	Reference		
		1500	0 (Control)		1,542	_	4.2			
Waste activated sludge	Power input	1500	10,000	1 1 337/ 3	2,412	21/4	5.7			
(TS: 3%)			28 200	1.1 W/mL	4 824	N/A	12.3	Khanal et al. (2006c)		
	Frequency	20	34,600		5,846		13.7			
			66,800		7,022		16.2			
				0 (Control)	410					
	Power input	N/A		0.1 W/mL	1,050					
Waste activated sludge with			N/A	0.2 W/mL	1,500	N/A	N/A	Zhang et al. (2006)		
nutrient removal				0.5 W/mL	3,150	14/24	11/11	Zhang et al. (2000)		
(TS: 1%)	Frequency	25		1.0 W/mL	4,500					
				1.5 W/mL	5,400					
	Douvon immut	1500		0 (Control)	775					
Waste activated sludge	Power input	1300	N/A	0.18 W/mL	950			$M_{\rm ext} = 1.(2005)$		
(TS: N/A)	5	20	14/21	0.33 W/mL	1,200	N/A	N/A	Mao et al. (2005)		
	Frequency	20		0.52 W/mL	1,500					
	<b>D</b>	750	0 (Control)		·		5.8			
Waste activated sludge	Power input	/50	660				10.5			
(TS: 1.85%)	Frequency			1,35	1,355	N/A	N/A	N/A	16.1	Bougrier et al. (2005)
(15. 1.6576)		20	2,700				22.3			
			6,951				33.1			
Thickened waste activated	Power input	N/A	0 (Control)		1,300					
sludge (TS: 2.45%)	Frequency	27	3,000	1.25 W/mL	2,600	N/A	N/A	Grönroos et al. (2005)		
studge (15. 2.4570)	Frequency	27	14,900		4,050					
Biological sludge from SBR	Power input	N/A		60W/cm <sup>2</sup>			12			
(TS: 0.5%)	Frequency	Frequency	Frequency	20	N/A	120W/cm <sup>2</sup>			18	Wang et al. (2005a)
		20		230W/cm <sup>2</sup>			30			
	Power input	500	8,000			4.2				
Waste activated sludge	rower input	500	24,000	NI/A	NT/A	8	NI/A	$\mathbf{D}_{ai}$ at al. (2004)		
(TS: 0.48%)	Enomen	NI/A	40,000	IN/A	IN/A	10	IN/A	Kai et al. (2004)		
	Frequency	IN/A	64,000			25				
	Douvon immut	750	0 (control)				5.8			
Waste activated sludge (TS)	Power input	730	1,355				16.1			
2%)			2,707	N/A	N/A	N/A	22.3	Bougrier et al. (2004)		
270)	Frequency	20	6,951				33.1			
			14,547				41.6			
	Douvoningut	NI/A	5,000							
Thickened waste activated	Power input	IN/A	10,000	]			]			
sludge (TS: 2 59%)			25,000	1.4W/cm <sup>2</sup>	N/A		N/A	Tiehm et al. (2001)		
Shudge (15. 2.5770)	Frequency	Frequency	Frequency	Frequency	Frequency	41	40,000	),000	1	
			60,000				]			

In evaluating the effects of sonication conditions on sludge disintegration, parameters such as pH and temperature also become equally important. The SCOD release was found to increase when the sludge was sonicated at a higher pH as shown in Figure 2.15 (Wang et al., 2005a).



Figure 2.15 SCOD release at different pH during 30 min of sonication

It is likely that the alkaline addition to raise the pH may have weakened the bacterial cell wall that facilitated better destruction during ultrasonic treatment. Therefore, alkaline treatment of sludge followed by ultrasonic application could lower the energy cost of ultrasonic systems to achieve a desired degree of sludge disintegration. However, a thorough study is needed to examine the effect of alkaline addition on ultrasonic sludge disintegration. Wang et al. (2005a) examined the effects of pH, TS content, ultrasonic intensity and density on disintegration of biological sludge based on a kinetic model using a multi-variable linear regression method. The authors found a first order disintegration in the order:

Sludge pH > sludge concentration > ultrasonic intensity > ultrasonic density

#### Temperature

Sonication of sludge results in an increase in the temperature of the aqueous phase. The temperature increase depends on both sonication time and sonication density. Tiehm et al. (1997) observed an increased in sludge temperature from 15 to about 45°C during 64 seconds of sonication in a flow-through-type ultrasonic unit at frequency 31 kHz. Chu et al. (2001) observed an appreciable increase in sludge temperature when the sludge was sonicated for 120 seconds. The respective temperatures were 30, 42, 51 and 56°C, at ultrasonic densities of 0.11, 0.22, 0.33 and 0.44W/mL. At a constant power density of 0.44W/mL, the sludge temperature increased from 19°C to 30, 50 and 56°C, when the sludge was sonicated for 0 (control), 20, 60 and 120s. Interestingly, the temperature increased at a rate almost proportional to the increase in ultrasonic density. The respective temperature increase rates were (c.) 0.15, 0.28, 0.43 and 0.51°C /sec at ultrasonic densities of 0.11, 0.22, 0.33 and 0.44W/mL. As a matter of fact, ultrasonic density plays a more prominent role in temperature increase than the sonication time.

The solubilization of sludge could also be due to thermal effects resulting from the increase in sludge temperature during sonication. It is often difficult to quantify the contribution of thermal effects on the degree of sludge disintegration. In one study, SCOD release increased nearly 2.4-fold during sonication for 60 min at an ultrasonic density of 0.33W/mL without temperature control compared to sludge samples sonicated at a controlled temperature of 15°C (Chu et al., 2001). However, there was no data on final temperature of sonicated sludge. Grönroos et al. (2005) also reported a significant contribution of temperature on ultrasonic sludge disintegration.

The authors adjusted the sludge temperature in their experiment, and the SCOD release during 30 min was measured for sludge with and without ultrasound treatment. The SCOD increased from (c.) 750 to 1,550 mg/L for unsonicated and (c.) 2,150 to 2,950 mg/L for sonicated sludge, when the sludge temperature was raised from 30 to 60oC. Some studies, however, concluded that temperature has no significant impact on ultrasonic disintegration (Wang, et al., 2005a; Shirgaonkar and Pandit, 1997; Cheung and Kurup, 1994). There is a need to conduct a systematic study to elucidate the contribution of temperature on overall sludge disintegration.

### 2.7.3 Design of ultrasonic components

While there are many different ultrasonic manufacturers and designs, nearly all systems consist of two major components; (1) the power supply and (2) the stack assembly (see Figure 2.4). In order to maximize the efficiency of operation, most systems operate at a particular frequency. The stack assembly is designed and manufactured to mechanically resonate at that frequency, similar to the ringing of a bell or strumming of a string, where the stored energy of the system is high compared to the energy loss of the system ("Q" of the system). The power supply then matches this frequency through an electro-mechanical system. The stack consists of three sub-components, the converter, the booster and the horn. As previously detailed (section 4.4), the converter is simply a linear motor. The maximum displacement of the converter is usually rated in peak-to-peak displacement and is inversely proportional to the operating frequency. For example, at 20 and 40 kHz, the typical maximum amplitude is 20 and 10 µmpp, respectively. The limitation of the amplitude at higher frequency is primarily due to design constraints because of the desire to have a resonant system. In most systems, the converter is designed to be half the wavelength ( $\lambda$ ) of the vibrations. It is important to note that because the converter consists of various components (i.e., back drive, piezo-electric ceramics and front drive that are manufactured from different materials), determining the amplitude of the entire converter is not a trivial task. In addition, the complexity of this problem is compounded by the fact that the piezo-electric ceramics have material constants (such as stiffness and displacement constants) which are load and voltage dependent.

Design of an efficient horn is extremely important to achieve an amplitude of 50  $\mu$ mpp or higher. This is because strong cavitation is generated at higher amplitudes. Horn configuration becomes a major limiting factor when dealing with high amplitudes. This is because high amplitudes with some horn designs may cause significant structural damage. The horn design could essentially limit its ability to achieve greater cavitation levels and power outputs. Thus, the design of all these units may significantly affect the efficacy of ultrasonic systems for sludge disintegration. Such information is often proprietary so manufacturers do not normally share this.
## 2.8 Effect of Ultrasonic Pretreatment on Sludge Digestibility

Ultrasound disintegration of waste activated sludge aims at enhancing the VS destruction during digestion (Khanal et al., 2006b; Bougrier et al., 2005; Hogan et al., 2004; Tiehm et al., 2001; Wang et al., 1999). The increased VS reduction directly translates into increased methane generation during anaerobic digestion and less stabilized biosolids to be disposed of. Additional methane generation provides an incentive for the waste generators to retrofit the existing digesters with ultrasonic systems. Such integration will not only result in additional revenue generation from excess bioenergy generation; but it will also help saving considerable sludge treatment and disposal costs.

# 2.9 Anaerobic Digestibility of Ultrasound Pretreated Sludge

Wang et al. (1999) studied the VS destruction, expressed as organic destruction efficiency or digestion efficiency of flotation-thickened WAS at a TS content of 3.3 to 4.0%. The sludge was sonicated using a 200 W ultrasonic unit at a frequency of 9 kHz. The authors reported that the organic destruction efficiency improved by 11, 20, 38 and 46% compared to a control on the 11th day of anaerobic digestion, when the WAS was sonicated for 10, 20, 30 and 40 min, respectively. The digesters appeared to reach steady state when the VS data were collected since the cumulative methane generation did not show much variation by 10th day of digester operation. The authors observed c. 15, 38, 68 and 75% improvement in cumulative methane yield for WAS sonicated for 10, 20, 30 and 40 min, respectively in comparison to control during 11 days of anaerobic digestion. Thus, the methane yield appears to be directly correlated with VS destruction. Tiehm et al. (1997) examined the effect of ultrasound pretreatment on VS destruction during anaerobic digestion of municipal sludge comprising of 53% primary sludge and 47% WAS on dry weight basis at different SRTs. The sonic treatment was conducted using a 3.6 kW ultrasound unit at a frequency of 31 kHz for 64 seconds. The authors observed nearly 9.8% higher VS destruction for an anaerobic digester fed with sonicated sludge compared to a control at an SRT of 22 days. Interestingly, the VS destruction efficiency did not deteriorate for a digester fed with sonicated sludge in comparison to a digester fed with unsonicated sludge, even when the operating SRT was reduced by one-third to 8 days. This finding apparently suggests that by integrating an ultrasonic system with an existing digester, the SRT could be reduced by as much as 3 times. A higher biogas yield was observed for sonicated WAS in comparison to control in this study. Based on serum bottle tests, the authors observed nearly c. 28% higher biogas yield for sonicated sludge in comparison to untreated sludge during 28 days of digestion. Interestingly, in a continuous study at an SRT of 22 days, the cumulative biogas production did not improve for sonicated sludge in comparison to unsonicated sludge during 100 days of digester operation. The authors explained that such observation could be due to a change in the biochemical fermentation process, which may not be a good reason. It is most likely that the longer SRT provided sufficient time even for the unsonicated sludge to achieve a better hydrolysis of particulate matter.

In another study, Tiehm et al. (2001) investigated the effect of sonication time on VS destruction during anaerobic digestion of WAS at an SRT of 8 days. The sonication test was conducted at a frequency of 41 kHz using a disk transducer of 25 cm<sup>2</sup> surface area. The VS removal efficiency is improved by 5.6%, 27%, 46% and 56.7%, when the sludge was sonicated for 7.5, 30, 60 and 150 minutes respectively. The authors reported cumulative biogas generation of 2.93, 2.79, 3.39, 3.38 and 4.15 L, respectively from five

completely mixed anaerobic digesters fed with WAS sonicated for 0 (control), 7.5, 30, 60 and 150 min. The biogas production declined slightly for 7.5 min of sonication time for unknown reasons. It is important to point out that even the shortest sonication time of 7.5 min is relatively long for full-scale applications. Lately, there has been a significant improvement in ultrasonic design, particularly horn and converter designs. These improvements made it possible to achieve high amplitudes and delivering more power to sludge in a short time.

Pilot-scale demonstration trials using V-shaped sonication chambers with donut horn was conducted at Avonmouth wastewater treatment plant in UK (Hogan et al., 2004). The sonication was carried-out at a frequency of 20 kHz using thickened municipal sludge with 70% TWAS (by weight). The authors obtained nearly 40% higher VS destruction compared to expected theoretical unsonicated TWAS. This study, however, did not have a control digester with 70% TWAS without sonication. Therefore, the comparison of VS destruction data may not be conclusive. The authors reported up to 100% more biogas production with sonicated sludge than with unsonicated sludge containing up to 70%TWAS. The authors also tested the VS removal efficiency of a mesophilic anaerobic digester fed with unsonicated TWAS (100%) followed by sonicated TWAS (100%) at Severn Trent Water (UK). An average of 54% VS destruction was achieved with sonicated TWAS in comparison to 38% for unsonicated TWAS. This study also found a higher biogas yield in accordance with VS destruction for sonicated sludge and the increase in biogas yield was about 40% after the full acclimation of digesters. However, no data on SRT, sonication time, and power input were given for above pilot studies. Hogan et al. (2004) conducted a demonstration trial to examine the effect of sonication on biogas generation in the Orange County Sanitation District (OCSD). The biogas production from sonicated sludge with 50-55% TWAS was about 50% higher than that without sonic pretreatment. It appears from the data that the authors compared the biogas data to pretest conditions, i.e. with feed TWAS of 20-30%. Also, there was no VS destruction data for sonicated and unsonicated sludge.

The biogas generation from WAS sonicated at different specific energy inputs was evaluated in a series of batch anaerobic digestion tests during 16 days of incubation (Bougrier et al., 2004). The WAS (2% TS content) was sonicated using an ultrasonic unit with a power supply of 225 W at a frequency of 20 kHz and different specific energy inputs. The authors found that the biogas yields were 1.48, 1.75, 1.88 and 1.84 times higher for the sonicated WAS in comparison to control (unsonicated) at specific energy inputs of 1,355, 2,707, 6,951 and 14,547 kJ/kgTS, respectively. The biogas yield clearly showed improvement with increase in specific energy inputs up to 6,951 kJ/kgTS. However, with further increase in energy input to 14,547 kJ/kgTS, the biogas yield did not improve further in spite of higher release in SCOD for unknown reasons. No data on VS destruction were presented.

Contrary to the above findings, Latitte-Trouqué and Forster (2002) reported no significant improvement in solids reduction during anaerobic digestion of ultrasound pretreated WAS. The WAS was sonicated for 90 seconds using an ultrasonic unit with power output of 47 W at a frequency of 23 kHz. However, the authors did not report data on DD<sub>COD</sub> or release of SCOD, and without such data it would be difficult to conclude whether the ultrasonic unit the authors employed was efficient enough for sludge disintegration.

One question that remains unanswered is: What could be the best way to judge the efficacy of ultrasonic system based on SCOD release before digestion or based on biogas production and VS destruction? So far, there is no well-defined protocol that could be used to effectively judge ultrasonic efficiency. Thus, more research is needed in this direction.

### 2.10 Effect of Ultrasonics on Sludge Dewaterability

Sludge dewatering essentially aims at reducing the liquid content of sludge by converting it into a solid cake through the use of physical forces. Sludge dewatering can be achieved by using filter press, belt filter press, centrifuge, vacuum filtration, etc. Even with the use of mechanical means, the maximum solids content achievable is still low in the range of 25 to 40%, and further lowering of the moisture content from the cake is relatively difficult due to the presence of water that is tightly bound by capillary forces between the sludge flocs (Kopp and Dichtl, 2001). Thus, any further improvement in the dewaterability of sludge could result in a considerable savings in sludge disposal costs. A few studies reported that the ultrasound pretreatment of sludge could enhance the dewaterability of sludge following digestion (Hogan et al., 2005; Bien and Wolny, 1997). One study conducted at OCSD concluded that anaerobic digestion of WAS following ultrasonic treatment improved the mean cake solids content by  $1.64 \pm 0.32\%$  in comparison to an unsonicated control.

Although, Wang et al. (2006) found the results which was different from others researcher observed that ultrasonication of waste activated sludge could improve the dewaterabilty. In this research, the CST of raw disintegrated sludge was 82s. Utilizing an ultrasonic density of 0.528w/mL to disintegrate the sludge for 5 min, the CST was 344s. However, when an ultrasonic density of 1.44w/mL was used to disintegrate the sludge for 5 min, the CST was 599s. Moreover author founds reasons for changes in dewaterability. It was governed by two phenomena, fist one is water retained by EPS and water inside the cells were released and interstitial water was transformed into free water during disintegration so that it will enhance dewaterability, second one is sludge flocs became smaller after disintegration, which increased effective adsorption surface area for water so that free water was transformed into interstitial water. It will decrease the dewaterability. The second phenomena dominated in the sludge dewaterability properties result. More research is needed to evaluate the sludge dewaterability of sonicated sludge after digestion.

# 2.11 Effect of Ultrasonics on Biochemical Methane Potential (BMP)

The BMP test is used to evaluate anaerobic biodegradability in batch mode in which cumulative methane calculates from the batch reactor. Wang et al. (2006) conducted the experiment to determine the BMP value for different specific energy applied to the raw WAS. Under both mesophilic and thermophilic conditions, the biogas production was improved with increasing specific energy inputs. At low specific energy, the total methane produced increased significantly between 12.5–17.5% and 11.0–19.7% under thermophilic and mesophilic conditions, respectively. However, the enhancement in the biogas yield was only 1.6% (thermophilic) and 2.5% (mesophilic) when the applied specific energy was changed from 11,000 to 15,000 kJ/kg TS. From recorded methane improvement at the tested specific energy, show that 11,000 kJ/kg TS was the optimum specific energy for the raw WAS.

#### 2.12 Soluble Organics in Digested Biosolids

Aerobic digestion studies conducted at Iowa State University reported that the residual SCOD of the ultrasound pretreated biosolids was significantly lower than the unsonicated biosolids at all SRTs as shown in Figure 2.16 (Khanal et al., 2006a). The SCOD removal showed an increasing trend with an increase in SRT for the digester fed with unsonicated sludge, except for a SRT of 10 days at which the efficiency declined slightly for unknown reasons. The respective mean SCOD removal efficiencies were 66, 62, 79 and 82% at SRTs of 8, 10, 12 and 15 days. The higher COD removal can be attributed to better mineralization of organics due to longer detention times. The digester fed with sonicated sludge showed consistently high SCOD removal of 92% even at a short SRT of 8 days.



Figure 2.16 SCOD removal for digesters fed with sonicated and unsonicated sludge at different SRTs, (Khanal et al., 2006b)

The removal efficiency further improved to 98%, and remained fairly constant when the SRTs were increased to 10, 12 and 15 days. The effluent SCOD concentration at these SRTs was below 120 mg/L. Such data are not readily available for anaerobic digestion to make a comparison, as much longer retention times are required.

#### 2.13 Effect of Ultrasonics on Biosolids Quality

The biosolids quality refers to residual organics and pathogen levels after digestion. As illustrated in earlier sections, ultrasonic pretreatment resulted in lower VS and SCOD levels in the digested biosolids. Khanal et al. (2006c) investigated the specific oxygen uptake rates (SOUR) of both sonicated and unsonicated aerobically digested sludge at different SRTs and the results are presented in Figure 2.17.



Figure 2.17 Specific oxygen uptake rate (SOUR) at different SRTs

Digested sludge from the digester fed with sonicated WAS was more stable than that from the control. This was evident from the fact that the former had a lower SOUR value than the latter as apparent from the figure. The SOUR data infers that the ultrasonic treated biosolids have less potential for vector attraction and odor emanation. This is particularly important when the biosolids are intended for land application.

Khanal et al. (2006a) determined bacteria levels, e.g. fecal coliform, E. coli and Salmonella sp. in sonicated and unsonicated, and digested and undigested sludge samples. The sludge samples were taken from the digesters operating at an SRT of 10 days. The results are presented in Table 2. The tested sludge had Salmonella sp. densities below detectable levels under all conditions. Fecal coliform and E. coli levels dropped by 42% and 70%, respectively for sonicated digested sludge compared to unsonicated ones. This clearly shows a positive impact of sonication on pathogen reduction. In order to be classified as Class-A biosolids as stipulated in the 40 CFR Part 503 regulation, the biosolids must meet one of the requirements: either density of fecal coliform less than 1,000 MPN/g total solids (dry weight basis); or density of Salmonella sp. less than 3 MPN/4 g total solids (dry weight basis).

# 2.14 Problem Identification

Ultrasonic application in sludge pretreatment is an emerging research frontier. Although a good number of publications are now available, there are a number of issues that need to be researched further. There are several inconsistencies in many of the previously published papers, particularly in research methodology, which needs to be standardized. Standardization will make comparisons between findings of different researchers easier. For example, Efficiency of ultrasonication depends on several parameters such as specific energy input and ultrasonic density; sonication period, TS contents of sludge, temperature needs to be controlled during ultrasound disintegration, and pH of sludge to maximize sludge disintegration prior to testing the sludge for anaerobic digestibility. Many researchers found the correlation between efficiency of ultrasonication and theses parameters, so far there is a lack of information as to how different degrees of sludge disintegration impact on the digestion process (Yoon et al., 2004; Tiehm et al., 2001). Moreover, there are contradiction results obtained from some researchers to maximize the sludge disintegration of WAS. Therefore, further research to be needed to clarify these

results. Until now no mathematical model which explains relationship between efficiency of ultrasonication and each parameter has been found. Therefore statistical analysis has to be applied to elucidate the relative significance of each factor.

Many of researchers had observed that ultrasonic disintegration of activated sludge could improve the dewaterability of sludge. Nevertheless Wang et al. (2006) had found that the dewaterability is increasingly reduced with increasing ultrasonic density and time. Therefore, dewaterability after sonication and digestion has to be determined further in this study to find out influence of ultrasonication on dewaterability of waste activated sludge. The digestion for sonicated sludge that causes the end product shows a substantially better biological stability than the digested sludge without sonication. Therefore, land-fill disposal and limited agricultural use as a fertilizer are possible. Ultrasonication controls pathogen in the digested sludge in terms of E. coli levels and density of Salmonella sp. Effects of sonication on biosolids quality have to be determined using optimum sonication condition. A cost-benefit analysis of ultrasonic integrated systems needs to be conducted to justify the economics of the process in full-scale applications.

# Chapter 3

### Methodology

## 3.1 Introduction

This study was focused on anaerobic digestibility of ultrasonic pretreated waste activated sludge from a local domestic wastewater plant. The optimum sonication conditions were investigated followed by anaerobic digestion. The research work is divided into three parts: (i) optimization of sonication conditions to maximize sludge disintegration; (ii) evaluation of anaerobic digestibility of ultrasound pretreated WAS at different solids retention times (SRTs); and (iii) determination of hydrolysis coefficient for both sonicated and nonsonicated sludge during anaerobic digestion. The research plan frame work is shown in the Figure 3.1.



Figure 3.2 Research Plan Framework

In the first part, sonication conditions were optimized with respect to specific energy input and sonication time to maximize sludge disintegration. The second part was examined the anaerobic digestibility of full-stream (100% sonicated) and part stream (50% sonicated and 50% unsonicated) WAS at different solids retention time. In the third part, the biokinetic coefficients were determined for both full and part stream sonicated and non sonicated sludge. In addition, the tests on dewaterability and pathogen counts were conducted on digested sludge.

## 3.2 Waste Activated Sludge Sample

The waste activated sludge (WAS) sample was collected at return sludge line from the Thammasat University domestic wastewater treatment plant located in Pathumthani, in which the activated sludge process is used to treat the wastewater. After the initial sampling process is completed, chemical parameters such as SCOD, TS, VS, and pH were analyzed immediately. The sludge sample was preserved in cold storage room at 4°C prior to use to prevent biodegradation. Sludge sample was concentrated to 3% by centrifugation (at 5000 rpm for 2-3 minutes) for sonication and subsequent digestion studies.

### 3.3 Ultrasonic Equipment

The WAS samples was sonicated using Sonics ultrasound unit (VC750 model, Newtown, CT, USA). The ultrasound unit has a maximum power output of 200W and operates at a constant frequency of 20 kHz. This unit is equipped with three different horns; small (1.2 cm), medium (2.5 cm) and large (3.8 cm). The power input can be set independently from 40 - 200 W. The amplitude can be also set independently from 20-100 %. The ultrasonic equipment is shown in Figure 3.2



Figure 3.3 Ultrasonic equipment

### 3.4 Sonication Chamber

A sonication chamber known as Rosett cell was employed for sonication using small horn. At the bottom of the chamber have three open loops to facilitate heat dissipation during sonication. The chamber is made up of glass with total volume of 300 ml. The pictorial view of Rosett cell is shown in Figure 3.3a. For sonication using medium and large horns, stainless steel sonication chamber (fabricated at Environmental Engineering Laboratory, AIT) were employed. The sonication chamber has a total volume of 600 ml. The pictorial view of the stainless steel is shown in Figure 3.3b. Design details of stainless steel chamber are shown in the Figure 3.4.





Figure 3.4a Rosett cooling cell

Figure 3.3b Stainless steel chamber



Figure 3. 5 Sonication chamber design with large horn and setup

### 3.5 Selection of Horn

Thickened WAS (TWAS) of 100 ml with TS content of 3% (as obtained from section 3.2) was sonicated in a batch mode using three different horns as discussed in Section 3.3 at different sonication times of 0 (control), 30, 60, 120, 240 and 480 seconds. The amplitude was kept constant at 95% for all sonication durations. The SCOD released at different sonication durations were determined at constant power input (~ 190 W). The SCOD was plotted against sonication time for all three horns. The horn that releases the highest SCOD was chosen for all subsequent sonication tests.

# 3.6 Ultrasound Pretreatment

Ultrasonic experiment was carried out with 100 ml TWAS (TS content 3%) in sonication chamber. The ultrasonic unit was operated at a constant frequency of 20 kHz. The sludge sample was sonicated at different power inputs (e.g., 50, 100, 150 and 190 W), and at different sonication durations of 0 (control), 30, 60, 120, 240 seconds for each power level. The detail operating conditions of ultrasonic unit are presented the Table 3.1.

Parameters	Range	Remarks
Frequency	20 kHz	Constant
Probe immersed	2 cm	Fixed
Sludge volume	100 mL	Fixed
TS content	3%	Fixed
Power input (P)	50, 100, 150, 190 W	Variable
Sonication duration	0, 30, 60, 120, 240 and 480 seconds	Variable

Table 3.1 Operating conditions for ultrasonication

# 3.7 Optimization of Sonication Conditions

# 3.7.1 Calculation of specific energy input and ultrasonic density

**Specific energy input:** The important parameters affecting the ultrasonic disintegration are the power input, TS content, sonication time and volume of sludge to be sonicated. These parameters can be lumped together into a single parameter, commonly known as "specific energy input." The specific energy input was calculated using the following equation:

$$SE = \frac{P.t}{V.TS}$$
 (Eq. 3.1)

Where,

SE : Specific energy input (kWs/g TS)
P : Power inputs (W)
t : Sonication times (second)
V : Volume of sludge used for sonication (ml)
TS : Total solids (g/l)

**Ultrasonic density (UD):** It relates to the power supplied per unit volume of sludge and has a unit of W/L or kW/L or W/ml [ML<sup>-1</sup>T<sup>-3</sup>]. As long as the solids content remains fairly constant, the ultrasound dose is a practical method of expressing the energy input for the disintegration of sludge. The ultrasonic density was estimated using the following relationship:

$$UD_{avg} = \frac{P_{avg}}{V} \qquad \Lambda \Lambda \Lambda \Lambda \Lambda$$
(Eq. 3.2)

Where,

UDavg:Average ultrasonic density (W/ml);Pavg:Power input (W)V:Volume of sludge used for sonication (ml)

#### 3.7.2 Selection of optimal specific energy input:

100 ml of TWAS obtained from Section 3.2 was sonicated with a selected horn as discussed above, at different power inputs (50, 100, 150 and 190 W). Different sonication times of 0, 30, 60, 120, 240 and 480 seconds were investigated at each power input. The SCOD release at each power input was determined at different sonication durations. The specific energy input was calculated using equation (3.1) and the SCOD release was plotted against specific energy input for each ultrasonic density (calculated using equation 3.2).



As above graph, Optimum point, A was selected to achieve maximum SCOD release at lowest energy input. Optimum sonication time was calculated using value of Specific energy and ultrasonic density corresponding to point, by applying the equation (3.1).

### **3.8** Evaluation of Sludge Disintegration Efficiency

SCOD, BMP and microscopic examination will be employed to evaluate the sludge disintegration efficiency. Some detail discussions are given in the following section.

#### 3.8.1 Soluble chemical oxygen demand (SCOD)

The SCOD release was used as a direct measurement of sludge disintegration. When WAS is sonicated, the intracellular materials from bacterial the cell was released into the aqueous phase. An increased SCOD after ultrasonic treatment of sludge is an indication of sludge disintegration efficiency. SCOD release for TWAS was determined for each operating condition listed as per Standard Methods (APHA, 2003). The samples were centrifuged at 5000 rpm for 30 minutes and the supernatant was filtered through 0.45  $\mu$ m pore size membrane filter.

#### 3.8.2 Biochemical Methane Potential (BMP)

BMP test was carried out to determine the anaerobic biodegradability by comparing methane production for different sonication conditions applied to the same substrate with the same inoculum under mesophilic condition  $(37^{\circ}C)$  in the batch anaerobic digester. Inoculum used was collected from the effluent line of lab scale semi-continuous mesophilic anaerobic digester in the ambient laboratory working under steady state conditions with SRT of 20 days. Substrate was sonicated at different power inputs (50, 100, 150, 150 W). Different sonication times of 0, 30, 60, 120, 240 and 480 seconds were investigated at each power input.



Figure 3.6 The serum bottle with capped with butyl rubber stopper used in BMP test

In BMP test, all the experiments were undertaken using 125 ml serum bottle capped with butyl rubber stoppers and wrapped in aluminum foil to prevent photolysis as shown in the Figure 3.5. An inoculum of 5 ml and substrate of substrate of 25 ml were added together into serum bottle. All the bottles were sealed after the addition of NaHCO<sub>3</sub> to achieve an alkalinity of 4000 mg/l (as CaCO<sub>3</sub>) and headspace was also purged with oxygen free nitrogen gas. Serum bottles were kept in 37°C incubator until they stopped producing biogas. Daily biogas was measured by inserting needle attached to a syringe (10ml). Methane composition was measured by Gas Chromatography with a packed column. The blank also was undertaken without adding substrate to determine the actual biome methane potential for only substrate.

In this study, cumulative methane production curves with respect to time were obtained first from the methane production experiments; then the modified Gompertz equation was applied to determine the methane production potential (Zwietering et al., 1990).

t,

<i>M(t)</i>	$= P \cdot ex$	$\exp\left\{-\exp\left(-\exp\left(-\exp\left(-\exp\left(-\exp\left(-\exp\left(-\exp\left(-\exp\left(-\exp\left(-\exp\left($	$\left[\frac{R \cdot e}{P}(\lambda - t) + 1\right] $ (Eq. 3.3)
where	,		
	M(t)	=	cumulative methane production (mL) at time
	λ	=	time of lag-phase (day),
	Р	=	methane production potential (mL),
	R	=	methane production rate (mL/day), and
	e	=	exponential (1) $(= 2.71828)$ .

These parameters in Eq. (3.3) were estimated by minimizing the sum square of errors (SSE) between experimental data and estimation from the models. This estimation was carried out by using the 'Solver' function in 'Tools' menu of Microsoft Excel 2002.

### 3.8.3 Microscopic evaluation

Bacterial cell and floc structure will be affected by ultrasonic treatment. To evaluate the effect of sonication on sludge disintegration, sonicated TWAS samples was examined at the cellular level using Light microscope. Sludge morphology of sonicated sludge was compared to control sludge.

### **3.9** Evaluation of Anaerobic Digestibility

The performance of anaerobic digester was observed based on biogas production and composition, total and individual VFA and TS and VS removal. In addition, the dewaterability and pathogen counts (fecal coliform, *E. coli* and *Salmonella* sp.) of the digested biosolids were also determined.

# 3.9.1 Anaerobic digester set-up

Three laboratory scale anaerobic digesters were fabricated using a transparent acrylic cylinder of 14 cm internal diameter and 28 cm heights, covered both ends by acrylic plate. Each digester had an approximate total volume of 4.3 L and working volume of 2L. Mechanical mixer is provided to achieve completely mixing the bioreactor. Each bioreactor was provided with three ports: one for feeding the sludge, second one for withdrawal of digested sludge and last one for biogas collection. The biogas generated during digestion was collected in a 3L Teflon bag, which was measured by 100 mL syringe. Digester design drawing is shown in Figure 3.6.

To establish the effect anaerobic digestibility of full-stream (100% sonicated), part stream (50% sonicated and 50% unsonicated) and control (non-sonicated) WAS at different solids retention time (SRT), a semi continues digestion experiment was performed. Three anaerobic reactors (named  $U_f$ ,  $U_p$ , and  $U_o$ ) were used for full-stream (100% sonicated), part stream (50% sonicated and 50% unsonicated) and non-sonicated WAS respectively. All three reactors were sealed in order to make them air-tight, and placed in a water bath. Experimental setup drawing is shown in Figure 3.7.



Figure 3.7 Anaerobic Digester Design



Figure 3.8 Experimental setup for anaerobic reactors used in this experiment

#### **3.9.2 Digester startup**

All three digesters ( $U_p$ ,  $U_f$ , and  $U_o$ ) were initially seeded with anaerobically digested sewage sludge obtained from a local full-scale anaerobic digester. Right after inoculation, head space in the reactor was purged with oxygen free nitrogen gas. The digesters were kept under completely mixed condition following nitrogen gas purging. The temperature of the digester was immediately increased to 37°C and maintained the mesophilic condition by using hot water bath throughout the testing. The biogas production, VFA, alkalinity and pH were monitored regularly for at least for the first 5 days before start feeding the fresh TWAS. The VS loading to the digester was kept around 20% of design loading rate and operated at SRT of 20 days. The loading was gradually increased to full design loading for the first 20 days. During the feeding, biogas production, total VFA, and pH were analyzed on a daily basis. When the digester performance deter, feeding was stopped and appropriate corrective measure (alkalinity addition) was taken. The digesters were operated until the steady state is reached. The steady state was believed to have reached when the collected data do not vary more that 5%.

#### 3.9.3 Digester operation

After steady state, all digesters were operated for a minimum of three weeks to collect enough steady state data. Initially first run will stared with SRT of 20 days. In this period, biogas production pH, biogas composition, total & individual VFA, VS and TS parameters were analyzed in three times per week. Dewaterability of digested biosolids was analyzed in three times per week after steady state and pathogen count was measured in one time after steady state. Operating condition is presented in the Table 3.2

Parameter	Optimum range
VSS	> 3%
SRT	20-30 days
VSS loading rate	1.6-6.4 kg VSS/(m <sup>3</sup> .day)
Temperature	35-37 ° C

Table 3.2 Typical operating conditions for anaerobic digester

Source: Peavy. H.S et al. (1985)

All three reactors were operated in a semi-continues mode with feeding and decanting, 2 times per day for every day. The digested sludge was withdrawn from digester immediately before feeding. Each run was manually controlled. The biogas produced was collected in a teflon bag connected to a gas outlet. Continuous mixing was applied to each digester by mechanical mixer.

Digester (U <sub>0</sub> ) :	was fed by nonsonicated TWAS
<b>Digester (U</b> <sub>p</sub> ) :	was fed by part stream (50 % sonicated and 50% non sonicated) TWAS
Digester (U <sub>f</sub> ) :	was fed by full stream sonicated TWAS.

The solid retention time (SRT) is the average time which solids are retained in a digester. SRT was changed from 20 to 15 days for corresponding experimental run. Feeding and decanting rate is shown in the table 3.3, and the parameter analyzed in anaerobic digestion is presented in the Table 3.4.

Solid	Control	Full stream	Part stream		Organic
retention	Non-sonicated	Sonicated	Non-sonicated	Sonicated	loading rate
time (days)	mL/day	mL/day	mL/day	mL/day	kg VS/m <sup>3</sup> /day
15	200	200	100	100	1.40
20	150	150	75	75	1.05

Table 3.3 Feeding and Decanting rate with different solid retention time

Following parameters were analyzed one time after steady state.

Parameter	Method		
Total coliform	EC medium test procedure (9222E) APHA standard		
E.coli	EC-MUG medium test procedure 9221F APHA standard		
Salmonellas	Quantitative salmonella procedure 9260D APHA standard		

Parameter	Interference	Frequen cy/week	Method
Biogas production	Solubility of CO <sub>2</sub> in water	1(*,**)	Water displacement method
Biogas composition	Instrumental operational calibration curve	2 (*) 3 (**)	Gas Chromatography equipped with TCD SHIMADZU GC14A, Column SUS, WG-100 mesh
VFA(Total)	Instrumental operational calibration curve	2(*) 3 (**)	Gas chromatograph equipped with a flame ionization detector.
Individual VFA (C2,-C5)	Instrumental operational calibration curve	3(**)	Gas chromatograph equipped with a flame ionization detector.
РН	-	7(*,**)	Standard methods part 4500 B: Electrometric method, pH meter (Glass electrode)
Alkalinity	-	3 (*) 3(**)	Volumetrically by titration with $0.02 \text{ N H}_2\text{SO}_4$
TS	Large, floating particles or submerged agglomerates of no homogenous materials, visible floating oil and grease etc	3 (*) 3 (**)	Standard Method Part 2540 B : TS dried at 105°C
VS	Loss of ammonium carbonate and volatile organic matter during drying	3 (*) 3 (**)	Standard Method Part 2540 B : TS incinerated at 550°C for 2.0 hours.

Table 3.4 Analysis parameters in anaerobic digestion

• \* = Start up period

• \*\* = After steady state

# 3.10 Sample Preparation and Analytical Methods

# 3.10.1 Biogas production and composition

The quality and quantity of digester gas produced can be used to evaluate digester performance. Gas production is directly related biochemically to the amount of volatile solids destroyed and is expressed as volume of gas per unit mass of volatile solids removed. Volume of gas collected in the Teflon bag was measured by measuring cylinder at S.T.P. The cumulative gas collected in Teflon bag was recorded with time in once in week.

When cumulative gas collection becomes to constant value, it was indication of the steady state to be achieved. Gas samples from the sampling point was taken by syringe to analyze the composition (CH<sub>4</sub>, CO<sub>2</sub>, H<sub>2</sub> and N<sub>2</sub>) by using Gas Chromatography attached with Thermal Conductivity Detector (TCD) and Helium as carrier gas. Table 3.5 provides the analytical conditions for Gas Chromatography. The flow of carrier gas was adjusted to 40 mL/ min. The gas sample of 200  $\mu$ L was Injected gases and it was recorded the elution time of the gas. Elution time is the time lapsed between injection of sample and response

of peak at tip. The components of the sample were identified by comparing to standard gas peaks.

Calculations:

Calculate area all peaks by formula area  $=\frac{Base \times height}{2}$ Calculate the concentration of component of interest, % Component in the sample  $=\frac{Area C}{Area T} \times 100$ 

Where,

Area C = Area of peak of component of interest Area T = Sum of areas all the peaks in the sample.

Condition	Digester gas
1. sample volume (mL)	0.2
2. Carrier gas	Helium
3. Working Pressure(kg/cm <sup>2</sup> )	0.75
4. injection/column/detector temp (°C)	50/50/100

# 3.10.2 Volatile Fatty Acids (VFA)

Sample was filled into glass vial for VFA analysis. Suitable column, carrier gas and conditions for gas chromatography is shown in the Table 3.6.

Parameters	Cloumn	Carrier gas	Detector	Column temperature
Acetic, Propionic	FFAP 10%	Не	FID	180°C Inj. Port-210°C Detector-210°C
n-Butyric, Isobuyric	On Chromosorb W	Не	FID	150°C Inj. Port-210°C Detector-210°C
Total VFA	FFAP 10%	Не	FID	180°C Inj. Port-210°C Detector-210°C

Table 3.6 Condition of gas chromatography

- TCD- Thermal Conductivity Detector
- FID- Flame Ionization Detector

The flow the carrier gas was adjusted to 40 mL/min and so also of hydrogen and air for flame to 30 and 300 mL/min respectively. Automatic injection was used to inject soluble sample and recorded elution time of all individual components.

> Inject a 2.0  $\mu$ L of standard mixture of acids and run the chromatograph.

> Inject several times solvent (acetone) to clean the columns.

➢ Inject 2.0 µL of the sample. Calculate area of all peaks of interest. Calculations

$$Rf_i = \frac{Area i}{X_i}$$

Where

 $Rf_i$  = response factor of component i Area i = Area of peak i  $X_i$  = Amount of component in calibration mixture. Calculate Rf of all components of interest,

Conc i = 
$$\frac{Areai}{Rf_i}$$

Where,

Conc i = Amount of component I in sample

Area i = Area of peak of component in sample

The accuracy of sample size is an important factor in the analysis.

# 3.10.3 Capillary Suction Time (CST)

The rate of dewaterability was determined by measuring the capillary suction time, using apparatus is shown in the Figure 3.8 (Yin et al., 2004).



Figure 3.9 Schematic representation of CST apparatus

This original circular setup consists of a sludge column contained in a sample cylinder (Dc = 1.07 cm), which is centered in the middle of two concentric electrodes located at diameter D1 (2.0 cm) and D2 (6 cm) resting on Whatman-17 filter paper. A timing device is started when the waterfront reaches the inner electrode (1) at D1, and is stopped when

the water reaches the outer electrode (2) at D2. The time elapsed will be the capillary suction time.

#### 3.11 Determination of Kinetic Coefficient of Volatile Solid Degradation

Aim of this section was to evaluate the effect of the ultrasound pre-treatment on the kinetics of the volatile solids (VS) destruction. First order kinetic equation is as follows:

$$\frac{dX_s}{dt} = -k_x X_s \tag{Eq. 3.4}$$

Where,

 $X_s$  : Anaerobically degradable fraction (mg/L)

 $X_s$  will be evaluated by assuming the degradable VS fraction ( $f_x = 0.3$ )

 $K_x$  : Hydrolysis constant (d<sup>-1</sup>)

T : Time (s)

The final VS concentration was evaluated using the following equation by the integration of the above equation:

$$X_{Tf} = X_{To} \left[ 1 - f_x \left( 1 - e^{-k_x t} \right) \right]$$
(Eq. 3.5)

Where,

 $XT_f$  = Final total VS concentration (mg/L)

 $XT_o$  = Initial total VS concentration (mg/L)

 $f_x$  = Degradable VS fraction

Final VS concentration was plotted in the graph with digestion period for Control unsonicated, fully stream (100% sonicated) and part stream (50% sonicated and 50% unsonicated). From this graph, Kinectic coefficient of volatile solid degradation was determined to fitting the experimental data with empirical data.

#### 3.12 Energy balance calculation



Assumptions

- 1. Temperature of fresh sludge  $(T_1)=20$  °C
- 2. Average temperature of ambient air temp $(T_2) = 30 \text{ °C}$
- 3. Temperature in the digester( $T_3$ ) = 37 °C
- 4. Specific heat capacity of  $sludge(C_p) = 4.2kJ/kg/^{\circ}C$
- 5. Overall heat transfer coefficient (U) =  $2.5 \text{ W/m}^2/^{\circ}\text{C}$  (Barber, 2005)
- 6. Calorific value of methane = 35.8 kJ/g (Barber, 2005)
- 7. Specific gravity of sludge = 1.02

8. Sludge flow rate (Q) = 150 mL/day and 200 mL/dayHeat requirement for the sludge (Q<sub>1</sub>)

$$= \sum mC_P(T_3 - T_1)$$
  
= Q\*1020\*4200\*(30-20) J/day

Rate of heat addition required to compensate for loss from the digester (Q<sub>2</sub>)  $= UA(T_3 - T_2)$ Where A= Cross-sectional area through which the losing is occurring, 0.153 m<sup>2</sup> = 2.5\*0.153\*(37-30) W = 231.336 kJ/day

#### Energy generation from methane (Q<sub>3</sub>)

Biogas gas mole calculation from biogas volume,

$$n = \frac{PV}{RT}$$

$$n = \frac{1.01325 * 10^5 * V * 10^{-6}}{8.314 * 300}$$

Where, V is measured in mL and ambient temperature is 300K.

$$n = 4.0624 * 10^{-5} * V mol$$

Methane production  $= n \mod/day$ 

Methane production = x g/day

 $Q_3 = x*35.8*kJ/day$ 

Ultrasonic energy input (Q<sub>5</sub>)

$$Q_5 = Q' * UD * t$$

Where,

Q' = Sonicated sludge flow rate (150ml/day for full stream and 75ml/day for part stream) UD = Ultrasonic density (power/ volume) t = Sonication duration

Full stream,  $Q_5 = Q^1 \text{ ml/day}^* \frac{190 \text{ W}}{100 \text{ ml}} *45 \text{ s}$ 

Part stream,  

$$Q_5 = Q^1 \text{ ml/day} * \frac{190 \text{ W}}{100 \text{ ml}} * 45 \text{ s}$$

**Excess energy gain (Q<sub>4</sub>)** =  $Q3 - (Q_1+Q_2-Q_5)$ 

# Chapter 4

### **Results and Discussion**

In this study, a series of sonication tests were conducted to determine the release of SCOD at the different sonication condition and hence to evaluate the impact of ultrasonic pretreatment on the digestibility of semi-continuous anaerobic digestion. The three anaerobic digesters were operated at the mesophilic temperature. The results obtained during the experiment are described and analyzed in the following two parts. The first one is the ultrasonication pretreatment and second one is the evaluation of digestion performance in all three digesters.

# 4.1 Ultrasonic pretreatment

The main aim of ultrasonication pretreatment is to destroy the cell wall of microbes and release the intercellular and extracellular substances into the aqueous phase. The optimization of the energy requirement for efficient sludge disintegration is important for cost-effective digestion of sonicated sludge in the field application. The feed sludge was routinely analyzed to determine important parameters as presented in the Table 4.1. The data obtained was used as the baseline for comparing with after digestion. The volatile solid in the sample sludge was 70% of total solid.

Parameters	Feed sludge
TS (%)	$3 \pm 0.1$
VS (%)	$2.1 \pm 0.1$
VS/TS	0.68 - 0.70
pH	$7.27 \pm 0.05$
CST (s)	$50.5 \pm 3.1$
SCOD (mg/l)	80-100

#### Table 4.1 Characteristics of raw sludge

# 4.1.1 Selection of horn

Thickened waste activated sludge (TWAS) of 100 ml with TS content of 3% was sonicated in a batch mode using three different horns (Large, Medium and Small) at different sonication times. The power input was kept constant at 190 W. The SCOD release at different sonication time was measured at constant power input of 190 W. The SCOD results are presented in the Table 4.2.

Table 4.2 SCOD	release at dif	ferent sonicat	tion time u	using three	e types of horn
				0	

Sonication time (s)	SCOD (mg/L)				
Someation time (s)	With large horn	With medium horn	With small horn		
0	80	80	80		
30	1280	840	1120		
60	2480	1280	2080		
120	4600	1600	3400		
240	7000	4640	6000		
480	9000	8320	9000		

The two sonication chambers were used in this experiment, bigger chamber was used for the large and medium horn and small chamber was used for the small horn. The SCOD release against the sonication time for each horn is shown in Figure 4.1.



Figure 4.1 SCOD release at different sonication time with three types of horn

The SCOD prior to sonication was around 80 mg/L. The SCOD release at any sonication time was found to be higher for the combination of large horn and large chamber. However, SCOD release was lower value for the combination of medium horn and large chamber, since propagation of ultrasonic energy inside the sludge was limited to the certain distance and also distance between horn outer surface and inner surface of chamber was higher. Therefore, in this case some part of sludge had not influence the creation of cavitation bubbles. The large horn gave high SCOD values than medium horn and small horn for any sonication time. Therefore, the large horn was selected for the subsequent experiments.

# 4.1.2 Effect of horn immersion depth on SCOD release

Figure 4.2 shows large horn and large sonication chamber. The ultrasound energy was released from horn surface into sludge. This horn contained two surfaces, such as the circular surface in bottom and the cylindrical surface in side face. The cylindrical surface, which released the sonication energy, changes with immersed depth into the sludge.



Figure 4.2 Large horn and large sonication chamber

In this experiment, the immersion depth of horn of 1cm, 2cm, and 3cm were selected to evaluate the effect of the immersion depth on the SCOD release. The results of this experiment are attached in appendix B1 and B2. SCOD/TS variation with specific energy is shown in Figure 4.3.



Figure 4.3 SCOD/gTS release against specific energy at different immersion depth

The statistical analysis was done using one way ANOVA, hypothesis defined as immersed depth of horn does not affect the SCOD release. F-value and significant level () were 0.009 and 0.991 (Appendix B3). The hypothesis could not be rejected. Therefore, could not conclude that immersed depth of horn affects the SCOD release by sonication pretreatment. It may be the reason that sonication energy released from horn mainly depends on circular bottom surface.

# 4.1.3 Sonication condition optimization

Sludge disintegration depends on many sonication conditions. Though, this experiment focused only on the ultrasonic density and specific energy (S.E). The Table 4.3 shows the SCOD release at different sonication times and at different power inputs. When the sludge sonicated at power input of 190 W for sonication time of 480s, The SCOD release was increased more than ten folds of the SCOD in nonsonicated sludge.

Sociation time (a)	SCOD (mg/L)					
Someation time (s)	P = 50 W	$\mathbf{P} = 100 \ \mathbf{W}$	P=150 W	$\mathbf{P} = 190 \ \mathbf{W}$		
0	80	80	80	80		
30	120	480	800	1360		
60	320	880	1520	1760		
120	640	2080	3360	3600		
240	1440	3800	6800	7200		
480	2000	4600	8200	9000		

Table 4.3 SCOD release at different sonication times and at different power inputs

#### (a) Effect of the ultrasonic density on the SCOD release

From Table 4.3, ultrasonic density was calculated using the equation 3.2 (Appendix B5). SCOD release at various sonication times for different ultrasonic densities is shown in the Figure 4.4.



Figure 4.4 SCOD release at various sonication times for different ultrasonic densities

From Figure 4.4, when the sonication duration was 30s, SCOD release for four different ultrasonic densities was found to be increased steadily with increasing ultrasonic density. When ultrasonic density was increased from 1.5 W/ml to 1.9 W/ml for sonication time of 60s, 120s and 240s, the SCOD release was increased slightly in comparison to other ultrasonic density changes (P, Q, R, S). Therefore, ultrasonic density of 1.5 W/ml was found to be appropriate value for effective SCOD release at sonication duration of 60, 120, 240s and 480s.

#### (b) Effect of the specific energy on the mgSCOD/gTS releasae

From Table 4.3, specific energy was calculated using the equation 3.1 (Appendix B6). The mgSCOD/gTS release at various specific energies for different ultrasonic densities is shown in the Figure 4.5. Form this graph, when the specific energy was increased up to a certain value of 2.3 kWs/gTS (X), the mgSCOD/gTS release was increased rapidly at all ultrasonic densities. Thereafter, increment in the mgSCOD/gTS release was slowed down upto 4 kWs/gTS of the specific energy. Again, the mgSCOD/gTS release was found to be increased rapidly with increasing specific energy from 4 kWs/gTS to12 kWs/gTS. The beyond 12 kWs/gTS (Y), increment in mgSCOD/gTS release were considered together, specific energies of 2.3 kWs/gTS and 12 kWs/gTS were found to be the critical values for the effective mgSCOD/gTS release. However, the specific energy should be controlled within 12 kWs/gTS for effective mgSCOD/gTS release.



Figure 4.5 mgSCOD/gTS release at various specific energies for different ultrasonic densities.

From both specific energy values, sonication duration was calculated using equation 3.1 in chapter 3. The sonication power input of 190 W was selected for this calculation to reduce the sonication duration.

$$SE = \frac{P.t}{V.TS}$$
For SE = 2.3 kWs/gTS,  $t = 36.3 \text{ s}$   
For SE = 12 kWs/gTS,  $t = 189.5 \text{ s}$ 
(Eq. 3.1)

#### 4.1.4 Effect of the sonication on temperature increase

When sludge is exposed to ultrasonic waves, an increase of temperature was observed. The increased temperature generated in the sonication chamber during the ultrasonic treatment was investigated. In each batch sonication, temperature was recorded in start and end of each sonication. Temperature increments ( $\Delta$ T) related to sonication time (t) is given in Appendix B4.

A significant increase of temperature was observed at long sonication duration. Since this research intended to study the effect of ultrasonic treatment itself, so experimental have to be dissociated from thermal effects. Considerable temperature increase should be avoided. The power input and sonication duration have a significant influence in the temperature of the sludge sample. These temperature increments were plotted against the sonication time and are shown in Figure 4.6.



Figure 4.6 Temperature increment with sonication time for different power input

From above figure 4.6, temperature increment for sonication time of 36.3s and 189.5s, which were selected as sonication duration for effective mgSCOD/gTS release, are 18°C and 51°C respectively.

For SE = $2.3 \text{ kWs/gTS}$ ,	t = 36.3 s	$\Delta T = 18 \ ^{\circ}C$
For SE = $12 \text{ kWs/gTS}$ ,	t = 189.5s	$\Delta T = 51 $ °C

In this research, SE of 2.3kWs/gTS and sonication duration of 36.3s were suitable to eliminate the thermal effect on sludge disintegration. The sonication duration of 45s was selected instead of 36.3s to investigate the anaerobic digestibility of control, full stream and part stream digester.

#### 4.1.5 Light microscopic image of untreated and tread WAS sample.

In this research, light microscope was used to observed sludge structure of ultrasonically treated and nonsonicated WAS sample. The Figure 4.7 shows the light microscope image of ultrasonically treated sludge with different sonication duration and nonsonicated WAS sample. In which, Figure 4.7 (a) reveals that many smaller sludge cell formed sludge floc, which was bounded by extra cellular substances. When sludge was disintegrated with ultrasonic power of 190 W for sonication duration of 30s, the sludge floc was deagglomerated into individual particles. When sonication time was increased further to 60s and 120s with same power input, size reduction of particle was observed from Figure 4.7 (c) and 4.7 (d). When the sonication time was increased to 240s for the same ultrasonic power, the filaments and flocs were almost completely disintegrated.







Microscopic image of (a) Non sonicated sludge (b) Sonicated sludge with sonication time of 30s at power input of 190 W (c) Sonicated sludge with sonication time of 60s at power input of 190 W (d) Sonicated sludge with sonication time of 120s at power input of 190 W

Figure 4.7 Light microscope image of ultrasonically treated with different sonication duration and untreated WAS sample

The Figure 4.8 shows the light microscopic image of ultrasonically treated sludge with different power input and nonsonicated WAS sample. Normally, WAS floc entangled within a large numbers of filaments were observed prior to sonication as shown in Figure 4.8 (a). When the sludge was sonicated with different power increments for the same sonication time, the different level of disintegration was observed as shown in the Figures 4.8 (b), 4.8 (c) and 4.8 (d). This Figures show the structural changes in flocs, disappearance of filaments, etc., during ultrasonic treatment



Figure 4.8 Light microscope image of ultrasonically treated with different power input and untreated WAS sample

# 4.1.6 Sonication condition optimization by BMP

The impact of the ultrasonic power and sonication duration on the anaerobic digestibility efficiency was evaluated by the methane accumulation in the BMP test. All the experiments were carried out in identical conditions such as temperature and pressure. The Figure 4.9 shows the accumulated methane production with the digestion period at different sonication durations at the power input of 190W. In the first two days, the methane production rate was low for all sonication times due to lag phase of anaerobic digestion. After second day, methane production rate was increased exponentially and after 20 days it was retarded due to substrate limitation or VFA accumulation. In the 30 days, the accumulated methane production at different sonication power inputs and sonication durations (Appendix C1 and C2).



Figure 4.9 Accumulated methane production with digestion time at different sonication times at the power input of 190W

Sonication time	Power input	BMP (mesophilic)	BMP (STP)
Solication time	(W)	Accumulated methane	Accumulated methane
(5)	(**)	volume (ml)	volume (ml)
	0	41.639	36.980
	50	56.022	49.755
30	100	58.970	52.373
	150	64.200	57.017
	190	66.990	59.495
	0	41.639	36.980
	50	60.747	53.951
60	100	67.856	60.264
	150	75.487	67.042
	190	83.476	74.137
120	0	41.639	36.980
	50	67.826	60.238
	100	74.435	66.107
	150	84.678	75.205
	190	96.261	85.491
240	0	41.639	36.980
	50	74.011	65.731
	100	82.965	73.683
	150	94.914	84.296
	190	104.715	93.000

Table 4.4 Accumulated methane production in different sonication conditions

The impact of the different specific energy applied to the raw substrate on the biochemical methane potential was evaluated; the Figure 4.10 shows the biochemical methane potential with the specific energy at the different ultrasonic density applied (Appendix C3). In this experiment, the control batch digester produced the total methane production of 36.98 ml at STP. When the specific energy was increased in all the ultrasonic density, biochemical methane potential also increased. However, after certain specific energy (12 kJ/gTS), the biochemical methane potential was not increased significantly. Therefore, the specific energy of 12kJ/gTS is appropriate specific energy for efficient biochemical methane potential at the mesophilic condition.



Figure 4.10 Biochemical methane potential with the specific energy

### 4.1.7 Kinetic analysis of methane production in BMP batch test

The cumulative methane production with digestion time obtained from the experiments with different power inputs and the sonication times are presented in Appendix C2. The power input and sonication time were found to affect the methane production significantly. The kinetic parameters methane production potential (P), methane production rate (R) and lag phase ( $\lambda$ ) estimated based on Equation 3.3 in the section 3.8.2 are presented in Table 4.5. Methane production was well correlated to the modified Gompertz equation (R2 > 0.98).

Power	Time	Methane potential	Methane production rate	Lag time	$\mathbb{R}^2$	Methane potential	Methane production
		P(mL)	R (mL/d)	$\lambda$ (d)		illerease (70)	Tate merease (70)
	0	41	2.53	1.50	0.9952		
	30	56	3.33	1.49	0.9960	35.4173	31.4390
50	60	60	3.68	1.60	0.9970	46.3211	45.2480
	120	68	3.95	1.53	0.9972	66.6798	55.8150
	240	74	4.45	1.82	0.9988	80.0343	75.7046
	0	41	2.54	1.46	0.9953		
	30	59	3.56	1.44	0.9966	42.6912	40.2765
100	60	68	3.97	1.48	0.9972	64.5971	56.6547
	120	76	4.38	1.54	0.9976	83.5083	72.8118
	240	82	5.04	2.00	0.9981	99.7855	98.5032
	0	41	2.54	1.41	0.9953		
	30	64	3.69	1.30	0.9967	55.6710	45.2458
150	60	77	4.46	1.65	0.9964	87.6596	75.5993
	120	86	5.13	1.72	0.9962	108.5534	101.9764
	240	95	5.68	2.32	0.9967	130.9681	123.7771
	0	41	2.54	1.37	0.9953		
190	30	68	3.95	1.34	0.9972	63.6138	55.5929
	60	86	4.91	1.80	0.9935	106.9885	93.2674
	120	98	5.99	2.03	0.9964	136.8235	135.7733
	240	105	6.50	2.60	0.9971	152.9886	155.7644

Table 4.5 The kinetic parameters estimated based on the Gompertz equation

# 4.2 Quasis-Steady State Condition and Digester Performance

The first set of digestion experiment was carried under identical conditions, such as temperature and mixing condition. In this experiment, all three digesters were fed with nonsonicated WAS at aa SRT of 20 days. The biogas production rate was increased during start-up period of the digestion process, because VS loading rate was gradually increased to the full design loading rate (1.05 kgVS/m<sup>3</sup>/day) for the first 20 days. All three digesters had reached a quasi-steady state value by 35 days from experimental initiation. The digester performance data under quasi-steady state condition are presented in the Table 4.6.

Parameters	Digester A	Digester B	Digester C
Alkalinity(mg/L as CaCO <sub>3</sub> )	1891.7±38.2	1916.7±28.9	1916.7±101.0
pH	6.99±0.08	6.99±0.07	6.97±0.05
TVFA (mg/L as acetic acid)	15.26±0.92	14.15±1.42	15.22±0.19
TS removal (%)	10.2±2.2	9.0±1.3	10.5±1.0
VS removal (%)	20.0±0.5	20.3±0.5	19.5±0.5
Biogas production (ml/week)	1151.7±48.8	1188.3±10.4	1205±13.2
Methane production (ml/week)	685.9±2.1	701.8±0.4	704.1±2.1
Methane content (%)	58.3±0.4	59.4±0.2	58.8±0

 Table 4.6 Performance data on anaerobic digester under quasi steady state conditions

\* Mean ± standard deviation of 3 samples under quasi-steady state condition.

These results reveal that all three digesters performances were nearly the same because all important parameters were not significantly different for all three digesters based on statistical analysis using one-way ANOVA (at = 0.01). The details of statistical analysis are presented in Appendix D13. The VS reductions of all three digesters were 19.5-20.5%.

# 4.3 Effect of Ultrasound Pretreatment on Digester Performance

Following quasi-steady state operation of all three digesters, one digester was fed with 100% sonicated WAS known as full stream  $AD(U_f)$  and another was fed with mixture of 50% sonicated and 50% nonsonicated WAS known as part stream  $AD(U_p)$ . The third digester was continuously fed with nonsonicated WAS know as part stream as control  $AD(U_o)$ .

# 4.3.1 TS and VS removal efficiency

The TS and VS of the digested and the feed sludge in both the start-up and experimental period are graphically represented in Figure 4.11 and Figure 4.12 (Appendix D1 and D2). During the digestion operation, the TS and VS content of the feed sludge were maintained almost constant around 30 and 20 g/L respectively. After steady state digestion, TS removal efficiencies of all three digesters  $AD(U_0)$ ,  $AD(U_f)$  and  $AD(U_p)$  were around 20%, 24.6% and 21.7% at an SRT of 20 days and 15%, 19.9% and 16.4% at an SRT of 15 days respectively (Appendix D3). The TS removal efficiency was increased in the full stream digester by 22.8% in a SRT of 20 days and 32.2% in a SRT of 15 days in comparison to the control digester. This reveals that the full stream digester shows better TS removal in comparison to the control digester in SRT of 15 days. Though, the part stream digester shows the same TS removal in comparison to the control digester in both SRTs of 15 and 20 days.



Figure 4.11 TS of digested sludge in all three reactors with digestion operation

VS removal efficiencies of all three digesters  $AD(U_0)$ ,  $AD(U_f)$  and  $AD(U_p)$  were 19.2%, 25.9% and 24% at a SRT of 20 days and 12.1%, 19.4% and 16.7 % at SRT of 15 days respectively (Appendix D4). Interestingly, for full stream digester, the VS removal improved by 34.6% with respect to the control digester in SRT of 20 days and 60.3% with respect to the control digester in a SRT of 15 days. VS removal with respect to the control digester.



Figure 4.12 VS of digested sludge in all three reactors with digestion operation

Moreover, results show that VS removal efficiency of full stream digester with a SRT of 15 days was almost the same as that in the control digester with SRT of 20 days. This shows the significance of ultrasonic pretreatment of WAS in the case of VS removal.

### 4.3.2 Biogas and methane production rate and biogas composition

The biogas production rate in all three digesters with the digestion period is presented in Table 4.7. The quasi steady state of the start-up period was reached in 35 days. It was confirmed that biogas production of all three digesters were 1151.7, 1188.3and 1205ml/week and the percentage change in biogas production was also less than 5% of the mean biogas production for each digester. By 42 days, sonicated sludge feeding was started for anaerobic digesters  $AD(U_f)$  and  $AD(U_p)$ . After sonicated sludge feeding, the biogas production rate in the full stream and part stream digester was increased rapidly for the first two weeks, and then the production rate was constant for one week, and it was repeatedly increased to reach steady state conditions. The biogas production rate was increased suddenly in 84 days, due to unexpected increment in temperature from 35°C to 45-50°C caused by a technical fault in the temperature controller.

Days	Control (ml/week)	Full stream(ml/week)	Part stream(ml/week)			
7	883	940	940			
14	1010	1170	1150			
21	1020	1170	1150			
28	1100	1200	1220			
35	1180	1180	1200			
42	1175	1185	1195			
S	onication sludge feedi	ng started with SRT of 20	0 days			
49	1205	1887	1582			
56	1250	2358	2077			
63	1260	2480	2110			
70	1320	2815	2552			
77	1350	2805	2684			
84	1612	3025	2816			
91	1395	2790	2645			
98	1415	2805	2650			
105	1410	2835	2640			
SRT of 15 days						
112	1750	3325	2810			
119	1890	3520	2885			
126	2015	3635	3139			
133	2010	3640	3180			
140	2000	3655	3150			

Table 4.7 Biogas production rate in all three reactors with digestion period

From Table 4.6, the steady state biogas production rate for all three digesters  $AD(U_o)$ ,  $AD(U_f)$  and  $AD(U_p)$  was found during digestion operation with a SRT of 20 days, which was 1387, 2800 and 2660mL/week respectively. The biogas production rate in the full stream and part stream digesters was increased by 102% and 91% respectively, for a SRT of 20 days.

However, the biogas production rate was increased in the full stream and part stream digesters by 81.4% and 57.1% respectively, for a SRT of 15 days in comparison to control digester. High biogas production was observed in full stream digester than that in part stream digester in both SRTs, though the specific biogas production (biogas volume/VS removed) for all three digesters  $AD(U_0)$ ,  $AD(U_f)$  and  $AD(U_p)$  was 0.328, 0.492, and 0.502 L/g VS removed at an SRT of 20 days and 0.565, 0.638 and 0.644 L/g VS removed at an SRT of 15 days respectively. These results reveal that specific biogas production was same in both full stream and part stream digesters for both SRTs, i.e. 0.500 L/g VS removed at an SRT of 20 days and 0.64 L/g VS removed in a SRT of 15 days respectively. Benabdallah El-Hadj et al. (2006) found that the specific biogas potential for control and sonicated sludge was 0.76 and 0.883 L/g VS removed respectively. This is a higher value than the value obtained in this research, since the feed sludge was composed by mixture of primary (75% on TS basis) and secondary (25% on TS basis) sludge. Specific biogas potential obtained in this research is opposite to the result found by Thehm el al. (2000), in which specific biogas potential was decreased in the anaerobic digester fed with sonicated sludge.



Figure 4.13 Methane production rate for all three reactors with digestion period

Figure 4.13 shows the methane production rate in all three digesters with the digestion period (Appendix D5). From this graph, the full stream digester shows the high methane production rate rather than the part stream and control digester. Those are 1712.6 ml/ week at an SRT of 20 days and 2203 ml/week at an SRT of 15 days. The methane production rate in the full stream digester was improved by as much as 110% at an SRT of 20 days and 88.7% at an SRT of 15 days in comparison to the control digester. However, the methane production rate in part stream digester was improved by as much as 94.5% at an SRT of 20 days and 61.8% at an SRT of 15 days in comparison to the control digester. The specific methane yield is presented in Table 4.8.
Specific Methane Yield	Control (L/gVS removed)	Full stream (L/gVS removed)	Part stream (L/gVS removed)
SRT = 20  days	0.192	0.300	0.300
SRT = 15 days	0.328	0.386	0.385

Table 4.8 Specific methane yield in all three reactors for different SRTs

Interestingly, specific methane yields were the same in the full stream and part stream digesters in both SRTs. It was improved by as much as 56% at an SRT of 20 days and 18% at an SRT of 15 days in both the full stream and part stream digesters in comparison to the control digester.



Figure 4.14 Biogas composition for all three reactors with digestion period

The biogas composition was analyzed in each week using gas chromatography (GC-TCD). Figure 4.14 shows methane content in biogas in all three digesters for both SRTs. The methane content in biogas for all three anaerobic digesters  $AD(U_o)$ ,  $AD(U_f)$  and  $AD(U_p)$  was 58.56%, 60.98% and 59.8 % at an SRT of 20 days and 58.13%, 60.47% and 59.83% at an SRT of 15 days (Appendix D5). This result indicated that the percentage of methane in the biogas was improved slightly in the full stream and part stream anaerobic digesters than the control digester. The reason could be a favorable condition for methanogenic bacteria in the full stream and part stream digesters. Thehm et al. (2000) found that the methane content of biogas in the sonicated and control sludge samples were 66% and 62.8% respectively, and these results were found to be the same as those in this research.

#### 4.3.3 Dewaterability measurement by CST

The capillary suction time (CST) was measured after steady-state to be achieved for each SRT. CSTs of raw and digested sludge in each digester are shown in Figure 4.13 (Appendix D6). The results of the CST obtained were concluded as follows:

Full stream	>	Part stream	>	Control digested	>	Raw Sludge
digested sludge		digested sludge		sludge		

During the ultrasonication, sludge flocs became smaller particles, which provided more absorbing surface area for water. On the other hand, water retained in sludge flocs and inside the cells was released into the aqueous phase. The former theory is predominant to determine the dewaterability (CST) of the sludge. Therefore, the CST of the full stream digested sludge is higher than the control digested sludge.



#### Capillary suction time for different SRTs

Figure 4.15 Capillary suction time of digested and raw sludge for all three reactors

From Figure 4.15, the capillary suction time of digested sludge in the each digester was increased with decreasing SRT. The reason is that when SRT was decreased, the TS of digested sludge were increased. It mean, the no of particle was increased in digested sludge for a SRT of 15 days. The high no of particles facilitate the high absorbing area for water.

#### 4.3.4 Total and individual Volatile Fatty Acid Results

Total and Individual VFA were measured in digested effluent sludge. The Figure 4.16 shows the Total and Individual VFA in all the three digesters for both SRTs of 20 and 15 days (Appendix D7-D11). Lower Total VFA was observed in the digested sludge from part stream digester rather than digested sludge from the full stream and control digesters for

both SRTs of 20 and 15 days. It is an indication that methanogenesis is predominant in the part stream condition. The TVFA of sonicated sludge was also high in comparison with the untreated sludge (Mao and Yeow Show, 2007), though the TVFA of the digested sludge in the full stream digester was slightly lower than the control digested sludge at an SRT of 20 days. The reason is that the TVFA in the sonicated sludge was available to the methanogenic bacteria than that of untreated sludge. However, the TVFA in digested sludge from the full stream digester was slightly higher than the digested sludge from the control digester at an SRT of 15 days. Since the SRT is not sufficient, that high amount of TVFA in the sonicated feed sludge has to be converted to biogas. Difference in the TVFA in the digested sludge between the full stream digester and the control digester in both SRTs was low, hence the full stream digester was well stabilized as the control digester. Considering the Individual VFA in sludge, specially acetic acid and iso-butyric acid concentration in the digested sludge was influenced by ultrasonic pretreatment. Low acetic acid in digested sludge was observed in all the three digesters for both SRTs of 20 and 15 days. When the SRT was decreased from 20 to 15 days, the acetic acid and the propionic acid in the digested sludge in the corresponding digester was increased. The valeric acid in the digested sludge was almost the same in all the three digesters for both SRTs.



Figure 4.16 Individual and Total VFA concentration of digested sludge effluent

Figure 4.17 shows the percentage distribution of the Individual VFA in the TVFA in all the three digesters for both SRTs of 20 and 15 days. When SRT was decreased from 20 to 15 days, the percentage of acetic acid in the Total VFA in all the three digesters was decreased significantly. However the percentage of propionic acid in the Total VFA in all three digesters was increased significantly. The percentage of valeric acid in the Total VFA remained constant in all the three digesters for both SRTs.



Figure 4.17 Percentage of Individual VFA in Total VFA of digested sludge effluent

#### 4.3.5 pH and Alkalinity of digested sludge



Figure 4.18 pH and Alkalinity of digested sludge with digestion period

The Figure 4.18 shows the pH and alkalinity of digested sludge in the all three digesters (Appendix D12). The pH of the digested sludge in each digester was generally slightly different, which varied in the range of 6.84-7.15. The pH of the digested sludge in the control digester was slightly slower than the other two digesters for a SRT of 20 days, as

the TVFA of the control sludge was higher than the digested sludge from the other two digesters as mentioned in Section 4.3.4. However, the pH of the digested sludge in the full stream digester was lower than the digested sludge from the other two digesters for a SRT of 15 days. The reason is that the high total VFA feeding, increased by sonication pretreatment, was not mineralized efficiently due to the short SRT of 15 days. The alkalinity of the digested sludge in all the three digester was in the range of 1850- 2150 mg/L as CaCO3.

#### 4.3.6 Energy balance of digester and specific energy recovery

The energy balance calculation procedure is attached in the appendix E1. All energy input and energy gained is shown in the Figure 4.19.



Figure 4.19 Energy input and output across the digester

The energy input and output in the digester is summarized in Table 4.9. The energy obtained from the biogas in the full stream digester was higher in comparison to the other two digesters, which was 5.894 kJ/day for a SRT of 20 days, and 7.324 kJ/day in SRT of 15 day. The ultrasonic energy input into the full stream digester was double the value of the ultrasonic energy input into the part stream digester. The energy required to heat the sludge for each digester was the same in the same SRT. The heat loss in the surroundings from the digester was considerably higher than the other energy inputs, which was constant for each digester. The specific energy gained from each digester was compared in the Table 4.9, in which the heat loss in the surroundings and the energy required to heat the sludge was the same in all three digesters for both SRTs and Heat required to heat the sludge was the same in each digester with same SRT. Therefore, In comparison of the specific (Net) energy gain, the heat required to heat the sludge and the heat loss in surrounding was not included in the calculation of specific energy gained from the biogas. The specific energy gained from biogas from full stream and part stream digester were negative value because the ultrasonic energy input was higher than the energy gained from the biogas. Figure 4.20 shows the energy gained from the biogas and the ultrasonic energy input in all the three digesters for both the SRTs.



Figure 4.20 Energy gained from biogas and Ultrasonic energy input in all three digesters

Based on the energy balance calculation, energy gained from biogas in the full stream digester was around two-fold compared to the energy gained from biogas in the control digester. In part stream digester, around 88% of sonication energy input was replenished in the form of biogas. This energy could be reused to generate electricity to run ultrasound system.

		SRT = 20 days		SRT = 15 days			
Parameters	Control	Full stream	Part stream	Control	Full stream	Part stream	
Biogas (ml/day)	198.929	401.257	379.257	286.900	520.471	450.900	
Biogas (mol/day)	0.0081	0.0163	0.0154	0.0117	0.0211	0.0183	
Methane content (%)	58.56	60.98	59.80	58.13	60.47	59.83	
Methane (mol/day)	0.0047	0.0099	0.0092	0.0068	0.0128	0.0110	
Methane (g/day)	0.0757	0.1590	0.1474	0.1084	0.2046	0.1753	
Energy gained from biogas,Q <sub>3</sub> (kJ/day)	2.711	5.694	5.277	3.881	7.324	6.277	
Ultrasonic energy input (Q <sub>5</sub> ) (kJ/day)	0	(12. 825)	(6. 4125)	0	(17.100)	(8.550)	
Energy required to heat the sludge (kJ/day)	4.4982	4.4982	4.4982	5.9976	5.9976	5.9976	
Heat loss in digester (kJ/day)	231.336	231.336	231.336	231.336	231.336	231.336	
VS removal (g/day)	0.606	0.816	0.756	0.508	0.816	0.700	
Specific energy gained from digester (kJ/gVS <sub>r</sub> )	4.473	-8.739	-1.501	7.639	-11.981	-3.246	

Table 4.9 Energy Balance in all three digester for both SRTs of 20 days and 15 days

\*  $VS_r - VS$  removal

#### 4.3.7 Determination of Hydrolysis coefficients

The derivation of equation of final VS concentration is given by section 3.11, which is,

$$X_{Tf} = X_{To} \left[ 1 - f_x \left( 1 - e^{-k_x t} \right) \right]_{\text{(Eq 4.1)}}$$

Where,

XTf	=	Final total VS concentration (mg/L)
ХТо	=	Initial total VS concentration (mg/L)
fx		= Degradable VS fraction ( $fx = 0.3$ )
kx	=	Hydrolysis constant (d-1)
Т	=	Time (s)

The equation was modified as follow,

$$k_x = -\frac{1}{T} \ln \left[ 1 - \left( \frac{1 - \frac{\lambda_{Tf}}{\lambda_{To}}}{f_x} \right) \right]$$

Hydrolysis constants for each digester for each SRT were listed in the Table 4.10.

Digester		X <sub>Tf</sub> (mg/L)	X <sub>To</sub> (mg/L)	(1- X <sub>Tf</sub> / X <sub>To)</sub>	$\left[1 - \left(\frac{1 - \frac{X_{Tf}}{X_{To}}}{f_x}\right)\right]$	$k_x (d^{-1})$			
		Hyd	rolysis cor	nstant (d <sup>-1</sup> ) with	SRT of 20 days				
Control	Max	16.9	21	0.1952	0.3492	0.053			
SRT=20	Min	17	21	0.1905	0.3651	0.050			
Full stream	Max	15.6	21	0.2571	0.1429	0.097			
SRT=20	Min	15.5	21	0.2619	0.1270	0.103			
Part	Max	16	21	0.2381	0.2063	0.079			
SRT=20	Min	15.9	21	0.2429	0.1905	0.083			
		Hydrolysis constant (d <sup>-1</sup> ) with SRT of 15 days							
Control	Max	18.4	21	0.1238	0.5873	0.035			
SRT=15	Min	18.5	21	0.1190	0.6032	0.034			
Full	Max	16.9	21	0.1952	0.3492	0.070			
SRT=15	Min	17	21	0.1905	0.3651	0.067			
Part	Max	17.4	21	0.1714	0.4286	0.056			
SRT=15	Min	17.6	21	0.1619	0.4603	0.052			

Table 4.10 Hydrolysis constants calculation in all three digester

From Table 4.10, the hydrolysis coefficient of digestion in the, full stream and part stream digesters was improved by 94% and 57% at an SRT of 20 days, and 99% and 57% at an SRT of 15 days respectively in comparison to the control digester. The ultrasonic pretreatment enhanced the VS hydrolysis coefficient in the digester.

# Table 4.11 Summary of Results

Devemeters		SRT = 20 days			SRT = 15 days		
rarameters	Control	Full stream	Part stream	Control	Full stream	Part stream	
Digester operating conditions							
Temperature (°C)		35-37			35-37		
Flow rate (mL/day)		150			200		
HRT (days)		20			15		
VS loading rate (kg VS/ m <sup>3</sup> /day)		1.05			1.40		
Feeding conditions							
TS (g/L)		$3.02 \pm 0$			$2.96 \pm 0$		
VS (g/L)		$21 \pm 0$			$21 \pm 0$		
pH		$7.27 \pm 0.05$			$7.27 \pm 0.05$		
Treated effluent Characteristics							
TS (g/L)	26.00±0.07	25.04±0.05	25.64±0.05	26.44±0.05	25.42±0.08	26.16±0.05	
VS (g/L)	16.96±0.05	15.56±0.05	15.90±0.05	18.46±0.05	16.92±0.04	17.5±0.07	
pH	6.84-7.08	6.91-7.14	6.91-7.14	7.05-7.12	7.00-7.15	7.01-7.15	
Alkalinity (mg/L as CaCO <sub>3</sub> )	1800-1950	1850-2050	1975-2150	1900-2050	1850-2000	1975-2150	
Capillary suction time (s)	69.1±7.6	95.0±13.6	84.6±11.7	72.9±5.8	138.8±15.8	112.8±10.3	
TVFA (mg/L)	10.25±2.06	9.28±1.73	7.98±0.57	8.4±0.24	10.74±0.95	6.46±0.99	
Removal efficiency							
TS removal (%)	20.00±0.34	24.57±0.26	21.71±0.26	15.05±0.26	19.90±0.40	16.38±0.26	
VS removal (%)	19.24±0.26	25.90±0.26	24.00±0.26	12.10±0.26	19.4±0.21	16.67±0.34	
<b>Biogas Characteristics</b>							
Biogas production rate (ml/week)	1392.5±29.6	2808.8±18.9	2654.8±19.1	2008.3±7.6	3643.3±10.4	3156.3±21.2	
Biogas yield (L/gVS removed)	$0.328 \pm 0.007$	$0.492 \pm 0.002$	$0.502 \pm 0.004$	$0.565 \pm 0.002$	0.638±0.002	$0.644 \pm 0.004$	
Methane Production rate(ml/week)	816.0±17.3	1712.6±14.0	1587.5±10.5	1167.5±0.23	2203.0±13.6	1888.6±28.9	
Methane composition (%)	58.56±0.09	$60.98 \pm 0.08$	59.80±0.07	58.13±0.21	60.47±0.23	59.83±0.51	
Ultrasonic energy input (kJ/day)	0	12.825	6. 4125	0	17.100	8.550	
Energy obtained from biogas (kJ/day)	2.711	5.694	5.277	3.881	7.324	6.277	
Kinetic coefficient for VS removal (d <sup>-1</sup> )	0.05-0.053	0.097-0.103	0.079-0.083	0.034-0.035	0.067-0.070	0.052-0.056	
Biogas energy gained/VS removed (kJ/gVS)	4.473	6.978	6.981	7.639	8.975	8.968	

### Chapter 5

#### **Conclusions and Recommendations**

#### 5.1 Conclusions

The ultrasonic pretreatment of waste activated sludge was examined in order to improve the anaerobic digestibility. This study examined, mainly the effect of ultrasonic density and specific energy input on disintegration efficiency. At longer sonication duration and high ultrasound power input, better disintegration was observed thereby resulting higher release of intercellular and extracellular polymeric substances in the aqueous phase, measured as SCOD. The important conclusions are as follows;

- 1. The immersed depth of horn did not affect the SCOD release. Therefore, the sonication energy input was mainly governed by bottom surface of the horn.
- 2. Considering both the power input and the cell disruption efficacy, ultrasonic density of 1.5 W/ml was found appropriate value for effective SCOD release. The specific energy inputs of 2.3 kWS/gTS and 12 kWs/gTS were two critical values for effective mgSCOD/gTS release. However, the specific energy input of 12 kWs/gTS was chosen for more effective SCOD release. The SCOD release was about 225 mgSCOD/gTS at the specific energy input of 12 kWs/gTS and TS content of 3%.
- 3. The significant increase of temperature was observed during long sonication duration. Based on light microscopy examination, the structural changes in flocs, particularly disappearance of filaments was observed at high power input during longer sonication times of 60 and 120s.
- 4. The specific energy of 12kJ/gTS was found to be critical for efficient disintegration, which was further evidenced by biochemical methane potential (BMP) test conducted at mesophilic condition.
- 5. The ultrasonic pretreatment enhanced the subsequent anaerobic digestibility with significantly high TS and VS removal with respect to control. The TS removal efficiency was increased in the full stream digester by 22.8% at an SRT of 20 days and 32.2% at an SRT of 15 days in comparison to the control digester. The part stream digester however showed the same TS removal in comparison to the control digester at both SRTs.
- 6. The VS removal in the full stream digester was improved by 34.6% at SRT of 20 days, and 60.3% at SRT of 15 days with respect to the control digester. Interestingly, the VS removal was higher at an SRT of 15 days rather than at 20 days for both full and part stream digesters with respect to the control digester. The VS removal efficiency of full stream digester at an SRT of 15 days was nearly the same as that of the control digester at an SRT of 20 days. This apparently showed that with ultrasound pretreatment smaller digester volume is needed to achieve same degree of VS removal.

- 7. The biogas production rate in the full stream and part stream digesters improved by 102% and 91%, respectively with respect to control at an SRT of 20 days. At an SRT of 15 days, the improvement in biogas production rate, however declined for both full stream and part stream digesters in comparison to SRT of 20 days. The respective improvements with respect to control digester were 81.4% and 57.1%. Higher biogas production was observed in full stream digester than that in part stream digester at both SRTs. Though, the biogas yield was nearly the same in full stream and part stream digesters which was around 0.50 L/g VS removed at an SRT of 20 days, and 0.64 L/g VS removed in an SRT of 15 days,
- 8. The methane content in the biogas improved slightly in both full stream and part stream anaerobic digesters in comparison to the control digester. Thus, the ultrasound pretreatment enhanced the methane content in the biogas.
- 9. The dewaterabilty of the sludge was ranked in the ascending order as full stream digested sludge, part stream digested sludge, control digester sludge and raw sludge.
- 10. The lowest total VFA was observed in the digested sludge from a part stream digester rather than digested sludge from the full stream and control digesters at both SRTs of 20 and 15 days. When the SRT was decreased from 20 to 15 days, the concentration of acetic acid and the propionic acid in the digested sludge was increased, while the valeric acid level was almost the same in all three digesters at both SRTs. When the SRT was reduced from 20 to 15 days, the percentage distribution of acetic acid in the total VFA was decreased significantly in contrast to the percentage of propionic acid which increased significantly in all three digesters. The percentage distribution of valeric acid in the total VFA remained fairly constant in all the three digesters for both SRTs.
- 11. Based on the energy balance calculation, energy gained from biogas in the full stream digester was around two-fold compared to the energy gained from biogas in the control digester. In part stream digester, around 88% of sonication energy input was replenished in the form of biogas. This energy could be reused to generate electricity to run ultrasound system.
- 12. The hydrolysis coefficient of digestion in the, full stream and part stream digesters improved by 94% and 57% at an SRT of 20 days, and 99% and 57% at an SRT of 15 days respectively in comparison to the control digester. The higher capital and operating costs of the ultrasonic system could be offset by significant reduction in foot print of anaerobic digester and ultimate reduction in amount of digested sludge to be disposed off.
- 13. The part stream digester was more recommended than the full stream digester, since part stream digester was gave nearly same performance with minimum sonication energy input.

#### 5.2 Recommendations for Further Research

Based on the results of this research, following recommendations are proposed for further studies.

- 1. The effect of ultrasonication with specific energy of 12 kWs/gTS on the anaerobic digestibility of sonicated and nonsonicated waste activated sludge (WAS) needs to be studied with controlling temperature of sludge using jacket-cooled water bath since this study concluded that the specific energy of 12 kWs/gTS is effective for better disintegration of the sludge. This setup facilitates the disintegration of the sludge only by sonication effect rather than the thermal effect during the sonication.
- 2. The sludge disintegration efficiency of WAS by the ultrasonic sound is increased with decreasing TS content. However the anaerobic digestibility efficiency is increased with increasing TS content. Therefore the TS contents should be optimized by considering both the disintegration efficiency and the anaerobic digestibility efficiency.
- 3. The efficiency comparison of the mesophilic and thermophilic digesters of the control, full stream and part stream digester should be studied.
- 4. The level of disintegration has to be evaluated by the mechanism and kinetic models for the ultrasonic waste activated sludge disintegration.
- 5. The evaluation of the change in microbial communities in digesters, when sludge is sonicated, should also be examined.
- 6. There is a need to examine the effect of ultrasound on rheological properties of digester sludge.
- 7. A cost-benefit analysis of ultrasonic integrated systems needs to be conducted to justify the economics of the process in full-scale applications.

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Appendices

## Appendix A

### Photographs



A2: BMP test experimental setup



A2: Serum bottle in BMP test



- A3: Ultrasonic device
- A4: Large and small sonication chamber



A5: Small, medium and large horn



A6: Anaerobic digester experimental setup



A7: Mixer, Air seal and Teflon biogas bag in Anaerobic digester experimental setup

### Appendix B

### Sonication optimization results

Sonication time (s)	SCOD (mg/L)							
Someation time (s)	d = 1cm	d = 2cm	d = 3cm					
0	80	80	80					
30	400	320	240					
60	640	560	560					
120	1120	1120	800					
240	2600	2800	2800					
480	5600	6600	6200					

Appendix B1: SCOD with sonication duration at different immersion depths

Appendix B2: SCOD released with specific energy at different immersion depths

		d = 1cm	d = 2cm	d = 3cm
Sonication time (s)	SE(kWs/gTS)	SCOD	SCOD	SCOD
		(mg/gTS)	(mg/gTS)	(mg/gTS)
0	0	2.7	2.7	2.7
30	1	13.3	10.7	8.0
60	2	21.3	18.7	18.7
120	4	37.3	37.3	26.7
240	8	86.7	93.3	93.3
480	16	186.7	220.0	206.7

Appendix B3: ANOVA Table for significant of immerged depth of horn

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	109.848	2	54.924	.009	.991
Within Groups	90205.823	15	6013.722		
Total	90315.671	17			

Appendix B4: Temperature increment with sonication time at different power inputs

Sonication time (s)				
	P = 50 W	$\mathbf{P} = 100 \ \mathbf{W}$	P=150 W	$\mathbf{P} = 190 \ \mathbf{W}$
0	0	0	0	0
30	11	13	15	17
60	13	18	21	24
120	16	28	30	39
240	25	37	49	56
480	38	52	63	67

Sonication	n UD :	= 1.9 W/ml	UD=	1.5 W/ml	UD =	1.0 W/ml	UD = 0.5 W/ml		
time (s)	SE(kWs/gT	S) SCOD(mg/L	) SE(kWs/gTS	) SCOD(mg/L)	SE(kWs/gTS	) SCOD(mg/L)	SE(kWs/gTS)	SCOD(mg/L)	
0	0 80 0 80		80	0	80	0	80		
30	1.9	1360	1.5	800	1	480	0.5	120	
60	3.8	1760	3	1520	2	880	1	320	
120	7.6	3600	6	3360	4	2080	2	640	
240	15.2	7200	12	6800	8	3800	4	1440	
480	30.4	9000	24	8200	16	4600	8	2000	
Appendix I	36: SCOD releas	e with sonication	times for differe	ent specific energ	ies				
Sonicatin	UD = 1	.9 W/ml	U <b>D</b> = 1.	UD= 1.5 W/ml		.0 W/ml	UD = 0.5 W/ml		
time (s)	SE(kWs/gTS)	mgSCOD/gTS	SE(kWs/gTS)	mgSCOD/gTS	SE(kWs/gTS)	mgSCOD/gTS	SE(kWs/gTS)	mgSCOD/gTS	
0	0	2.7	0	2.7	0	2.7	0	2.7	
30	1.9	45.3	1.5	26.7	1	16.0	0.5	4.0	
60	3.8	58.7	3	50.7	2	29.3	1	10.7	
120	7.6	120.0	6	112.0	4	69.3	2	21.3	
240	15.2	240.0	12	226.7	8	126.7	4	48.0	

Appendix B5 SCOD release with sonication times at different power densities

## Appendix C Biochemical Methane Potential (BMP) results related to different sonication conditions

D		25/03/2007				27/03/2007				29/03/2007			
(W)	(s)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)
	0	1.8437	4.0	0.074	0.074	8.7645	6.2	0.543	0.617	11.3473	6.8	0.772	1.389
	30	2.380	4.5	0.107	0.107	10.196	8.9	0.907	1.015	14.681	9.1	1.336	2.351
50	60	3.013	11.1	0.334	0.334	10.229	11.3	1.156	1.490	15.344	10.8	1.657	3.147
	120	3.083	12.1	0.373	0.373	11.304	11.5	1.300	1.673	16.364	11.3	1.849	3.522
	240	6.035	16.6	1.002	1.002	12.188	12.5	1.523	2.525	17.911	12.1	2.167	4.692
	0	1.8437	4.0	0.074	0.074	8.7645	6.2	0.543	0.617	11.3473	6.8	0.772	1.389
	30	2.520	8.5	0.214	0.214	11.365	9.1	1.034	1.248	15.339	10.9	1.672	2.920
100	60	3.342	16.0	0.535	0.535	11.246	11.7	1.316	1.850	16.834	11.5	1.936	3.786
	120	5.545	17.1	0.948	0.948	12.039	11.9	1.433	2.381	17.694	12.3	2.176	4.557
	240	6.822	18.9	1.289	1.289	12.711	13.1	1.665	2.954	19.214	12.5	2.402	5.356
	0	1.8437	4.0	0.074	0.074	8.7645	6.2	0.543	0.617	11.3473	6.8	0.772	1.389
	30	2.767	12.7	0.351	0.351	11.660	9.7	1.131	1.482	16.002	11.3	1.808	3.291
150	60	4.994	20.7	1.034	1.034	12.611	12.5	1.576	2.610	17.472	11.9	2.079	4.689
	120	6.166	23.3	1.437	1.437	13.782	13.5	1.861	3.297	19.613	12.9	2.530	5.827
	240	7.054	24.1	1.700	1.700	14.176	14.5	2.055	3.755	20.294	13.1	2.659	6.414
-	0	1.844	4.0	0.074	0.074	8.765	6.2	0.543	0.617	11.347	6.8	0.772	1.389
-	30	2.911	15.5	0.451	0.451	12.410	10.7	1.328	1.779	16.900	11.8	1.994	3.773
190	60	5.380	24.3	1.307	1.307	13.915	13.1	1.823	3.130	18.144	12.8	2.322	5.453
	120	6.776	26.2	1.775	1.775	14.300	13.9	1.988	3.763	20.336	13.6	2.766	6.529
	240	7.603	29.1	2.212	2.212	14.650	14.9	2.183	4.395	21.071	14.5	3.055	7.451

Appendix C1: Biochemical methane potential calculation

D	<b>—</b> •	31/03/2007				02/04/2007					04/04/2007			
Power (W)	(s)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)	
	0	17.1345	6.4	1.097	2.485	19.3489	7.1	1.374	3.859	24.5465	4.6	1.129	4.988	
	30	21.166	8.7	1.841	4.192	24.133	9.0	2.172	6.364	30.277	5.7	1.726	8.090	
50	60	22.747	9.1	2.070	5.217	24.999	10.1	2.525	7.742	31.673	6.3	1.995	9.738	
	120	22.929	9.3	2.132	5.655	25.924	10.9	2.826	8.480	32.955	10.1	3.328	11.809	
	240	23.003	10.5	2.415	7.108	28.831	14.1	4.065	11.173	33.845	12.7	4.298	15.471	
	0	17.1345	6.4	1.097	2.485	19.3489	7.1	1.374	3.859	24.5465	4.6	1.129	4.988	
	30	21.837	9.5	2.075	4.995	24.735	9.5	2.350	7.345	31.046	6.7	2.080	9.425	
100	60	23.457	9.7	2.275	6.062	25.914	12.0	3.110	9.171	32.144	8.5	2.732	11.904	
	120	23.890	10.3	2.461	7.018	27.127	11.7	3.174	10.192	34.677	12.2	4.231	14.422	
-	240	25.111	11.5	2.888	8.244	30.844	16.7	5.151	13.395	34.523	14.8	5.109	18.504	
	0	17.1345	6.4	1.097	2.485	19.3489	7.1	1.374	3.859	24.5465	4.6	1.129	4.988	
	30	22.472	9.7	2.180	5.470	25.745	9.8	2.523	7.993	31.473	8.5	2.675	10.669	
150	60	23.921	10.3	2.464	7.153	26.141	14.1	3.686	10.839	32.887	9.5	3.124	13.963	
	120	24.973	10.9	2.722	8.549	28.974	13.2	3.825	12.374	37.701	16.2	6.108	18.482	
	240	26.262	12.1	3.178	9.592	31.183	17.1	5.332	14.924	35.357	19.1	6.753	21.677	
	0	17.135	6.4	1.097	2.485	19.349	7.1	1.374	3.859	24.547	4.6	1.129	4.988	
	30	22.819	10.1	2.305	6.078	26.151	11.2	2.929	9.007	31.833	9.3	2.960	11.967	
190	60	24.786	10.5	2.602	8.055	26.688	15.1	4.030	12.085	33.391	12.3	4.107	16.192	
	120	26.056	11.1	2.892	9.421	32.779	15.9	5.212	14.633	40.864	19.7	8.050	22.683	
	240	27.811	14.1	3.921	11.372	33.338	18.1	6.034	17.406	38.907	21.3	8.287	25.693	

		07/04/2007				10	10/04/2007			14	/04/2007		
Power (W)	Time (s)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)
	0	26.8497	7.5	2.014	7.002	28.3218	5.8	1.643	8.645	29.7493	5.2	1.547	10.192
	30	32.548	10.6	3.450	11.540	34.596	7.5	2.595	14.134	36.150	6.8	2.458	16.593
50	60	34.572	13.2	4.563	14.301	35.882	8.4	3.014	17.315	37.404	7.6	2.843	20.158
	120	35.761	15.3	5.471	17.280	37.241	9.7	3.612	20.892	39.638	8.2	3.250	24.143
	240	37.137	20.2	7.502	22.973	38.507	11.3	4.351	27.324	41.030	8.6	3.529	30.853
	0	26.8497	7.5	2.014	7.002	28.3218	5.8	1.643	8.645	29.7493	5.2	1.547	10.192
	30	33.165	12.7	4.212	13.637	35.678	8.7	3.104	16.741	36.663	7.1	2.603	19.344
100	60	35.445	17.7	6.274	18.177	37.782	9.1	3.438	21.616	38.661	8.1	3.132	24.747
	120	38.869	18.3	7.113	21.535	39.419	10.3	4.060	25.595	42.302	8.6	3.638	29.233
	240	40.562	23.5	9.532	28.036	40.770	13.0	5.300	33.336	42.269	9.1	3.846	37.183
	0	26.8497	7.5	2.014	7.002	28.3218	5.8	1.643	8.645	29.7493	5.2	1.547	10.192
	30	34.241	13.6	4.657	15.325	36.301	9.6	3.485	18.810	37.906	8.6	3.260	22.070
150	60	38.925	23.5	9.147	23.111	40.290	10.5	4.230	27.341	42.123	8.8	3.707	31.048
	120	40.915	25.2	10.311	28.792	43.047	10.5	4.520	33.312	44.390	9.4	4.173	37.485
	240	43.704	27.3	11.931	33.608	45.360	14.5	6.577	40.185	47.514	9.5	4.514	44.699
	0	26.850	7.5	2.014	7.002	28.322	5.8	1.643	8.645	29.749	5.2	1.547	10.192
	30	35.548	15.2	5.403	17.371	37.989	10.8	4.103	21.474	38.584	8.7	3.357	24.830
190	60	41.895	26.9	11.270	27.462	42.800	11.3	4.836	32.298	44.017	9.1	4.006	36.304
	120	44.300	27.7	12.271	34.954	46.533	10.8	5.026	39.980	48.806	9.7	4.734	44.714
	240	45.863	28.8	13.208	38.902	49.387	16.9	8.346	47.248	49.981	10.3	5.148	52.396

D	<b>T</b> .	28/04/2007				22/04/2007					26/04/2007			
Power (W)	Time (s)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)	Methane (%)	Biogas (ml)	Methane (ml)	Accumulated (ml)	
	0	31.0061	5.0	1.550	11.742	31.5973	4.3	1.359	13.101	31.8763	2.0	0.638	13.738	
	30	37.675	6.4	2.411	19.004	38.754	4.8	1.860	20.864	39.030	2.1	0.820	21.684	
50	60	39.130	5.9	2.309	22.467	39.874	4.2	1.675	24.141	40.252	2.9	1.167	25.309	
	120	41.948	5.7	2.391	26.534	43.478	3.4	1.478	28.012	44.373	1.5	0.666	28.678	
	240	42.553	6.6	2.809	33.661	42.945	3.3	1.417	35.078	43.411	1.5	0.651	35.730	
	0	31.0061	5.0	1.550	11.742	31.5973	4.3	1.359	13.101	31.8763	2.0	0.638	13.738	
	30	37.866	6.7	2.537	21.881	38.997	3.8	1.482	23.363	39.733	1.6	0.636	23.999	
100	60	40.753	5.4	2.201	26.948	42.523	3.5	1.488	28.436	43.374	2.7	1.171	29.607	
	120	43.788	6.2	2.715	31.948	44.880	3.7	1.661	33.609	45.086	2.3	1.037	34.646	
	240	43.787	6.8	2.978	40.160	44.997	3.3	1.485	41.645	45.869	1.8	0.826	42.471	
	0	31.0061	5.0	1.550	11.742	31.5973	4.3	1.359	13.101	31.8763	2.0	0.638	13.738	
	30	39.028	7.5	2.927	24.997	40.137	4.6	1.846	26.844	40.840	3.4	1.389	28.232	
150	60	44.048	6.1	2.687	33.735	44.658	2.9	1.295	35.030	45.077	1.5	0.676	35.706	
	120	45.876	6.3	2.890	40.375	46.736	3.2	1.496	41.871	47.130	2.5	1.178	43.049	
	240	48.748	7.1	3.461	48.160	49.138	3.7	1.818	49.979	49.431	2.5	1.236	51.214	
	0	31.006	5.0	1.550	11.742	31.597	4.3	1.359	13.101	31.876	2.0	0.638	13.738	
	30	39.824	7.2	2.867	27.698	40.936	3.8	1.556	29.253	41.558	2.7	1.122	30.375	
190	60	45.794	6.0	2.748	39.052	46.633	3.2	1.492	40.544	47.574	1.9	0.904	41.448	
	120	49.254	7.6	3.743	48.457	49.755	4.3	2.139	50.597	50.491	2.0	1.010	51.606	
	240	51.143	6.6	3.375	55.772	51.637	3.5	1.807	57.579	52.035	2.1	1.093	58.672	

Power (W)	Time (s)	Methane (%)	Head space Volume (ml)	Methane In head space	Total methane (ml)	Gross methane (ml) (Ambient condition)	Gross methane (ml) (STP)
	0	31.8763	90	28.689	42.427	41.639	36.980
	30	39.030	90	35.127	56.810	56.022	49.755
50	60	40.252	90	36.227	61.535	60.747	53.951
	120	44.373	90	39.936	68.614	67.826	60.238
	240	43.411	90	39.070	74.799	74.011	65.731
	0	31.8763	90	28.689	42.427	41.639	36.980
	30	39.733	90	35.760	59.758	58.970	52.373
100	60	43.374	90	39.037	68.644	67.856	60.264
	120	45.086	90	40.577	75.223	74.435	66.107
	240	45.869	90	41.282	83.753	82.965	73.683
	0	31.8763	90	28.689	42.427	41.639	36.980
	30	40.840	90	36.756	64.988	64.200	57.017
150	60	45.077	90	40.569	76.275	75.487	67.042
	120	47.130	90	42.417	85.466	84.678	75.205
	240	49.431	90	44.488	95.702	94.914	84.296
	0	31.876	90	28.689	42.427	41.639	36.980
	30	41.558	90	37.402	67.778	66.990	59.495
190	60	47.574	90	42.816	84.264	83.476	74.137
	120	50.491	90	45.442	97.049	96.261	85.491
	240	52.035	90	46.831	105.503	104.715	93.000

The	cumulative	methane	producti	ion at th	e mesoph	ilic condi	ition, ml	related t	o digestio	on period	, days (A	fter blanl	k adjustn	nent)
Power (W)	Sonication time (s)	0	2	4	6	8	10	12	15	18	22	26	30	34
	0	0	1.598	8.195	11.196	17.440	20.789	26.574	31.145	33.501	36.266	38.909	40.792	41.675
	30	0	2.308	10.074	15.352	22.969	27.794	35.027	41.005	44.831	48.622	52.366	55.190	56.253
50	60	0	2.917	10.391	16.556	25.228	29.763	37.743	45.399	48.981	53.127	56.950	59.286	60.789
	120	0	3.608	12.132	18.440	26.419	31.924	41.558	50.038	54.371	59.713	64.143	66.991	68.457
	240	0	5.337	12.222	19.445	26.382	35.676	44.465	55.413	60.386	66.119	70.259	72.020	73.086
	0	0	1.706	8.302	11.303	17.547	20.897	26.682	31.252	33.608	36.373	39.016	40.899	41.782
	30	0	2.634	11.454	16.607	24.469	29.410	37.148	43.751	48.504	51.928	55.509	58.000	59.294
100	60	0	3.788	12.042	18.912	27.087	32.391	40.708	50.436	55.366	59.222	63.267	66.341	68.272
	120	0	6.112	13.213	20.383	28.360	34.430	45.434	56.803	60.747	66.913	70.927	73.562	74.779
	240	0	6.045	12.835	20.994	29.128	39.422	47.820	63.271	68.146	73.276	77.581	80.147	81.752
	0	0	1.843	8.439	11.440	17.684	21.034	26.819	31.389	33.745	36.511	39.153	41.036	41.919
	30	0	3.356	12.314	17.936	25.877	31.329	39.138	46.770	51.496	56.135	60.033	62.869	64.885
150	60	0	5.763	14.019	20.378	28.585	34.252	43.425	58.491	63.338	68.628	73.009	74.845	75.893
	120	0	7.082	15.620	23.303	30.788	38.197	52.137	65.824	71.651	76.966	81.154	83.416	84.944
	240	0	6.254	14.543	22.613	31.101	40.846	51.333	71.260	78.716	85.103	89.636	91.796	93.291
	0	0	1.943	8.539	11.540	17.784	21.134	26.919	31.489	33.845	36.610	39.253	41.136	42.019
	30	0	3.759	13.461	19.400	26.971	32.882	40.934	50.165	55.853	59.679	63.624	66.171	67.849
190	60	0	6.450	15.778	21.811	30.330	36.055	46.173	65.580	70.619	75.655	79.962	82.201	83.947
	120	0	8.143	16.726	24.829	32.809	44.054	59.359	75.206	81.629	88.343	92.451	95.033	96.701
	240	0	6.677	15.025	23.765	33.691	44.683	57.960	77.912	88.819	94.436	98.818	101.062	102.508

Appendix C2: Accumulative methane production with digestion period in BMP batch digester

Sonication	UD = 1.9 W/ml		<b>UD</b> = 1.5 W/ml		UD = 1.0 W/ml		UD = 0.5 W/ml	
time (s)	SE(kWs/gTS)	SCOD(mg/L)	SE(kWs/gTS)	SCOD(mg/L)	SE(kWs/gTS)	SCOD(mg/L)	SE(kWs/gTS)	SCOD(mg/L)
0	0	36.980	0	36.980	0	36.980	0	36.980
30	1.9	59.495	1.5	57.017	1	52.373	0.5	49.755
60	3.8	74.137	3	67.042	2	60.264	1	53.951
120	7.6	85.491	6	75.205	4	66.107	2	60.238
240	15.2	93.000	12	84.296	8	73.683	4	65.731

Appendix C3: Biochemical methane potential calculation

## Appendix D

## Digestion performance results with digestion period

	Digestion		TS						
Dates	operation (days)	Feeding (%)	Control (%)	Full stream (%)	Part stream (%)				
1/12/2006	1	3	2.95	3.02	3.01				
6/12/2006	6	3	2.86	2.95	2.93				
11/12/2006	11	3	2.85	2.9	2.9				
18/12/2006	18	3	2.79	2.84	2.82				
25/12/2006	25	3	2.76	2.79	2.76				
2/1/2007	33	2.92	2.76	2.76	2.7				
11/1/2007	42	2.92	2.68	2.72	2.72				
18/01/2007	49	2.92	2.68	2.63	2.67				
2/2/2007	64	2.92	2.61	2.59	2.65				
5/2/2007	67	2.92	2.61	2.55	2.65				
8/2/2007	70	3.02	2.61	2.56	2.63				
11/2/2007	73	3.02	2.62	2.53	2.61				
14/02/2007	76	3.02	2.62	2.53	2.61				
17/02/2007	79	3.02	2.6	2.52	2.58				
20/02/2007	82	3.02	2.59	2.52	2.55				
23/02/2007	85	3.02	2.6	2.5	2.56				
26/02/2007	88	3.02	2.59	2.5	2.56				
1/3/2007	91	3.02	2.61	2.51	2.56				
8/3./2007	98	3.02	2.60	2.51	2.57				
15/3/2007	105	3.02	2.60	2.50	2.57				
19/03/2007	109	2.96	2.62	2.51	2.6				
25/03/2007	115	2.96	2.64	2.51	2.6				
29/03/2007	119	2.96	2.64	2.53	2.61				
1/3/2007	122	2.96	2.65	2.52	2.61				
4/4/2007	125	2.96	2.64	2.53	2.61				
7/4/2007	128	2.96	2.64	2.53	2.62				
10/4/2007	131	2.96	2.65	2.54	2.62				
13/4/2007	134	2.96	2.65	2.54	2.61				
16/04/2007	137	2.96	2.64	2.55	2.61				
19/04/2007	140	2.96	2.64	2.55	2.62				

Appendix D1: TS of feed sludge and digested sludge in all three reactors

	Digestion			VS	
Dates	operation (days)	Feeding (%)	Control	Full stream	Part stream
1/12/2006	1	2.1	1.67	1.7	1.71
6/12/2006	6	2.1	1.67	1.69	1.7
11/12/2006	11	2.1	1.65	1.64	1.69
18/12/2006	18	2.1	1.67	1.67	1.67
25/12/2006	25	2.1	1.67	1.66	1.68
2/1/2007	33	2.08	1.67	1.66	1.68
11/1/2007	42	2.08	1.66	1.66	1.67
18/01/2007	49	2.08	1.66	1.58	1.59
2/2/2007	64	2.08	1.66	1.55	1.6
5/2/2007	67	2.08	1.65	1.56	1.61
8/2/2007	70	2.1	1.66	1.56	1.6
11/2/2007	73	2.1	1.68	1.57	1.6
14/02/2007	76	2.1	1.68	1.56	1.6
17/02/2007	79	2.1	1.68	1.56	1.58
20/02/2007	82	2.1	1.68	1.55	1.58
23/02/2007	85	2.1	1.69	1.55	1.59
26/02/2007	88	2.1	1.69	1.56	1.6
1/3/2007	91	2.1	1.7	1.56	1.59
8/3./2007	98	2.1	1.70	1.55	1.60
15/3/2007	105	2.1	1.70	1.56	1.60
19/03/2007	109	2.1	1.75	1.60	1.67
25/03/2007	115	2.1	1.81	1.65	1.71
29/03/2007	119	2.1	1.83	1.68	1.74
1/3/2007	122	2.1	1.84	1.68	1.74
4/4/2007	125	2.1	1.85	1.69	1.75
7/4/2007	128	2.1	1.84	1.69	1.74
10/4/2007	131	2.1	1.84	1.69	1.75
13/4/2007	134	2.1	1.85	1.69	1.75
16/04/2007	137	2.1	1.85	1.70	1.76
19/04/2007	140	2.1	1.85	1.69	1.75

Appendix D2: VS of feed sludge and digested sludge in all three reactors

Digostion operation		TS removal effici	ency
(days)	Control (%)	Full stream (%)	Part stream (%)
1	2.4	-1.0	-0.5
6	6.7	2.4	3.3
11	7.1	4.8	4.8
18	10.0	7.6	8.6
25	11.4	10.0	11.4
33	7.6	7.6	10.5
42	11.4	9.5	9.5
49	11.4	13.8	11.9
64	14.8	15.7	12.9
67	14.8	17.6	12.9
70	19.5	21.9	18.6
73	19.0	23.3	19.5
76	19.0	23.3	19.5
79	20.0	23.8	21.0
82	20.5	23.8	22.4
85	20.0	24.8	21.9
88	20.5	24.8	21.9
91	19.5	24.3	21.9
98	20.0	24.3	21.4
105	20.0	24.8	21.4
109	16.2	21.4	17.1
115	15.2	21.4	17.1
119	15.2	20.5	16.7
122	14.8	21.0	16.7
125	15.2	20.5	16.7
128	15.2	20.5	16.2
131	14.8	20.0	16.2
134	14.8	20.0	16.7
137	15.2	19.5	16.7
140	15.2	19.5	16.2

Appendix D3: TS removal efficiency in all three digesters

		VS removal effic	iency
Digestion operation (days)	Control (%)	Full stream (%)	Part stream (%)
1	20.5	19.0	18.6
6	20.5	19.5	19.0
11	21.4	21.9	19.5
18	20.5	20.5	20.5
25	20.5	21.0	20.0
33	19.5	20.0	19.0
42	20.0	20.0	19.5
49	20.0	23.8	23.3
64	20.0	25.2	22.9
67	20.5	24.8	22.4
70	21.0	25.7	23.8
73	20.0	25.2	23.8
76	20.0	25.7	23.8
79	20.0	25.7	24.8
82	20.0	26.2	24.8
85	19.5	26.2	24.3
88	19.5	25.7	23.8
91	19.0	25.7	24.3
98	19.0	26.2	23.8
105	19.0	25.7	23.8
109	16.7	23.8	20.5
115	13.8	21.4	18.6
119	12.9	20.0	17.1
122	12.4	20.0	17.1
125	11.9	19.5	16.7
128	12.4	19.5	17.1
131	12.4	19.5	16.7
134	11.9	19.5	16.7
137	11.9	19.0	16.2
140	11.9	19.5	16.7

Appendix D4: VS removal efficiency in all three digesters

		AD0				AD1			AD2			
Date	Days	Biogas Production ml/week	Composition (%)	Methane Production ml/week	Biogas Production ml/week	Composition (%)	Methane Production ml/week	Biogas Production ml/week	Composition (%)	Methane Production ml/week		
07/12/2007	7	883	31.4	277	940	46.4	436	940	42.9	403		
14/12/2007	14	1010	28.8	291	1170	53.6	627	1150	51	587		
21/12/2007	21	1020	39.8	406	1170	56.7	663	1150	53	610		
28/12/2007	28	1100	47.2	519	1200	58.5	702	1220	56.2	686		
04/1/2007	35	1180	58	684	1180	59.5	702	1200	58.8	706		
11/1/2007	42	1175	58.5	687	1185	59.2	702	1195	58.8	703		
18/01/2007	49	1205	58.3	703	1887	59.7	1127	1582	59.4	940		
25/01/2007	56	1250	58.8	735	2358	61.0	1438	2077	59.6	1238		
01/2/2007	63	1260	58.6	738	2480	60.5	1500	2110	59.6	1258		
08/2/2007	70	1320	58.4	771	2815	60.6	1706	2552	59.9	1529		
15/02/2007	77	1350	58.6	791	2805	60.9	1708	2684	59.7	1602		
22/02/2007	84	1612	58.4	941	3025	61.0	1845	2816	59.8	1684		
29/02/2007	91	1395	58.6	817	2790	60.9	1699	2645	59.8	1582		
08/3/2007	98	1415	58.6	829	2805	61.0	1711	2650	59.9	1587		
15/3/2007	105	1410	58.6	826	2835	61.1	1732	2640	59.8	1579		
22/3/2007	112	1750	59.0	1033	3325	60.9	2024	2810	60.5	1701		
29/3/2007	119	1890	58.9	1113	3520	60.8	2139	2885	60.3	1740		
05/4/2007	126	2015	58.2	1173	3635	60.2	2188	3139	59.4	1865		
12/4/2007	133	2010	57.9	1164	3640	60.6	2206	3180	60.4	1921		
19/4/2007	140	2000	58.3	1166	3655	60.6	2215	3150	59.7	1881		

Appendix D5: Biogas and methane production rate and methane composition

Data		Capillary	Suction Time	
Date	Raw sludge	Control	Full stream	Part stream
1/1/2007	42.5	52.9	47.3	46.2
4/1/2007	42.5	57.5	62.8	52.8
7/1/2007	46.2	53.3	50.2	51.7
11/1/2007	50.5	52.9	57.3	56.2
16/01/2007	48.5	57.5	62.8	52.8
24/01/2007	46.2	53.3	50.2	51.7
19/2/2007	46	52.9	60.8	57
23/2/2007	49	66.6	75.7	70
27/02/2007	45	73	107.1	87.8
3/3/2007	51	59.8	95.8	82.5
7/3/2007	48.5	77.1	101.2	98.1
2/4/2007	48.7	75.2	143.8	110.6
5/4/2007	55.5	79.9	122.3	128.8
8/4/2007	51.2	75.3	150.8	115.9
11/4/2007	48.4	74.4	158.4	109.8
14/04/2007	49.7	64.5	124.6	101.8
17/04/2007	47.6	70.3	138.1	107.6

Appendix D6: Capillary suction time fed sludge and digested sludge in all three reactors

Appendix D7: TVFA in digested sludge in all three reactors

Date	Days	Control	Full stream	Part stream
07/12/2006	7	13.24	15.41	14.45
14/12/2006	14	13.42	15.82	15.96
21/12/2006	21	16.36	15.45	14.79
28/12/2006	28	14.54	13.53	15.40
04/1/2007	35	14.95	13.14	15.23
11/1/2007	42	16.29	15.77	15.02
18/01/2007	49	16.24	12.41	9.56
25/01/2007	56	14.40	12.00	10.50
01/2/2007	63	8.25	7.78	7.45
08/2/2007	70	9.86	8.89	8.59
15/02/2007	77	10.47	9.77	9.25
22/02/2007	84	9.61	8.63	8.04
29/02/2007	91	10.56	9.52	8.67
08/3/2007	98	11.72	11.20	7.90
15/3/2007	105	11.54	10.97	8.22
22/3/2007	112	23.86	20.65	13.28
29/3/2007	119	26.20	19.33	18.21
05/4/2007	126	19.03	15.70	13.11
12/4/2007	133	8.66	11.80	7.57
19/4/2007	140	8.32	10.44	6.13
26/4/2007	147	8.20	9.88	5.67



Appendix D8: TVFA of digested effluent with digestion period

VEA	Control						
٧ſA	mg/L	mg/L	mg/L	AVG	STDEV		
Acetic acid	5.38	6.85	5.78	6.00	0.76		
Propionic acid	2.35	0.22	0.23	0.94	1.22		
Iso-butyric acid	0.45	0.27	0.24	0.32	0.11		
n-butyric acid	3.54	1.94	1.70	2.39	1.00		
Valeric acid	0.63	0.58	0.30	0.50	0.17		
TVFA	12.35	9.86	8.25	10.15	2.06		
	Full stream						
Acetic acid	5.05	6.09	5.59	5.58	0.52		
Propionic acid	1.66	0.32	0.17	0.72	0.82		
Iso-butyric acid	0.00	0.25	0.21	0.15	0.13		
n-butyric acid	3.55	1.72	1.44	2.24	1.14		
Valeric acid	0.91	0.50	0.36	0.59	0.28		
TVFA	11.16	8.89	7.78	9.28	1.73		
	Part stream						
Acetic acid	3.40	5.54	5.29	4.74	1.17		
Propionic acid	1.47	0.39	0.12	0.66	0.71		
Iso-butyric acid	0.00	0.31	0.21	0.17	0.16		
n-butyric acid	2.59	1.66	1.35	1.87	0.64		
Valeric acid	0.45	0.68	0.48	0.54	0.12		
TVFA	7.90	8.59	7.45	7.98	0.57		

Appendix D9: Individual VFA in digested sludge in all three reactors for SRT of 20 days
VEA	Control					
VFA	mg/L	mg/L	mg/L	AVG	STDEV	
Acetic acid	3.92	3.97	4.08	3.99	0.08	
Propionic acid	1.84	1.91	1.52	1.75	0.21	
Iso-butyric acid	0.33	0.33	0.33	0.33	0.00	
n-butyric acid	1.56	1.59	2.30	1.82	0.42	
Valeric acid	0.54	0.53	0.43	0.50	0.06	
TVFA	8.20	8.32	8.66	8.40	0.24	
			Full strea	m		
Acetic acid	4.65	4.87	5.85	5.12	0.64	
Propionic acid	2.31	2.40	2.40	2.37	0.05	
Iso-butyric acid	0.78	0.84	0.62	0.75	0.12	
n-butyric acid	1.73	1.78	2.07	1.86	0.19	
Valeric acid	0.51	0.56	0.85	0.64	0.18	
TVFA	9.98	10.44	11.80	10.74	0.95	
	Part stream					
Acetic acid	2.09	2.67	4.85	3.20	1.46	
Propionic acid	1.62	1.50	1.62	1.58	0.07	
Iso-butyric acid	0.44	0.44	0.00	0.29	0.25	
n-butyric acid	0.80	0.80	1.10	0.90	0.17	
Valeric acid	0.72	0.72	0.00	0.48	0.42	
TVFA	5.67	6.13	7.57	6.46	0.99	

Appendix D10: Individual VFA in digested sludge in all three reactors for SRT of 15 days

Appendix D11: VFA standard curve





	рН			Alkalinity (mg/L)		
Digestion period (days)	Control	Full stream	Part stream	Control	Full stream	Part stream
1	7.16	7.12	7.13	2100	2100	2100
19	7.16	7.11	7.1	1700	1850	1750
21	7.06	7.08	7.05	1750	1850	1800
26	7.1	7.15	7.05	1850	1850	1700
29	7.06	7.02	7	1850	1900	1800
40	7	7.05	7	1900	1900	1975
42	6.91	6.91	6.91	1925	1950	1975
55	6.84	6.91	7.13	1800	1850	2000
61	6.94	7.02	7.1	1950	2000	2150
63	6.98	7.05	7.11	1950	1950	2050
70	7.05	7.14	7.11	1950	2050	2150
81	7.08	7.1	7.14	1950	1975	2025
105	7.13	7.06	7.09	1975	2000	2050
108	7.05	7.02	7.01	1900	1950	1975
117	7.11	7	7.08	1950	1950	2000
121	7.12	7.05	7.15	1950	2000	2150
124	7.05	7.01	7.08	2000	1850	2100
131	7.12	7.02	7.14	2025	1950	2150
140	7.1	7.06	7.15	2050	1900	2150

Appendix D12: pH and Alkalinity of the digested sludge in all three digesters

Appendix D13: One-way ANOVA for Biogas production between the digesters

	Sum of Squares	Degree of Freedom	Mean Square	F	Sig.
Between Groups	24093.333	2	12046.667	4.651	.032
Within Groups	31080.000	12	2590.000		
Total	55173.333	14			

#### **Appendix E**

#### **Specimen Calculations**

Appendix E1: Energy balance calculations

Assumptions

- 1. Temperature of fresh sludge  $(T_1)=20^{\circ}C$
- 2. Average temperature of ambient air temp $(T_2) = 27^{\circ}C$
- 3. Temperature in the digester( $T_3$ ) = 37°C
- 4. Specific heat capacity of  $sludge(C_p) = 4.2 kJ/kg/^{\circ}C$
- 5. Overall heat transfer coefficient (U) =  $2.5 \text{ W/m}^2/^{\circ}\text{C}$  (Barber, 2005)
- 6. Calorific value of methane = 35.8 kJ/g (Barber, 2005)
- 7. Specific gravity of sludge = 1.02
- 8. Sludge flow rate (Q) = 150 mL/day and 200 mL/day

#### Heat requirement for the sludge (Q<sub>1</sub>)

SRT = 20 days,	SRT = 15 days,
$=\sum mC_{P}(T_{3}-T_{1})$	$=\sum mC_{P}(T_{3}-T_{1})$
= Q*1020*4200*(27-20) J/day	$= Q^{1020} + 4200 + (27 - 20) J/day$
$= 150*10^{-6}*1020*4200*(30-20)/1000 \text{ kJ/day}$	$= 200*10^{-6}*1020*4200*(30-20)/1000 \text{ kJ/day}$
= 4.4982  kJ/day	= 5.9976 kJ/day

Rate of heat addition required to compensate for loss from the digester (Q<sub>2</sub>) =  $UA(T_3 - T_2)$ 

Where A= Cross-sectional area through which the losing is occurring, 0.153 m<sup>2</sup> = 2.5\*0.153\*(37-27) W = 231.336 kJ/day

#### Energy generation from methane (Q<sub>3</sub>)

Biogas gas mole calculation from biogas volume,

$$n = \frac{PV}{RT}$$
$$n = \frac{1.01325 * 10^5 * V * 10^{-6}}{8.314 * 300}$$

Where, V is measured in mL and ambient temperature is 300K.  $n = 4.0624*10^{-5}*V \text{ mol}$ 

 $n = 4.0624 * 10^{\circ} * V mo$ 

Methane production  $= n \mod/day$ Methane production = x g/day

 $Q_3 = x*35.8*kJ/day$ 

#### Ultrasonic energy input (Q<sub>5</sub>)

$$Q_5 = Q' * UD * t$$

Where,

Q' = Sonicated sludge flow rate (150ml/day for full stream and 75ml/day for part stream for SRT = 20 days) (200ml/day for full stream and 100ml/day for part stream for SRT = 20 days) UD = Ultrasonic density (power/ volume) t = Sonication duration

**Excess energy gain**  $(Q_4) = Q_3 - (Q_1 + Q_2 - Q_5)$ 

Full stream,  $Q_5 = 150 \text{ml/day} * \frac{190 \text{ W}}{100 \text{ ml}} * 45 \text{ s}$   $Q_5 = 12, 825 \text{ J/day}$   $Q_5 = 12. 825 \text{ kJ/day}$ Part stream,  $Q_5 = 75 \text{ml/day} * \frac{190 \text{ W}}{100 \text{ ml}} * 45 \text{ s}$   $Q_5 = 6, 412.5 \text{ J/day}$  $Q_5 = 6. 4125 \text{ kJ/day}$ 

Full stream,  

$$Q_5 = 200 \text{ml/day} * \frac{190 \text{ W}}{100 \text{ ml}} * 45 \text{ s}$$
  
 $Q_5 = 17, 100 \text{ J/day}$   
 $Q_5 = 17. 1 \text{ kJ/day}$   
Part stream,  
 $Q_5 = 100 \text{ml/day} * \frac{190 \text{ W}}{100 \text{ ml}} * 45 \text{ s}$   
 $Q_5 = 8,550 \text{ J/day}$   
 $Q_5 = 8.55 \text{ kJ/day}$ 

**Excess energy gain (Q<sub>4</sub>)** =  $Q3 - (Q_1 + Q_2 - Q_5)$ 

Biogas gas mole calculation from biogas volume,

$$n = \frac{PV}{RT}$$
$$n = \frac{1.01325 * 10^5 * V * 10^{-6}}{8.314 * 300}$$

Where, V is measured in mL and ambient temperature is 300K.

$$n = 4.0624 * 10^{-5} * V mol$$

#### Appendix F: Occupational health and safety in ultrasonic pretreatment

(Source: http://www2.worksafebc.com/Publications/OHSRegulation)

#### F1: Noise physical hazard

The ultrasound is high frequency sound wave ( $\geq 20$  kHz). Ultrasound with frequency 20 kHz is used for sludge disintegration as a pretreatment. The cavitation bubbles created by ultrasound energy in sludge medium are collapsed suddenly when they reach the critical size. It causes high level of noise. The noise exposure limits is that an employer must ensure that a worker is not exposed to noise levels above either of the following exposure limits:

- (a) 85 dBA Lex daily noise exposure level;
- (b) 140 dBC peak sound level.

#### F1.1: Noise measurement required

- 1. If a worker is or may be exposed to potentially harmful levels of noise, or if information indicates that a worker may be exposed to a level exceeding 85 dBA Lex, the employer must measure the noise exposure.
- 2. The employer must inform affected workers of the results of any noise exposure measurement and the significance of the measurement to risk of hearing loss.

### F.1.2: Exemption

An employer is not required to measure the noise exposure of a worker if

- a) based on other information, the employer identifies the worker as being exposed to noise in excess of an exposure limit, and
- b) the employer establishes an effective noise control and hearing conservation program for that worker.

#### F1.3: Noise control and hearing conservation program

If noise in the workplace exceeds either of the noise exposure limits, the employer must develop and implement an effective noise control and hearing conservation program with the following elements:

- (a) noise measurement;
- (b) education and training;
- (c) engineered noise control;
- (d) hearing protection;
- (e) posting of noise hazard areas;
- (f) hearing tests;
- (g) annual program review.

#### (i) Engineered noise control

If a worker is exposed to noise above a noise exposure limit, the employer must

- a) investigate options for engineered noise control, and
- b) when practicable, implement one or more of those options to reduce noise exposure of workers to or below the exposure limits.

### (ii) Hearing protection and warning signs

- 1. If it is not practicable to reduce noise levels to or below noise exposure limits, the employer must
  - (a) reduce noise exposure to the lowest level practicable,
  - (b) post warning signs in the noise hazard areas,
  - (c) ensure that hearing protection is worn effectively in noise hazard areas.
- 2. Workers in a posted noise hazard area must wear hearing protection.

### (iii) Hearing tests

- 1. The employer must give workers who are exposed to noise that exceeds noise exposure limits
  - (a) an initial hearing test as soon as practicable after employment starts, but not later than 6 months after the start of employment, and
  - (b) a test at least once every 12 months after the initial test.
- 2. Hearing tests must be administered by a hearing tester authorized by the board of health department.
- 3. The employer must ensure that the authorized hearing tester sends the test results to the Board.

### F2: Vibration physical hazard

The ultrasound wave is a longitudinal wave. It travels in the media by vibration. Therefore, Vibration is one of physical hazard to human. There are two possible way to be transmitted the vibration.

- I. "hand-arm vibration" means vibration that is transmitted from vibrating surfaces of objects, such as hand tools, through the hands and arms;
- II. "whole-body vibration" means vibration that is transmitted to a worker's body from vibrating surfaces on which a worker stands or sits.

### **F2.1:** Vibration exposure limits

An employer must ensure, to the extent practicable, that workers are not exposed to vibration in excess of the limits specified in

- a) for hand-arm vibration, the American Conference of Governmental Industrial Hygienists publication entitled Threshold Limit Values and Biological Exposure Indices, dated 2003, as amended from time to time;
- b) for whole-body vibration, ANSI Standard S3.18-2002/ISO 2631-1-1997, Mechanical Vibration and Shock – Evaluation of Human Exposure to Whole Body Vibration – Part 1: General Requirements, as amended from time to time;

### **F2.2:** Vibration exposure control obligations

The employer must, if a worker is or may be exposed to vibration in excess of the vibration exposure limits, develop and implement an exposure control plan that meets the requirements.

#### **F2.3: Information about vibration hazards**

The employer must, if a worker is exposed to levels of vibration above the vibration exposure limits, inform the worker of the nature of the hazard and possible adverse effects.

#### F2.4: Labels

If the manufacturer of equipment that produces levels of vibration in excess of the vibration exposure limits does not label the equipment to identify the hazard, the employer is responsible for doing so.

#### F2.5: Exposure to cold and hand-arm vibration

When a worker is exposed to hand-arm vibration, the employer, to the extent practicable, must ensure that the worker's hands or arms are not exposed to cold, either

- a) from the environment in which the worker is working or as a result of using equipment, or
- b) from coming into contact with cold objects.

#### Appendix G: Pathogen count in the digested sludge

Table G1 shows the pathogen count in the digested sludge at an SRT of 20 days. These results reveal that effect of the ultrasonication pretreatment on pathogen reduction in the digested sludge. Which was not acceptable, since thought, ultrasonication was destroyed the pathogens. The pathogen level in the control digested sludge was not in typical range which was found in the other research and full scale plant. Total coliform should be higher than E. *coli* in any sample. However results show that opposite way. The analysis used in experiment should be wrong. Therefore, It may be reason that sample analyzed was not represent effect of sonication pretreatment on digested sludge since initial pathogens in digester were multiplied inside the sonication digester even after feeding of sonicated sludge was done. Therefore pathogen analysis should be done after long period of digester operation. It will be make sure that initial sludge inside the sonicated digester will be replaced by sonicated sludge.

Digested Sludge	Total coliforms (MPN/g)	<i>E.coli</i> (MPN/g)	Salmonella spp.* (CFU/g)
Control	93	15	< 100
Full stream	230	230	< 100
Part stream	430	20	< 100

#### Appendix G1 Pathogen count in the digested sludge at an SRT of 20 days

\* Analysis by direct count



# Anaerobic Digestion of Waste Activated Sludge with Ultrasonic Pretreatment

by N. Navaneethan

**Examination Committee:** 

Prof. C. Visvanathan (Chairperson) Prof. Chongrak Polprasert Prof. Ajit P. Annachhatre Dr. Samir Kumar Khanal

AIT, 08 May 2007

### **Outlines**





## Introduction



- Activated sludge process is the most widely used biological process for domestic wastewater treatment
- The treatment, handling and disposal of excess sludge amounts cause up to 60% of the total operating cost of wastewater treatment plant



## **Ultrasonic pretreatment**



**Aim of pretreatment**: Break the cell wall and membrane of biological cell to release the intracellular matter into the aqueous phase for subsequent biological degradation.



### **Ultrasonication**





## **Objectives**



- Optimize ultrasonic pretreatment to maximize waste activated sludge (WAS) disintegration
- Examine the anaerobic digestibility of full-stream (100% sonicated) and part stream (50% sonicated and 50% non-sonicated) WAS at different solids retention times
- Evaluate the effluent quality of ultrasonic pretreated WAS following digestion with respect to dewaterability and pathogen count
- Determine the hydrolysis coefficient for both sonicated and nonsonicated sludge during anaerobic digestion

### Scope of the Study

- WAS sample were obtained from the Thammasat university domestic WWTP located in Pathumthani, and Total solid (TS) of 3% was used.
- Semi-continuous operation at SRT = 20 and 15 days

## Methodology





### **Experimental set-up**





## **Pictorial view of experimental set-up**





Mixer (max capacity 110 rpm) Biogas bag (3L) Water bath (36  $\pm$  1°C) Air seal (Saturated with NaCl + 4% H<sub>2</sub>SO<sub>4</sub>) Converter

6 Booster
7 Horn
8 Sonication chamber
9 Ultrasonic power supply

## **Operational conditions for digesters**



Davamatava	SRT (days)			
Parameters	20	10		
Temperature (°C)	36 ± 1			
Working volume (L)	3			
Mixer speed (rpm)	40-45			
TS (%)	3.0± 0.1			
VS (%)	2.1± 1			
Feeding rate (mL/day)	150	200		
Volumetric organic loading rate (kg VS/m <sup>3</sup> /day)	1.05	1.40		

### **Results and Discussion**



Characteristics of Feed sludge			
Parameters	Feed sludge		
TS (%)	3 ± 0.1		
VS (%)	2.1 ± 0.1		
VS/TS ratio	0.68 - 0.70		
рН	$7.27 \pm 0.05$		
CST (s)	50.5 ± 3.1		
SCOD (mg/L)	80-100		

Horn selection







### **Horn selection**





Large horn gets high SCOD release than medium and small horn

Large horn was selected for the subsequent experiment



## Effect of horn immersed depth on SCOD release





## Effect of ultrasonic density on SCOD release





### Effect of specific energy on SCOD release





Specific energy inputs of 2.3 kWs/gTS and 12 kWs/gTS were critical value for effective mgSCOD/gTS release.

Specific energy input should to be contriled within 12 kWs/gTS for effective SCOD release

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SCOD/TS, (mg/g)

### Effect of sonication on temperature increase







## Microscopic examination with sonication times





### **Effect of sonication time on BMP**





### **Effect of specific energy on BMP**





Specific energy of 12 kWs/gTS was found to be critical for efficient disintegration



## **Kinetic analysis of methane production**





- P = Methane production potential (mL)
- R = Methane production rate (mL/day)
- e = Exponential (1) (= 2.71828)



## **Digester performance data at start-up**



Parameters	Digester A	Digester B	Digester C
Alkalinity (mg/L as CaCO <sub>3</sub> )	1891.7±38.2	1916.7±28.9	1916.7±101.0
рН	7.0±0.1	7.0±0.07	7.0±0.05
TVFA (mg/L as acetic acid)	15.3±0.9	14.2±1.4	15.2±0.2
TS removal (%)	10.2±2.2	9.0±1.3	10.5±1.0
VS removal (%)	20.0±0.5	20.3±0.5	19.5±0.5
Biogas production (mL/week)	1151.7±48.8	1188.3±10.4	1205±13.2
Methane production (mL/week)	685.9±2.1	701.8±0.4	704.1±2.1
Methane content (%)	58.3±0.4	59.4±0.2	58.8±0

### TS and VS removal Efficiency after sonicated sludge feeding





## **Methane production rate**





### **Methane content in Biogas**



Methane content was improved slightly in the full stream and part stream anaerobic digesters than the control digester.



## **Capillary suction time (CST)**



### **Total and Individual VFA**



Specially, concentration of acetic acid and the propionic acid in the digested sludge was influenced by ultrasonic pretreatment and valeric acid level was almost the same



## % of individual VFA in TVFA



Percentage distribution of acetic acid in the total VFA was decreased and percentage of propionic acid increased significantly at SRT = 15 days. percentage distribution of valeric acid in the total VFA remained fairly constant


## **pH and Alkalinity**





pH and alkalinity of the digested sludge in all three digester was in the range of 6.84-7.15 and 1850- 2150 mg/L as  $CaCO_3$  respectively.

#### **Energy balance across the digester**





In part stream digester, around 88% of sonication energy input was replenished in the form of biogas. This energy could be reused to generate electricity to run ultrasound system.





## Conclusions



- Sonication energy input was mainly governed by bottom surface of the horn
- Specific energy inputs of 2.3 kWS/gTS and 12 kWs/gTS were two critical values for effective mgSCOD/gTS release. which was further evidenced by biochemical methane potential (BMP) test conducted at mesophilic condition
- VS removal in the full stream digester was improved by 34.6% at SRT of 20days, and 60.3% at SRT of 15 days with respect to the control digester
- Biogas production rate in the full stream and part stream digesters improved by 102% and 91%, respectively with respect to control at an SRT of 20 days

## Conclusions



- Biogas yield was nearly the same in full stream and part stream digesters which was around 0.50 L/g VS removed at an SRT of 20 days and 0.64 L/g VS removed in an SRT of 15 days
- Methane content in the biogas improved slightly in both full stream and part stream anaerobic digesters in comparison to the control digester
- In part stream digester, around 88% of sonication energy input was replenished in the form of biogas. This energy could be reused to generate electricity to run ultrasound system
- Hydrolysis coefficient of digestion in the, full stream and part stream digesters improved by 94% and 57% at an SRT of 20 days, and 99% and 57% at an SRT of 15 days respectively in comparison to the control digester
- Part stream digester was more recommended than the full stream digester

#### **Recommendations**



- Effect of ultrasonication with specific energy of 12 kWs/gTS on the anaerobic digestibility of sonicated and nonsonicated WAS needs to be studied with controlling temperature of sludge using jacket-cooled water bath
- TS contents should be optimized by considering both the disintegration efficiency and the anaerobic digestibility efficiency
- Evaluation of the change in microbial communities in digesters, when sludge is sonicated, should also be examined
- A cost-benefit analysis of ultrasonic integrated systems needs to be conducted to justify the economics of the process in full-scale applications



# Thanks for your kind attention

