ANAEROBIC DIGESTIBILITY OF ULTRASOUND AND CHEMICALLY PRETREATED WASTE ACTIVATED SLUDGE

by

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Abstract

Proper management of ‘excess sludge’, more precisely, ‘waste activated sludge (WAS)’, is a big challenge to wastewater treatment operators due to implementation of the stringent disposal regulations. To reduce time period of the rate limiting cell lysis step at the first phase of anaerobic digestion, pretreatment processes were commonly adopted prior to anaerobic digestion. Ultrasonic, chemical and chemical-ultrasonic techniques were investigated in this study for disintegrating the sludge. The results revealed that chemical-ultrasonic gave a better efficiency on sludge disintegration compared to individual chemical and ultrasonic techniques. The optimum condition of chemical-ultrasonic was found at 10 mg/g TS chemical dose and 3.8 kJ/g TS specific energy input, whereas chemical dose of 50 mg/g TS and specific energy of 3.8 kJ/g TS were the optimum operating condition of individual chemical and ultrasonic, respectively. The results from anaerobic digester indicated that pretreated sludge could enhance the performance of anaerobic digester by increasing the methane production, and also by TS and VS removal. With three (25, 15 and 10 days) operated Sludge Retention Time (SRT), 15 days SRT was found to be a suitable digestion time for both ultrasonicated and chemical-ultrasonicated sludge. Compared to control digester, the methane production of ultrasonic and chemical-ultrasonic digester at 15 days SRT increased by 23 and 31%, respectively. Beside methane production improvement, degradation rate of chemical ultrasonicated sludge was also faster than ultrasonicated and non-pretreated sludge at all SRTs. At 15 days SRT, the degradation rate of the chemical ultrasonicated sludge improved by 52%, while ultrasonic digester improved by around 26% when compared to control digester. Similarly, dewaterability of digested sludge was also found to be improved by 62 and 66% from ultrasonic and chemical-ultrasonic digester respectively compared to feed sludge. Based on energy balance and economic analysis, control digester operated at 25 days SRT was economically viable. It was found that the income from bioenergy recovery and landfill cost reduction were almost the same as expenses incurred for chemical and energy consumption. However, the 15 days SRT with ultrasonic pretreatment would be more efficient and beneficial as it requires low capital and maintenance cost, smaller reactor volume and reduce the waste volume; consequently reducing the cost for landfill, equipments, energy consumptions, land for treatment plant, etc.

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List of Abbreviations

AD	Anaerobic Digestion
ASP	Activated Sludge Process
BMP	Biochemical Methane Potential
C/N	Carbon to Nitrogen ratio
COD	Chemical Oxygen Demand
CST	Capillary Suction Time
d_{50}	Cut Diameter
DD_{COD}	Degree of Disintegration
DD_{OUR}	Degree of Inactivation
DNA	Deoxyribonucleic Acid
DS	Dry Solids
dS	Sauter Diameter
Es	Specific Energy
EPS	Extracellular Polymeric Substance
FID	Flame Ionization Detector
GC	Gas Chromatography
HRT	Hydraulic Retention Time
KHz	Kilohertz
KWh	Kilowatt Hour
OLR	Organic Loading Rate
OUR	Oxygen Uptake Rate
RF	Radio Frequency
SCOD	Soluble Chemical Oxygen Demand
SOUR	Specific Oxygen Uptake Rate
SRT	Solids Retention Time
STP	Standard Temperature and Pressure
TCD	Thermal Conductivity Detector
TKN	Kjeldahl Nitrogen
TS	Total Solids
TWAS	Thickened Waste Activated Sludge
UD	Ultrasonic Density
VFA	Volatile Fatty Acid
VS	Volatile Solids
WAS	Waste Activated Sludge

Chapter 1

Introduction

1.1 Background

One of the major problems facing the biological wastewater treatment of both domestic and industrial wastewaters is the generation of high amount of biological sludge known as waste activated sludge (WAS). Due to the stringent disposal regulations, proper management of excess sludge poses considerable challenge to wastewater treatment operators. Moreover, the costs of treatment and disposal of WAS account for nearly 60% of the total wastewater treatment plant operating cost. Therefore, many available techniques for WAS treatment have been developed, particularly anaerobic digestion (AD).

Anaerobic digestion of WAS is widely applied for mass reduction, bioenergy production and stabilization. AD offers several merits such as renewable energy production in the form of methane gas, energy saving due to no aeration requirement and pathogens reduction. Hydrolysis phase of AD of WAS, however, is a rate-limiting step due to complex cellular structure of biological cells. This limitation requires a long solids retention time for effective digestion. Thus, pretreatment is often required to accelerate the biological decomposition of WAS and to enhance the biogas production during anaerobic digestion. Various pretreatments, such as thermal, chemical, ultrasonic, and biological have been studied by many researchers. Amongst them, ultrasonic is becoming popular due to several inherent merits, such as no chemical requirement, efficient sludge disintegration, and improvement in digester's stability.

Ultrasonic energy disintegrates flocs and disrupts microbial cells due to the formation and implosion of cavitation bubbles. This leads to the release of intra-cellular matter into the aqueous phase for faster subsequent degradation during digestion. AD of ultrasound pretreated sludge improves volatile solids destruction and biogas production. It, however, requires substantial energy input for effective disintegration of high solid wastes. Small dosing of chemical, especially alkali prior to sonication may potentially enhance the sludge disintegration with significantly less energy input.

Chemical pretreatment can be carried out at ambient temperature. Sodium hydroxide (NaOH) is often used due to the ease of its solubilization in comparison to dibasic reagents. At a relatively low dosage, disintegration of WAS can be improved during sonication, which may significantly increase methane production. Chemical treatment (alkali) alone may require significantly high dosage for solubilization and it may impose sodium toxicity to methanogens during digestion. Therefore, combination of chemical and ultrasonic (chemical-ultrasonic) pretreatment could significantly improve sludge disintegration with relatively low chemical dose and energy inputs. Ultrasonic and chemical disintegration of WAS can also improve the dewaterability of the digested sludge which could reduce the dewatering cost. Hence, the sludge handling cost could be reduced.

1.2 Objectives of the Study

The main goal of this study is to investigate the performances of anaerobic digestibility of WAS following ultrasonic and chemical-ultrasonic pretreatments. The specific objectives are as follow:

1. To optimize ultrasonic, chemical and chemical-ultrasonic pretreatment to maximize WAS disintegration.
2. To investigate performances of anaerobic digestibility of the pretreated and non-pretreated WAS (control) at different solids retention times (SRTs).
3. To determine the rate constant of the hydrolysis step for both pretreated and non-pretreated sludge during anaerobic digestion.
4. To conduct an economic analysis of various pretreatment options: ultrasonic and chemical-ultrasonic.

1.3 Scope of the Study

The study was conducted at AIT based on laboratory scale experiment with the overall operation as bellow:

1. The sewage waste activated sludge used in the research was collected from Future Park Rangsit wastewater treatment plant located in Pathumthani, Thailand.
2. TS content of sample sludge was increased to 3% (30,000 mg/L) by centrifugation.
3. Semi-continuous feeding was applied to anaerobic reactors two times per day in equal time intervals.
4. Solids retention times of anaerobic digesters were 10, 15 and 25 days.

Chapter 2

Literature Review

2.1 Introduction

Domestic wastewater is one of the big sources of water pollution in the environment and need to be treated. Biological processes have been widely used to treat wastewater to meet the discharge standard for preventing the public health effect and associated environmental problems. One of the most commonly used techniques is activated sludge process (ASP). ASP is a potential method of treating wastewater but it produces high amount of biomass as a big concern of sludge disposal and management. Wei et al. (2003) was estimated that the spending cost of the excess sludge treatment and disposal is up to 60% of the total cost of wastewater treatment plant. To overcome this problem, anaerobic digestion of WAS was used to minimize around 40 to 50% of sludge production and to gain the biogas as a renewable energy (Kim et al., 2003). Though, anaerobic gives a great interest of its end products, the complexities of the process were found and need solving further. Pretreatment processes are involved to tackle the problem of long digestion time requirement due to the hydrolysis step which is considered as a rate limiting step in anaerobic process (Wang et al., 1999b). As a result, various pretreatment were investigated with different aspects. Similarly, ultrasonic, chemical and chemical-ultrasonic pretreatment of WAS followed by anaerobic digestion was investigated in this study with respect to various conditions.

In order to get the clear understanding of the study, type and characteristic of sludge, anaerobic process, ultrasonic, chemical and chemical-ultrasonic pretreatment are given in details in this chapter.

2.2 Type and Characteristics of Sludge

Normally, sludge is divided into three different categories namely primary, secondary or waste activated sludge and digested sludge.

2.2.1 Primary sludge

Primary sludge is produced through the mechanical wastewater treatment process. It occurs after the screen and the grit chamber and consists of unsolved wastewater contaminations. The sludge amassing at the bottom of the primary sedimentation basin is also called primary sludge. The composition of this sludge depends on the characteristics of the catchment area. Primary sludge consists to a high portion of organic matters, as faeces, vegetables, fruits, textiles, paper etc. The consistence is a thick fluid with a water percentage between 93% and 97%.

2.2.2 Secondary sludge or waste activated sludge

Waste activated sludge is generally coming from the secondary wastewater treatment process. In the secondary treatment, different types of bacteria and microorganisms consume oxygen to live, grow and multiply in order to biodegrade the organic matter. The resulting sludge from this process is called waste activated sludge. Normally, a part of the WAS is return back to the system call return activated sludge and the remaining is removed at the bottom of secondary clarifier called excess sludge or secondary sludge. Overall, the sludge is the same properties but different calling regarding to their usage.

WAS consists largely of biological mass, mainly protein (30%), carbohydrate (40%) and lipids (30%) in particulate form (Lin et al., 1999). Normally, WAS contains large amount of pathogens and causes odor problem. Therefore, sludge has to be stabilized to prevent public health and odor nuisance.

2.2.3 Digested sludge

Digested sludge accrues during the anaerobic digestion process. When primary or WAS is used as raw material of anaerobic reactor, it will be stabilized by anaerobic microorganisms and the remaining waste after the process is called digested sludge.

2.3 Anaerobic Digestion

Anaerobic digestion (AD) is the biochemical process which is used to decompose the organic matter by various microorganisms with the absence of oxygen. Anaerobic treatment processes include anaerobic suspended growth, upflow and downflow anaerobic attached growth, fluidized-bed attached growth, upflow anaerobic sludge blanket, anaerobic lagoons and membrane separation anaerobic processes. Amongst them, anaerobic suspended growth is selected to use in this study. Before going to detail of the AD process, it would be better to know the advantages and the weakness of its operation. The advantages and disadvantages of the AD are shown in Table 2.1.

Table 2.1 Advantage and disadvantage of anaerobic processes

Advantages	Disadvantages
Less energy required: no oxygen has to be supplied, or extensive mixing has to take place	Longer start-up time to develop necessary biomass inventory
Less biological sludge production: sludge processing and disposal costs are reduced greatly	Might need alkalinity addition: Alkalinity concentration of 2000 to 3000 mg/L as $CaCO_3$ may be needed in anaerobic process to maintain an acceptable pH with the high gas phase CO_2 concentration
Fewer nutrients required	May require further treatment: anaerobic processes can also be followed by aerobic treatment process to meet discharge requirement
Methane production, a potential of energy source	Biological N and P removal is not possible
Smaller reactor volume required: high organic loading rate	Much more sensitive to the adverse effect of lower temperature on reaction rates
Elimination of off-gas air pollution	May be more susceptible to upsets due to toxic substances
Rapid response to substrate addition after long periods without feeding	Potential for production of odors and corrosive gases

Source: Modified from Metcalf & Eddy, 2003

2.3.1 Process description

Anaerobic digestion is a complex biochemical process in which several groups of facultative and anaerobic organisms simultaneously assimilate and break down organic matter. It is well known that there are four main steps take place in AD namely hydrolysis, acidogenesis, acetogenesis and methanogesis (Lin et al., 1999). Figure 3.1 illustrates the process of AD.

a. Hydrolysis

During hydrolysis, complex insoluble organic polymers, such as carbohydrates, cellulose, proteins and fats, are broken down and liquefied by the extracellular enzymes produced by

hydrolytic bacteria. This makes them more easily available for use by the acidogenic bacteria of the next stage. In general, proteins present in the waste are converted into amino acids, fats into long-chain fatty acids and carbohydrates into simple sugars. The liquefaction of complex compounds, and especially cellulose, to simple, soluble substance is often the rate limiting step in digestion (Evans, 2001). The rate at which hydrolysis takes place is governed by substrate availability, bacterial population density, temperature and pH.

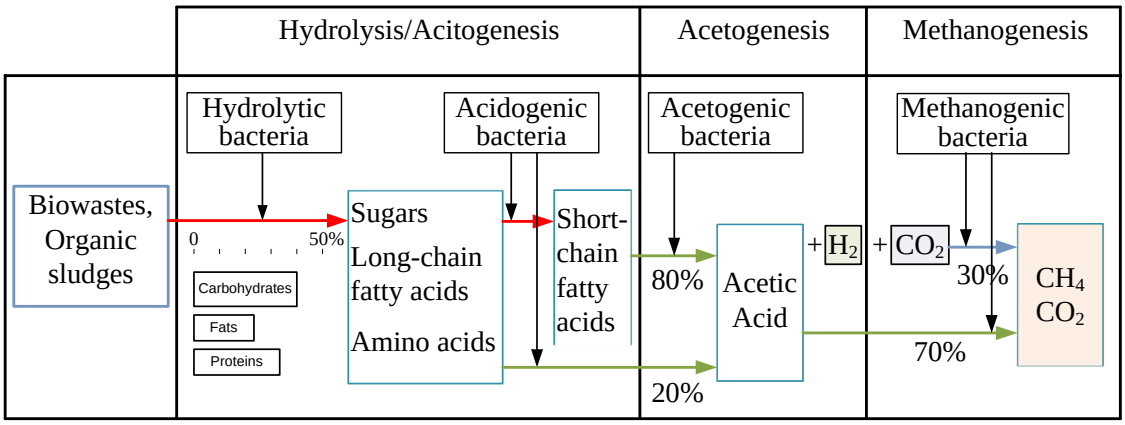


Figure 2.1 Metabolic chain of anaerobic digestion (Modified from Metcalf & Eddy, 2003)

Due to the complex cell structure of waste, particularly WAS, various pretreatments are normally used to break down the microbial cell for releasing the intra and extra-cellular into the aqueous phase which results in accelerating degradation rate of AD. The use of physical (Ultrasonic, Bill milling), chemical (Alkali, Acid, Ozonation), thermal (Pyrolysis) and biological (Enzyme) pretreatment is to accelerate the solubilization (hydrolysis) of WAS.

b. Acidogenesis and Acetogenesis

Acidogenesis, sometimes, splits into acidogenesis and acetogenesis. In this stage, amino acids, sugars and some fatty acids are degraded further to acetate, hydrogen, CO_2, and propionate and butyrate. The propionate and butyrate are fermented further to also produce hydrogen, CO_2 and acetate. Thus the final products of the stage are acetate, hydrogen and CO_2. The free energy change associated with the conversion of propionate and butyrate to acetate and hydrogen requires that hydrogen be at low concentrations in the system ($H_2 < 10^{-4}$ atm) or the reaction will not proceed (Metcalf & Eddy, 2003). The pH falls down as the level of organic acids increase and the proportion of the different by-products produced depends on the environmental conditions, to some extent, and more largely on the particular bacteria species present.

c. Methanogenesis

Methanogenesis is the last step of AD. Two groups of methanogenic organisms convert the end products from the preceding stages to methane gas. Methane is mainly converted from acetic acid (approximately 75%) by aceticlastic methanogens and hydrogen and CO_2 (approximately 25%) by hydrogen-utilizing methanogens. Methanogens are pH sensitive; pH should be monitored properly as methanogenesis is the most important process of sludge stabilization.

2.3.2 Factors affecting anaerobic digestion process

a. Biological-physical factors

To achieve high levels of sludge stabilization certain biological and physical requirements of the methane-forming microorganisms must be met. These factors are summarized in Table 2.2.

Table 2.2 Factors in anaerobic digestion

Physical factors	Chemical factors
Temperature	pH
Hydraulic Retention Time	Alkalinity
Solids Retention Time	Volatile Acids
Solids loading	Nutrients
Mixing	Trace Elements
Solids Concentration	Toxic compounds
Sludge Type	
Volatile Solids Loading	

Source: Cook and Boening, 1987

Solids loading rate. The solids retention time, hydraulic retention, volume and solids concentration determine the solids loading to the digester. These factors determine the amount of sludge "food" the microorganisms must stabilize and the amount of time the microorganisms must stabilize the sludge. Microorganisms growth and stabilization rate are the main factors which determine the maximum loading rates possible for stable operation. To control a proper operation, biologically volatile solids are the most important element to control because they are degradable solids.

Mixing. It plays an important role in AD. It helps to homogenize the digesting material, thereby avoiding any localized concentrations of any given substance, dead zones or scum formation. In addition, it improves contact between the material itself and the digester's resident bacteria, thereby increasing their ease of access to the available nutrients and facilitating the desired breakdown of the biowaste feedstock. Moreover, mixing can also release the gas generation and maintain a more uniform temperature within the digester.

Temperature. It is another main factor for monitoring anaerobic digester. AD is commonly operated in both mesophilic (35^oC) and thermophilic (55^oC) due to the optimum condition of methanogens. Normally, microorganisms grow faster at higher temperature which leads to digest more organic matters (high organic loading rate). Thus, thermophilic AD can decompose the organic substances faster which result in more biogas generation compared to mesophilic condition. Due to the high energy consumption for controlling temperature, very sensitive of methanogenic bacterias to temperature variation ($<0.5^oC$), and comparable biogas yield to mosophilic, thermophilic is not economically viable. Mesophilic, therefore is selected and operate in the temperature range of $35\text{-}37^oC$ (36 ± 1^oC). The comparison of thermophilic and mesophilic condition is presented in Table 2.3.

b. Chemical factors

The anaerobic process is biochemical in nature. The proper chemical environment is required for microorganisms to function properly. Some of the chemical factors are listed in Table 2.2.

Alkalinity, Volatile Acids and pH. These three factors and their effects on AD are interdependent and accordingly, are best considered together. Monitoring the pH is required to enable adequate process control in order to provide the optimum conditions for the balanced growth of microorganisms. VFAs are important intermediary compounds in the metabolic pathway of methane fermentation and cause microbial stress if present in high concentrations, resulting in a decrease of pH, and ultimately leading to failure of the digester. Thus, the concentration of VFAs is an important consideration for good performance of a digester (Hill and Holmberg, 1988, Hill and Bolte, 1989 and Ahring et al., 1995). It was found that VFAs decrease throughout the reactor from bottom to top (Buyukkamaci and Filibeli, 2004). The main acids are acetate, propionate and n-butyrate (Kim et al., 2002a). Among them, n-butyric acid degradation rate was found to be the highest (Wang et al., 1999a). The ratio of propionic acid to acetic acid can also be used as an indicator of digester imbalance. When the acetic acid level in excess of 800 mg/L or a propionic acid to acetic acid ratio greater than 1.4 indicated digester failure (Buyukkamaci and Filibeli, 2004). Alkalinity plays an important role of neutralizing VFAs in the digester in order to maintain the pH in the range of 6 to 8 for methane-forming microorganisms. However, the optimum pH range of methanogenic bacteria is between 6.8 and 7.2. If volatile acids increase, they are neutralized by bicarbonate alkalinity. Digester should have a bicarbonate alkalinity concentration of 2500 to 5000 mg/L to neutralize volatile acids and prevent a drop of pH (Cook and Boening, 1987). Table 2.4 represents the healthy and failure digestion with respect to individual VFA.

Table 2.3 Comparison of mesophilic and thermophilic conditions

Parameter	Mesophilic	Thermophilic
Temperature	20 – 45 ^{o}C	> 45 ^{o}C
Residence Time	15 – 30 days	10 – 20 days
Benefits	- More robust and tolerance process - Less sensitive to the temperature change (within 2 ^{o}C) - Less energy consumption due to low temperature supplied	- High gas production - Faster throughput - Short residence time - Small digester volume - High organic loading rate
Limitations	- Low gas production rate - Large digester volume - Long residence time	- Need effective control - Very sensitive to temperature change (<0.5 ^{o}C) - High energy consumption

Nutrient. The major nutrients required in anaerobic digestion are phosphorus and nitrogen. These elements are building blocks for the cells of microorganisms responsible for sludge stabilization. The amount of each nutrient required is directly proportional to the amount of microorganisms grown. An average cell contains approximately 12.5% nitrogen and 2%

phosphorus (Cook and Boening, 1987). Overall the optimum range of C:N ratio for AD is 20-30.

Table 2.4 FVA conditions for anaerobic digestion

Individual VFA	Healthy digestion	Failure of digestion
Acetic acid	< 8000 mg/L	> 8000 mg/L
Iso-butyric	< 5 mg/L	> 15 mg/L
Iso-valeric	< 5 mg/L	> 15 mg/L
Propionic/Acetic ratio	< 1.4	> 1.4

Toxicity. The anaerobic treatment process is sensitive to certain compounds including sulfides, volatile acids, heavy mentals, calcium, sodium, potassium, dissolved oxygen, ammonia and chlorinated organic compounds. The inhibitory concentration of a substance depends on many variables, including pH, organic loading, temperature, hydraulic loading, the presence of other materials, and the ratio of the toxic substance concentration to the biomass concentration. Table 2.5 summarizes inhibitory levels of several compounds.

Table 2.5 Inhibitory levels of several compounds in anaerobic digester

Ammonia Nitrogen	
NH_3-N concentration (mg/L)	Effect
50-200	Beneficial
200-1000	No adverse effects
1500-3000	Inhibitory at pH over 7.4-7.6
Above 3000	Toxic

Total concentration of individual metals required to severed inhibit anaerobic digestion			
Metal	Percent dry solids	M mole metal/kg dry solids	Soluble metal (mg/L)
Copper	0.93	150	0.5
Cadmium	1.08	100	-
Zinc	0.97	150	1.0
Iron	9.56	1710	-
Chromium			
+6	2.20	420	3.0
+3	2.60	500	-
Nickel	-	-	2.0

Stimulating and inhibitory concentrations of light metal cations			
Cation	Stimulatory	Moderately inhibitory	Strongly inhibitory
Calcium	100-200	2500-4500	8 000
Magnesium	75-150	1000-1500	3 000
Potassium	200-400	2500-4500	12 000
Sodium	100-200	3500-5500	8 000

Source: Cook and Boening, 1987

2.4 Pretreatments

Municipal wastewater sludge, particularly WAS, is more difficult to digest than primary solids due to the rate limiting cell lysis step which result in long digestion time and large fermenters. Typical digestion times of anaerobic WAS are generally more or less 20 days. Therefore, pretreatments are needed to increase the degradation rate and improve the

biogas quantity and quality. Chemical pretreatment (Wei et al., 2003; Lin et al., 1999 and Lin et al., 1997) and ultrasonic pretreatment (Sangave et al., 2007; Nickel and Neis., 2007 Bougrier et al., 2006, 2005; Wang et al., 2006) had been investigated on sludge disintegration and improvement of AD performance. Similarly, thermal and ozone pretreatment were also studied to find out the degree of disintegration (Bougrier et al., 2006; Sangave et al., 2007). Moreover, pretreatment can be done by using enzyme and thermophilic aerobe/anaerobe. Though, individual pretreatments give a good effect in sludge solubilization, the combination of thermal and chemical pretreatment (thermochemical pretreatment) were studied by Vlyssides and Karlis, (2004), and Kim et al. (2003) using lime and alkali reagents, respectively, at high temperature. Till now, there is a very few study on the effects of combining chemical and ultrasonic (chemical-ultrasonic) technique in the field of WAS disintegration. However, Chiu et al. (1997) conducted a detailed study of the combination of these two processes.

The main purpose of various pretreatments are to solubilize and/or to reduce the size of organic compounds, and especially refractory compounds, in order to make them more easily biodegradable (Bougrier et al., 2006). Hence, improvement accessibility to soluble organic substances resulted in faster, more extensive rates of VFA and methane generation (Wang et al., 1999a). Table 2.6 summaries the options of sludge disintegration.

Table 2.6 Options for the disintegration of sewage sludge

Type	Process
Mechanical	Bill milling
	High pressure homogenization
	Shear gap homogenization
	Lysate centrifugation
Electrical	Electro impulse discharge
Thermal	Pyrolysis
Thermal-Biological	Aerobic digestion
Chemical	Acid/Base reaction
	Ozone oxidation
Biological	Addition of enzymes
Acoustic	Cavitation/Sonochemical reaction

Source: Neis and Tiehm, 2007

2.5 Ultrasonic Pretreatment

Ultrasonic pretreatment of WAS is the process of supplying pressure wave which leads to cavitations bubble formation in the liquid phase. These bubbles grow and then violently collapse when they reach a critical size. Cavitational collapse produces intense local heating and high pressure on liquid-gas interface, turbulence, and high shearing phenomena in the liquid phase. Thus, sonication is a combination of different phenomena: chemical reactions using radicals, pyrolysis, combustion and shearing (Bougrier et al., 2005). However, it was found that hydro-mechanical shear forces are predominantly responsible for ultrasonic activated sludge disintegration (Wang et al., 2005).

Ultrasonic pretreatment give a great effect on the physical, chemical and biological characteristic of WAS (Chu et al., 2001). The efficiency of sonication depends on many factors during the process such as sonication duration, temperature increment, ultrasonic frequency, energy supplied, total solids (TS) content of WAS, the nature of the influent and the ultrasound system (booster, converter, horn design). All of these effects and the influent factors are given in details in this section as well as its mechanisms and its usefulness.

2.5.1 Mechanisms of ultrasound

Ultrasound is a cyclic sound pressure with a frequency greater than the upper limit of human hearing, this limit being approximately 20 kilohertz (20,000 hertz). Figure 2.2 shows the range of frequency range from infrasound to ultrasound.

Even through ultrasound vibrations are above the human audible range, ultrasonic processing produces a high pitched noise in the form of harmonics, which emanate from the vessel walls and the fluid surface. The sound abating enclosure permits extended processing without discomfort by reducing the sound by 35 db. The probe/converter assembly is supported by the converter clamp, and the converter cable is fed through the opening at the top.

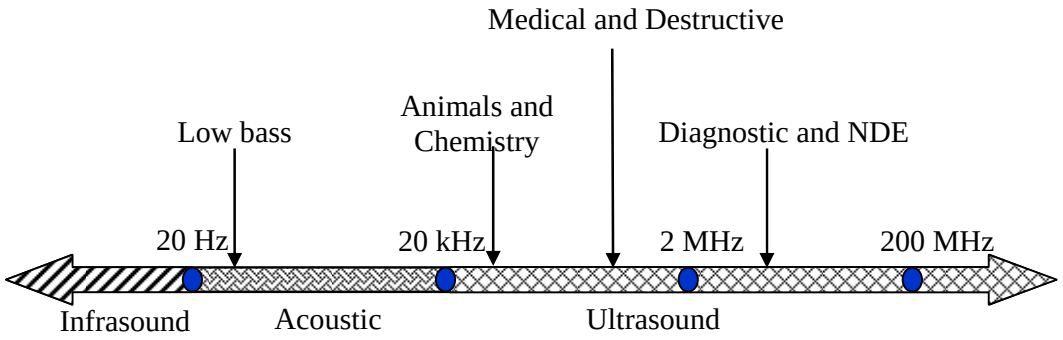


Figure 2.2 Ultrasound range diagram

The unit is faced on the exterior with white laminate, and lined on the interior with white waterproof noise abating material. The access door permits observation during treatment and protects the operator against accidental splashing. The details configuration of the sonicator is given in Figure 2.3.

2.5.2 Usefulness of ultrasound

Ultrasound offers a great potential for improving water, wastewater and sludge treatment processes. The application of ultrasound technology in environmental engineering still is not widely applicable as it is a new technology. Many studies were conducted to find out the effectiveness of ultrasound but a number of scientific and technical questions exist addressing, for example the influence of frequency, of dissolved gases and of suspended solids on cavitation, optimal reactor design, economy, reliability and life expectation of ultrasound equipment. An overview is given on current ultrasound applications in water, wastewater and sludge systems (Table 2.7).

2.5.3 Energy or power input to sludge

Energy (J or kJ) or power (W or kW) input supplied by ultrasound plays a very important consideration with respect to economic aspect. Normally, ultrasound gives a good effect to sludge disintegration at high energy input which leads to increase the operation cost of the treatment. Thus, quantification of energy/power input has to be properly supplied to get the effective sludge disintegration. According to Bougrier et al (2005), specific supplied energy lower than 1000 kJ/kg TS is used to reduce flocs size and supplementary energy will be used to break down flocs or cells. It is not necessary to supply energy higher than 7000 kJ/kg TS as it does not give significant increase in biogas production. The degree of disintegration does not depend on only power input but also the treatment time (Tiehm et al., 2001). Total energy requirement depends on the operation duration. Thus, optimum energy supplied and operation duration needs to be investigated for higher degree of disintegration. Hence, the operating cost will be reduced. The power or energy supplied for sludge disintegration can be expressed in a number of ways as elucidated below.

a. Specific energy input

The important parameters affecting the ultrasonic disintegration are 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 (Es). The specific energy input is a function of ultrasonic power, sonication duration, sample volume and initial total solids concentration (Bougrier et al., 2006). It can be calculated using the following equation:

$$Es = \frac{P}{V \,.\, TS} \; x \; t \qquad \text{Eq. 2.1}$$

Where,

Es : Specific energy in kWs/kg TS (KJ/kg TS)
P : Power input in kW
t : Sonication time in second (s)
V : Volume of sludge used for sonication in liter (L)
TS : Total solids concentration in kg/L

Specific energy is one of the most commonly used parameters amongst ultrasonic dose, ultrasonic density and ultrasonic intensity for the correlation of energy supplied and sludge disintegration because it can be applied to every sludge characteristics.

b. Ultrasonic dose

Ultrasonic dose is the energy supplied per sample volume and expresses in Wsl^{-1} or Jl^{-1} (Tiehm et al., 2001). It can be calculated by below equation:

$$Ultrasonic\ dose = \frac{P}{V} \; x \; t \qquad \text{Eq. 2.2}$$

The ultrasonic dose cannot be used to compare the power input to different TS content of the sludge. As long as the TS content remains fairly constant, the ultrasonic dose is a practical method of expressing power input for the disintegration of sludge on a volume basis.

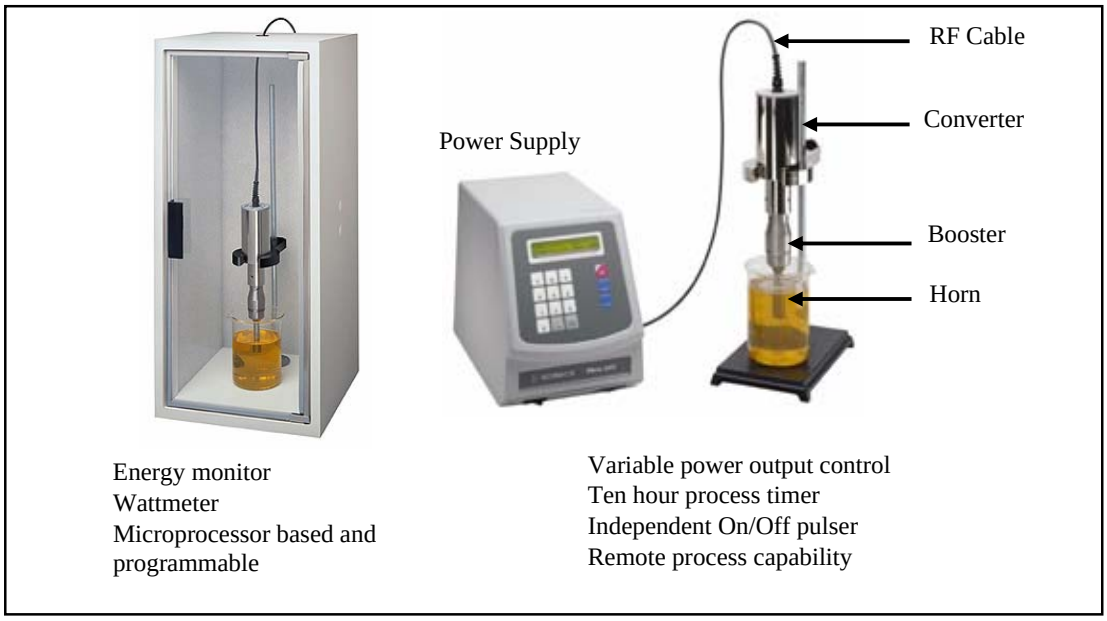


Figure 2.3 Ultrasonic equipment

Table 2.7 Ultrasound applications in environmental engineering

Domain	Objective
Potable water	Inactivate bacteria (disintegration)
	Improve separation of solids
	Improve filter regeneration
	Remove incrustations in pipes and wells
Wastewater	Sonochemical pollutant degradation
	Improve biological degradation
	Membrane fouling control
Sludge	Disintegrate biosolids
	Decompose bulking activated sludge
	Flocs to allow sedimentation
	Improve dewatering

Source: Modified from Neis and Tiehm, 2007

c. Ultrasonic density

Ultrasonic density relates to the power supplied per sample volume and expresses in Wl^{-1} (Tiehm et al., 2000). It can be calculated by below formula:

$$Ultrasonic\ density = \frac{P}{V} \qquad \text{Eq. 2.3}$$

d. Ultrasonic intensity

Ultrasonic intensity relates to the power supplied per transducer area and expresses in Wcm^{-2}. It can be calculated by below formula:

$$Ultrasonic\ \text{int}ensity = \frac{P}{A} \qquad \text{Eq. 2.4}$$

Where,

A : Transducer surface area (cm^2)

2.5.4 Evaluation of ultrasonic disintegration

Ultrasound pretreatment is believed to change the physical, chemical and biological properties of WAS, and improved and stabilized the AD which lead to increase the biogas production. A high pressure wave of ultrasound will produce a high sharing force which breaks down bacterial cell wall and releases the intracellular into aqueous phase. In addition, ultrasound also helps to deagglomerate the biological flocs and disrupts large organic particles into smaller size particles. Thus, the degree of sludge disintegration has to be evaluated base on physical (particle size distribution and microscopic examination), chemical (increase in SCOD and protein concentration, and release of NH_3) and biological (heterotrophic count and specific oxygen uptake rate) properties. Detailed discussion of each property is presented in the following section.

a. 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. Particle size distribution is one of the factors affecting the AD. The smaller particle size gives a large surface area which leads to increase the AD efficiency. Therefore, ultrasound was found to be very effective to break down particles size in WAS. Particle size distribution during sonication operation depends upon on power input, sonication frequency, sonication duration and sludge characteristic. Bougrier et al. (2005) performed an ultrasonic treatment of WAS using 20 kHz frequency and different specific energy inputs. The study was investigated the particle size distribution within the ranged from 0.4 to 1000 µm. The volume occupied by small particles increased with the increasing of specific energy supplied: for Es = 14, 550 kJ/kg TS particles of 1µm occupied 1.5% of the whole volume, whereas they occupied 0.1% in the untreated sample. Table 2.8 presents the distribution of particle size obtained for cut diameter (d_{50}) and for Sauter means diameter (dS).

Similarly, Chu et al. (2001) found that the floc size of WAS reduced accordingly to the sonication density and duration times. At the sonication density of 0.11 W/mL, there is almost no effect on the floc size. Only when the sonication density has exceeded 0.22 W/mL would the particle size apparently decrease. The higher sonication densities of 0.33 and 0.44 W/mL reduce particle size from 98.9 µm (mean diameter) to 22 and 3 µm after 20 min sonication duration, respectively. The decrease trend of particle size after sonication seems less effective even higher sonication density is supplied. Figure 2.4 shows the correlation of floc size and sonication duration. Again, Bougrier et al. (2006) compared the

particle size reduction amongst sonication, ozonation and thermal treatment. Ultrasound was operated at 20 kHz frequency and about 225 W supplied power. It was found that the flocs size is reduced from 36 µm to 10.7 and 9.6 µm with Es of 6250 and 9350 kJ/kg TS, respectively. Tiehm et al. (2001) was concluded that the lower ultrasonic frequency (20 KHz) gives higher sludge disintegration efficiency which leads to reduce median sludge particle size as well as to increase in turbidity of the sludge sample.

Table 2.8 Cut diameter and Sauter diameter for different supplied energies

	Specific Energy (kJ/kg TS)				
	0	660	1350	6950	14550
d_{50} (µm)	31.99	19.6	18.5	17.6	12.7
dS (µm)	18.5	11.2	8.3	5. 8	3. 7

Note: d_{50} : 50% of particles volume having a diameter lower or equal to d_{50}
dS : corresponds to the diameter of a sphere of the same surface area.

Source: Bougrier et al. (2005)

Microscopic image evaluation. The sludge disintegration has widely examined based on visual observation using light and electron microscopes. Basically, the architecture of floc after sonication within 40 min at 0.11 W/mL is the same as the original sludge even the floc structure becomes somewhat looser and some filamentous bacteria have been exposed outside. However, the structure integrity of floc has almost completely broken down after 40 min sonication at 0.33 W/mL (Chu et al., 2001). Khanal et al. (2006b) was investigated on structural changes of WAS at a constant power input of 1.5 kW and a frequency of 20 kHz with respect to different sonication times. During 2 min of sonication, the srructural integrity of flocs as well as filaments was significantly disrupted without appreciable destruction of bacterial cells. Up to 10 min sonication, nearly complete disintegration of flocs and filament-like structures with a few scattered bacterial cells was observed. When the sludge was sonicated for 30 min, more or less complete break-up of cell walls was observed with several punctured cells.

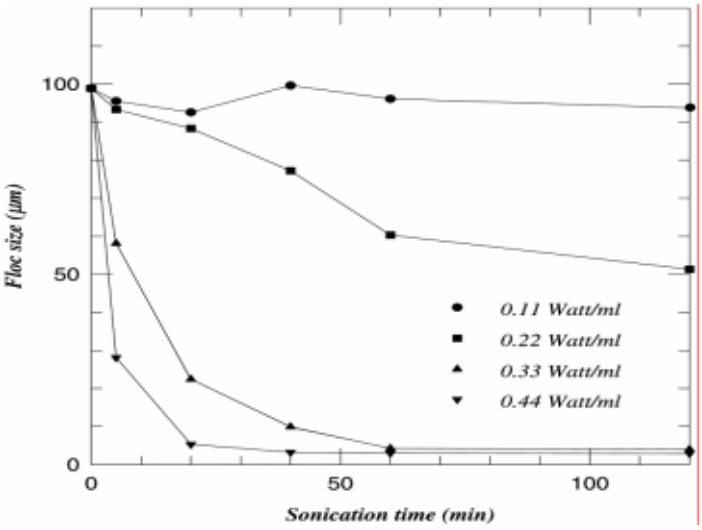


Figure 2.4 Floc size vs. sonication time (Chu et al., 2001)

b. Chemical evaluation

Chemical evaluation of ultrasonic pretreatment mainly focuses on sludge disintegration efficiency. It primarily measure the solubilization of WAS in the aqueous phase in term of SCOD increment. SCOD plays an important role for sludge disintegration evaluation, and Protein and Ammonia (NH_3) are also the important parameters investigated after pretreatment.

SCOD assessment

After ultrasonic pretreatment, microbial cells are broken down and the organic matters are released to the aqueous phase which leads to increase the soluble organic substances measured in term of SCOD. Ultrasonic also disintegrates extracellular matter including organic debris and extracellular polymeric substances (EPS), which become part of SCOD. Therefore, SCOD is the main parameter of the evaluation.

Ultrasonic pretreatment efficiency depends on several factors such as sonication frequency, TS content, influent sludge characteristics, sonication duration, temperature, and power supply. Thus, to find out the unity of sludge disintegration evaluation, SCOD released with respect to specific energy input is commonly applied. Khanal et al. (2006a) investigated the sludge disintegration efficiency with several specific energy inputs. Specific energy of 35 kJ/g TS was found to be the optimum power input for the highest SCOD released. Moreover, Wang et al. (2006) investigated the SCOD release at ultrasonic density of 0.768 W/mL with different sonication times. The author found that the increment speed of SCOD was slow down after 20 min disintegration time.

In order to evaluate the sludge disintegration efficiency regarding the COD data, one parameter known as "degree of disintegration (DD)" was commonly used for many researchers. Tiehm et al. (2001), Rai et al. (2004), Bougrier et al. (2005) and Nickel and Neis. (2007) used degree of disintegration (DD_{COD}) modified by Müller and Pelletier (Modified version from Kunz and Wagner). DD_{COD} is the comparison between SCOD release by ultrasonic disintegration and a maximum SCOD release obtained by alkaline addition (chemical disintegration).DD_{COD} can be calculated as bellow equation:

$$DD_{COD} = \frac{(SCOD - SCOD_o)}{(SCOD_{NaOH} - SCOD_o)} x\,100 \qquad \text{Eq. 2.5}$$

Where,

SCOD : Soluble COD of sonicated sample (mg/L)
$SCOD_o$: Soluble COD of untreated sample (mg/L)
$SCOD_{NaOH}$: Soluble COD of reference sample alkaline disintegration (mg/L)

$SCOD_{NaOH}$ is believed to be the maximum COD release of the complete disintegration of sludge and use as a reference COD. It normally carries out by treating the sludge sample with 1 M NaOH in the ratio of 1:2 for 10 min at 90 ^oC. However, it is varied depending on researcher's modification.

Protein assessment

According to the procedure of Kunz and Wagner, and of Müller, six or three COD analyses are respectively required in order to get a reliable result. Due to time consuming and

analytical cost of COD analysis, protein measurement was found to be a reliable technique of assessing the sludge disintegration (Schmitz et al., 2000 and Khanal et al., 2007). However, for field application, protein measurement is still not common as none of the published studies employed protein measurement to assess the efficiency of ultrasonic sludge disintegration. The COD measurement will continue to be the method of choice for daily operation due to its simplicity.

Wang et al. (2006) examined the release of protein, polysaccharide and deoxyribonucleic acid (DNA) of WAS with various specific energy inputs. It was found that protein was predominant in the aqueous phase of the sonicated sludge. Protein was released very fast during the first 20 min, while polysaccharide was fluctuated and DNA dropped a little after 20 min. Figure 2.5 represents the release of protein, polysaccharide and DNA versus specific energy inputs. The release of soluble protein and carbohydrate in the aqueous phase during different sonication times was also investigated by Wang et al. (1999a).

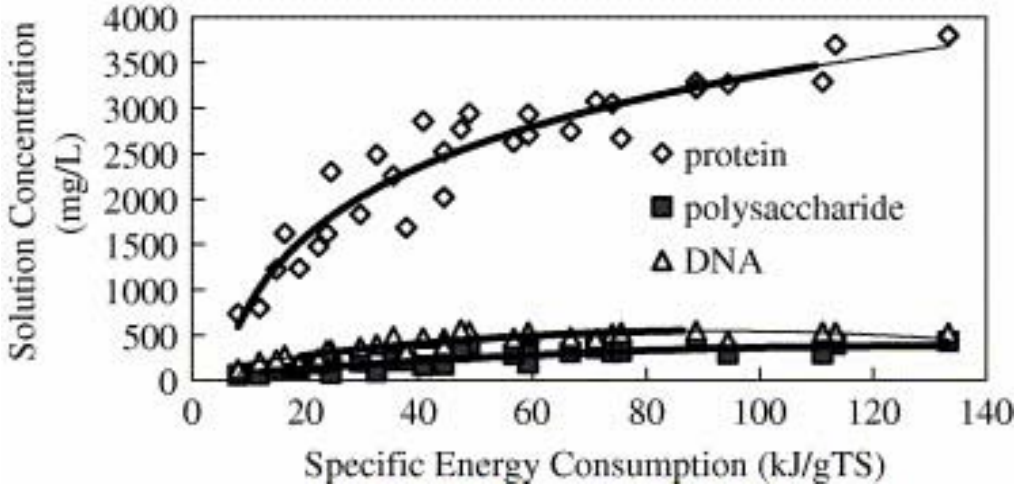


Figure 2.5 Specific energy consumption versus solution concentration of protein, polysaccharide and DNA (Wang et al., 2006)

NH_3 assessment

Ammonia nitrogen (NH_3-N) is also one of the parameters investigated during sludge disintegration. Khanal et al. (2006b) studied the NH_3-N released of WAS after sonication with several TS contents and specific energy inputs. The author found that NH_3-N concentration reached a fairly constant level at lower specific energy input compared to SOCD released, for example, 20 kWs/g for 2.0, 2.5, and 3% TS, and 10 kWs/g for 1.5% TS. Figure 2.6 shows the release of NH_3-N of different TS content WAS with respect to specific energy inputs.

Bougrier et al. (2005) investigated nitrogen solubilization of WAS after ultrasonic treatment which performed at different specific energy inputs. The total Kjeldahl nitrogen (TKN) was found to be constant whatever the specific energy. It can be concluded that ultrasound did not contribute to nitrogen mineralization or vitalization. On the other hand, the organic nitrogen and ammonia concentration in the aqueous phase was increased while organic nitrogen in particle was decreased. The maximum nitrogen solubilization was obtained for a supplied energy of 10,000 kJ/kg TS.

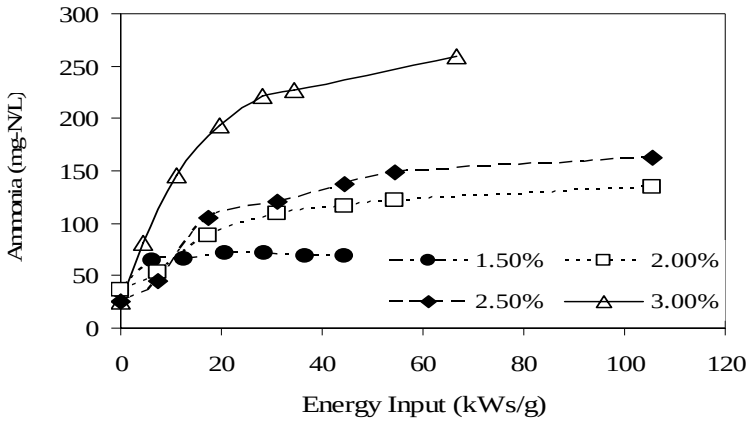


Figure 2.6 Ammonia released versus specific energy input (Khanal et al., 2006b)

c. Biological evaluation

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) observed the survival ratio of heterotrophic bacteria and total coliform against the sonication times. It was reported that the survival ratio (ratio of viable bacteria density levels after sonication to those of original sample) of heterotrophic bacteria decreased with sonication time and reached a value of 44% at a sonication density of 0.33W/mL during 120 min of sonication (Figure 2.7). However, heterotrophic plate count is not a pragmatic method for judging the sludge disintegration efficiency in field applications.

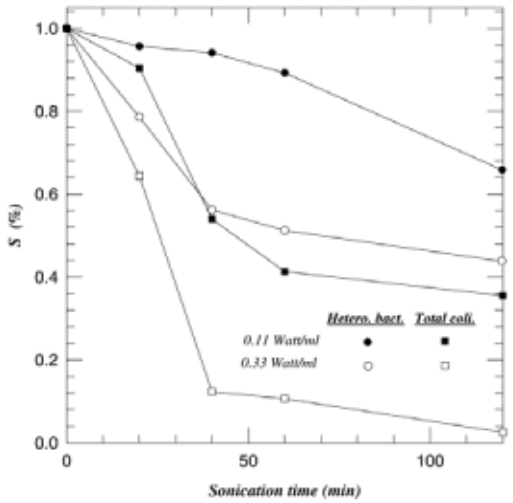


Figure 2.7 Survival ratio (S) vs sonication time (Chu et al., 2001)

Specific oxygen uptake rate

Normally, WAS consists of aerobic and facultative bacteria. They use oxygen in their metabolic processes. Therefore, measurement of oxygen uptake rate (OUR) is a good indicator of bioactivity of WAS. Since ultrasonic treatment disrupts the bacterial cells, which result in loss of their ability to consume oxygen, the measurement of specific oxygen uptake rate (SOUR) of sonicated WAS could be used to assess the effectiveness of

sludge disintegration. Based on this premise, Rai et al. (2004) used a parameter known as "degree of inactivation" to evaluate the sludge disintegration which can be calculated as follows:

$$DD_{OUR} = \left[1 - \frac{OUR_{sonicated}}{OUR_{original}}\right] x100 \qquad \text{Eq. 2.6}$$

Where,

$OUR_{sonicated}$: Oxygent uptake rate of sonicated sludge
$OUR_{origine}$: Oxygen uptake rate of untreated sludge (original sample)

OUR can be computed by measuring the decrease in dissolved oxygen over time. And the equation is as follow:

$$OUR = -\frac{d[O_2]}{dt} \qquad \text{Eq. 2.7}$$

The DD_{OUR} increased rapidly with increase in specific energy input up to 40 kJ/g TS, after that the increase slowed down. In contrast, at a low specific energy input of 8 kJ/g TS, DD_{OUR} was found to be negative because ultrasound improved the biological activities of WAS (Rai et al., 2004).

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 2.8.

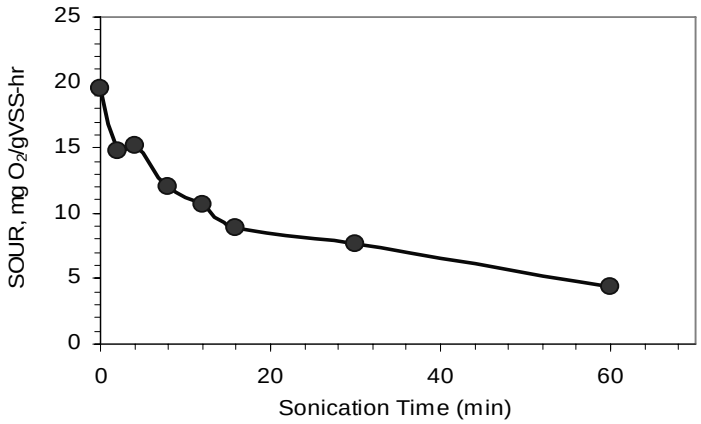


Figure 2.8 SOUR of WAS at different sonication durations (Khanal et al., 2006c)

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). In contrary, Zhang et al. (2008) found that sonicated sludge at 25 kHz frequency, 0.2 W/ml power density, and 30 min sonication duration could improve biological activity of sequential biological reactor (SBR) which resulted in increase of OUR up to 28%.

2.5.5 Factors affecting efficiency of ultrasonic disintegration

The efficiency of ultrasonic disintegration is affected by many factors. These factors can be broadly classified into three categories:

a. Sludge characteristics (solid content)
b. Sonication conditions (frequency, intersity and density, temperature, pH, frequency, amplitude and power input)
c. Design of ultrasonic components

a. Sludge characteristics

The sludge characteristics such as type of sludge (primary solids, waste activated sludge or animal manure, etc.), TS content, and particle size affect significantly to the sludge disintegration efficiency. Amongst them, TS content gives significant effects on sludge disintegration which was investigated by Dewil et al. (2006), Khanal et al. (2006c), Wang et al. (2005), Grönroos et al. (2005) and Onueche et al (2002).

The high efficiency of ultrasonic disintegration is observed at high dry solid (DS) concentration due to the fact that (1) more DS creates 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. Dewil et al. (2006) conducted a thorough study to evaluate the effect of DS concentration on SCOD release at different specific energy inputs. The results are presented in Figure 2.9.

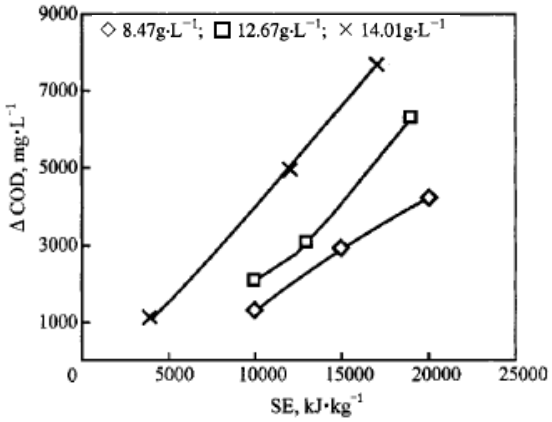


Figure 2.9 Effect of DS-concentration on the release of COD (Dewil et al., 2006)

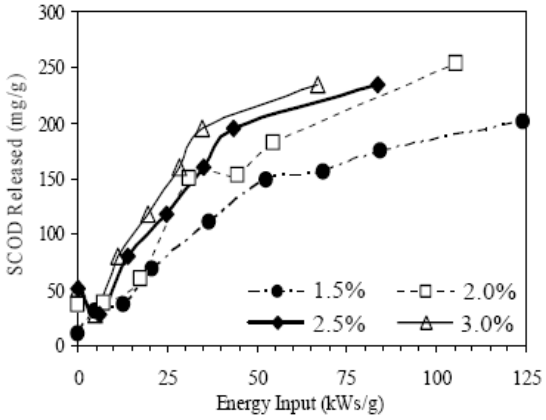


Figure 2.10 Sludge disintegration at different TS contents (Khanal et al., 2006c)

According to the graph, it clearly shows that the high ΔSCOD was corresponded to the high DS concentration at an equal Es. However, the author found also that the ΔSCOD decreased dramatically at higher DS concentration because it 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.

Khanal et al. (2006c) investigated the effect of TS content and energy input on SCOD release. The results are presented in Figure 2.10. SCOD release showed an increasing trend with increase in both TS and energy input. However, the release in SCOD slowed sown at an energy input of over 35 kWs/g TS for all TS contents. Based on linear regression analysis, SCOD releases were 1.6, 2.2, 2.5 and 3.2 mg/kWs at TS content 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%.

Wang et al. (2005) also observed the sludge disintegration at ultrasonic density of 1.44 W/mL for different TS contents and sonication durations. SCOD can reach 2785 and 2261 mg/L for the TS content of 1% and 0.5%, respectively for 10 min sonication duration. When the disintegration time is 30 min, the SOCD can reach 9019 and 3966 mg/L for the solid content of 1% and 0.5%, respectively.

Grönroos et al. (2005) and Onyeche et al. (2002) reported also that the sludge disintegration efficiency increased with increase of TS content of the sludge. Though, there is no clearly SCOD release data with respect to TS contents, the authors directly compared the methane production with different sludge TS contents.

According to the mentioned findings, overall, high TS content of WAS gave better sludge disintegration efficiency. However, it seemed to decrease when TS content was too high as it contributes to high viscosity.

b. 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.

Frequency

Sludge disintegration using ultrasound is well known to be effective due to combination of phenomena taken place at the same time. It is believed that hydro-mechanical shear forces are the main contributor to microbial cell break down due to the cavitation phenomenon. Cavitation phenomenon occurs whenever the bubbles reach a critical size and then violently collapse. The bubble radius is inversely proportional to the ultrasound frequency. Thus, hydro-mechanical shear force predominantly takes place at low frequency resulting in high sludge disintegration efficiency. However, sonochemical reactions were most significant at frequencies between 200 to 1000 kHz (Tiehm et al., 2001).

Tiehm et al. (2001) studied the effect of sludge disintegration at frequency range of 41-1068 kHz. The DD_{COD} were found to be 13.9, 3.6, 3.1 and 1.0%, respectively at frequencies of 41, 207, 360 and 1,068 kHz. The author expected the best disintegration efficiency with the lowest ultrasound frequency of 20 kHz.

Due to this finding, nearly all studies of ultrasonic pretreatment of WAS, afterward, were operated at the frequency of 20 kHz (Wang et al., 2005, Bougrier et al., 2005; Khanal et al., 2006a, b, c, Wang et al., 2006).

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 2.9.

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

As evident from Figure 2.10 (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/g TS for all TS contents. Based on linear regression analysis ($R^2 > 0.90$), 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%.

Sonication duration

As evident from equation 2.1, 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. (2006) 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., 2005; Khanal et al., 2006c). 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_{COD} 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_{COD} 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_{COD} of 9.2%, whereas an ultrasonic density of 0.1W/mL (sonicated for 30 min) yielded DD_{COD} of 7.3% (Zhang et al., 2007). Along the same line, the authors reported DD_{COD} 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.

pH and Temperature

pH and Temperature are equally important parameters affecting sludge disintegration efficiency. The SCOD release of sonicated sludge was gradually increased with increase in sludge pH. Figure 2.10 presents the effect of pH on SCOD release.

The details of the effect of alkaline addition will be given in section 2.4.2. The same author 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 with the relative magnitude of the effect of each parameter on ultrasonic disintegration in the order:

Sludge pH > sludge concentration > ultrasonic intensity > ultrasonic density

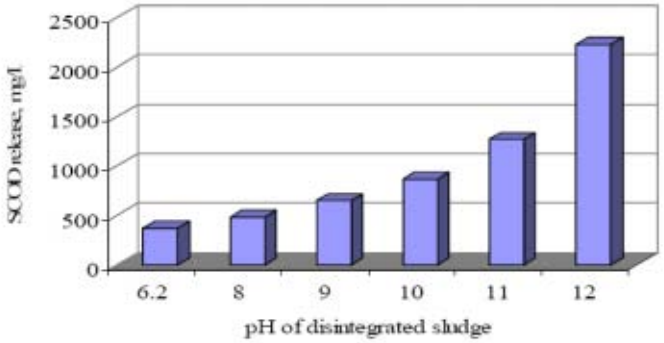


Figure 2.11 SCOD, at different pH within 30 min of disintegration time

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 early 2.4-fold during sonication for 60 min at an ultrasonic density of 0.33W/mL without emperature 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., 2005). There is a need to conduct a systematic study to elucidate the contribution of temperature on overall sludge disintegration.

2.5.6 Anaerobic digestibility of ultrasound pretreated sludge

Using ultrasound for sludge disintegration aims to enhance the VS destruction during digestion. The increased VS reduction eventually increases in methane production and less stabilized biosolid to dispose of. Thus, VS reduction and methane generation were used to investigate the anaerobic digester performance of sonicated sludge.

Wang et al. (1999a) 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 11^{th} 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 10^{th} 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^2 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.

Table 2.9 Organic solids solubilization at different sonication conditions

Type of sludge	Power input (W) Frequency (kHz)		Specific energy input (kJ/kg TS)	Ultrasonic density or intensity	Degree of solubilization			Reference
					SCOD release (mg/L)	DD_{COD} (%)	SCOD/COD (%)	
Waste activated sludge (TS: 3%)	Power input	1500	0 (Control)	1.1 W/mL	1,542	N/A	4.2	Khanal et al. (2006c)
			11,000		2,412		5.7	
			19,600		3,540		8.7	
	Frequency	20	28,200		4,824		12.3	
			34,600		5,846		13.7	
			66,800		7,022		16.2	
Waste activated sludge with nutrient removal (TS: 1%)	Power input	N/A	N/A	0 (Control)	410	N/A	N/A	Zhang et al. (2006)
				0.1 W/mL	1,050			
				0.2 W/mL	1,500			
	Frequency	25		0.5 W/mL	3,150			
				1.0 W/mL	4,500			
				1.5 W/mL	5,400			
Waste activated sludge (TS: N/A)	Power input	1500	N/A	0 (Control)	775	N/A	N/A	Mao et al. (2005)
				0.18 W/mL	950			
	Frequency	20		0.33 W/mL	1,200			
				0.52 W/mL	1,500			
Waste activated sludge (TS: 1.85%)	Power input	750	0 (Control)	N/A	N/A	N/A	5.8	Bougrier et al. (2005)
			660				10.5	
			1,355				16.1	
	Frequency	20	2,700				22.3	
			6,951				33.1	
Thickened waste activated sludge (TS: 2.45%)	Power input	N/A	0 (Control)	1.25 W/mL	1,300	N/A	N/A	Grönroos et al. (2005)
	Frequency	27	3,000		2,600			
			14,900		4,050			
Biological sludge from SBR (TS: 0.5%)	Power input	N/A	N/A	60W/cm^2			12	Wang et al. (2005)
				120W/cm^2			18	
	Frequency	20		230W/cm^2			30	
Waste activated sludge	Power input	500	8,000	N/A	N/A	4.2	N/A	Rai et al. (2004)
			24,000			8		

(TS: 0.48%)	Frequency	N/A	40,000			10		
			64,000			25		
Type of sludge	**Power input (W) Frequency (kHz)**		**Specific energy input (kJ/kg TS)**	**Ultrasonic density or intensity**	**Degree of solubilization**			**Reference**
					SCOD release (mg/L)	**DD_{COD} (%)**	**SCOD/COD (%)**	
Waste activated sludge (TS: 2%)	Power input	750	0 (control)	N/A	N/A	N/A	5.8	Waste activated sludge (TS: 2%)
			1,355				16.1	
			2,707				22.3	
	Frequency	20	6,951				33.1	
			14,547				41.6	
Waste activated sludge (TS: 2%)	Power input	N/A	5,000	$1.4W/cm^2$	N/A	5.5	N/A	Tiehm et al. (2001)
			10,000			10		
	Frequency	41	25,000			22		
			40,000			35		
			60,000			40		
	Power input	190	0	N/A	80	N/A	N/A	Navaneethan, (2007)
			1,900		1,360			
			3,800		1,760			
			7,600		3,600			
			15,200		7,200			
	Frequency	20	30,400		9,000			

Source: Modified from Navaneethan. (2007)

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.

A pilot scale study was performed by Kickel and Neis (2007) at frequency of 31 kHz and acoustic intensities range of 5-18 W/cm^2. WAS with TS content of 0.5-4% was sonicated immediately before feeding for 90 seconds. The authors found that the VS degradation rate of the sonicated biosolids at 16 days SRT increased by more than 30% compared to conventional digestion. At an SRT of 8 days, ultrasonic disintegration of WAS enhanced the degree of anaerobic degradation by more than 40%. However, the highest rate of VS degradation was obtained at the shortest SRT (4 days).

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/kg TS, respectively. The biogas yield clearly showed improvement with increase in specific energy inputs up to 6,951 kJ/kg TS. However, with further increase in energy input to 14,547 kJ/kg TS, 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 during anaerobic digestion of ultrasound pretreated WAS, either at the mesophilic or at the thermophilic temperature. 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_{COD} 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.

2.5.7 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. Thus, any further improvement in the dewaterability of sludge could result in a considerable savings in sludge disposal costs. Ultrasonic energy was reported useful to dewater digested sludge (Hogan et al., 2007; Chu et al., 2001; Sarabia et al., 2000). However, few studies were indicated that ultrasound inhibited sludge dewatering.

Sarabia et al. (2000) investigated a dewatering of particle suspension (TiO_2) using ultrasound at the frequency range of 10-20 kHz and power supplied of 10-100W (0.075 to 0.75 W/cm^2). The authors detected that the dewatering increased with power until a certain saturation value. There was no clearly mentioned the saturated value which probably related to the filtration system behavior. The dewaterability of digested sludge was improved to 1.64 ±0.32% compared to the control digester (Hogan et al., 2007).

Chu et al. (2001) thoroughly investigated the dewaterability of sonicated sludge at different ultrasonic densities. According to the CST data, sonication at 0.11 W/mL only slightly deteriorated the sludge's filterability (from 197 s for the original sludge to 218 s after 60 min treatment). However, the CST for 0.33 W/mL (60 min sonication) treated sludge had been significantly increased up to 490 s. Dewil et al. (2006) confirmed also that the rate of dewatering of sonicated sludge decreased as a function of specific energy supplied. The dewatering data expressed in CST is shown in Figure 2.11.

The average flocs size decreased with longer treatment times, thus leading to a reduced dewaterability due to the fact that (1) smaller flocs cause clogging of the cake; (2) offer an extended surface area, hence binding more surface water which is difficult to remove. More details of sonicated sludge dewaterability refer to Yin et al. (2004).

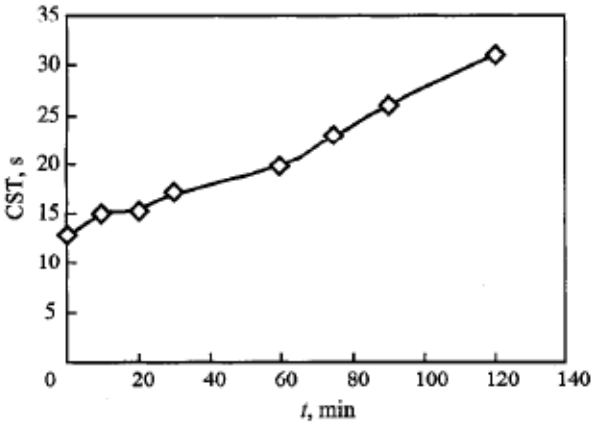


Figure 2.12 CST-values of sludge after given durations of ultrasonic sludge treatment (Dewil et al., 1006)

2.6 Chemical Pretreatment

Chemical pretreatment of WAS is not a new method for sludge disintegration. It is well known to be the effective technique for breaking down the microbial cell wall and membrane. The additives used for chemical pretreatment are acidic reagent, basic reagent and ozone. Amongst them, basic reagent was commonly used for sludge disintegration, known as alkaline treatment, due to its high efficiency. Alkaline treatment is an easy and effective technique which can be operated at ambient temperature. Moreover, it was found to be a cost effective of sludge solubilization as it gives higher release of organic substances into aqueous phase at relatively low dosage level.

Alkaline sludge treatment depends on dissolution or destruction of flocs structure and cell wall by hydroxyl radical. An extreme of high pH causes protein to loose their natural shapes, saponification of lipid and hydrolysis of RNA. Strong alkali solubilizes gels not only because of chemical degradation but also ionization of the hydroxyl groups, which

leads to extensive swelling and subsequent solubilization. After the destruction of EPS and gels, cells are exposed to environment with extremes of pH thereby cannot keep the appropriate turgor pressure. Due to aforementioned reason and saponification of lipid, cells are disrupted and the inner matters are released. Therefore, alkaline treatment can solubilize sludge and release intracellular (Li et al., 2007; Neyen et al., 2004). Factors affecting efficiency of alkaline treatment, anaerobic digestibility of alkaline pretreated sludge and effect of alkaline on sludge dewaterability are given in the follow section:

2.6.1 Factors affecting efficiency of alkaline treatment

Alkaline nature, alkaline dose and pH, temperature, treatment time and sludge characteristics are some of the important parameters that affect the alkaline pretreatment efficiency.

Alkaline nature

Alkaline nature plays a significant effect to the sludge disintegration efficiency due to its fast solubilization into aqueous solution. The alkaline agents which can be used are NaOH, KOH, $Ca(OH)_2$, $Ma(OH)_2$, etc. Almost every published paper had reported that monobasic agents are the most preference because it solutes very fast into the sludge. As a result, sludge was significantly disintegrated. Alkaline treatment of WAS using NaOH were thoroughly investigated by many researchers: Kim et al. (2002), Lin et al. (1997), Lin et al. (1999) and Chiu et al. (1997). However, few studies were investigated the effect of different alkaline agents in WAS disintegration (Kim et al., 2003 and Li et al., 2007). Moreover, lime agent was also studied by Vlyssides and Karlis, (2004), and Torres and Lloréns. (2007) due to its cheap cost compared to NaOH.

Kim et al. (2003) investigated the efficiency of alkaline pretreatment on WAS solubilization with various alkaline agents: NaOH, KOH, $Mg(OH)_2$ and $Ca(OH)_2$. The experiment was performed at a constant pH of 12. The authors found that monobasic agents, particularly NaOH, resulted in higher solubilization percentages than dibasic agents either at ambient or thermal temperature (Figure 2.12). For example, SCOD release from adding NaOH was 39.8% and 51.8% while 15.3% and 17.1% of that from adding $Ca(OH)_2$ at ambient and at 121 ^oC temperature, respectively.

Li et al. (2007) observed the behavior of WAS solubilization using NaOH and $Ca(OH)_2$ with various alkaline doses. The authors concluded that NaOH was more effective than $Ca(OH)_2$ for sludge solubilization due to the reason that bivalent cation (Ca^{2+}) is the key matter connecting cell with EPS. Hence, calcium cation helps the dissolved organic polymers to re-flocculate the fragments produced by alkaline treatment.

Alkaline dose and pH

Alkaline dose is the amount of alkaline agent added per unit volume of sludge. It normally express in mg/L, meq/L or mol/L. Alkaline dose has a very close correlation to pH. As long as the alkaline dose increases, it, eventually, increases in pH. Thus, these two affecting factors were used to investigate the efficiency of sludge disintegration.

Kim et al. (2002) performed the alkaline pretreatment of WAS in the pH range of 9, 10, 11 and 12 using 2M NaOH solution at constant treatment time. The results showed that the pH of 11 is the critical value for the optimum of sludge disintegration. Vlyssides and Karlis,

(2004) conducted a thermal alkaline solubilization of WAS at various pH of 8, 9, 10 and 11. The authors found that at pH ≥ 10 and T ≥ 80 ^{o}C, SCOD increased significantly until the 8th hour of hydrolysis, where about 80% of the solubilization had been achieved. However, at pH =10 and T = 90 ^{o}C and after 10 h of hydrolysis, the solubilizaation rate was still increased significantly which is difficult to get a clear picture of which pH is the optimum one as temperature was involved.

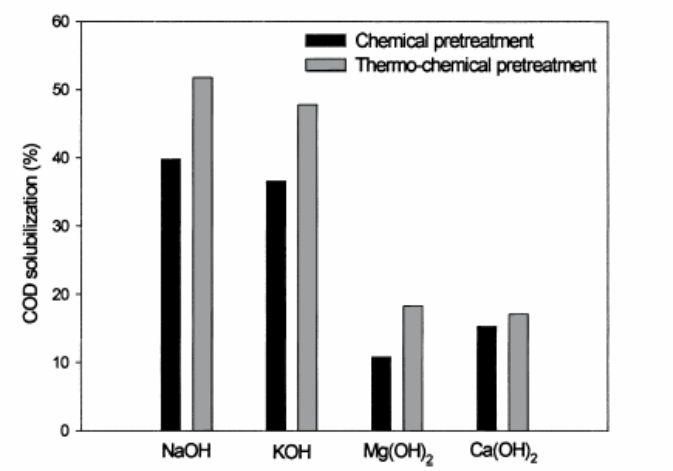


Figure 2.13 Effects of various alkali agents on COD solubilization (Kim et al., 2003)

In contrary, Kim et al. (2003) investigated the efficiency of WAS pretreatment to enhance anaerobic digestion using NaOH at concentration ranging from 0 to 21 g/L. The authors found that COD solubilization increased as the dose of NaOH increased, reaching 43.5% when 7 g/L NaOH were added. From 7 to 21 g/L NaOH, a slower rate of COD solubilization increase was observed. Li et al. (2007) studied the effects of sludge solubilization using NaOH dose of 0.05-1 mol/L and $Ca(OH)_2$ dose of 0.02-0.5 mol/L at temperature range of 0-40 ^{o}C. It was found that NaOH dose of 0.05 mol/L (0.16g/g DS) was the most efficient in sludge disintegration. However, $Ca(OH)_2$ dose of 0.02 mol/L gave higher SCOD release compared to higher doses.

Similarly, Lin et al. (1999) investigated the COD change in particle and soluble portion of WAS using NaOH of 20 and 40 meq/L NaOH. With increased NaOH concentration, the total COD remained constant, but the particulate portion showed a decreasing COD with a corresponding increasing COD in the soluble portion. When the NaOH was increased from 20 to 40 meq/L, the COD in the soluble portion increased from 830 to 1190 mg/L (SCOD of untreated sample was 70 mg/L).

Till now, there is no a clear judgment whether pH or alkaline dose should be considered for the efficient sludge solubilization. So far, researches have been done depending on individual judgment. However, alkaline dose is likely to be the most appropriate factor compared to pH because sludge solubilization depends not only alkaline natures but also sludge characteristics.

Temperature

Operating temperature is one of the important factors affecting sludge disintegration during alkaline treatment. High temperature helps chemical reaction faster and leads to thermal phenomena when temperature increases higher. It, eventually, give a high effects to sludge

pretreatment by the combination of both chemical and high temperature phenomena known as thermochemical. Few studies had been focused on WAS disintegration by controlling temperature at ambient air (Lin et al., 1999; 1997), at temperature range of 0-40 ^{o}C (Li et al., 2007), and at high range temperature (Kim et al., 2003; Vlyssides and Karlis, 2004).

Kim et al. (2003) investigated WAS solubilization using alkali agents at ambient and thermal temperature. The author found that thermochemical was more efficient than chemical alone as result presented in Figure 2.13. At same alkaline dose of 7 mg/L, SCOD release increased by 43.5% and 85.4% at ambient temperature and 121 ^{o}C, respectively.

Treatment time

Treatment time is an equally important factor on sludge solubilization. Treatment time is becoming more concerned because it correlates with capital cost of the treatment plant. In the full scale application, when long treatment time is needed it eventually requires large reactor volume. Thus, the cost of reactor construction will be automatically high that leads to uneconomically viable. Kim et al. (2002b) investigated the pretreatment times of WAS solubilization. The experiments were carried out at duration of 10, 20, 30 and 40 min and the results showed that 10 min was identified as the optimal pretreatment time. Li et al. (2007) also studied the effects of treatment duration on sludge disintegration. The authors found that in first 30 min, the solubilization quantity was 60-71% of total solubilization organic matters in 24 h. Thus, 30 min was the most efficient treatment duration. Figure 2.15 represented the variation of sludge SCOD with duration time during NaOH treatment. Similarly, Vlyssides and Karlis, (2004) observed the effect of treatment times of WAS hydrolysis after adding alkali agent.

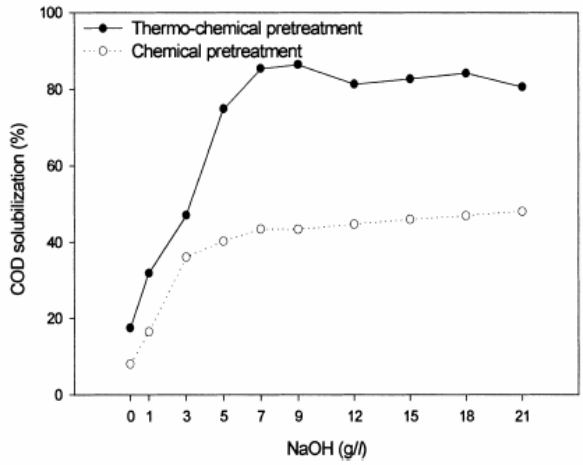


Figure 2.14 Effects of temperature on COD solubilization (Kim et al., 2003)

Sludge characteristics

Sludge characteristics, particularly TS content, play a role in sludge disintegration efficiency. A big portion of COD in WAS is within the particulate form which is the target of alkaline pretreatment to break down in order to release the intracellular into the aqueous phase. So far, there is no a thorough study had been focused on this parameter. However, Lin et al. (1997) observed the anaerobic digester performances of two different sludge TS contents (TS = 1 and 2%). The authors found that organic removal increased as the concentration of either NaOH or sludge solids increased. Moreover, it was concluded that

increasing the concentration of sludge solids was more effective for organic removal than increasing the concentration of NaOH. These findings were not sufficient enough to evaluate the optimum TS content for alkaline WAS disintegration; hence further studies on sludge characteristics are needed.

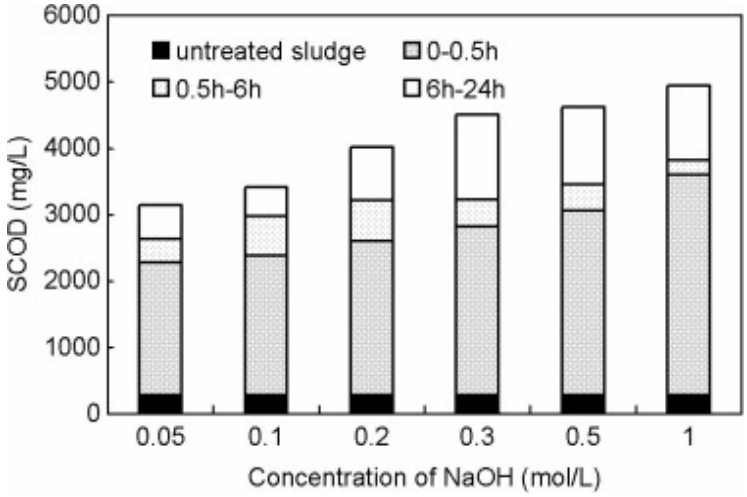


Figure 2.15 Variation of sludge SCOD with duration times (Li et al., 2007)

2.6.2 Anaerobic digestibility of alkaline pretreated sludge

The aims of anaerobic of alkaline pretreated sludge are exactly the same as anaerobic of ultrasonic pretreated sludge (section 2.4.1.6).

Lin et al. (1999) studied the effects of chemical pretreatment of WAS using BMP test in term of VS and COD removal, and methane production. The authors investigated also the different efficiencies of unfiltered sludge, supernatant and particulate. The results showed that the methane gas production obtained at 30 days digestion time for sample treated with 20 and 40 meq/L NaOH was higher than that for sludge without pretreatment (control). With 40 meq/L NaOH, the observed gas production, VS removal and COD removal increased by 34, 41 and 30% over the control, respectively.

Vlyssides and Karlis, (2004) investigated the SCOD destruction and biogas production from anaerobic digester using thermal alkaline pretreated WAS. WAS was pretreated with NaOH for 10 h with different pH and temperatures before BMP experiment was started. According to the obtained results, for 50 ^{o}C temperature and pH 8, 90 % of the SCOD was decreased at thermophilic anaerobic digestion, while for 90 ^{o}C temperature and pH 11 only 80% of the SCOD was decreased. Moreover, the gas production rate was found to be 0.07 l CH_4/g VSS and 0.21 l CH_4/g VSS for those two conditions, respectively.

Lin et al. (1997) conducted a laboratory scale experiment of alkaline pretreated sludge at four different HRTs (20, 13, 10 and 7.5 days). Four reactors were fed with unpretreated sludge (A), 20 meq/L and TS of 1% (B), 40 meq/L and TS of 1% (C), and 20 meq/L and TS of 2% (D). Amongst the HRTs operated, 7.5 days was the most effective HRT which gave 72, 76 and 86% of VS removal, COD removal, and gas production increase from reactor C comparing to reactor A, respectively.

Overall, alkaline pretreated sludge improves anaerobic digestion performances but many factors have to be taken into consideration such as pH of pretreated sludge, alkaline nature,

alkaline dosage, sludge characteristics, anaerobic operating conditions (thermophilic, mesophilic, SRT). Alkaline sludge disintegration for anaerobic digestion can be used up to some extend, beyond that it would not be useful because of the toxicity of cations and/or pH to methanogenic bacteria.

2.6.3 Effect of alkaline treatment on sludge dewaterability

The purpose of sludge dewatering using chemical pretreatment is exactly the same as ultrasonic pretreatment, reduce cost of sludge disposal. A recent study (Li et al., 2007) was investigated the effects of alkaline pretreatment on sludge dewaterability. The results indicated that sludge dewatering ability deteriorated at first and then improved gradually when NaOH dose was increased from 0.05 to 0.5 mol/L. When NaOH dose was lower than 0.1 mol/L, sludge dewatering ability deteriorated obviously because of disruption of sludge flocs and cells which eventually increased the hydrophilic organic polymers and vicinal water content. However, at NaOH dose higher than 0.2 mol/L, sludge dewatering ability was improved compared with the effect of low dose NaOH treatment, but still worse than untreated sludge. At the same time of NaOH investigation, $Ca(OH)_2$ was also investigated. It was found that sludge dewatering ability using $Ca(OH)_2$ was much more efficient than NaOH but the weight of sludge cake increased by 24.3% at 0.05 mol/L (26% decrease for NaOH treatment with the same dose). With the increase in sludge cake weight, there will be a proportional increase of sludge disposal cost.

Though alkaline pretreatment does not improve dewatering ability of pretreated sludge, it could improve anaerobic digested sludge dewaterability (Lin et al., 1997). As long as anaerobic digestion performs well, particles of all sizes are destroyed, but there is a preferential removal of particles of small sizes. It consequently losses of specific surface area and therefore, improves in dewateribility.

2.7 Chemical-ultrasonic Pretreatment

Chemical-ultrasonic pretreatment is the combination of chemical followed by ultrasonic treatment. Overall processes of this field are exactly the same as individual chemical and ultrasonic treatment. All the affecting factors of both treatments have to be taken into consideration for effective sludge disintegration. Sludge disintegration evaluation is also the same as that of ultrasonic disintegration (SCOD, NH_3, particle size distribution, etc.). The main aim of using chemical-ultrasonic pretreatment is to increase the efficiency of sludge disintegration which eventually improves sludge stabilization and increases biogas production prior anaerobic digestion.

The research on this field is still very limited. Chiu et al. (1997) investigated the hydrolysis rate of alkaline, ultrasonic, chemical-ultrasonic and simultaneous ultrasonic and alkaline pretreatment on WAS. The experiment was carried out with WAS of 1% TS contend at ambient temperature. There were three set of experiments; (1) pretreated with 40 meq/L NaOH for 24 h, (2) pretreated with 40 meq/L NaOH for 24 h followed by ultrasonic vibration for 24 sec/mL and (3) simultaneous ultrasonic (14.4 sec/mL) applied to samples dosed with 40 meq/L NaOH. The authors found that amongst the three pretreatment schemes, the initial hydrolysis rate of the simultaneous ultrasonic and alkaline treatment was the highest being 211.9 mg/L/min. The pretreatment of alkaline followed by ultrasonic was found to be more effective in SCOD release and soluble organic nitrogen compared to alkaline pretreatment alone but to be closed to simultaneous one. However, it was

concluded that the process of simultaneous ultrasonic and alkaline treatment could shorten the WAS pretreatment time and resulted in a prolific production of SCOD.

The combination of chemical and ultrasonic is still unclear on its effectiveness in the field of WAS disintegration as well as in the anaerobic digestion. The studies should be focused more on sludge disintegration efficiency with various aspects (ultrasonic application and chemical operating condition) and subsequent anaerobic digestion in term of biogas production and biosolids to be disposed of.

2.8 Further research trend

Ultrasonic technology is recognized to be an effective sludge disintegration technique. The anaerobic stabilization of sonicated sludge eventually improves which result in high bioenergy recovery and less amount of sludge to be disposed of. However, ultrasonic, particularly in full scale application, consumes a large amount of energy which subjects to high pretreatment cost. Therefore, ultrasonic of WAS at low power input should be investigated for effective subsequent anaerobic digestion. Moreover, the combination of chemical and ultrasonic is another attractive technique on sludge disintegration. At a relatively low chemical dose and low energy input, WAS could be effectively disintegrated in a short period of time. As a result, it will automatically reduce the reactor volume which reflects to low capital cost of the treatment. Nevertheless, the performances of anaerobic digester using chemical-ultrasonic pretreated sludge should be investigated in order to get a clear picture of its efficiency.

Chapter 3

Methodology

3.1 Experimental Plan

The study was carried out with ultrasonic, chemical and chemical-ultrasonic pretreatments of WAS from a local domestic wastewater treatment plant. The optimum conditions of ultrasonic, chemical and chemical-ultrasonic pretreatment were investigated before feeding to anaerobic digester. The research work was divided into three steps; (i) optimization of the pretreatment with respect to various conditions (holding time, power input and alkali dose), (ii) operation of anaerobic digester with pretreated and fresh WAS (control) at three different SRTs and (iii) conducting an economic analysis of all pretreatment options by comparing the income from bioenergy recovery and landfill cost reduction to the expense of energy consumption and chemical cost. The outline of research plan is presented in Figure 3.1.

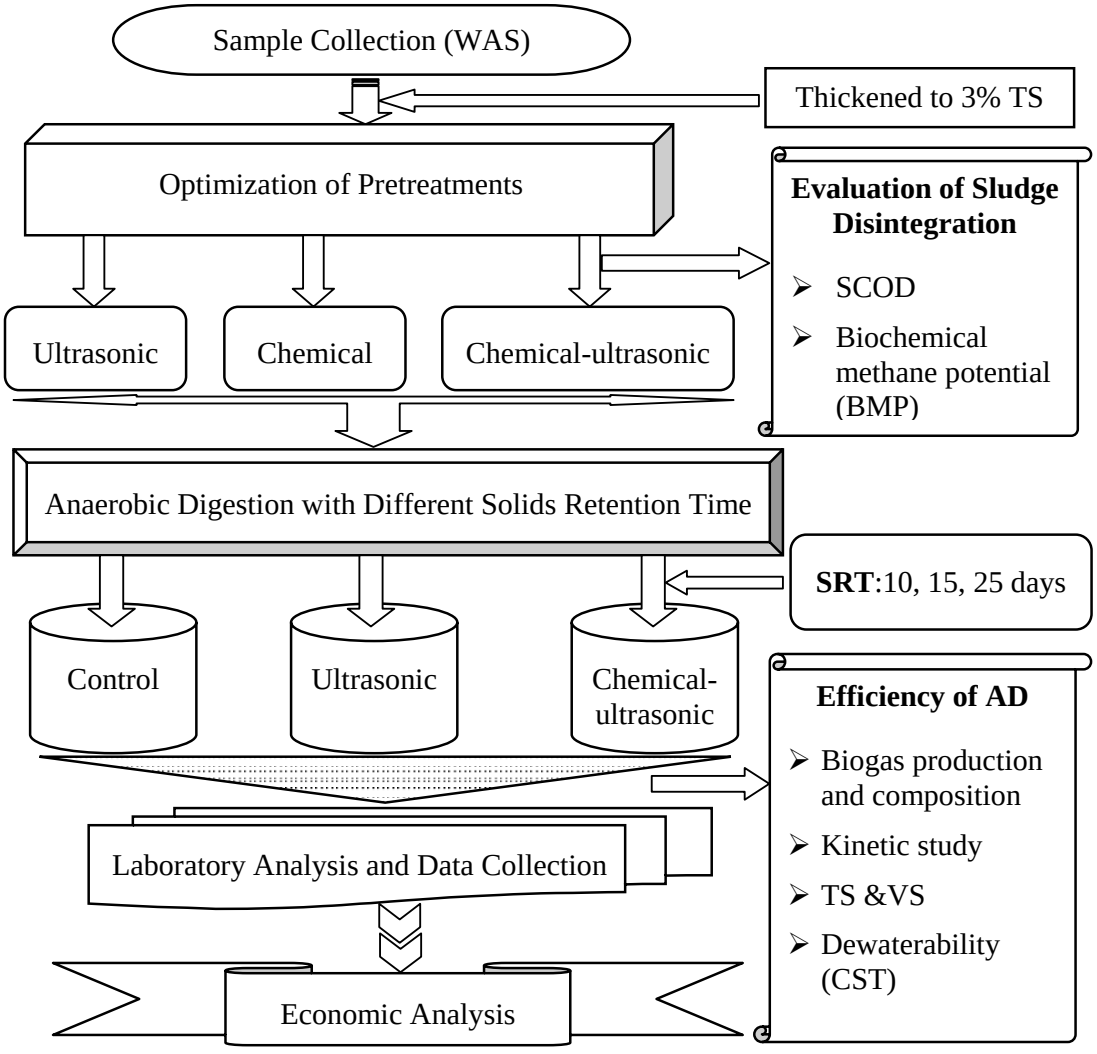


Figure 3.1 Outline of research plan

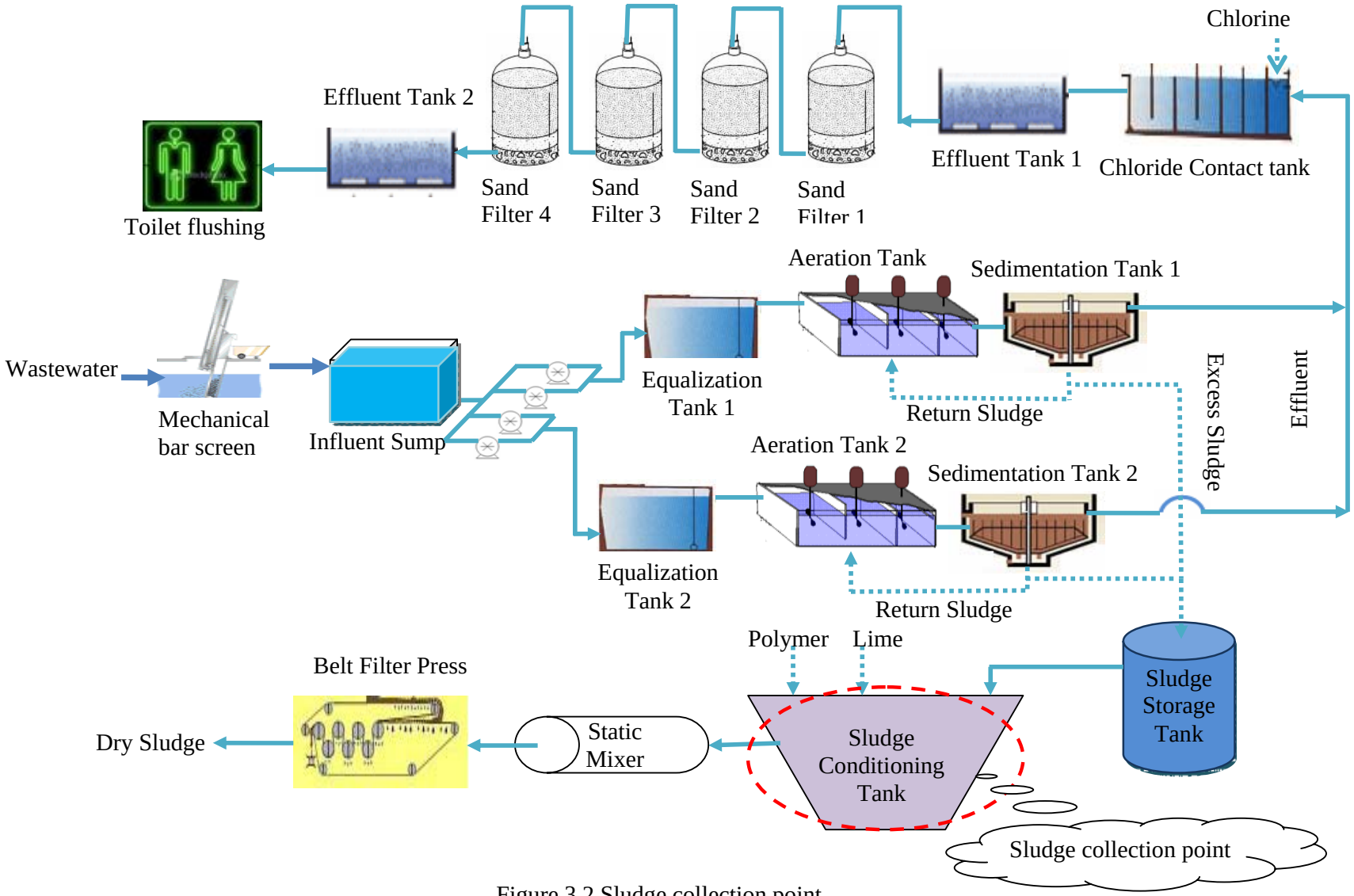


Figure 3.2 Sludge collection point

3.2 Waste Activated Sludge Sample

The WAS from the secondary treatment was selected as a representative sample. The secondary sludge was collected from the sludge conditioning tank (***prior to polymer and lime addition***) from Future Park Rangsit (Thailand) domestic wastewater treatment plant. The location of sample collection is shown in Figure 3.2. After collection, the sludge was stored in a cold room at 4 ^{o}C at AIT ambient laboratory prior to use to prevent biodegradation. Normally, WAS from secondary sedimentation tank has a low solid concentration. Therefore, the sludge was centrifuged at 4500 rpm for 4 min to increase the total solids (TS) content by 3%, and SCOD, TS, VS, and pH were analyzed. The centrifuged sludge was used to examine the optimum condition of ultrasonic, chemical, and chemical-ultrasonic pretreatment and subsequent anaerobic digesters.

3.3 Ultrasonic Pretreatment

Ultrasonic pretreatment of this research is a continuous study from previous research work (Navaneethan, 2007). The sonication unit and sonication chamber were exactly the same as previous work. However, the selection of horn and horn immersion depth were based on previous finding, and few changes in operating condition were made. The details of ultrasonic equipments, ultrasonic operation and optimization of ultrasonic pretreatment are given in this section:

3.3.1 Ultrasonic equipment

The WAS was sonicated using sonic ultrasound unit (VC750 model, Newtown, CT, USA). The ultrasound unit has a maximum power output of 750W 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) from which the large horn was selected for ultrasonic pretreatment as it gave higher efficiency compared to small and medium horn (Navaneethan, 2007). The power input can be set independently from 40 - 200 W. The amplitude can be also set independently from 20-100%. A sonication chamber with the total volume of 600 ml fabricated at Environmental Engineering Laboratory of AIT was used. The ultrasonic equipment is displayed in Figure 3.3 and the pictorial view and designed details of stainless steel chamber are shown in Figure 3.4 and Figure 3.5, respectively.

3.3.2 Ultrasonic operation

Ultrasonic experiment was carryout at the ambient temperature of 30^{o}C. 100 mL of thicken waste activated sludge (TWAS) with 3% TS content was used to run the sonicator with respect to variation of power input and sonication duration. The operating condition of sonication experiment is shown in Table 3.1. After every run, SCOD and pH were analyzed immediately.

3.3.3 Optimization of sonication condition

The optimum condition of ultrasonic depends on many factors. However, in this research mainly sonication duration and power input were investigated. Ultrasound was operated at the power input of 50, 100, 150 and 190 W with respect to various operation duration of 0, 30, 60 120 seconds. SCOD released after sonication was recorded and used as a key

parameter to judge the optimum condition with respect to ultrasonic density (UD) and specific energy (Es) input.

Figure 3.3 Ultrasonic equipment

Figure 3.4 Stainless steel chambers

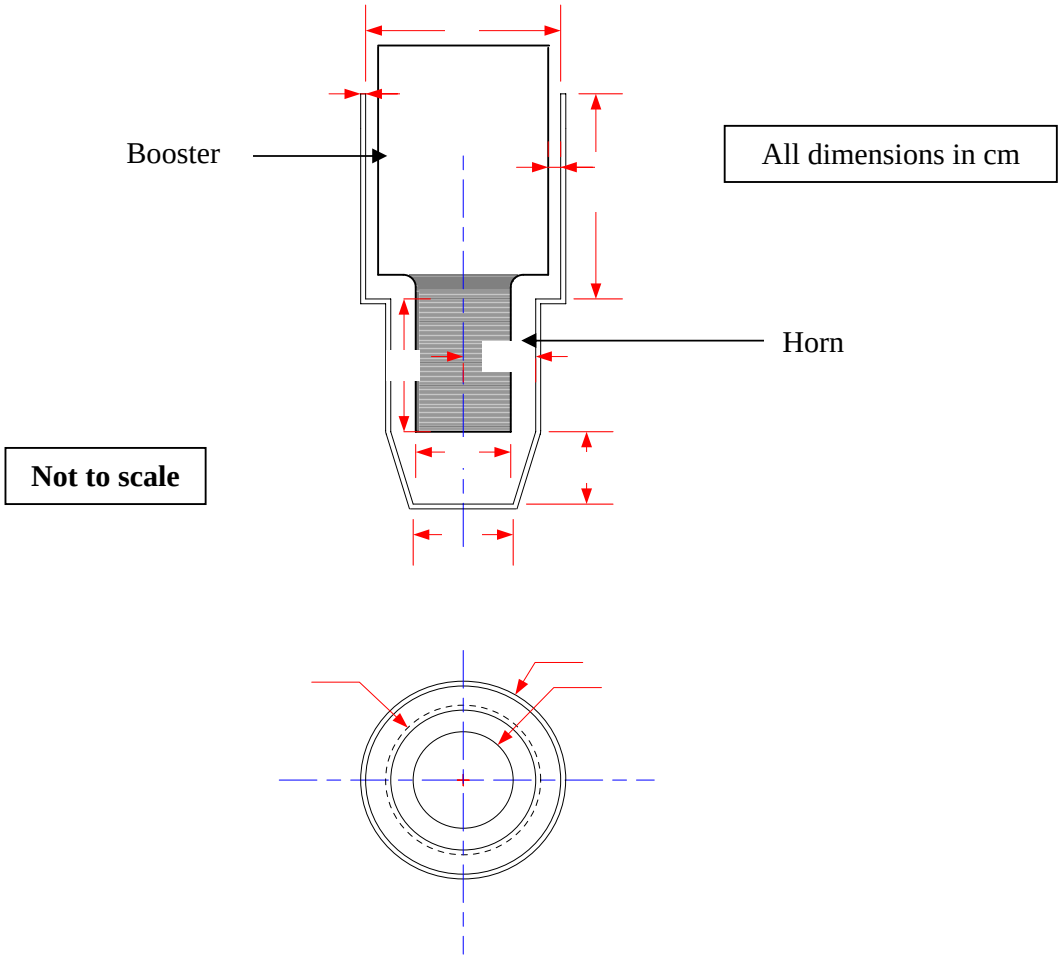


Figure 3.5 Sonication chamber design with large horn

The Es and UD can be calculated by the following equations:

$$Es = \frac{P}{V \,.\, TS}\ x\ t \qquad \text{Eq. 3.1}$$

Where,

Es: Specific energy (KJ/kg TS)
P : Power input (kW)
t : Sonication duration (s)
V : Volume of sludge used for sonication (L)
TS: Total solids (kg/L)

$$UD_{avg} = \frac{P_{avg}}{V} \qquad \text{Eq. 3.2}$$

Where,

UD_{avg} : Ultrasonic density (W/mL)
P_{avg} : Power input (W)
V : Volume of sludge used for sonication (mL)

The optimum condition of ultrasonic was selected according to the high SCOD released at a low specific energy input. To get this, the graph of specific energy input and SCOD released was needed to plot with four different UDs.

3.4 Chemical Pretreatment

Chemical pretreatment was carried out at ambient temperature using NaOH as basic agent. NaOH in pallet form (commercial grade) was grinded into power form and weighed to the desired weight. The following section presents the details of chemical pretreatment step.

3.4.1 Chemical pretreatment set-up

Chemical pretreatment was operated in batch with different holding times. The centrifuged sludge (3% TS) of 350 mL was put into beaker and NaOH was added at different doses. Complete mixing was provided in order to prevent sludge settling and dispersed the alkali added using mechanical mixer. The details of operating condition and procedure of chemical pretreatment are presented in Table 3.1 and Figure 3.6, respectively.

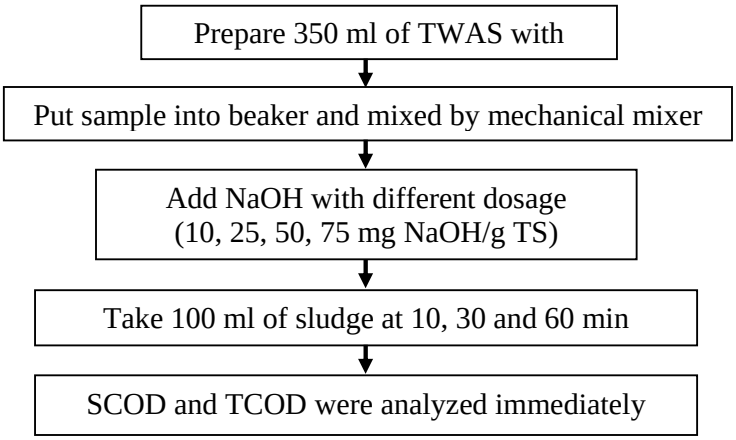


Figure 3.6 Procedure of chemical pretreatment

Sedi

3.4.2 Optimization of chemical pretreatment

Solubilization of WAS by alkaline addition depends mainly on the amount of basic agent utilized and the holding time. These two parameters were investigated to find out the optimum condition in term of SOCD release and alkaline dose. SCOD was examined and used as an indicator for sludge disintegration optimization. To achieve this, the graph of SCOD released of different dosage versus holding times was plotted. Finally, the optimum value of alkaline dose and holding time were derived.

3.5 Chemical-ultrasonic Pretreatment

Chemical-ultrasonic is the combination of chemical and ultrasonic processes. The sludge sample was pretreated with alkaline followed by ultrasound at ambient temperature. NaOH (commercial grade) was used as alkaline agent and ultrasound unit was exactly the same as mentioned in section 3.3.1. The experimental set-up and optimization of chemical-ultrasonic pretreatment are described as follow:

3.5.1 Chemical-ultrasonic set-up

The treatment was divided into two steps. First, sample was treated with alkaline solution and then with ultrasound. The experiment was carried out at alkaline dose of 10, 15, 20 and 25 mg/g TS with a constant holding time of 5 min. The ultrasound was operated at power input of 190 W and sonication duration of 30, 60 and 120 min. The sample volume of 130 mL was put into a beaker and mixed (magnetic stirrer) with addition of alkaline solution to meet the desired alkaline doses. After 6 min mixing, sample was immediately sonicated with the details operating condition shown in Table 3.1.

Table 3.1 Operating condition of chemical-ultrasonic pretreatment

Parameters	Ultrasonic	Chemical	Chemical-ultrasonic
Sludge volume	100 ml	350 ml	130 ml
Duration	30, 60, 120s	5, 10, 30, and 60 min	Optimum holding time of chemical + variable sonication times
NaOH dose	-	10, 25, 50 and 75 mg NaOH/g TS	10, 15, 20 and 25 mg NaOH/g TS
Frequency	20 kHz	-	20 kHz
Probe immersed	2 cm	-	2 cm
Power input	50, 100, 150 and 190 W	-	190 W

3.5.2 Optimization of chemical-ultrasonic pretreatment

The optimization condition of chemical-ultrasonic was quite similar to ultrasonic pretreatment. After completing the pretreatment processes, SCOD was measured and used as an indicator to find out the optimum condition. The optimum condition was selected based on less chemical consumption and low energy input with respect to high SCOD released. A graph of SCOD released with respect to different chemical doses was plotted against specific energy input in order to investigate the optimum point of the pretreatment.

3.6 Evaluation of Sludge Disintegration Efficiency

Evaluation of sludge disintegration efficiency based on COD solubilization into aqueous phase. SCOD and total COD (TCOD) of untreated sludge were analyzed before pretreatment and SCOD of pretreated sludge was also analyzed. The increment of SCOD was computed in percentage increase compared to TCOD which can be calculated by equation 3.3. The comparison of % SCOD before and after the run was made to clarify the high efficiency amongst pretreatments.

$$\%\ SCOD_{soluble} = \frac{SCOD_{after\ pre.}}{TCOD} x\ 100 \qquad \text{Eq. 3.3}$$

3.7 Anaerobic Digestion

As mention earlier, the research was carried out at AIT based on laboratory scale experiment. The evaluation of the performance of anaerobic digesters was observed based on biogas production and composition, total and individual VFA, and TS and VS removal. Moreover, the dewaterability of the digested sludge was also examined after steady state was believed to reach. The experimental set-up, the procedure of digester startup, and the operating conditions of the digester for the whole study are given in detail in this section.

3.7.1 Experimental set-up

Three transparent acrylic cylinders of 15 cm internal diameter and 30 cm heights with the total volume of 5.3 L were used as anaerobic digesters. 3 L of the total volume of each reactor was fed by different sludge. Mechanical mixer was provided to achieve completely mixing. The reactors consist of three ports equipped with valve which are used for feeding, withdrawing, and biogas collection. The biogas generated was collected in 3 L sampling biogas bag (SKC sampling bag). The digester design drawing is shown in Figure 3.8.

Due to the small working volume of the digester, semi-continuous mode was performed for feeding and decanting the pretreated sludge and digested sludge, respectively. Three different types of feed sludge (100% WAS, 50% sonicated sludge and 50% WAS, 50% chemical-sonicated sludge and 50% WAS) were fed to the reactors namely control (Do), ultrasonic (Du) and chemical-ultrasonic (Dcu), respectively. All reactors were sealed in order to make them air-tight and placed in a hot water bath maintaining the temperature at 37±1 ^{o}C. The temperature was controlled by temperature controller and the hot water was circulated using circulation pump. Experimental set up drawing is illustrated in Figure 3.7.

3.7.2 Anaerobic digester startup

The anaerobic digested sewage sludge collected from a local full scale anaerobic digester was initially seeded to the reactors as seed sludge in order to reduce the start up time. After inoculation, head space of the reactors was purged with nitrogen gas to take out the air inside the reactors. Mechanical mixing was provided, and temperature was increased immediately up to 37 ^{o}C and maintained the mesophilic condition using hot water bath. The anaerobic digesters performance was monitored regularly in term of pH, alkalinity, and VFA during the startup period. The fresh TWAS was fed at least after the first 5 days of operation and the amount of feeding should not excess 20% of the design loading capacity for volatile solids of the SRT of 25 days. After 15 to 20 days of light feeding, the loading was gradually increased to achieve normal design loading in 60 to 70 days

afterward. Sludge was fed two times a day in equal interval (12 AM and 12PM). Biogas production and composition, pH, alkalinity, and VFA were analyzed and kept monitoring until the steady state is believed to achieve. The steady state is reached when the collected data do not vary more than 5%.

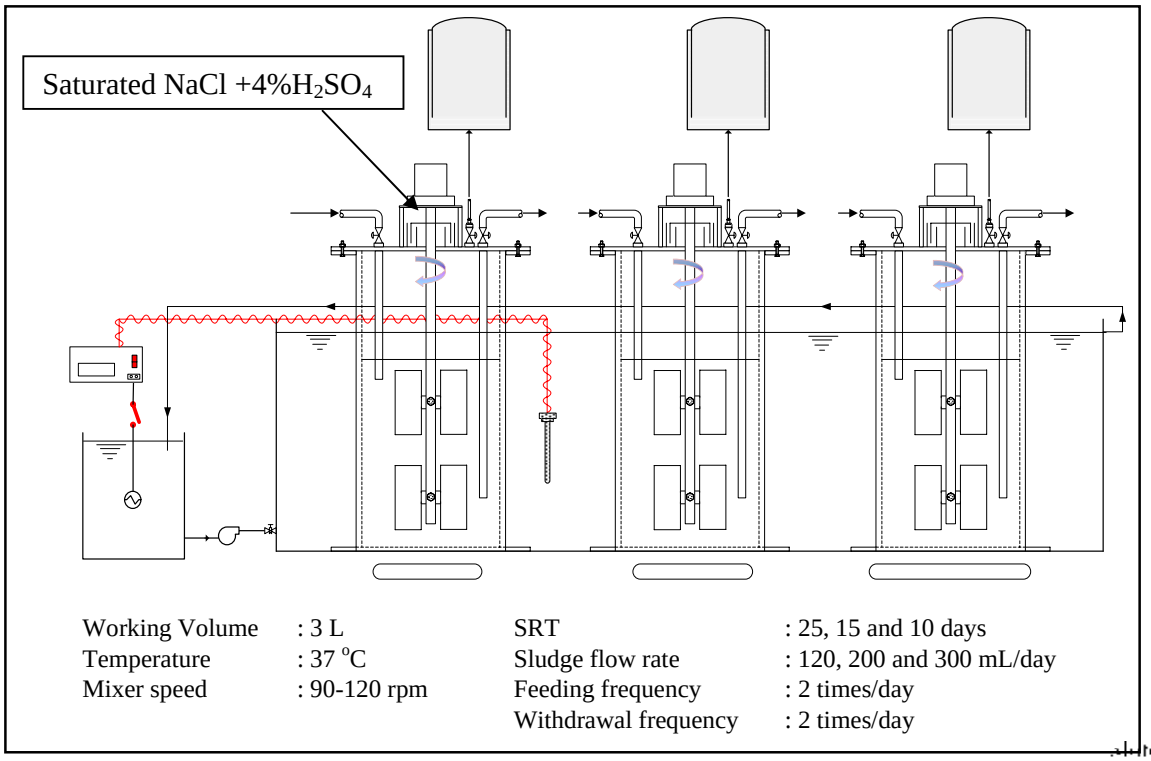


Figure 3.7 Experimental setup for anaerobic digesters

3.7.3 Experimental operation

After steady state was reached, the bioreactors were operated for a minimum of two weeks to collect enough steady state data. The three reactors were fed by three different pretreated sludge, as mentioned earlier, with respect to three SRTs. Feeding and withdrawal were performed manually in a semi-continuous mode. The reactors were fed two times a day and withdrawn immediately before each feeding. The biogas generated was collected into a sampling biogas bag and used for biogas analysis. Continuous mixing and temperature controlling were continued monitoring for the whole experiment. The digesters were operated at SRT of 25, 15 and 10 days. The longer SRT (25 days) was initially conducted followed by 15 and 10 days, respectively. Normally, the amount of feeding rate is equal to decanting rate in order to maintain the constant working volume. The feeding, decanting and solid loading rate (SLR) of different sludge types are shown in Table 3.2.

During the run, biogas production and composition, pH, total and individual VFA, TS, and VS were analyzed in three times per week. The dewaterability of digested biosolid was also examined three times per week after steady state (Table 3.3)

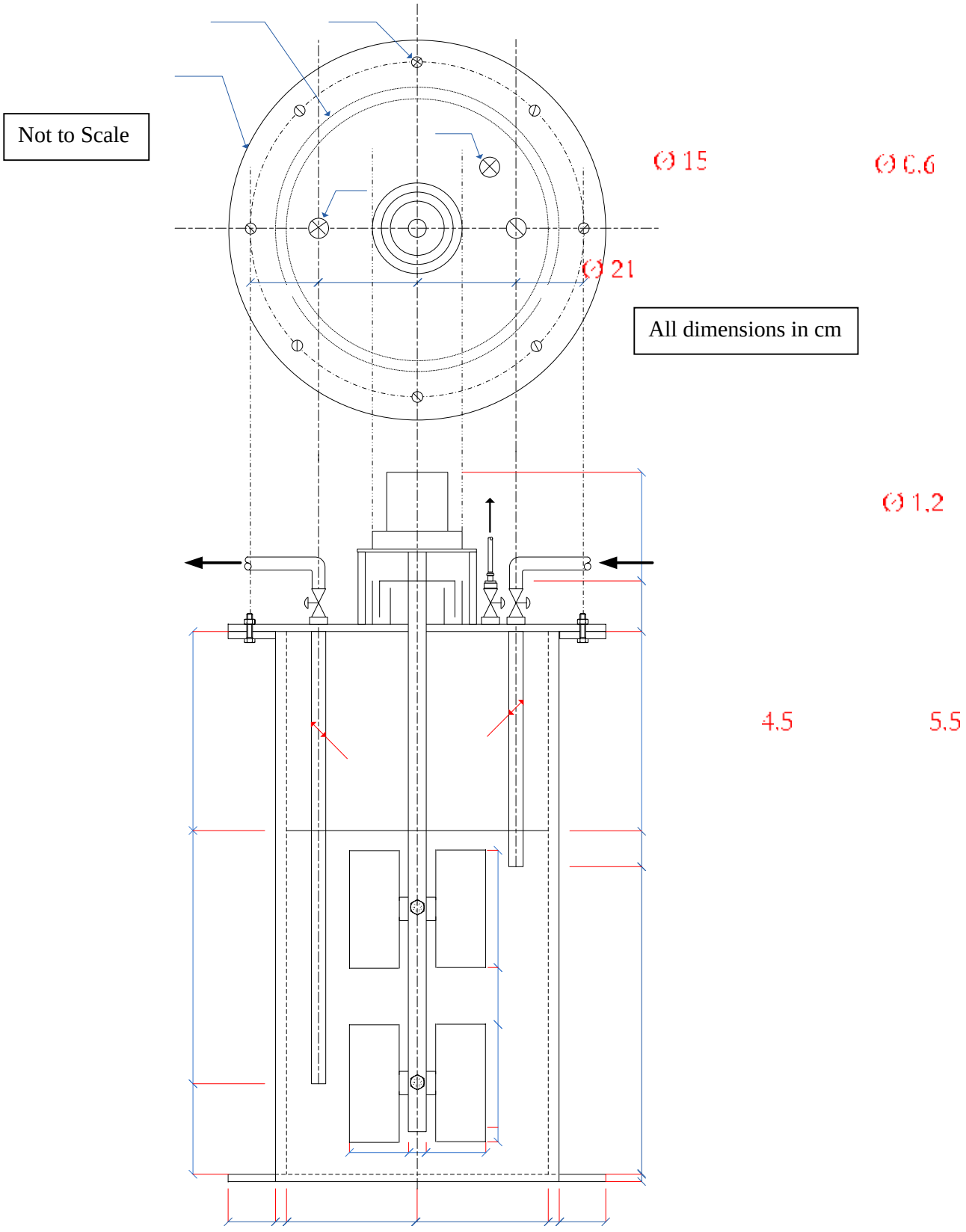


Figure 3.8 Anaerobic Digester Design

Table 3.2 Feeding and organic loading rate with respect to SRT

SRT days	**Control (D_0)**	**Ultrasonic (D_u)**		**Chemical-ultrasonic (D_{cu})**		**SLR** Kg TS/m^3days
	100% Fresh WAS mL/day	50% Sonicated mL/day	50% Fresh WAS mL/day	50% Chemical-ultrasonic mL/day	50% Fresh WAS mL/day	
25	120	60	60	60	60	1.2
15	200	100	100	100	100	2
10	300	150	150	150	150	3

3.8 Analytical Methods

3.8.1 Total solids (TS) and Volatile solids (VS)

Total solids and volatile solids were measured according to standard method (APHA, 1998) The TS content of semisolid or sludge is calculated in percentage by weight compared to the total weight of the sludge. Similarly, VS is also calculated in percentage by weight compared to the TS content.

3.8.2 Chemical oxygen demand (COD)

COD was analyzed according to standard method (APHA, 1998). SCOD is the measurement of COD in the soluble form. Sonicated sludge was centrifuged with 5000 rpm for 30 min. The supernatant was collected and filtered with membrane filter (pore size of 0.45 μm). The filtrate was used to analyze SCOD.

3.8.3 Biochemical methane potential (BMP)

Biochemical methane potential (BMP) test was carried out to determine the biodegradability of fresh and pretreated sludge (ultrasonic, chemical and chemical-ultrasonic) under mesophilic condition (37 ^oC) in the batch anaerobic digester for around 30 days. In BMP test, the serum bottle of 120 ml capped with butyl rubber stoppers was used. The total working volume was 30 mL in which 5 mL was inoculums and 25 mL was substrate. Before purging oxygen free nitrogen gas to the headspace, $NaHCO_3$ was added to increase the alkalinity up to 4000 mg/L. Then, serum bottles were kept in 37 ^oC incubator till they stop producing biogas. Daily biogas was measured by inserting needle attached to a syringe (10 and 25 mL). Methane composition was also examined by Gas Chromatograph with a packed column.

3.8.4 Biogas production and composition

Biogas production is the measurement of the quantity of biogas generated from WAS. 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. The gas in sampling biogas bag was sucked out by using a syringe at standard temperature and pressure (STP). The cumulative gas in gas sampling bag was collected two or three times a week depending on gas generated. The steady state is believed to reach whenever the daily biogas production remains constant.

Biogas composition was measured by Gas Chromatograph (GC) equipped with thermal conductivity detector (TCD) using Helium as carrier gas. The sample was collected by a graduated syringe with the volume of 0.2 mL and injected to the sampling column of GC. The result shows within 15 minutes with the component of H_2, CO_2, O_2, N_2 and CH_4. The details of operating condition of GC are shown in Table 3.4.

Table 3.3 Analytical parameters and methods of anaerobic digester performance

Parameter	Range/ Accuracy	Interference	Frequency/ week	Method/Instrument
Biogas production	0-100 mL	Solubility of CO2 in water	2 (a) 2 (b)	Graduated syringe
Biogas composition	0-100 %	Instrumental operational calibration curve	2 (a) 3 (b)	Gas Chromatograph equipped with TCD SHIMADZU GC14A, Column SUS, WG-100
VFA	95% accuracy	Instrumental operational calibration curve	2 (a) 3 (b)	Gas Chromatograph equipped with FID HP 5890 series II plus
pH	(1-14) ±0.1	-	7 (a) 7 (b)	Electrometric method, pH meter (Glass electrode)
Alkalinity	Standard deviation; 5 mg/L	-	3 (a) 3 (b)	Volumetrically by titration with 0.02 N H2SO4
TS	-	Large, floating particles or submerged agglomerates of no homogenous materials, visible floating oil and grease	3 (a) 3 (b)	Standard Method TS dried at 105˚C for 24 hours.
VS	-	Loss of ammonium carbonate and volatile organic matter during drying	3 (a) 3 (b)	Standard Method: TS incinerated at 550˚C for 1 hour.
CST	-	-	3 (b)	C.S.T apparatus, TRITON, England

a : Start up period b : After steady state

3.8.5 Volatile fatty acid (VFA)

Sample taken from the digester was centrifuged and then filtered before filling into glass vial for volatile fatty acid (VFA) analysis. The sample volume of 2 µL was automatically injected into the column and elution time of all individual components was recorded. The details of operating condition are given in Table 3.4.

3.8.6 Alkalinity

Alkalinity of digested sludge was measured three times per week. Due to the difficulty of color judgment, direct measurement by pH meter was used instead. 10 mL sample was taken into a plastic bottle and titrated with H_2SO_4 (0.05 N) till pH reduces to 4.5. Figure 3.10 illustrates the procedure of alkalinity measurement.

3.8.7 Capillary suction time

CST apparatus, TRITON model, England was used for this measurement.

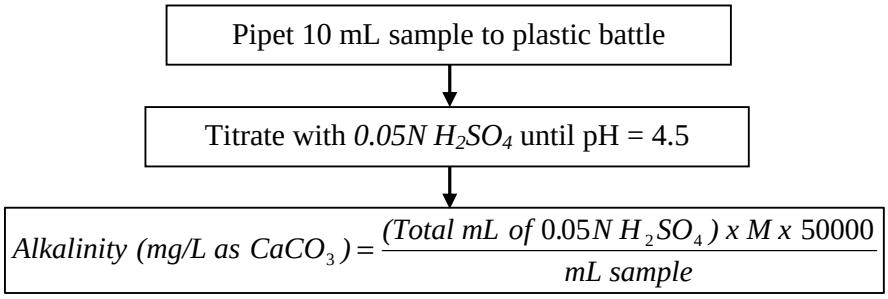


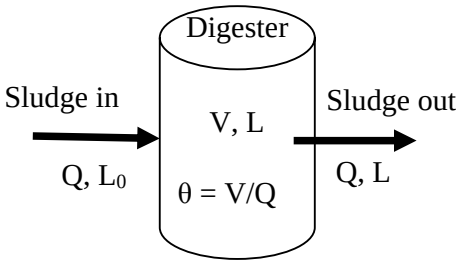
Figure 3.9 Alkalinity determination

Table 3.4 Analytical condition of Gas Chromatography

Description	Biogas Composition	Volatile Fatty Acid (VFA)
Detector	Thermal Conductivity Detector (TCD)	Flame Ionization Detector (FID)
Carrier	Helium (He)	Nitrogen (N_2)
Flow rate	40 mL/min	40 mL/min
Injection/Detector Temperature	50/100 ^oC	210/210
Column Temperature	50 ^oC	180 ^oC
Column	Pack (WG-100, SUS col.1/4', Inner diameter 1.8 mm)	Capillary, DBFFAP (30 mm x innerdimeter 0.32 mm x thickness 0.25 μm)
Working Pressure	0.75 kg/cm^2	-
Sample Volume	0.2 mL	0.002 mL

3.9 Kinetic Study

In order to determine the improvement of the process kinetics a first-order degradation model is used together with a mass balance over the anaerobic digestion system.



L_0 : Concentration of the WAS organic material input (kg/m^3)
L : Concentration of organic matter in the digester and effluent (kg/m^3)
Q : Volumetric sludge flow (m^3/d)
V : Volume of the digester (m^3)
θ : Sludge retention time (d)

Figure 3.10 Mass balance of anaerobic digestion system

The first-order reaction rate is written as follows:

$$\frac{dL}{dt} = -k.L \qquad \text{Eq. 3.4}$$

Where,

L : Concentration of organics compounds of WAS (kg/m^3)
k : Rate constant of the hydrolysis step (d^{-1})
t : Time (d)

Combining the mass balance of a continuous stirred tank reactor (CSTR) with Eq. 3.4, we get

$$k = \frac{1}{\theta}\left(\frac{L_o}{L} - 1\right) \qquad \text{Eq. 3.5}$$

Where,

L_o : Concentration of organics compounds of feed sludge (kg/m^3)
L : Concentration of organic compounds of digested sludge at steady state (kg/m^3)
θ : Digestion time (day)

3.10 Economic Analysis

Economic analysis was conducted in term of energy and chemical cost. The biogas generated from anaerobic digester was converted to energy, and then to monetary value (Income). Finally, the comparison of expense and income was conducted. The cost of energy supplied for anaerobic digester operation and for sonicating sludge was considered to be the expense for ultrasonic system, and the cost of energy supplied for anaerobic digestion operation and for sonicating sludge with chemical cost was considered to be the expense for chemical-ultrasonic system.

3.10.1 Energy consumption of anaerobic digester

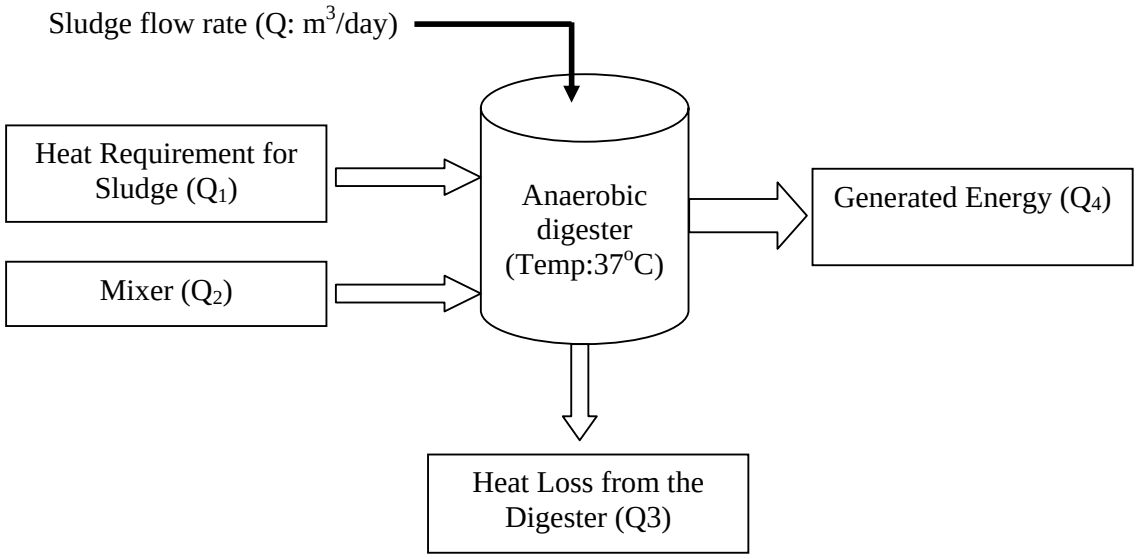


Figure 3.11 Energy balance of anaerobic digestion system

Assumptions:

- Temperature of fresh sludge (T_1) = 20 °C
- Average temperature of ambient air temp(T_2) = 30 °C
- Temperature in the digester(T_3) = 37 °C
- Specific heat capacity of sludge(C_p) = 4.2 kJ/kg/°C
- Overall heat transfer coefficient (U) = 2.5 W/m^2/°C
- Calorific value of methane = 35.8 kJ/g
- Specific gravity of sludge (m) = 1.02
- Sludge flow rate (Q) = 120, 200 and 300 mL/day

a. Heat requirement for the sludge

$$Q_1 = \sum m\, C_P (T_3 - T_1) \qquad \text{Eq. 3.6}$$

$= Q$*1.02*4.2*(37-20)

$= 72.828\, Q$ J/day

b. Energy requirement of mechanical mixer

Mechanical mixer used depends on the digester volume and the characteristic of the raw sludge. In this case, all the reactors were equipped with 9W motor. Therefore, the energy requirement for mechanical mixer was assumed regarding to this value.

$$Q_2 = 9\ W = 777.6\ \text{kJ/day}$$

c. Rate of heat addition required to compensate for loss from the digester

$$Q_3 = UA(T_3 - T_1) \qquad \text{Eq. 3.7}$$

Where, A= Cross-sectional area through which the losing is occurring, 0.1154 m^2

Q_3 = 2.5*0.1154*(37-30)

= 2.02 W

= 174.485 kJ/day

d. Energy consumption of anaerobic digester

Energy consumption of anaerobic digester operation is the sumps of energy supplied for heating sludge, for mixing, and for compensating the heat loss from the digester. It can be calculated as below equation:

$$Q_4 = Q_1 + Q_2 + Q_3 \qquad \text{Eq. 3.8}$$

3.10.2 Energy gain from anaerobic digester

Energy gain from anaerobic digester was the conversion of methane gas generated to energy. From the relation of Ideal Gas Law, mass of methane was derived and energy was calculated from the calorific value of methane.

Ideal Gas Law: $PV = nRT$ Eq. 3.9

Where,

- n : number of moles (mole)
- R : universal gas constant (8.3145 J/mole.K)
- P : absolute pressure (Pa)
- V : Volume of the gas (m^3)
- T : Temperature (^oK)

$$\Rightarrow \quad n = \frac{1.01325 \times 10^5 \, x \, V}{8.3145 \times 303} = 40.219 \, x \, V \ \text{mol}$$

V is measured in mL/day

Methane production: $M_{CH4} = 40.219\ 10^{-6} \times V \times 16 = 643.504\ V$ g/day

$$Q_5 = M_{CH4} \times 35.8 \text{ kJ/day}$$

Energy gain

Energy gain (Q_6) is the difference energy generated and energy consumption. Thus, energy gain was calculated as below equation:

$$Q_6 = Q_5 - Q_4$$ Eq. 3.10

3.10.3 Ultrasonic energy input

Ultrasonic energy input (Q_7) was the power requirement for optimum condition of ultrasonic and chemical-ultrasonic pretreatment. The ultrasonic energy input can be calculated using below formula:

$$Q_7 = Q \, x \, UD \, x \, t$$ Eq. 3.11

Where,

- Q : Sonication sludge flow rate (ml/day)
- UD : Ultrasonic density (W/mL)
- T : Sonication duration (s)

3.10.4 Energy cost

As the research was conducted in Thailand, the energy cost analysis was computed with respect to Thailand electricity cost. Overall, the energy cost in Thailand is around 2.5 Baht per kWh. Thus, the cost of energy was derived regarding to power and duration used.

$$Electricity\ cost = M_{energy} x\ 23.2\ x\ 24\ hr/day \quad Baht/day \qquad \text{Eq. 3.12}$$

Where,

M_{energy} : amount of energy used (kW/day)

3.10.5 Chemical cost

Chemical pretreatment uses only NaOH (commercial grade). Therefore, NaOH cost from the market price was used. According to AIT laboratory, NaOH costs around 0.05 Baht/g.

$$Chemical\ cost = M_{NaOH}\ x\ 0.05 \quad Baht/day \qquad \text{Eq. 3.13}$$

Where,

M_{NaOH} : amount of NaOH used (g/day)

3.10.6 Economic analysis

Economic analysis was focused on the income or benefit from AD in term of biogas production into energy cost. The income calculation is presented in Table 3.5.

Table 3.5 Economic analysis of pretreatments

	Ultrasonic	Chemical-ultrasonic
Energy cost	$E_1 = Q_6 x\ 2.5\ x\ 24$	$E_1 = Q_6 x\ 2.5\ x\ 24$
	$E_2 = Q_7 x\ 2.5\ x\ 24$	$E_2 = Q_7 x\ 2.5\ x\ 24$
Chemical cost	-	$C = M_{NaOH}\ x\ 0.4$
Benefit/Income	$Income = E_1 - E_2$	$Income = E_1 - (E_2 + C_1)$

Chapter 4

Results and Discussion

This chapter presents the findings obtained from laboratory scale experiment of a series of sludge disintegration, the performance of anaerobic digestibility of pretreated sludge and the economic analysis of the treatment options. The first part of the chapter describes the optimum operating condition of ultrasonic, chemical and chemical-ultrasonic pretreatment on WAS disintegration. The second part presents the results obtained from anaerobic digestion a long with the comparison of digesters performance and kinetic study. The last part is the economic analysis of anaerobic digestion with pretreated sludge compared to non-pretreated sludge.

4.1 Pretreatments

The main aim of pretreatments is to break down the microbial cell wall for releasing the intracellular and extracellular substances into the aqueous phase. It eventually accelerates the degradation rate of anaerobic digestion. WAS collected from a local wastewater treatment plant (Raw Sludge) was concentrated before using as representative sample (Feed Sludge). The characteristics of raw and feed sludges are presented in Table 4.1.

Table 4.1 Characteristics of raw sludge and feed sludge

Parameters	Raw Sludge	Feed Sludge
TS (%)	1.01±0.08	3 ± 0.1
VS (%)	0.84±0.07	2.62 ± 0.01
TCOD (mg/L)	11719±333	42196±875
SCOD (mg/L)	77±14	458±35
TKN (mg/L)	814±104	3136±150
NH_3 (mg/L)	26±3	245±1
CST (min)	55±1	992±23
pH	6.67±0.2	6.97±0.24

4.2 Ultrasonic Pretreatment

Optimization of sludge disintegration using ultrasound depends on many factors. However, ultrasonic density and specific energy input were used as the main parameters to evaluate the optimum condition of the pretreatment in term of SCOD released. Ultrasonic density is the measurement of power input per unit volume of sludge used and specific energy input is measured by energy input per unit mass of dry solids. The SCOD of non-sonicated and sonicated sludge at different power inputs are presented in Table 4.2, and at different ultrasonic densities and specific energy inputs in Appendix A3. During ultrasonic experiment, pH of the sludge decreased by less than 0.04 and was not adjusted for subsequent anaerobic digester.

Table 4.2 SCOD release at different sonication times and power input

Sonication Time (s)	SCOD (mg/L)			
	P = 50 W	P = 100 W	P = 150 W	P = 190 W
0	458	458	458	458
30	525	975	2007	2007
60	1200	1650	3927	5498
120	1800	3600	8116	7418

4.2.1 Effect of ultrasonic density on the SCOD release

In this study, ultrasonic density was calculated using the equation 3.2 (Appendix A1). The results of SCOD release at different ultrasonic densities are summarized in Figure 4.1.

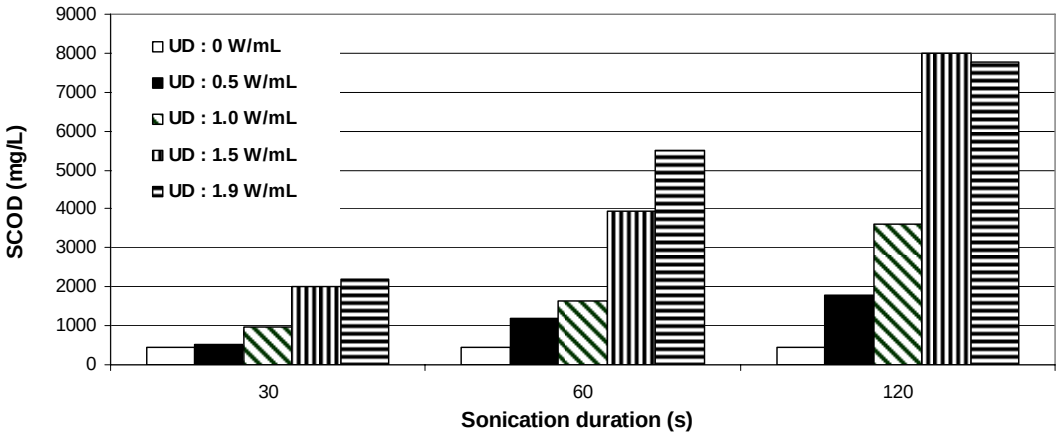


Figure 4.1 SCOD release at various sonication durations with different ultrasonic densities

SCOD gradually increased with increase of sonication duration and ultrasonic density. At sonication time of 30 s, the releases of SCOD of all UDs were relatively comparable; however they were considerable difference at sonication time of 120 s. Most interestingly, at the same duration of 120 s, the UD of 1.5 W/mL gave a big gap of SCOD released compared to UD of 1 W/mL but more or less similar to UD of 1.9 W/mL. Thus, UD of 1.5 W/mL was considered to be the optimum value for effective SCOD release at sonication duration within 120s, or in other word 1.5 W/mL was the optimum ultrasonic density on sludge disintegration. The same result was also found from previous study (Navaneethan, 2007). Using ultrasonic density cannot be used as a representative parameter to evaluate the effects of every ultrasonic pretreatment once the sludge concentration was changed. Therefore, specific energy input was also conducted as it was the most reliable parameter.

4.2.2 Effect of specific energy input on the mg SCOD/ g TS release

SCOD release per unit weight of TS concentration slowly increased at low specific energy input (Lower than 1 kJ/g TS) and significantly increased afterward. This low SCOD release is due to low energy supplied which can only reduce the flocs size, but not to the cells. Hence, the intracellular cannot release into the aqueous phase but only extracellular which contribute to the SCOD. This finding was also proved by Bougrier et al. (2005) that at 1000 kJ/kg TS was a minimum energy supplied to break down the cells. Figure 4.2

presents the increase trend of the mg SCOD/g TS with different ultrasonic densities against specific energy input.

From Figure 4.2, it was clearly shown that mg SCOD/g TS of UD 1.9 W/mL extensively increased at the specific energy input range of 2-3.8 kJ/g TS compared to other UDs, thereafter the increment slowed down. In contrary, the mg SCOD/g TS of UD 1.5 W/mL steadily increased to higher than that of UD 1.9 W/mL starting from 4.7 kJ/g TS. With respect to economic aspect, higher SCOD release at lower specific energy input was considered to be the critical value for effective sludge disintegration. Therefore, sonicating at 3.8 kJ/g TS of specific energy input with 1.9 W/mL of ultrasonic density was considered to be the optimum condition on sludge disintegration. This finding corresponds to the result found by Dewil et al. (2006). The author concluded that the minimum energy requirement for breaking cells should be at least 1500 kJ/kg TS. However, Khanal et al. (2006c) found the optimum specific energy input at 35 kWs/g TS. This value is quite high compared to the finding in this study due to the difference of investigating duration.

The specific energy input was calculated using equation 3.1 (Appendix A1). According to the optimum condition found from the graph, the sonication duration was derived to be 60 s. Thus, sonicated sludge at power input of 190 W for 60 s was selected to feed the subsequent anaerobic digester.

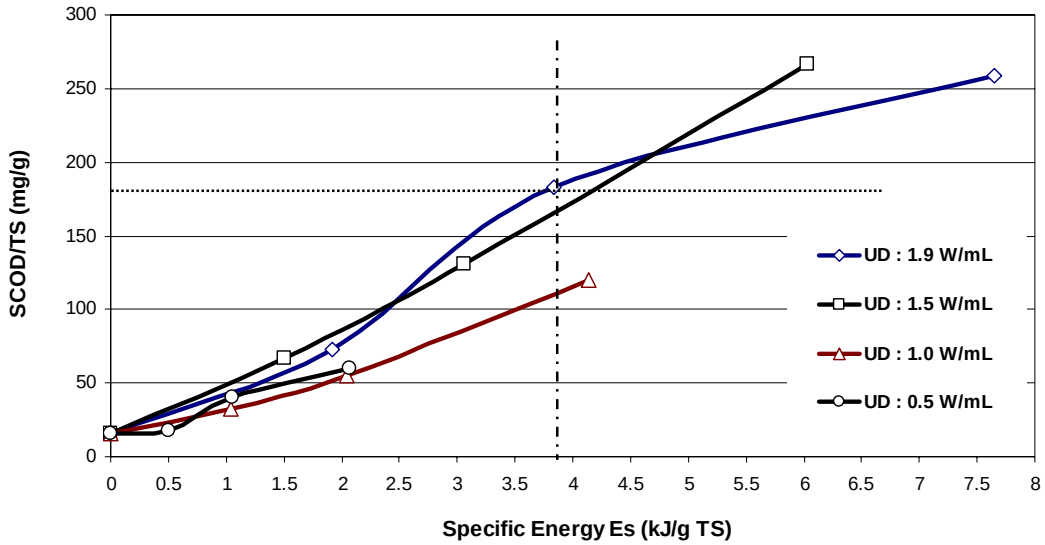


Figure 4.2 mg SCOD/g TS release at various specific energy input with different ultrasonic densities

4.3 Chemical Pretreatment

Optimization of chemical pretreatment using NaOH was based on SCOD released coupled with holding time. pH of pretreated sludge was also investigated with respect to chemical doses in order to compare with other published papers. The SCOD released of different chemical doses over holding time, and pH of pretreated sludge are summarized in Table 4.3 (Appendix A4).

Table 4.3 SCOD release and pH variation at different chemical doses

Holding Time (min)	SCOD (mg/L)			
	10 mg/g TS	25 mg/g TS	50 mg/g TS	75 mg/g TS
0	458	458	458	458
5	950	2004	5703	7414
10	1250	2863	6286	7984
30	1375	2672	6463	9695
60	1900	2863	6539	10645
pH	7.43	9.15	11.08	11.63

4.3.1 Effect of holding time on the mg SCOD/g TS release

The effect of holding time on the SCOD release of chemical pretreatment was investigated within 60 min. the increasing trend of SCOD a long with holding time is presented in Figure 4.3. Initially, the SCOD significantly increased for the first 6 min pretreatment duration and then the increment slowed down over the time for all chemical doses. Starting from 10 min holding time onward, the increment of SCOD of 10, 25 and 50 mg/g TS was more or less constant. However, the SCOD of 75 mg/g TS kept gradually increasing up to 30 min and then slowed down afterward. As the time was prolonged, the increment slowed down which could be due to the limitation of microbial cells population in the raw sludge to solubilize and/or the limitation of chemical used. According to the purpose of this study is to find out the higher SCOD release at shot holding time, 6 min of pretreatment was a critical value for chemical pretreatment. This holding time was selected as the effective pretreatment duration for subsequent chemical-ultrasonic pretreatment.

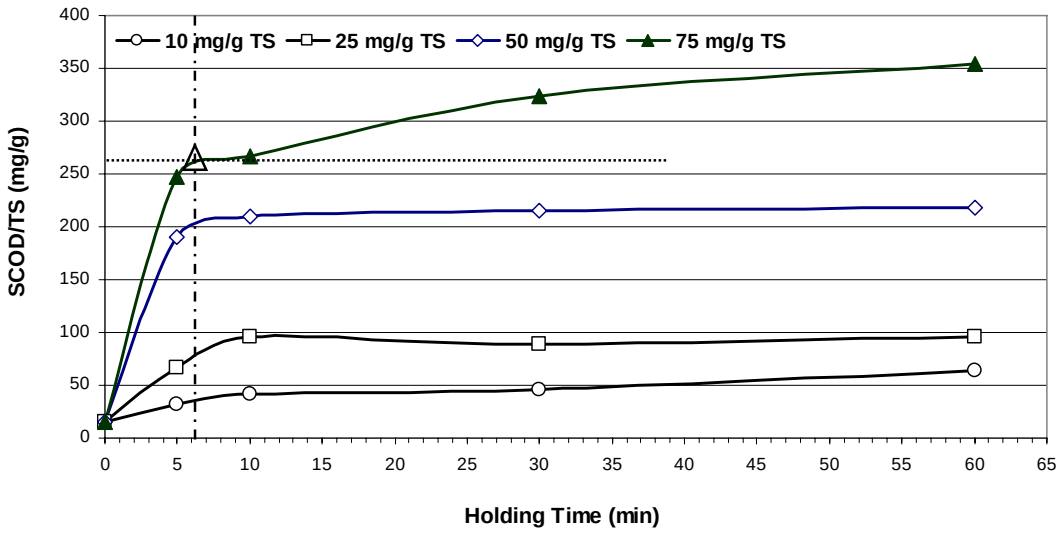


Figure 4.3 Effect of holding time on the SCOD release

4.3.2 Effect of chemical dose on the SCOD release

The effect of NaOH dose on sludge disintegration is evaluated by SCOD change (Figure 4.4). SCOD of the chemical pretreated sludge increased with increase of chemical dose. At chemical dose of 10 and 25 mg/g TS, the increment of SCOD was slightly improved. Whereas at 50 and 75 mg/g TS, the SCOD release was significantly increased up to more than double compared to previous two doses at all holding times. However, the SCOD increment of 50 mg/g TS was much higher than 25 mg/g TS and slightly lower than 75 mg/g TS during 5 and 10 min pretreatment duration. The increment trend kept remaining the same at 30 and 60 min pretreatment times. The increment was expected not to change from this trend when holding time was extended due to the fact that 23% of SCOD was achieved at 75 mg/g TS for 30 min and it was reported to reach up to 43.5% COD solubilization at chemical dose of 7g/L NaOH (180 mg/g TS) (Kim et al., 2003). Moreover, the solubilization rate was extensively slowed down after 30 min pretreatment and even down to 10 min pretreatment (Kim et al., 2002b). According to this result, 50 mg/g TS corresponding to pH of around 11 was considered to be the optimum chemical dose on sludge disintegration. The optimum pH of 11 was also found by Kim et al. (2002b). However, Li et al. (2007) found the optimum chemical dose of 0.05 mol/L (0.16 g/g DS) which is triple higher than the current study found. The reason of this big difference could be due to the different sludge characteristics (TS: 2%) and the limitation of low chemical dose to be investigated (0.05 mol/L was the lowest chemical dose).

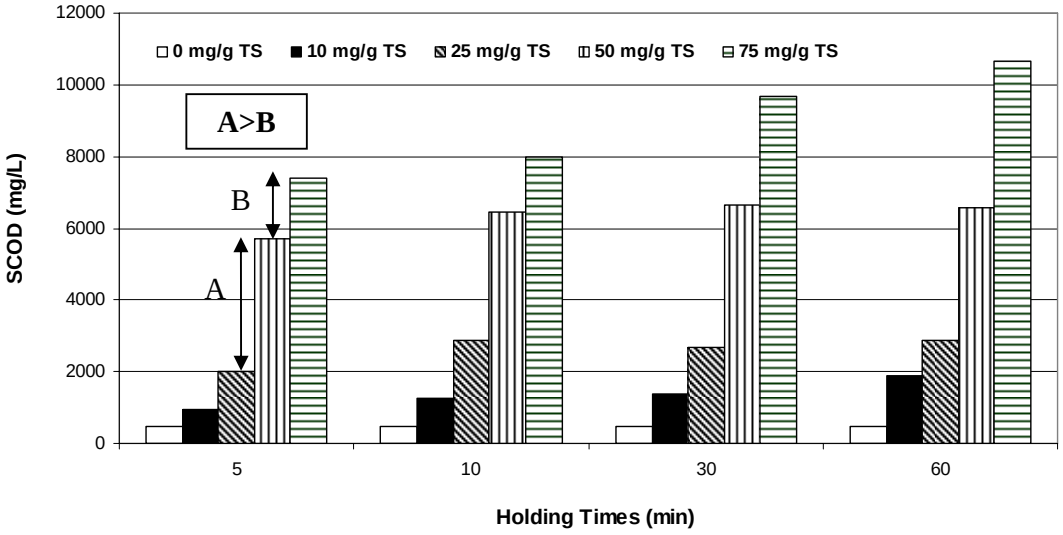


Figure 4.4 Effect of chemical dose on the SCOD release

4.4 Chemical-ultrasonic Pretreatment

Chemical-ultrasonic pretreatment is the combination of chemical and ultrasonic processes. Chemical pretreatment was performed first and then ultrasonic was followed afterward. Due to the optimum chemical dose was found at high pH which probably inhibits methanogenic bacteria activities, a series of chemical dose at low pH was investigated. The experiment was performed with dosage range of 10-25 mg/g TS with constant holding time of 6 min and constant power input of 190 W for 1 min (Appendix A2). The results of SCOD release with different sonication durations and pH variation after sonication are summarized in Table 4.4 (Appendix A5).

Table 4.4 SCOD release and pH variation of chemical-ultrasonic pretreatment

Sonication duration (s)	SCOD (mg/L)			
	10 mg/g TS	15 mg/g TS	20 mg/g TS	25 mg/g TS
30	3938	4827	5738	6249
60	7621	7560	9017	9183
120	10178	11567	12478	12118
pH	7.03	7.36	7.80	8.12

4.4.1 Effect of chemical dose on the SCOD release

SCOD increased with increase of chemical dose and sonication duration. At all sonication durations, SCOD releases of pretreated sludges with all chemical doses were relatively comparable. According to result presented in Figure 4.5, it was clearly shown that 10 mg/g TS was the effective chemical dose for chemical-ultrasonic pretreatment as it gave significant SCOD increment compared to non-pretreated sludge and relatively comparable SCOD to high doses (15, 20 and 25 mg/g TS). Therefore, 10 mg/g TS was selected as an effective chemical dose to treat WAS before feeding to anaerobic digester.

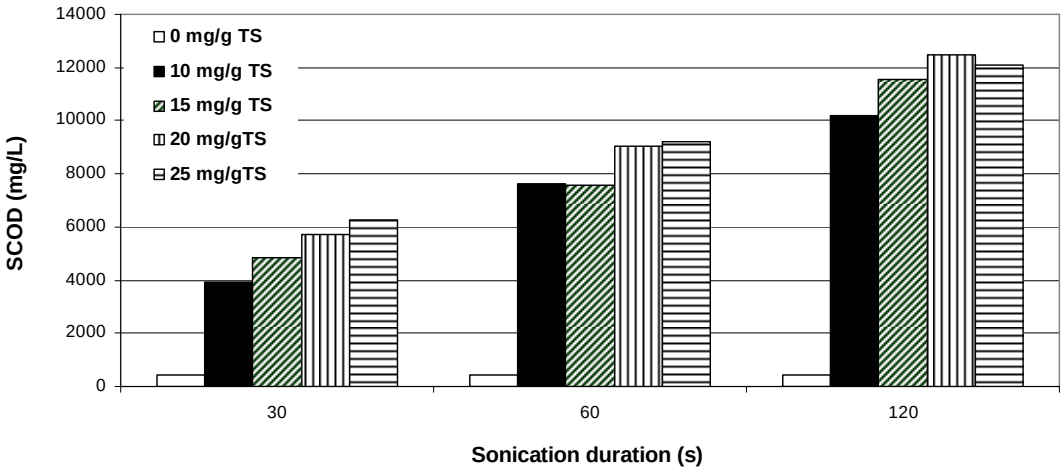


Figure 4.5 Effect of chemical dose of chemical-ultrasonic pretreatment on SCOD release

4.4.2 Effect of specific energy input on the mg SCOD/g TS release

The SCOD concentration increased with high specific energy supplied. The SCOD release rate significantly increased at the first stage of energy input range of 0-3.8 kJ/g TS, thereafter the increase rate slowly decreased (Figure 4.6). The study was attended to find out the effective pretreatment with low operating cost which corresponds to low chemical dose and low energy input. Therefore, chemical dose of 10 mg/g TS and specific energy input of 3.8 kJ/g TS were the critical values of chemical-ultrasonic pretreatment on sludge disintegration. This condition was selected to treat WAS for subsequent anaerobic digestion.

The COD solubilization of 10 mg/g TS was found to be 18% at specific energy input of 3.8 kJ/g TS which is much lower than result found by Chiu et al. (1997), reached 89.3% at chemical dose of 40 meq/L NaOH (0.16 g/g TS) for 24 hours and sonicated at 24 s/mL (288 kJ/g TS). From this result, it is quite difficult to give a clear comparison due to big different of pretreatment conditions and sludge characteristics.

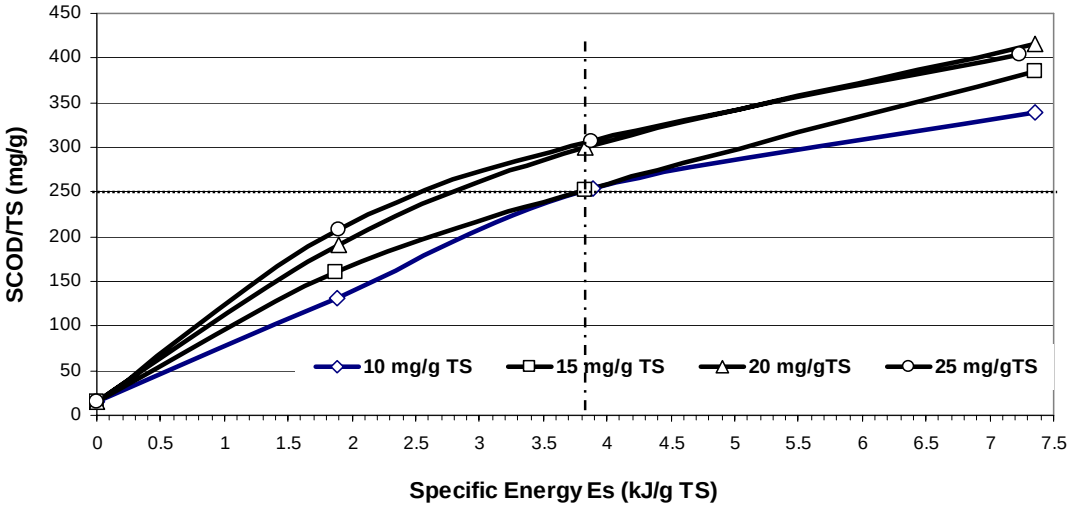


Figure 4.6 Effect of specific energy input on SCOD/TS release of chemical-ultrasonic pretreatment

4.5 Evaluation of Sludge Disintegration Efficiency

This section focused on the comparison of effect of pretreatments on WAS in term of SCOD release (Figure 4.7). The evaluation of sludge disintegration efficiency of all pretreatment processes was based on equation 3.3. %SCOD release from ultrasonic treatment at 1.9 W/mL increased from 1.1% to 17.6%, in which 13.0% was the %SCOD release at optimum condition (1.9 W/mL; 60 s). Similarly, it increased from 1.1% to 15.5 and 24.1% for chemical and chemical-ultrasonic treatment, respectively. 13.5 and 18% were found to be the %SCOD release at selected optimum condition of these two treatments. By comparing to ultrasonic treatment, the SCOD release of chemical and chemical-ultrasonic treatment was improved by 3.7 and 38.6%, respectively. The highest improvement of SCOD release into the aqueous phase was with chemical-ultrasonic which is due to the combination effects of mechanical shear force and radical OH^- reaction.

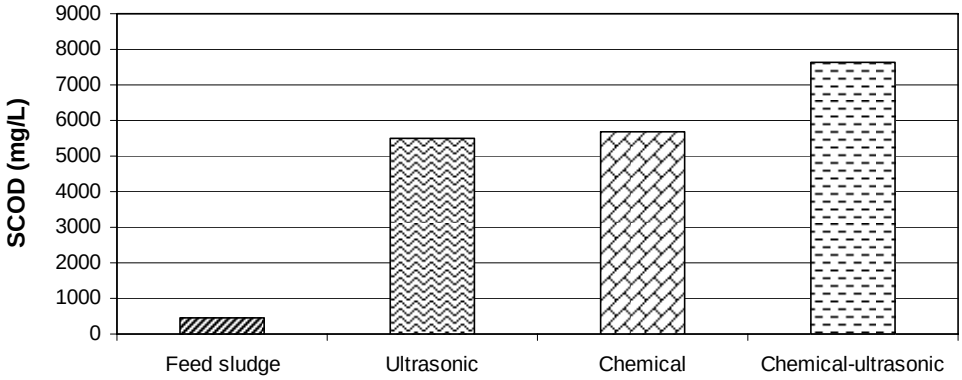


Figure 4.7 SCOD release of all pretreatment techniques at optimum condition

4.6 BMP Test Analysis

Due to the uncertain toxicity of NaOH addition, BMP test was conducted before anaerobic digesters. The performance of anaerobic digestion with different chemical doses was investigated in order to select the suitable chemical dose for semi-continuous digester. The experiment was carried out in identical conditions such as temperature and pressure. Figure 4.8 shows the cumulative methane production a long with digestion period at different chemical doses and power input of 190 W for 1 min (Appendix B1 and B2). The methane production rate started increasing potentially since the first day of operation. It is due to the ease of hydrolyzing the compounds and the availability of substrates which are facilitated by chemical-ultrasonic pretreatment. The increase rate slowed down after 10 days of digestion period due to the substrates limitation.

At the first 5 days of digestion period, the methane production of all chemical doses was more or less the same. Afterward, methane production of pretreated sludge with 10, 15 and 25 mg/g TS progressively increased to higher than that of non-pretreated sludge and it reached almost same at 80 days of digestion period (Table 4.5). It clearly indicated that chemical-ultrasonic pretreatment enhanced the biogas production at a shorter digestion time compared to non-pretreatment. Although, all chemical doses gave higher methane production, 20 mg/g TS was observed to be lower than non-pretreated sludge. According to the TS and VS reduction results, it evidently indicated that there was a leak of biogas from the serum bottles as it gave a comparable performance in term of TS and VS removal efficiency (Figure 4.9). With respect to the result shown in Figure 4.8, chemical-ultrasonic pretreated sludge with 10 mg/g TS gave a comparable methane production with relatively low chemical dose (triple lower) compared to other doses. Therefore, chemical dose of 10 mg/g TS was considered to be the suitable dose for anaerobic digester of chemical-ultrasonic pretreated sludge.

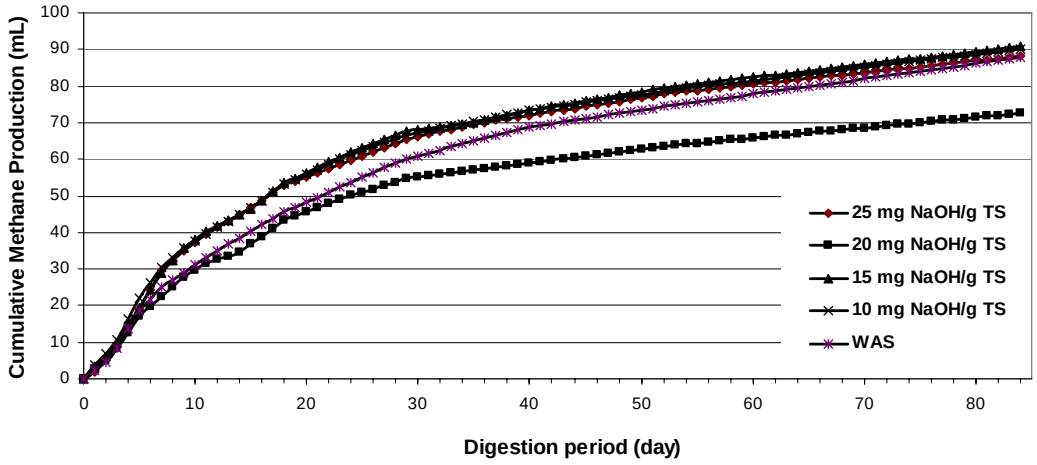


Figure 4.8 Cumulative methane production with digestion period at different chemical doses

The BMP experiment was carried out with total volume of 30 mL, from which 25 mL was feed sludge and 5 mL was seed sludge (inoculums). The TS and VS contents of feed and seed sludge, and the digested sludge after the experimental run are presented in Table 4.6. Figure 4.9 shows the TS and VS removal efficiency of sludge pretreated with different chemical doses. It clearly shows that the TS and VS removal efficiency increased

significantly within the dosage range of 0-15 mg/g TS, thereafter it slowed down to almost constant. Thus, chemical-ultrasonic pretreatment with 15 mg/g TS of chemical dose was considered to be the optimum dose in term of TS and VS destruction. Although, chemical dose of 15 mg/g TS gave higher TS and VS destruction, 10 mg/g TS was selected for semi-continuous anaerobic digester as it gave higher biogas production and lower chemical dose.

Table 4.5 Cumulative CH_4 production of different chemical doses at meshophilic and STP

Chemical Dose (mg/g TS)	Cumulative Methane Volume (Meshophilic) (mL)	Cumulative Methane Volume (STP) (mL)
Control (0)	122.74	108.91
10	124.73	110.67
15	125.92	111.73
20	107.60	95.48
25	122.58	108.77

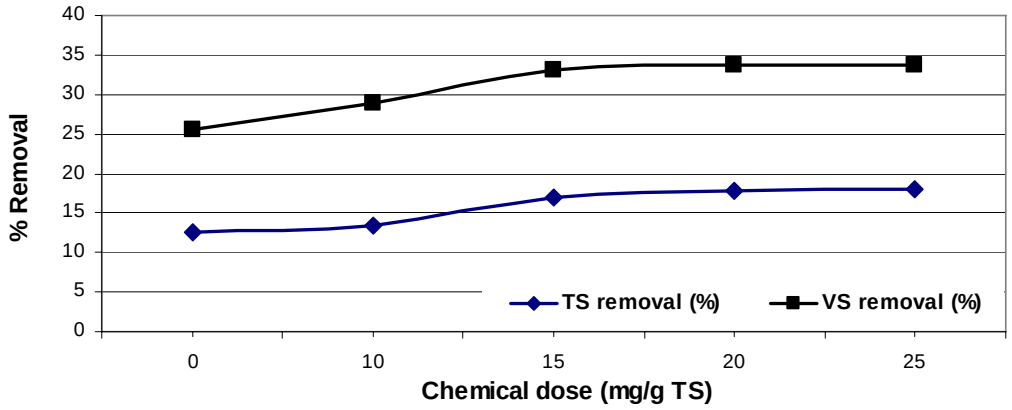


Figure 4.9 TS and VS removal of chemical-ultrasonic digested sludge at different chemical doses

Table 4.6 TS and VS removal of digested sludge at different chemical doses

	TS (%)		VS (%)	
Feed sludge	2.99		2.59	
Seed sludge	9.12		3.90	
Influent sludge (Feed + Seed)	4.01		2.81	
Digested Sludge				
	TS (%)	TS removal (%)	VS (%)	VS removal (%)
WAS	3.51	12.5	2.09	25.6
10 mg/g TS	3.47	13.5	2.00	28.8
15 mg/g TS	3.33	17.0	1.88	33.1
20 mg/g TS	3.30	17.7	1.86	33.8
25 mg/g TS	3.29	18.0	1.86	33.8

4.7 Quasi-Steady State Condition and Digester Performance

The digesters were operated under identical condition such as temperature and mixing speed. They were fed with non-pretreated sludge in the first stage and then with pretreated sludge, feeding sludge characteristics are summarized in Table 4.7. The digesters were first performed with 25 days SRT with feeding rate of 120 mL/day (1.2 kg TS/m^3.day). After steady state was reached, second and third SRTs (15 and 10 days) were continued with same procedure. The steady state was believed to reach when biogas production was stable for at least two weeks. The first two SRTs of all digesters took around two months, while the last SRT look around 1 month to get stable condition, and their performance is presented in Table 4.8.

Table 4.7 Characteristics of feed sludges

Parameter	Control	Ultrasonic	Chemical-ultrasonic
Total solids (%)	3±0.1	3±0.1	3±0.1
Volatile solids (%)	2.62±0.06	2.62±0.06	2.62±0.06
TCOD (mg/L)	42196±875	42196±875	42196±875
SCOD (mg/L)	458±35	3701±20	4110±30
pH	6.97±0.24	6.91±0.02	7.19±0.23
Alkalinity (mg/L)	1288±18	1338±35	1544±27
Dewaterability, CST (s)	1004±20	5325±40	5695±238

Table 4.8 Reactor performance at steady state condition

Parameters	Control			Ultrasonic			Chemical-ultrasonic		
SRT (day)	25	15	10	25	15	10	25	15	10
OLR (g VS/L.d)	1.04	1.73	2.60	1.04	1.73	2.60	1.04	1.73	2.60
Experimental Run (day)	54	57	32	54	57	32	54	57	32
pH	6.96	6.92	6.82	7.04	7.02	7	7.06	7.06	7
Alkalinity (mg/L)	3350	3075	2756	3753	3500	3169	4009	3941	3528
CST (s)	1369	1283	635	2172	2042	1404	2065	1964	1345
TS removal (%)	3.6	10.9	7.4	12.5	16.6	16.3	2.4	7.7	8.6
VS removal (%)	19.6	17.8	17.4	22.2	21.5	19.6	24.8	24.8	22.2
Biogas production (mL/day)	618	816	835	706	1016	1073	722	1078	1167
CH_4 production (mL/day)	358	478	485	404	589	636	420	626	688
Specific Methane Yield (L/g $VS_{removed}$)	0.56	0.52	0.34	0.56	0.54	0.4	0.52	0.49	0.38
Methane content (%)	58	58	58	57	58	59	58	58	59

4.8 TS and VS removal

WAS of 3% TS content and 2.62% VS content was used as a representative sample. It was fed into control reactor, and pretreated with ultrasonic and chemical-ultrasonic before

feeding to other two reactors. The variation of TS and VS removal are graphically presented in figure 4.10 and Figure 4.11, respectively (Appendix C3 and C4). As for TS removal, digester Do was in the range 3.6-10.9%, digester Du 12.5-16.6% and digester Dcu 2.4-8.6%. Pretreatment with ultrasound gave a great advantage in TS removal improvement compared to non-pretreatment and chemical-ultrasonic pretreatment. 16.6% was the highest TS removal achieving from ultrasonic digester at 15 days SRT, while the other two digesters were less than 11%. By comparing to non-pretreated sludge digester, it was found that there was little TS removal improvement from chemical-ultrasonicated sludge digester at short SRT, while significant improvement from ultrasonic pretreated digester, reached to 242% at 15 days SRT. It revealed that ultrasound is the effective technique for breaking down the microbial cells or difficult hydrolyzed compounds to easy biodegradable compounds. It eventually facilitates the decomposition reaction which leads to biodegrade more compounds in the digester. Regarding the low TS removal improvement from chemical-ultrasonicated digester, it does not mean the pretreatment could not break down the compounds but it is due to the addition of NaOH. Additional Na^+ into the feed sludge resulted in increasing TS content while sludge solubilization was taken place. Therefore, the final TS of digested sludge was found to be relatively high.

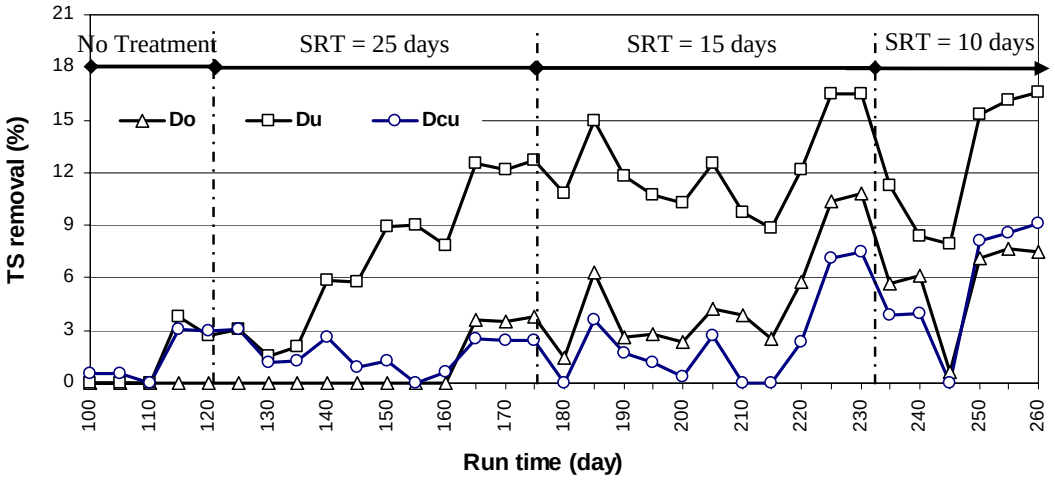


Figure 4.10 TS removal trend of all digesters against digestion duration

For VS removal, digester Do was in the range 17.4-19.6%, digester Du 19.6-22.2% and digester Dcu 22.2-24.8%. Digester Dcu gave a better performance in term of VS removal; reached to 27, 39 and 30% improvement compared to Do at SRT of 25, 15 and 10 days, respectively, whereas Du could reached to 13, 20.6 and 14% at the same SRTs. The VS removal of chemical-ultrasonic digester at 25 and 15 days SRT was constant and slightly reduced at 10 days SRT. However, it reduced for other two digesters from long to short SRT. This indicated that chemical-ultrasonic pretreatment could stabilize WAS more efficient than non-pretreatment and ultrasonic pretreatment alone at all SRTs. This higher performance is due to the combination effects of chemical and ultrasonic which help to break down the microbial cells for faster subsequent degradation.

At all SRTs, there was a slightly difference of TS removal between non-pretreatment and chemical-ultrasonic pretreatment. However a significant improvement was found with ultrasonic pretreatment. At the shortest SRT, the TS removal efficiency was relatively low, 12.5%, but it reached to a maximum value of 16.6% at 15 days SRT. Thereafter, it slightly reduced to a lower value of 16.3%. With respect to VS removal, it was less than 2%

difference from one SRT to another. The results obtained in this study let to the conclusion that 15 days SRT was the optimum digestion time for both pretreated sludges.

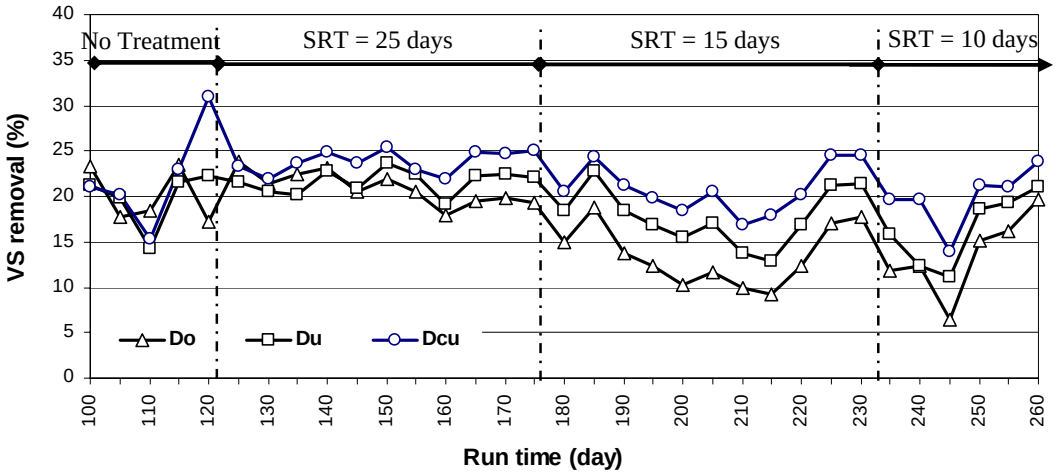


Figure 4.11 VS removal trend of all digesters against digestion duration

4.9 Biogas Production and Methane Production Rate

All digesters were first operated with non-pretreated sludge till a stable condition and then pretreated sludges were fed. The first SRT (25 days) was started at digestion run time of 121 days till 174 days, then second SRT (15 days) till 231 days, and finally the thirst SRT (10 days) till 262 days (Figure 4.12). The methane content in biogas from all digesters was around 59%, while 28% and 13% were CO_2 and other compounds (H_2, N_2, and O_2) from all SRTs, respectively (Appendix C2). The results revealed that there was no improvement of methane content in the biogas though pretreatments were used. The methane production rate at 25 days SRT were not significantly improved with all feed sludges. In contrast, it was considerable increased at 15 days SRT. Thereafter, the increase trend slowed down at 10 days SRT. By comparing to Do digester, the methane production of Du increased by 13, 23 and 31%, Dcu increased by 17, 31 and 42% at 25, 15 and 10 days SRT, respectively. The higher gas production in digester Dcu compared to Du and Do evidently indicated that the chemical-ultrasonic pretreated sludge was hydrolyzed more organic material into solution which is immediate used by anaerobic bacteria and eventually facilitates the digestion processes.

The specific methane yield of all digesters showed the same trend at all SRTs. The specific methane yield was high at long SRT and low at short SRT. At 25 days SRT, the methane yield of Do and Du was found to be same at the value of 0.56 L/g $VS_{removed}$, while a bit lower in Dcu digester at the value of 0.52 L/g $VS_{removed}$. However, at 15 days SRT, it slightly reduced to 0.52, 0.54 and 0.49 L/g $VS_{removed}$ from Do, Du and Dcu, respectively. Afterward, it continued reducing at 10 days SRT to 0.34, 0.4 and 0.38 L/g $VS_{removed}$ from same digesters. The reducing of methane yield could be due to high organic loading rate. Once the SRT was reduced, the organic loading rate automatically increases which eventually come to over loading phenomenon. It was clearly showed by dropping pH at 10 days SRT to lower than 6.8 from Do, and lower than 7 from Du and Dcu digesters. Another reason of reducing methane yield from Dcu digester is probably due to the combination effects of VFA accumulation and high Na^+ concentration once the feeding rate was increased.

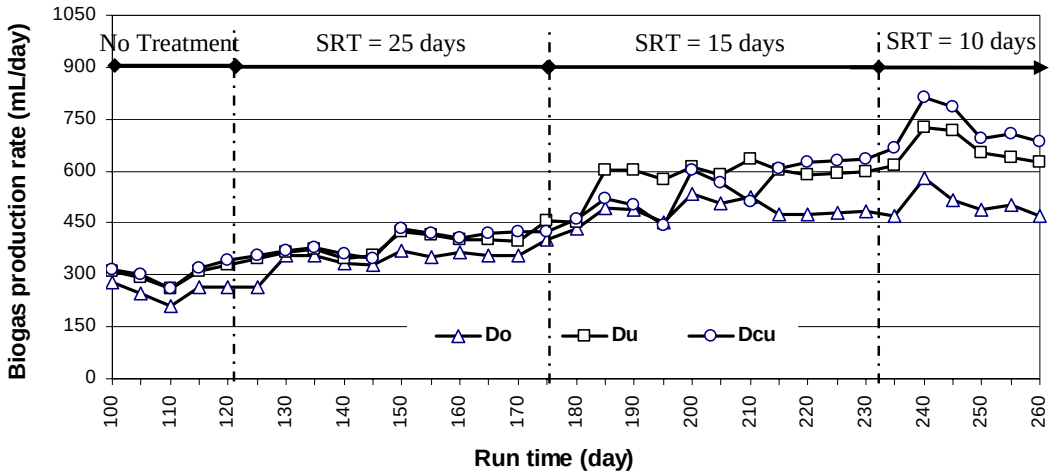


Figure 4.12 Methane production rate for all three digesters with digestion period

Figure 4.13 shows the comparison of methane production of all digesters at different SRTs. It clearly showed that methane production appreciably increased from 25 to 15 days SRT, while minor improvement was observed from 15 to 10 days SRT. Therefore, by considering both methane production and specific methane yield, SRT of 15 days was found to be the suitable detention time for effective sludge degradation.

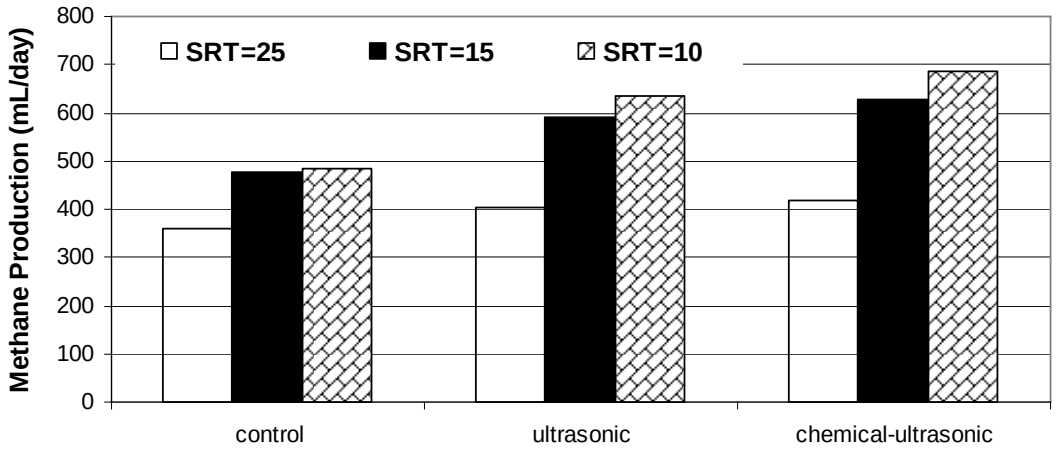


Figure 4.13 Comparison of average methane production at steady state condition with different SRTs

4.10 pH values and Alkalinity

The pH of feed sludge varied for the three digesters as the feed sludge was pretreated with ultrasound and NaOH (Table 4.5). The pH of digested sludge from Do digester was lower than Du and Dcu with all SRTs, while it was almost the same between Du and Dcu digester (Appendix C1). At every steady state condition, the pH of digested sludge from individual digester was almost the same at 25 and 15 days SRT, while it dropped down at 10 days SRT (Table 4.6). Although, pH dropped down at 10 days SRT, Du and Dcu digester could maintain their pH themselves within the range of 6.8 to 7.2, while addition

of $NaHCO_3$ was used for Do digester as its pH dropped to lower than 6.8 for some times. The dropping down of pH at shorter SRT is due to the high organic loading rate which results in high VFA concentration during the acidogenesis phase. The results obtained revealed that ultrasonicated and chemical-ultrasonicated sludge digester could maintain their pH themselves though higher organic loading was supplied.

The alkalinity of feed sludge and digested sludge varied significantly as pretreatments were used. Compared between the feed sludge and digested sludge, the alkalinity in all digesters increased during anaerobic digestion. The Dcu digester had higher alkalinity compared to Do and Du due to the addition of NaOH. The alkalinity of feed sludge was shown in Table 4.5 and of digested sludge in Table 4.6. Similarly to pH, the alkalinity reduced at shorter SRT which is due to the fact that more organic matter was supplied, it eventually increased the acid generation and subsequently neutralize the alkalinity capacity. The variation of pH and alkalinity with digestion time is graphically presented in Figure 4.14 (Appendix C1).

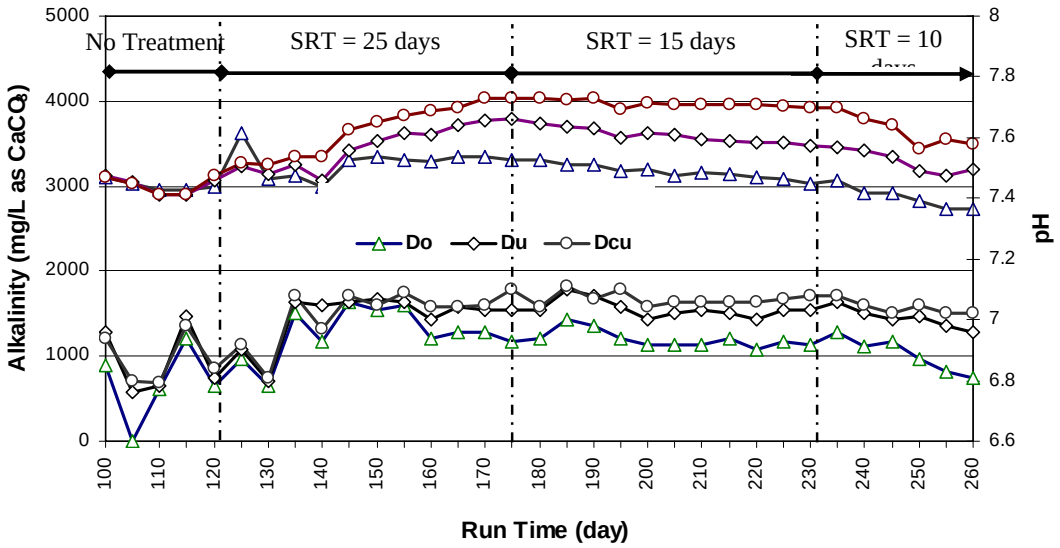


Figure 4.14 pH and alkalinity of digested sludge with digestion period

4.11 Dewaterability

The dewaterability evaluation was based on CST measurement. The CSTs of feed and digested sludges at different SRTs are shown in Table 4.5 and Table 4.6, respectively (Appendix C5). Figure 4.15 graphically presents the CST of fresh WAS and digested sludge from all digesters. The CST of feed sludges was found to be higher than original sludge (Fresh WAS). It indicated that ultrasonic and chemical-ultrasonic pretreatment reduced sludge dewaterability which due to the fact that pretreatment breaks the flocs to smaller size, it consequently clogs the cake during dewatering. Another reason is due to the increase of surface area, hence binding more surface water which is difficult to remove. This finding was also found by Chu et al. (2001) and Dewil et al. (2006).

The dewaterability of digested sludge from digester Du and Dcu improved by 59, 62 and 53% and by 64, 66 and 59% compared to feed sludge at 25, 15 and 10 days SRT, respectively. However, The CST of control digester increased to higher than fresh WAS as it was similarly found by Lin et al. (1997). According to the results obtained from this study, it was concluded that chemical-ultrasonic pretreatment gave higher dewaterability

improvement after digestion, followed by ultrasonic pretreatment and non-pretreatment. For all digesters, the descendant order of dewaterability of digested sludge occurred at 15, 25 and 10 days SRT. The lower dewaterabily at shorter SRT is due to insufficient time for biodegrading the smaller particle size which is solubilized by pretreatment processes. It consequently gives higher CST value.

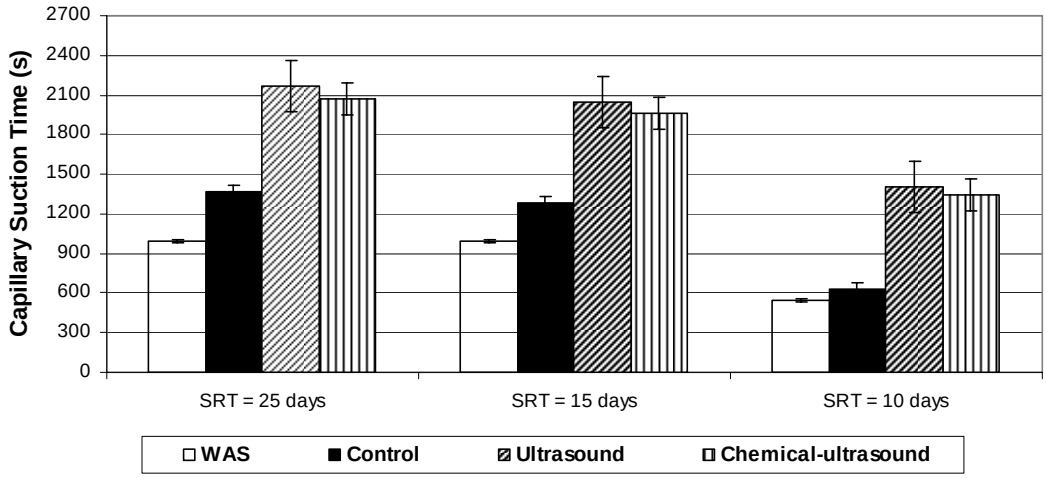


Figure 4.15 Dewaterability of digested sludges at different SRTs

4.12 Kinetic Study of Anaerobic Digestion

The kinetic study of anaerobic digestion was based on the degradation of biodegradable organic mass in the digester during the steady state condition. By combining the first order kinetic and mass balance from the digester, rate constant of the hydrolysis step can be expressed using the following formula:

$$k = \frac{1}{\theta}\left(\frac{L_o}{L} - 1\right) \qquad \text{Eq. 4.1}$$

Where,

L_o : Concentration of organics compounds of feed sludge (kg/m^3)
L : Concentration of organic compounds of digested sludge at steady state (kg/m^3)
k : Rate constant of the hydrolysis step (d^{-1})
θ : Time (d)

The rate constants of the hydrolysis step of each digester at each SRT were presented in Table 4.7.

Clearly, we can see that the degradation rate of all anaerobic digesters increased with reduction of SRT (Table 4.9). At all SRTs, the rate constant of chemical-ultrasonic digester was always higher than ultrasonic and control digester. This indicated that chemical-ultrasonic pretreatment makes more organic mass available for biological digestion. Hence, the organic mass was digested fast and higher biogas was produced. Amongst the three SRTs, the highest improvement of degradation rate was found at 15 days SRT from chemical-ultrasonic digester with the improvement of more than 52%, while ultrasonic digester improved by 26% compared to control digester, respectively. The rate constant improvement of ultrasonic and chemical-ultrasonic digester at 25 and 10 days SRT was

more or less same with lower than 38%. From results found, SRT of 15 days was considered to be the suitable digestion time for chemical-ultrasonicated sludge as well as ultrasonicated sludge because its improvement was more than 80% compared to the longest SRT and only 30% increased from second SRT to the shortest SRT (Figure 4.16).

Table 4.9 Rate constant of the hydrolysis step of anaerobic digesters

SRT		*Lo*	*L*			*k*		
			Do	Du	Dcu	Do	Du	Dcu
25	Max	3.24	2.68	2.61	2.50	0.010	0.012	0.013
	Min	3.20	2.59	2.51	2.44	0.008	0.009	0.011
15	Max	5.18	4.25	4.04	3.87	0.015	0.019	0.022
	Min	5.12	4.20	4.02	3.86	0.014	0.018	0.022
10	Max	8.12	6.77	6.53	6.35	0.023	0.024	0.029
	Min	7.86	6.51	6.40	6.19	0.018	0.023	0.027

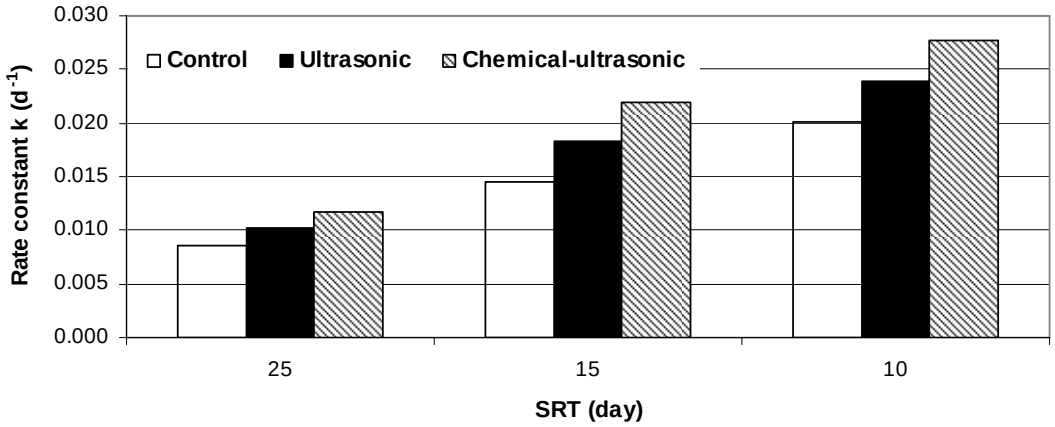


Figure 4.16 Rate constant of the hydrolysis step of anaerobic digesters

4.13 Energy Balance of Anaerobic Digester

The energy balance calculation procedure is attached in Appendix D1. Figure 4.17 shows the energy balance of anaerobic digester without inputting the energy consumption from pretreatments.

Energy balance of anaerobic digester was evaluated considering the energy required for heating sludge, for mixer and for heat loss. The energy balance calculation is summarized in Table 4.10. The results revealed that energy supply for mixer was the big consumer followed by required energy for heat loss. However, these two energy requirements were the same with all digesters. Therefore, they were not included in the specific energy gained calculation. According to three SRTs operation, it was found that digester of chemical-ultrasonicated sludge gave higher advantage of energy gained compared to other digesters. However, only energy gained from biogas at 25 days SRT of both pretreatment processes was sufficient enough to compensate the energy requirement for heating the sludge to

meshophilic temperature, whereas the untreated sludge digester always needed supplement energy (Figure 4.18).

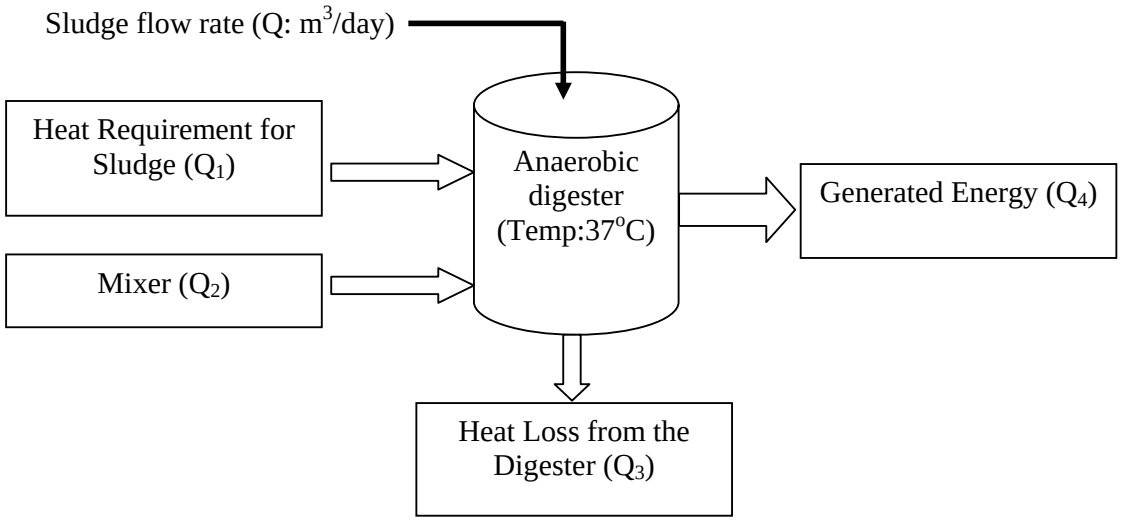


Figure 4.17 Energy Balance of anaerobic digester without pretreatment

From results found in Table 4.10, the energy obtained from biogas at 25 and 15 days SRT could totally supply to ultrasound unit (100%), whereas around 90% could achieve at 10 days SRT. However, it was not enough if we considered both energy requirements for heating sludge and ultrasound unit (Table 4.11). Figure 4.19 shows the different of energy obtained from biogas and energy consumption. At 25 days SRT, energy obtained from produced biogas from control, ultrasonic, chemical-ultrasonic digester could supply up to 95, 60 and 63%, respectively. At 15 days SRT, it was about 76, 53 and 56%, whereas around 52, 38 and 41% at 10 days SRT from same digester, respectively. By considering energy balance alone, control digester at 25 days SRT was better compared to other digesters as it could operate the system itself using the bioenergy recovery by more than 95%. The results found in this study were similar to result found by Braguglia and Gianico, 2008. The author found that the biogas produced with sonicated sludge can supply the energy requirement only at low OLR, 20 days SRT. Though, operating at longer SRT can tolerate the system, shorter SRT would be recommended if cost benefit analysis was taken into account. The economic analysis of the system with different pretreatment options is given in the next section.

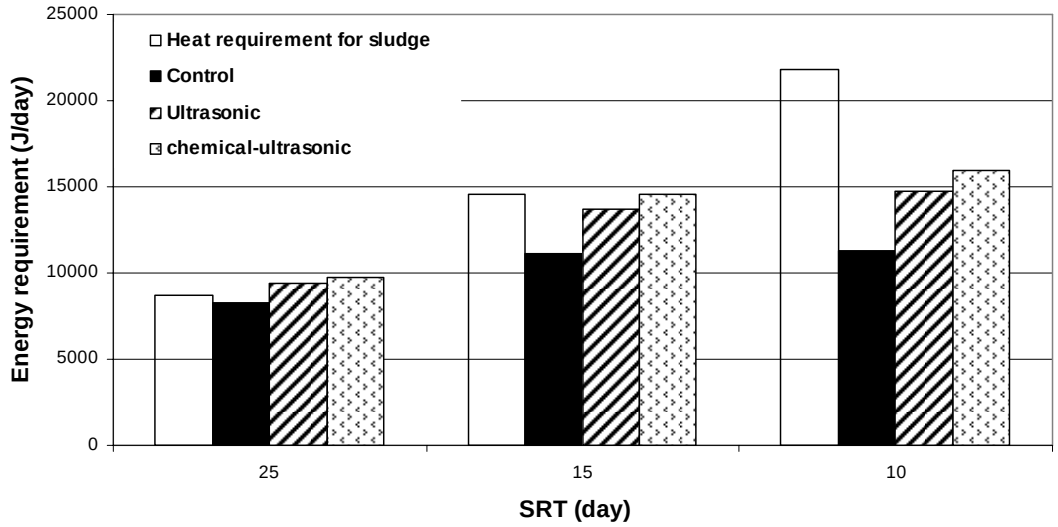


Figure 4.18 Comparison of energy from biogas and energy required for heating sludge

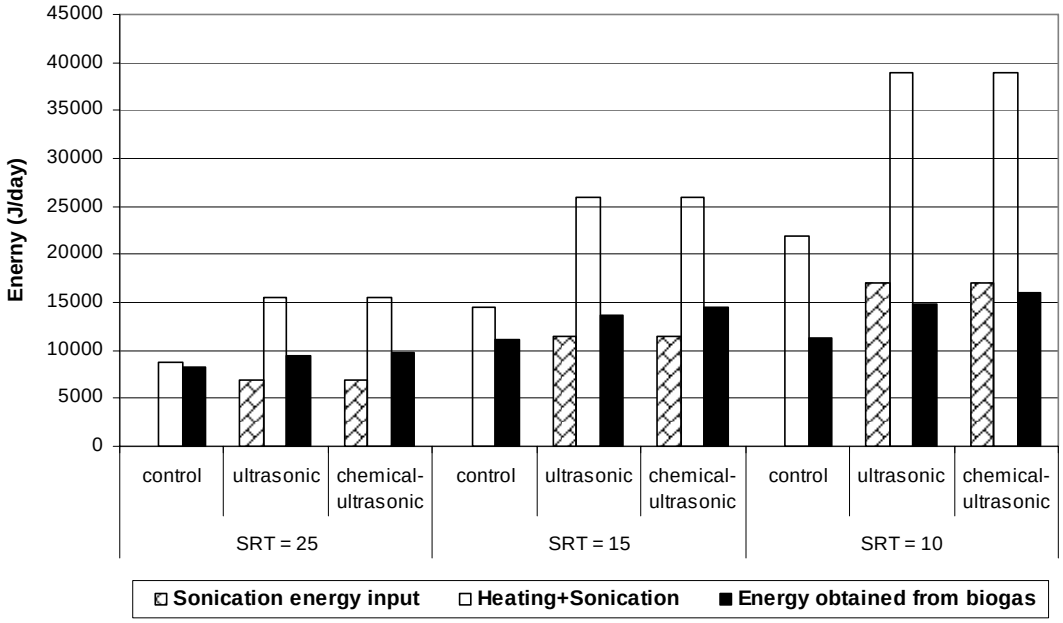


Figure 4.19 Compensation of energy gained from biogas for energy consumption

4.14 Economic Analysis

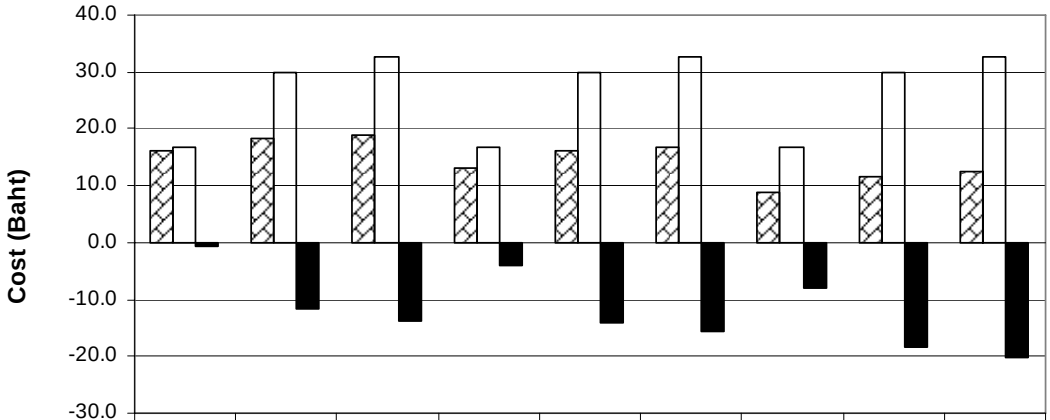
Economic analysis of anaerobic digestion with three different feed sludges was evaluated considering energy consumption cost, chemical cost, bioenergy recovery cost and cost from reducing sludge to landfill. The energy consumption cost includes energy cost for heating sludge and for ultrasonic unit, whereas the energy cost for mixer and heat loss were not taken into account for this evaluation. The sludge disposal cost, electricity cost and chemical cost used in this study was based on local data in Thailand. The detail calculation is presented in Appendix D2.

Table 4.10 Energy balance of anaerobic digester with pretreatment

Operating condition		Heat requirement for sludge (J/day)	Mixer (J/day)	Heat loss (J/day)	Ultrasound Unit (J/day)	Total Energy requirement (J/day)	Energy gained from CH_4 (J/day)
Digesters	SRT (day)	a	b	c	d	e = a+b+c	f
Control	25	8739	777600	174485	-	960824	8310
	15	14566	777600	174485	-	966650	11095
	10	21848	777600	174485	-	973933	11258
Ultrasonic	25	8739	777600	174485	6840	960824	9378
	15	14566	777600	174485	11400	966650	13672
	10	21848	777600	174485	17100	973933	14763
Chemical-ultrasonic	25	8739	777600	174485	6840	960824	9749
	15	14566	777600	174485	11400	966650	14531
	10	21848	777600	174485	17100	973933	15970

Table 4.11 Energy compensation of all digesters at different SRTs

Energy	SRT = 25 day			SRT = 15 day			SRT = 10 day		
	Control	Ultrasonic	Chemical-ultrasonic	Control	Ultrasonic	Chemical-ultrasonic	Control	Ultrasonic	Chemical-ultrasonic
Energy for heating sludge		8739			14566			21848	
Energy for ultrasound unit	0	6840	6840	0	11400	11400	0	17100	17100
Energy produced from biogas	8310	9378	9749	11095	13672	14531	11258	14763	15970
Balance	-429	-6202	-5830	-3470	-12294	-11435	-10591	-24186	-22979



	control	ultrasonic	che-ult	control	ultrasonic	che-ult	control	ultrasonic	che-ult
	SRT = 25			SRT = 15			SRT = 10		
☑ Income	16.1	18.3	18.9	13.0	16.0	16.9	8.7	11.5	12.4
□ Expense	16.9	30.1	32.6	16.9	30.1	32.6	16.9	30.1	32.6
■ Benefit	-0.8	-11.7	-13.7	-3.9	-14.0	-15.6	-8.1	-18.5	-20.2

Figure 4.20 Economic analysis of anaerobic digesters with different pretreatment options

Figure 4.20 illustrated the economic analysis of treating sludge 1L by anaerobic digestion with three different pretreatment options. The term income was the sum of bioenergy recovery cost and cost of reducing sludge to landfill, while the term expense was the sum of energy cost for heating sludge and for ultrasonic unit, and of chemical used. According to the results obtained, it was found that the energy cost for heating sludge was the first highest value followed by ultrasonic unit and chemical used. Hence, operating cost at shorter SRT was relatively high compared to long SRT, due to more sludge was fed into the digesters. At longest SRT of 25 days, the income was almost same the expenses from control digester, while the income from ultrasonic and chemical-ultrasonic digester was less than 40% compared to expense. At 15 days SRT, the income of control, ultrasonic and chemical-ultrasonic digester was around 77, 53 and 52% compared to the expense, respectively. The worse case was at shortest SRT which was less than 52% and around 38% from control and other two digesters, respectively. With respect to this economic analysis, anaerobic digester without pretreatment at 25 days SRT was the most feasible one compared to other two digesters. However, it was expected to be better at 15 days SRT with ultrasonic pretreatment once capital and maintenance costs of the system were taken into account. Operating the digester at 15 days SRT, the reactor volume will be reduced, it consequently reduced the investment cost (land, equipments, construction and energy consumption) compared to 25 days SRT. It would be more benefit when landfilling becomes tougher and tougher as it produces less sludge volume after digestion.

Chapter 5

Conclusions and Recommendations

5.1 Conclusions

The study mainly focused on the effectiveness of pretreatment options for the improvement of anaerobic digestion. The effects of ultrasonic, chemical and chemical-ultrasonic on sludge disintegration were investigated in different conditions. The results were found that the pretreatments gave significant effects on sludge solubilization which resulted in high soluble organic substances in the aqueous phase. Hence, it improved the performance of subsequent anaerobic digestion in term of biogas production, and TS and VS destruction. The important findings are summarized as followed:

1. The better sludge disintegration was observed at longer sonication time and high power input. However, 190 W of power input and 60 s of sonication duration which correspond to 3.8 kJ/g TS was considered to be the optimum condition since it gave relatively high SCOD at low specific energy input.

2. Chemical pretreatment using NaOH gave a significant effect on sludge disintegration at short holding time of 6 min. The optimum chemical dose was found at 50 mg/g TS since the increasing trend of SCOD started slowing down when higher dose was supplied.

3. Chemical-ultrasonic pretreatment gave higher SCOD release at high chemical dose and high energy input. However, it was found that operating with 10 mg/g TS chemical dose at specific energy input of 3.8 kJ/g TS was effective on sludge disintegration.

4. Amongst three pretreatment options, chemical-ultrasonic was found to be the most effective technique on sludge disintegration. It could release the %SCOD by 18%, while 13.5 and 13% could be achieved from chemical and ultrasonic treatment respectively. The higher efficiency of chemical-ultrasonic is due to the combination effects of hydro-mechanical shear force and OH^- radical reaction.

5. The pretreatments enhanced the subsequent anaerobic digestibility of WAS with significantly high TS and VS destruction, and biogas production. The highest TS removal improvement was found from ultrasonic digester with the value of 16.6%. However, the highest VS removal was achieved in chemical-ultrasonic digester with the value of 24.8%.

6. From all digesters, the biogas production significantly increased from 25 to 15 days SRT. Thereafter, the increasing trend slowed down at shorter SRT. At 15 days SRT, the methane production of ultrasonic and chemical-ultrasonic digester increased by 23 and 31%, respectively when compared to control digester.

7. The methane content of all digesters was observed to be more or less same with the value of 59%. The results revealed that there was no improvement of methane content in the biogas though pretreatments were used.

8. Amongst three operated SRTs, 15 days was considered to be the suitable digestion time for all digesters in term of biogas production, and TS and VS removal.

9. According to kinetic study result, it was found that the rate constant of the hydrolysis step of chemical-ultrasonicated sludge was higher than ultrasonicated and non-pretreated sludge. It indicated that chemical-ultrasonic pretreatment make more organic mass available for biological digestion. Hence, the degradation rate was faster compared to others which eventually reduce the digester volume for same digestion efficiency.

10. The results revealed that the dewaterability of pretreated sludge was significantly higher than non-pretreated sludge. However, it improved after anaerobic digestion. At the optimum of 15 days SRT, the digested sludge from chemical-ultrasonic and ultrasonic digester improved by 66 and 62% compared to feed sludge.

11. According to the energy calculation, energy requirement for mixer was found to be the first highest energy consumer followed by heat loss for maintaining the temperature of the digester. However, heat loss during digestion could be minimized by modifying the configuration of the system and/or using any techniques for preventing the heat.

12. Based on energy balance, energy obtained from methane gas from all digesters was sufficient enough for either heating sludge to meshophilic temperature or supplying to ultrasonic unit at 25 days SRT. However, it was not enough to compensate both energy used for heating sludge and ultrasonic unit. Only at 25 days SRT, energy obtained from biogas in Do digester could replace by 95%, whereas Du and Dcu digesters could replace only 60 and 63% of the energy used respectively. At 15 and 10 days SRT, the percentage replacement of energy was even lower than 25 days SRT. Thus, operating at 25 days SRT of non-pretreated sludge was the best in term of energy balance alone.

13. Economic analysis revealed that only control digester at 25 days SRT was economically viable since the income and expense was almost the same. At the same SRT, the income of ultrasonic and chemical-ultrasonic digester was less than 30% compared to expense. However, 15 days SRT with chemical-ultrasonicated sludge would be better once capital and maintenance cost of the system were taken into account.

5.2 Recommendations for Further Research

Based on these research experimental results, the following recommendations are proposed for further research:

1. TS content affects ultrasonication efficiency and anaerobic digester performance, therefore the effects of TS of WAS should be thoroughly studied. Different TS content gives different optimum specific energy input. Thus, optimum Es of different TS content should be compared and evaluated. Similarly, anaerobic digestion of different TS content feed sludge should also be investigated in order to select the correct TS content for ultrasonic operation.

2. The factors affecting the efficiency of ultrasonication were pH, sludge concentration, ultrasonic intensity and specific energy input. Thus, the mathematical modeling of the combination effects of these factors should be further focused and find out the reliable correlation for evaluating sludge disintegration for all type of sludge.

3. EPS of WAS affect the efficiency of the ultrasonication. It should be thoroughly investigated in order to see the effects of EPS on sludge disintegration couple with energy input.

4. The ratio of fresh and pretreated WAS for anaerobic digester should be investigated to find out the suitable ratio. BMP test is a useful method to achieve this goal.

5. The chemical-ultrasonic pretreatment is affected by many factors such as chemical pretreatment duration, chemical dose, and ultrasonic operating conditions. Therefore, optimization of each parameter should be investigated, particularly at relatively high chemical dose and varying specific energy inputs.

6. According to practical aspect of chemical-ultrasonic pretreatment, simultaneous chemical and ultrasonic process should be investigated in order to compare the efficiency of sludge disintegration to chemical followed by ultrasound process. This pretreatment process might be more effective and incur low operating cost as it does not need the mixer.

7. SCOD measurement is performed with the filtrate through membrane filter of 0.45μm pore size. The bio-polymer (pore size >0.45μm) which releases within intracellular substances during pretreatment retains on the filter. Therefore, SCOD release will be low which does not fully represent the effect of pretreatment. To get a clear evaluation of sludge disintegration, combination SCOD, NH_3-N, protein release should be investigated.

8. Acid reagent can also use to disintegrate the sludge. The investigation on acid disintegration should be investigated to compare its efficiency to other disintegration techniques.

9. The rate constant of the hydrolysis step in this study is a simple kinetic study. It should be further studied in a deep detail of the biodegradable organic fraction of WAS during the digestion.

10. In order to get a reliable cost benefit analysis of ultrasonic and chemical ultrasonic application, pilot scale experiment should be conducted.

11. Digested sludge be should further studied. It would be better to investigate the quality of the digested sludge whether it can meet the discharge standard of the country or can be used as a raw material for co-composting. It is also necessary to study the calorific value of the digested sludge in case further treatment by incineration is used.

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Appendices

Appendix A

Pretreatment Optimization

Appendix A1 Ultrasonic density and specific energy input calculation (Ultrasonic pretreatment)

Sonication Time (s)	Sample Volume (mL)	TS content (%)	Energy Input (J)	Power Input (W)	Ultrasonic Density (W/mL)	Specific Energy Input (kJ/g TS)
a	b	c	d	e= d/a	f = e/b	g = d/(b*c*10)
30	100	3	5746	191.5	1.9	1.92
60	100	3	11508	191.8		3.84
120	100	3	22964	191.4		7.65
30	100	3	4522	150.7	1.5	1.51
60	100	3	9178	153.0		3.06
120	100	3	18101	150.8		6.03
30	100	3	3116	103.9	1.0	1.04
60	100	3	6113	101.9		2.04
120	100	3	12413	103.4		4.14
30	100	3	1493	49.8	0.5	0.50
60	100	3	3173	52.9		1.06
120	100	3	6204	51.7		2.07

Ultrasonic Density: $UD_{avg} = \frac{P_{avg}}{V}$ Specific Energy Input: $E_S = \frac{P}{V.TS} xt$

Appendix A2 Specific energy input calculation for chemical-ultrasonic pretreatment

Sonication times (s)	Sludge Volume (mL)	TS Content (%)	Chemical Dose (mg/g TS)	Energy Input (J)	Power Input (W)	Specific Energy Input (kJ/g TS)
a	b	c	d	e	f = e/a	g = e/(b*c*10)
30	100	3	10	5656	189	1.89
	100	3	15	5620	187	1.87
	100	3	20	5662	189	1.89
	100	3	25	5692	190	1.90
60	100	3	10	11680	195	3.89
	100	3	15	11478	191	3.83
	100	3	20	11480	191	3.83
	100	3	25	11644	194	3.88
120	100	3	10	22070	184	7.36
	100	3	15	22040	184	7.35
	100	3	20	22040	184	7.35
	100	3	25	21705	181	7.24

Ultrasonic Density: $UD_{avg} = \frac{P_{avg}}{V}$

Specific Energy Input: $E_S = \frac{P}{V.TS} xt$

Appendix A3 SCOD release with sonication times at different ultrasonic densities

Sonication Duration	SCOD	SCOD/TS	Specific Energy Input
[s]	[mg/L]	[mg/g]	[kJ/g TS]
UD : 1.9 W/mL			
0	458	15	0
30	2182	73	1.9
60	5498	183	3.8
120	7767	259	7.7
UD : 1.5 W/mL			
0	458	15	0
30	2007	67	1.5
60	3927	131	3.1
120	7986	266	6.0
UD : 1.0 W/mL			
0	458	15	0
30	975	33	1.0
60	1650	55	2.0
120	3600	120	4.1
UD : 0.5 W/mL			
0	458	15	0
30	525	18	0.5
60	1200	40	1.1
120	1800	60	2.1

Appendix A4 SCOD release with holding times at different chemical doses

Holding Time	SCOD	SCOD/TS	pH
[min]	[mg/L]	[mg/g]	-
Chemical dose : 75 mg/g TS			
0	458	15	-
5	7414	247	11.65
10	7984	266	11.6
30	9695	323	11.7
60	10646	355	11.68
Chemical dose : 50 mg/g TS			
0	458	15	-
5	5703	190	11
10	6463	210	11.19
30	6653	215	11.05
60	6584	218	11.1
Chemical dose : 25 mg/g TS			
0	458	15	-
5	2004	67	9.14
10	2863	95	9.12
30	2672	89	9.25
60	2863	95	9.09
Chemical dose : 10 mg/g TS			
0	458	15	-
5	950	32	7.24
10	1250	42	7.5
30	1375	46	7.32
60	1900	63	7.64

Appendix A5 SCOD release with sonication times at different chemical doses

Sonication Duration	SCOD	SCOD/TS	Specific Energy Input	pH	
[s]	[mg/L]	[mg/g]	[kJ/g TS]	Before sonication	After sonication
Chemical dose: 25 mg/g TS					
0	458	15	0	-	-
30	6249	208	1.90	8.83	8.39
60	9183	306	3.88	8.76	8.16
120	12118	404	7.24	8.9	7.82
Chemical dose: 20 mg/g TS					
0	458	15	0	-	-
30	5738	191	1.90	8.83	8.39
60	9017	301	3.83	8.36	7.99
120	12478	416	7.35	8.27	7.39
Chemical dose: 15 mg/g TS					
0	458	15	0	-	-
30	4827	161	1.87	7.63	7.45
60	7560	252	3.83	7.62	7.4
120	11567	386	7.35	7.81	7.24
Chemical dose: 10 mg/g TS					
0	458	15	0	-	-
30	3938	131	1.89	7.24	7.13
60	7621	254	3.89	7.32	7.01
120	10178	339	7.36	7.5	6.95

Appendix B

Biochemical Methane Potential (BMP) results

Appendix B1: Biochemical methane potential along with digestion time of chemical-ultrasonicated sludge with different chemical doses

Run Time (day)	25 mg/g TS			20 mg/g TS			15 mg/g TS			10 mg/g TS			WAS		
	CH_4 (%)	CH_4 (mL)	Cumulative CH_4 (mL)	CH_4 (%)	CH_4 (mL)	Cumulative CH_4 (mL)	CH_4 (%)	CH_4 (mL)	Cumulative CH_4 (mL)	CH_4 (%)	CH_4 (mL)	Cumulative CH_4 (mL)	CH_4 (%)	CH_4 (mL)	Cumulative CH_4 (mL)
3	24.7493	3.30	8.58	25.5021	3.50	8.37	25.4524	3.89	9.90	24.8994	3.90	10.81	24.3137	3.88	8.42
6	42.5921	5.18	24.36	42.8623	2.63	19.65	43.0821	4.67	24.24	42.5521	4.32	26.29	39.1992	3.14	21.81
9	44.3080	2.81	35.16	44.3906	2.67	27.62	45.6961	3.40	35.72	44.5643	2.60	35.80	41.2877	1.99	28.92
12	46.5278	1.75	41.32	48.2020	1.02	32.60	46.9660	1.55	41.89	47.6185	1.79	41.32	43.4533	1.80	34.90
15	49.0336	2.00	46.83	49.7727	2.14	36.79	46.9660	1.55	46.55	47.6185	1.79	46.68	43.4533	1.80	40.30
18	49.0336	2.00	52.85	49.7727	2.14	43.21	49.5701	2.30	53.44	47.6060	2.16	53.14	45.3553	1.72	45.45
21	49.6703	1.16	56.32	51.4087	1.14	46.63	50.8153	1.43	57.73	49.6519	1.35	57.20	47.1143	1.39	49.60
24	49.6703	1.16	59.80	51.4087	1.14	50.05	50.8153	1.43	62.01	49.6519	1.35	61.26	47.1143	1.39	53.76
27	50.4317	1.14	63.23	51.3855	0.92	52.81	49.6813	1.13	65.39	50.4165	1.08	64.49	49.3929	1.28	57.61
30	50.9156	0.68	66.20	51.5839	0.42	55.08	34.3452	0.39	68.03	51.8428	0.60	67.24	50.9543	0.79	60.97
33	50.9156	0.68	68.24	51.5839	0.42	56.35	34.3452	0.39	69.21	51.8428	0.60	69.06	50.9543	0.79	63.33
36	51.9054	0.48	70.07	53.4200	0.37	57.57	34.6797	0.68	70.68	51.8428	0.60	70.87	50.9543	0.79	65.69
39	51.9054	0.48	71.51	53.4200	0.37	58.68	34.6797	0.68	72.72	51.8428	0.60	72.68	50.9543	0.79	68.05
42	51.9054	0.48	72.96	53.4200	0.37	59.78	50.5146	0.50	74.40	51.7460	0.44	74.16	51.4059	0.46	69.77
45	51.9054	0.48	74.40	53.4200	0.37	60.89	50.5146	0.50	75.90	51.7460	0.44	75.48	51.4059	0.46	71.15
48	51.9054	0.48	75.84	53.4200	0.37	61.99	50.5146	0.50	77.40	51.7460	0.44	76.79	51.4059	0.46	72.54

51	51.9054	0.48	77.28	53.4200	0.37	63.10	50.5146	0.50	78.90	51.7460	0.44	78.11	51.4059	0.46	73.92
54	52.2900	0.32	78.56	53.2874	0.28	64.11	53.1786	0.35	80.25	52.4550	0.36	79.34	52.8606	0.42	75.26
57	52.2900	0.32	79.52	53.2874	0.28	64.96	53.1786	0.35	81.31	52.4550	0.36	80.43	52.8606	0.42	76.52
60	52.2900	0.32	80.49	53.2874	0.28	65.81	53.1786	0.35	82.36	52.4550	0.36	81.51	52.8606	0.42	77.78
63	52.2900	0.32	81.45	53.2874	0.28	66.65	53.1786	0.35	83.41	52.4550	0.36	82.59	52.8606	0.42	79.03
66	52.2900	0.32	82.41	53.2874	0.28	67.50	53.1786	0.35	84.47	52.4550	0.36	83.68	52.8606	0.42	80.29
69	52.2900	0.32	83.37	53.2874	0.28	68.35	53.1786	0.35	85.52	52.4550	0.36	84.76	52.8606	0.42	81.55
72	52.2900	0.32	84.33	53.2874	0.28	69.19	53.1786	0.35	86.57	52.4550	0.36	85.85	52.8606	0.42	82.80
75	52.2900	0.32	85.29	53.2874	0.28	70.04	53.1786	0.35	87.63	52.4550	0.36	86.93	52.8606	0.42	84.06
78	52.2900	0.32	86.26	53.2874	0.28	70.88	53.1786	0.35	88.68	52.4550	0.36	88.01	52.8606	0.42	85.32
81	52.2900	0.32	87.22	53.2874	0.28	71.73	53.1786	0.35	89.73	52.4550	0.36	89.10	52.8606	0.42	86.57
84	52.2900	0.32	88.18	53.2874	0.28	72.58	53.1786	0.35	90.79	52.4550	0.36	90.18	52.8606	0.42	87.83

Appendix B2: Cumulative methane production after blank adjustment at mesophilic and STP

Chemical-dose (mg/g TS)	Methane (%)	Head space volume (mL)	Methane in head space (mL)	Cumulative Methane (mL)	Methane of Blank (mL)	Total Methane (mL)	Methane (STP) (mL)
control	52.8606	90	47.57	87.83	12.66	122.74	108.91
10	52.4550	90	47.21	90.18	12.66	124.73	110.67
15	53.0983	90	47.79	90.79	12.66	125.92	111.73
20	52.9793	90	47.68	72.58	12.66	107.60	95.48
25	52.2900	90	47.06	88.18	12.66	122.58	108.77

Appendix C

Digester Performance

Appendix C1 Feeding, Withdrawal, pH and Alkalinity of digested sludge with digestion times

Date	Run times	Feeding	Withdrawal	pH			Alkalinity		
				Do	Du	Dcu	Do	Du	Dcu
	[day]	[mL]	[mL]	-	-	-	[mg/L]	[mg/L]	[mg/L]
14-Jul-07	0	0	0	7.37	7.53	7.57	-	-	-
18-Jul-07	5	10	0	7.21	7.25	7.28	-	-	-
23-Jul-07	10	20	0	7.21	7.21	7.19	-	-	-
28-Jul-07	15	20	0	7.12	7.08	7.06	-	-	-
2-Aug-07	20	30	0	6.98	6.95	7.02	1860	1780	1800
7-Aug-07	25	30	0	6.96	6.98	7.07	1910	1780	1900
12-Aug-07	30	40	0	6.98	6.97	7.14	2850	3050	3850
17-Aug-07	35	46	46	7.06	7.08	7.17	2719	3175	3900
22-Aug-07	40	50	50	7.02	6.91	7.07	3350	3300	3875
27-Aug-07	45	55	50	7.01	7.00	7.38	3250	3050	2638
1-Sep-07	50	70	70	6.92	6.93	7.20	2950	3263	3925
6-Sep-07	55	80	80	7.06	6.9	7.17	2650	3075	3500
11-Sep-07	60	90	90	7.02	6.89	7.12	2725	3100	3600
16-Sep-07	65	90	90	6.75	6.85	6.86	2850	3075	3438
21-Sep-07	70	90	90	6.84	6.87	6.92	2825	3038	3275
26-Sep-07	75	100	100	6.83	6.83	6.97	3050	3025	3425
1-Oct-07	80	100	100	6.8	6.84	6.93	3025	3200	3375
6-Oct-07	85	110	110	6.75	6.79	6.85	3100	3100	3300
11-Oct-07	90	110	110	6.69	6.79	6.87	3175	3200	3300
16-Oct-07	95	110	110	6.65	6.85	7.03	3000	2850	3200
21-Oct-07	100	110	110	6.85	6.96	6.94	3100	3125	3100
26-Oct-07	105	120	120	6.60	6.76	6.8	3025	3050	3025
31-Oct-07	110	120	120	6.77	6.78	6.79	2950	2900	2900

5-Nov-07	115	120	120	6.94	7.01	6.98	2950	2900	2900
10-Nov-07	120	120	120	6.78	6.81	6.84	3000	3075	3125
15-Nov-07	125	120	120	6.87	6.90	6.92	3625	3238	3275
20-Nov-07	130	120	120	6.78	6.80	6.81	3088	3150	3250
25-Nov-07	135	120	120	7.02	7.06	7.08	3125	3250	3350
30-Nov-07	140	120	120	6.93	7.05	6.97	3000	3075	3350
5-Dec-07	145	120	120	7.06	7.06	7.08	3313	3413	3663
10-Dec-07	150	120	120	7.03	7.07	7.05	3338	3538	3750
15-Dec-07	155	120	120	7.05	7.06	7.09	3300	3625	3825
20-Dec-07	160	120	120	6.94	7.00	7.04	3288	3600	3888
25-Dec-07	165	120	120	6.96	7.04	7.04	3338	3713	3925
30-Dec-07	170	120	120	6.96	7.03	7.05	3350	3775	4038
4-Jan-08	175	150	150	6.93	7.03	7.1	3313	3788	4025
9-Jan-08	180	185	185	6.94	7.03	7.04	3300	3738	4038
14-Jan-08	185	200	200	7.00	7.10	7.11	3250	3700	4013
19-Jan-08	190	200	200	6.98	7.08	7.07	3250	3688	4025
24-Jan-08	195	200	200	6.94	7.04	7.10	3175	3575	3900
29-Jan-08	200	200	200	6.92	7.00	7.04	3188	3625	3975
3-Feb-08	205	200	200	6.92	7.02	7.06	3125	3600	3963
8-Feb-08	210	200	200	6.92	7.03	7.06	3163	3550	3950
13-Feb-08	215	200	200	6.94	7.02	7.06	3150	3525	3950
18-Feb-08	220	200	200	6.9	7.00	7.06	3100	3513	3950
23-Feb-08	225	200	200	6.93	7.03	7.07	3088	3513	3938
28-Feb-08	230	200	200	6.92	7.03	7.08	3025	3475	3925
4-Mar-08	235	240	240	6.96	7.06	7.08	3075	3463	3913
9-Mar-08	240	300	300	6.91	7.02	7.05	2925	3425	3800
14-Mar-08	245	300	300	6.93	7.00	7.02	2913	3350	3725
19-Mar-08	250	300	300	6.87	7.01	7.05	2825	3175	3438
24-Mar-08	255	300	300	6.83	6.98	7.02	2725	3125	3550
29-Mar-08	260	300	300	6.81	6.96	7.02	2725	3200	3500
31-Mar-08	262	300	300	6.77	6.95	6.98	2638	3100	3475

Appendix C2 Biogas and methane production and methane composition

Date	Run times (days)	Biogas Production (mL/day)								
		Do			Du			Dcu		
		Biogas Production	Methane Content	Methane production	Biogas Production	Methane Content	Methane production	Biogas Production	Methane Content	Methane production
		[mL/day]	[%]	[mL/day]	[mL/day]	[%]	[mL/day]	[mL/day]	[%]	[mL/day]
18-Jul-07	5	89.0	4.7125	4.2	110.6	4.7261	5.2	0	0	0
23-Jul-07	10	90.0	11.6688	10.5	91.9	12.5130	11.5	94.9	2.0011	1.9
28-Jul-07	15	88.3	17.8104	15.7	89.5	18.9457	16.9	138.0	9.5267	2.8
2-Aug-07	20	143.6	27.0720	42.3	141.7	27.2914	45.1	154.3	22.6770	44.6
7-Aug-07	25	142.9	46.4516	66.4	142.9	33.9017	66.0	162.9	35.9393	73.2
12-Aug-07	30	142.9	46.4516	66.4	142.9	46.1652	66.0	162.9	44.9400	73.2
17-Aug-07	35	260.5	52.7436	137.4	269.5	52.1022	140.4	286.8	52.0446	149.2
22-Aug-07	40	250.1	53.8852	134.8	282.1	56.5626	159.6	289.5	58.1338	168.3
27-Aug-07	45	250.1	53.8852	134.8	282.1	56.5626	159.6	289.5	58.1338	168.3
1-Sep-07	50	307.6	58.4513	179.8	342.1	57.4436	196.5	345.7	34.0549	117.7
6-Sep-07	55	403.8	58.7626	237.3	420.0	56.0547	235.4	426.2	49.8214	212.3
11-Sep-07	60	420.8	51.2180	245.7	431.3	56.0557	243.2	439.2	55.9719	248.0
16-Sep-07	65	429.8	51.2180	220.1	432.1	55.3188	239.0	436.3	53.1391	231.9
21-Sep-07	70	429.8	51.2180	220.1	432.1	55.3188	239.0	436.3	53.1391	231.9
26-Sep-07	75	500.8	49.4189	247.5	542.5	55.0185	302.3	534.8	55.2366	300.9
1-Oct-07	80	521.6	51.2984	267.6	536.4	55.6841	298.7	524.0	57.0845	299.1
6-Oct-07	85	548.6	48.7708	267.6	576.4	54.7964	315.8	546.6	57.0083	311.6

11-Oct-07	90	551.0	46.0274	253.6	557.6	56.7095	316.2	516.0	56.7095	292.6
16-Oct-07	95	555.8	44.7804	248.9	575.0	52.1702	300.0	558.0	56.6551	316.1
21-Oct-07	100	606.7	45.8190	278.0	573.3	54.2392	311.0	569.6	55.1018	313.8
26-Oct-07	105	540.0	45.3935	245.1	551.0	53.3810	294.1	553.6	54.2209	300.2
31-Oct-07	110	500.0	44.1346	209.7	500.0	52.4969	262.5	500.0	52.4898	262.4
5-Nov-07	115	621.1	42.6783	265.1	573.9	53.3470	310.3	630.5	51.4589	320.4
10-Nov-07	120	584.0	44.9854	262.7	630.6	52.5946	328.7	658.8	51.7908	341.6
15-Nov-07	125	557.0	47.7727	266.1	645.3	53.6867	346.5	681.5	52.0229	354.5
20-Nov-07	130	652.1	49.4029	356.5	655.8	55.8033	365.9	678.8	54.3035	368.6
25-Nov-07	135	633.3	56.2324	356.1	661.0	56.2958	372.1	700.0	54.3597	380.5
30-Nov-07	140	597.5	55.9533	334.3	618.9	55.9363	346.2	667.7	54.3176	362.7
5-Dec-07	145	587.6	56.2969	330.8	633.8	56.4538	357.8	623.3	55.6866	347.1
10-Dec-07	150	651.7	56.9415	371.1	751.0	56.8292	426.8	773.4	56.0248	433.3
15-Dec-07	155	612.3	57.2253	350.4	740.3	55.8931	413.8	755.5	55.4234	418.7
20-Dec-07	160	633.3	57.5044	364.2	706.9	56.6139	400.2	706.6	57.6841	407.6
25-Dec-07	165	617.1	57.8912	357.3	712.0	56.6139	403.1	726.2	57.6841	418.9
30-Dec-07	170	608.8	58.2016	354.3	692.5	56.6139	396.9	721.1	57.6841	423.1
4-Jan-08	175	691.9	57.7710	399.7	806.8	56.7567	457.9	737.9	57.7976	426.5
9-Jan-08	180	738.0	58.6924	433.1	776.6	58.1619	451.7	788.9	58.4367	461.0
14-Jan-08	185	844.5	58.4833	493.9	1043.0	57.7981	602.8	892.3	58.2322	519.6
19-Jan-08	190	845.1	57.8639	489.0	1046.5	57.4168	600.9	865.4	58.0802	502.6
24-Jan-08	195	778.3	58.2970	453.7	997.6	57.4611	573.2	764.4	58.1237	444.3
29-Jan-08	200	914.3	58.2029	532.1	1056.4	57.7265	609.8	1033.3	58.2767	602.2

3-Feb-08	205	867.9	58.5549	508.2	1022.7	57.7265	590.4	974.6	58.2767	568.0
8-Feb-08	210	891.8	58.6976	523.4	1102.5	57.7169	636.3	880.5	58.2270	512.7
13-Feb-08	215	831.5	57.1730	475.4	1046.4	57.4137	600.8	1063.6	57.1559	607.9
18-Feb-08	220	818.0	58.0232	474.6	1012.2	58.1699	588.8	1077.6	57.8608	623.5
23-Feb-08	225	826.1	58.2784	481.1	1024.7	57.8045	592.3	1097.0	57.4258	630.0
28-Feb-08	230	815.0	59.1219	481.8	1032.5	57.7943	596.7	1069.8	59.1717	633.0
4-Mar-08	235	808.5	58.3588	471.9	1070.7	57.7024	617.8	1120.6	59.3607	665.2
9-Mar-08	240	980.7	59.0569	579.2	1255.8	57.9658	727.9	1363.5	59.5849	812.4
14-Mar-08	245	878.2	58.8947	517.2	1214.3	58.9602	716.0	1318.8	59.4709	784.3
19-Mar-08	250	840.6	58.0562	488.0	1106.2	59.2248	655.1	1178.4	58.9792	695.0
24-Mar-08	255	867.8	58.0562	503.8	1083.0	59.2248	641.4	1200.8	58.9792	708.2
29-Mar-08	260	811.5	58.0562	471.1	1058.8	59.2248	627.1	1161.5	58.9792	685.0
30-Mar-08	261	811.5	58.0562	471.1	1058.8	59.2248	627.1	1161.5	58.9792	685.0
31-Mar-08	262	826.8	58.0562	480.0	1038.0	59.2248	615.0	1169.9	58.9792	690.0

Appendix C3 TS of feed and digested sludge and its removal efficiency

Run times (days)	Feeding (%)	Digested sludge (%)			% TS removal		
		Do	Du	Dcu	Do	Du	Dcu
35	3.01	3.36	3.16	3.24	-11.7	-5.1	-7.6
40	3.01	3.01	3.38	3.23	-0.1	-12.3	-7.3
45	3.01	3.09	3.28	3.23	-2.6	-8.9	-7.2
50	3.02	3.03	3.23	3.09	-0.4	-6.9	-2.3
55	3.02	2.94	3.20	3.03	2.7	-6.1	-0.3
60	3.02	2.97	3.19	3.04	1.8	-5.7	-0.6
65	3.02	3.12	3.18	3.19	-3.4	-5.2	-5.6
70	3.02	3.12	3.18	3.19	-3.45	-5.22	-5.63
75	3.02	3.38	3.11	3.17	-11.86	-2.91	-4.83
80	3.02	3.35	3.12	3.16	-10.9	-3.2	-4.7
85	3.02	3.41	3.09	3.12	-13.6	-3.0	-3.8
90	3.00	3.53	3.14	3.15	-17.6	-4.8	-5.0
95	3.00	3.62	3.12	3.09	-20.8	-3.9	-3.1
100	3.00	3.58	3.03	2.99	-19.2	-0.9	0.5
105	3.00	3.74	3.05	2.98	-24.8	-1.7	0.5
110	3.00	3.79	3.16	3.10	-26.3	-5.4	-3.2
115	3.00	3.56	2.88	2.90	-19.2	3.8	3.1
120	2.99	3.49	2.91	2.90	-16.7	2.7	3.0
125	2.99	3.43	2.90	2.90	-14.6	3.1	3.1
130	2.94	3.38	2.90	2.90	-14.9	1.5	1.2
135	2.94	3.23	2.88	2.90	-9.7	2.1	1.3
140	3.01	3.15	2.83	2.93	-4.6	5.9	2.6
145	3.02	3.15	2.85	2.99	-4.2	5.8	0.9
150	3.01	3.05	2.74	2.97	-1.2	8.9	1.3
155	3.01	3.01	2.74	3.03	0.0	9.0	-0.7
160	3.10	3.12	2.86	3.08	-0.5	7.8	0.6
165	3.10	2.98	2.71	3.02	3.6	12.5	2.5
170	3.10	2.99	2.72	3.03	3.5	12.2	2.4
175	3.10	2.98	2.70	3.02	3.8	12.7	2.4
180	3.00	2.96	2.68	3.03	1.4	10.8	-1.0
185	3.00	2.81	2.55	2.89	6.3	15.0	3.6
190	3.00	2.97	2.69	3.00	2.6	11.8	1.7
195	3.05	2.97	2.72	3.01	2.8	10.7	1.2
200	3.05	2.94	2.70	3.00	2.3	10.3	0.4
205	3.06	2.93	2.68	2.98	4.2	12.5	2.7
210	3.00	2.88	2.71	3.01	3.9	9.7	-0.4
215	2.96	2.89	2.70	2.98	2.5	8.8	-0.6
220	3.00	2.83	2.63	2.93	5.8	12.2	2.3
225	2.94	2.64	2.46	2.73	10.4	16.5	7.1
230	2.98	2.62	2.46	2.72	10.8	16.5	7.5
235	2.98	2.81	2.64	2.86	5.7	11.3	3.9
240	2.89	2.71	2.65	2.77	6.1	8.4	4.0
245	2.95	2.93	2.71	2.96	0.6	7.9	-0.6
250	3.05	2.83	2.58	2.80	7.1	15.3	8.1
255	3.10	2.86	2.60	2.83	7.7	16.1	8.6
260	3.15	2.91	2.59	2.86	7.5	17.6	9.1
262	3.12	2.90	2.62	2.85	7.2	16.1	8.5

Appendix C4 VS of feeding and digested sludge and its removal efficiency

Run times (days)	Feeding (%)	Digested sludge (%)			% VS removal		
		Do	Du	Dcu	Do	Du	Dcu
35	2.60	1.59	1.62	1.61	39.0	37.6	38.3
40	2.60	1.56	1.69	1.71	39.9	35.0	34.5
45	2.60	1.71	1.65	1.73	34.3	36.8	33.6
50	2.62	1.72	1.67	1.67	34.3	36.2	36.0
55	2.62	1.72	1.68	1.69	34.1	35.7	35.4
60	2.62	1.76	1.73	1.70	32.8	33.9	34.9
65	2.62	1.78	1.79	1.73	32.1	31.5	33.7
70	2.62	1.78	1.79	1.73	32.07	31.47	33.72
75	2.62	1.88	1.86	1.88	28.10	29.04	28.15
80	2.62	1.88	1.90	1.94	28.1	27.5	25.7
85	2.62	1.91	1.94	1.91	26.5	25.4	26.3
90	2.60	2.06	2.05	2.07	20.7	21.2	20.2
95	2.60	2.04	2.08	2.12	21.3	19.9	18.4
100	2.60	1.99	2.04	2.05	23.3	21.3	21.0
105	2.61	2.15	2.09	2.09	17.8	19.8	20.1
110	2.61	2.13	2.24	2.21	18.5	14.3	15.3
115	2.59	1.98	2.03	2.00	23.5	21.5	22.9
120	2.59	1.96	2.02	1.96	17.2	22.2	31.0
125	2.59	1.97	2.03	1.99	23.8	21.5	23.3
130	2.56	2.01	2.03	2.00	21.4	20.5	22.0
135	2.56	1.98	2.04	1.95	22.5	20.2	23.6
140	2.59	1.99	2.00	1.95	23.1	22.7	24.8
145	2.60	2.07	2.06	1.98	20.5	20.8	23.7
150	2.59	2.02	1.98	1.93	22.0	23.6	25.4
155	2.59	2.06	2.00	2.00	20.6	22.5	22.9
160	2.70	2.19	2.15	2.08	17.9	19.2	22.0
165	2.67	2.17	2.10	2.04	19.5	22.2	24.8
170	2.70	2.16	2.09	2.03	19.9	22.4	24.7
175	2.61	2.18	2.10	2.02	19.3	22.1	25.0
180	2.61	2.22	2.13	2.08	14.9	18.4	20.5
185	2.62	2.13	2.02	1.98	18.8	22.7	24.3
190	2.66	2.30	2.17	2.10	13.7	18.4	21.2
195	2.66	2.33	2.21	2.13	12.3	16.8	19.8
200	2.61	2.35	2.21	2.13	10.2	15.4	18.5
205	2.66	2.35	2.20	2.11	11.6	17.1	20.5
210	2.59	2.33	2.24	2.15	9.9	13.7	16.9
215	2.56	2.33	2.23	2.11	9.2	12.9	17.9
220	2.61	2.29	2.17	2.08	12.4	16.9	20.1
225	2.52	2.12	2.02	1.93	17.0	21.2	24.6
230	2.56	2.10	2.01	1.93	17.8	21.4	24.6
235	2.51	2.28	2.18	2.08	11.9	15.9	19.6
240	2.51	2.20	2.19	2.02	12.3	12.4	19.6
245	2.55	2.39	2.27	2.20	6.5	11.2	13.9
250	2.62	2.23	2.13	2.06	15.1	18.6	21.3
255	2.71	2.23	2.15	2.10	16.1	19.3	21.1
260	2.71	2.18	2.14	2.06	19.6	21.1	23.9
262	2.68	2.17	2.15	2.08	19.0	19.5	22.4

Appendix C5 Capillary suction time of digested sludge with digestion time

Date	Capillary Suction Time (min)			
	WAS	Control	Ultrasonic	Chemical-ultrasonic
SRT: 25 days				
12-Dec-07	-	21.70	24.84	24.90
14-Dec-07	-	19.03	24.97	25.56
17-Dec-07	-	18.44	27.80	23.73
21-Dec-07	-	22.98	29.46	31.40
23-Dec-07	-	22.58	32.21	30.57
25-Dec-07	16.34	23.18	29.01	33.36
27-Dec-07	16.60	22.22	33.60	32.20
31-Dec-07	16.81	21.68	37.83	33.44
2-Jan-08	17.15	24.18	44.36	38.64
SRT: 15 days				
25-Feb-08	16.11	22.09	36.61	32.28
27-Feb-08	16.86	20.73	34.37	32.76
29-Feb-08	16.65	21.35	31.10	33.16
SRT: 10 days				
23-Mar-08	10.09	11.35	24.18	23.43
26-Mar-08	9.92	10.83	23.31	21.51
29-Mar-08	8.24	10.01	23.09	20.17
31-Mar-08	8.01	10.11	23.00	24.55

Appendix D

Specimen Calculation

Appendix D1: Energy balance Calculation

Assumptions:

- Temperature of fresh sludge (T_1) = 20°C
- Average temperature of ambient air temp(T_2) = 30°C
- Temperature in the digester(T_3) = 37°C
- Specific heat capacity of sludge(C_p) = 4.2 kJ/kg/°C
- Overall heat transfer coefficient (U) = 2.5 W/m^2/°C
- Calorific value of methane = 35.8 kJ/g
- Specific gravity of sludge (m) = 1.02
- Sludge flow rate (Q) = 120, 200 and 300 mL/day

1. Heat requirement for the sludge (Q_1)

$$Q_1 = \sum m\,C_p(T_3 - T_1)$$

- **SRT of 25 days**

$$Q_1 = 120x1.02x4.2x(37 - 20)J/day$$
$$= 8739.36J/day$$

- **SRT of 15 days**

$$Q_1 = 200x1.02x4.2x(37 - 20)J/day$$
$$= 14565.6J/day$$

- **SRT of 15 days**

$$Q_1 = 300x1.02x4.2x(37 - 20)J/day$$
$$= 21848.4J/day$$

2. Energy requirement for mechanical mixer (Q_2)

$$Q_2 = 9\ W$$
$$Q_2 = 9x24x3600 = 777600J/day$$

3. Energy to compensate the heat loss from digester (Q_3)

$$Q_3 = UA(T_3 - T_1)$$

Where, A : Cross sectional area through which the losing is occurring (A = 0.1154 m^2)

$$Q_3 = 2.5x0.1154x(37 - 30)J/day$$

$$= 174484.8J/day$$

4. Energy generation from methane (Q_5)

Methane gas mole calculation from methane gas volume

Ideal Gas Law $PV = nRT$

$$n = \frac{PV}{RT} = \frac{1.02091x10^5 xV}{8.3145x303}$$

$$n = 40.52.Vmol$$

Where, V is measured in mL at ambient prepressure (1.02091Pa) and temperature (303K)

$$m_{CH4} = 40.52xV.10^{-6}x16g$$

$$m_{CH4} = 648.32xV.10^{-6}g$$

Thus, $Q_5 = m_{CH4}x35.8$

$$Q_5 = 23.21xVJ/day$$

SRT (day)	Digesters	Methane gas volume (mL)	Energy obtained from biogas (J/say)
25	control	358	8310
	ultrasonic	404	9378
	chemical-ultrasonic	420	9749
15	control	478	11095
	ultrasonic	589	13672
	chemical-ultrasonic	626	14531
10	control	485	9749
	ultrasonic	636	14531
	chemical-ultrasonic	688	15970

5. Energy requirement for ultrasonic unit (Q_7)

$$Q_7 = Q\ x\ UD\ x\ t$$

The pretreated sludge was mixed with WAS at ratio of 1:1 (50% sonicated sludge and 50% WAS), thus

$$Q_7 = \frac{Q\ x\ UD\ x\ t}{2}$$

- **SRT of 25 days**

$$Q_7 = \frac{120x1.9x60}{2} J/day$$

$$= 6840 J/day$$

- **SRT of 15 days**

$$Q_7 = \frac{200x1.9x60}{2} J/day$$

$$= 11400 J/day$$

- **SRT of 15 days**

$$Q_7 = \frac{300x1.9x60}{2} J/day$$

$$= 17100 J/day$$

Appendix D2: Economic Analysis of anaerobic digester with different pretreatment options

- Disposal cost : 200 Baht/ton
- Electricity cost: 2.5 Baht/kW.h
- Chemical cost: 0.05 Baht/g

	SRT	Energy cost for heating sludge	Energy cost for mixer	Energy cost for heat loss	Energy consumption cost	Ultrasonic operating cost	chemical cost	Cost of sludge reduction to landfill	Methane recovery cost
Control	25	0.006	0.540	0.121	0.67	0	0	0.00003	0.006
	15	0.010	0.540	0.121	0.67	0	0	0.00008	0.008
	10	0.015	0.540	0.121	0.68	0	0	0.00006	0.008
Ultrasound	25	0.006	0.540	0.121	0.67	0.005	0	0.00009	0.007
	15	0.010	0.540	0.121	0.67	0.008	0	0.00012	0.009
	10	0.015	0.540	0.121	0.68	0.012	0	0.00012	0.010
Chemical-ultrasound	25	0.006	0.540	0.121	0.67	0.005	0.001	0.00002	0.007
	15	0.010	0.540	0.121	0.67	0.008	0.002	0.00006	0.010
	10	0.015	0.540	0.121	0.68	0.012	0.002	0.00007	0.011

	Cost (Baht)								
	SRT = 25 day			SRT = 15 day			SRT = 10 day		
	Control	Ultrasonic	Chemical-ultrasonic	Control	Ultrasonic	Chemical-ultrasonic	Control	Ultrasonic	Chemical-ultrasonic
Heating sludge	0.006			0.010			0.015		
Ultrasound unit	0	0.005	0.005	0	0.005	0.005	0	0.005	0.005
Chemical (NaOH)	0	0	0.001	0	0	0.001	0	0	0.001
Bioenergy recovery	0.006	0.007	0.007	0.006	0.007	0.007	0.006	0.007	0.007
Benefit from Landfill	0.00003	0.00009	0.00002	0.00003	0.00009	0.00002	0.00003	0.00009	0.00002
Balance	-0.0003	-0.0042	-0.0049	-0.0003	-0.0042	-0.0049	-0.0003	-0.0042	-0.0049

Economic analysis of treating 1L sludge

	Digester	Income	Expense	Benefit
SRT = 25	control	16.1	16.9	-0.8
	ultrasonic	18.3	30.1	-11.7
	chemical-ultrasonic	18.9	32.6	-13.7
SRT = 15	control	13.0	16.9	-3.9
	ultrasonic	16.0	30.1	-14.0
	chemical-ultrasonic	16.9	32.6	-15.6
SRT = 10	control	8.7	16.9	-8.1
	ultrasonic	11.5	30.1	-18.5
	chemical-ultrasonic	12.4	32.6	-20.2

Anaerobic Digestibility of Ultrasound and Chemical Pretreated Waste Activated Sludge

Mr. Seng Bunrith

Examination Committee:

Prof. C. Visvanathan (Chairperson)

Prof. Ajit P. Annachhatre

Dr. Thammarat Koottatep

Dr. Samir Kumar Khanal

April 25, 2008

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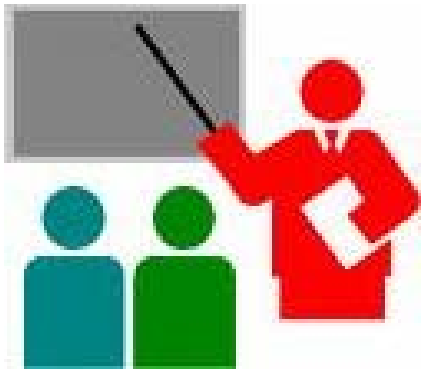


BACKGROUND

Activated Sludge Process

WAS

1- High BOD/COD
Pathogen count

Sludge Management

- 60% of operating cost of WWTP
- Treatment
  Incineration, Aerobic and
  Anaerobic

Anaerobic Digestion

- Long digestion
- Large digester

Pretreatments

1- Mechanical
2- Thermal
3- Chemical
4- Biological

Final Exam

OBJECTIVES

- To optimize ultrasonic, chemical and chemical-ultrasonic pretreatment to maximize Waste Activated Sludge (WAS) disintegration.
- To investigate the anaerobic digestibility of various pretreated WAS compared to non pretreated sludge (control) at different solid retention times (SRTs).

- To determine the rate constant of the hydrolysis step for both pretreated and non-pretreated sludge during anaerobic digestion.
- To conduct the economic analysis of the control, ultrasonic, and chemical-ultrasonic anaerobic digesters.

PREVIOUS AND CURRENT STUDY

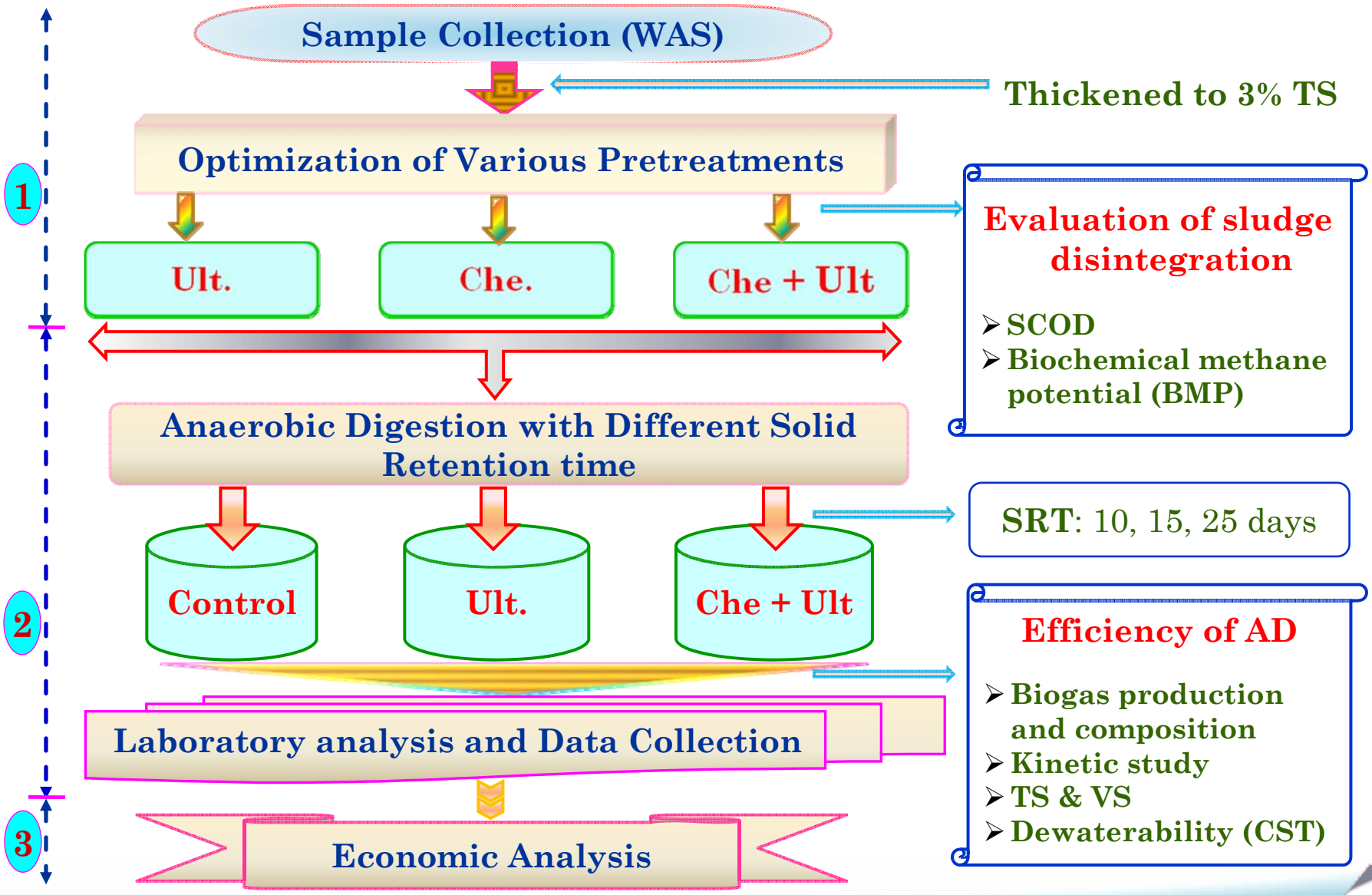
Previous Study

- **Pretreatment**
  - Ultrasonic alone
  - Horn : 1.2, 2.5 and 3.8 cm
  - Horn immersion depth: 1, 2 and 3 cm
- **Anaerobic Digestion**
  - Control
  - Full stream (100% sonicated sludge)
  - Part stream (50% WAS + 50% sonicated sludge)
  - SRT: 15 and 20 days

Current Study

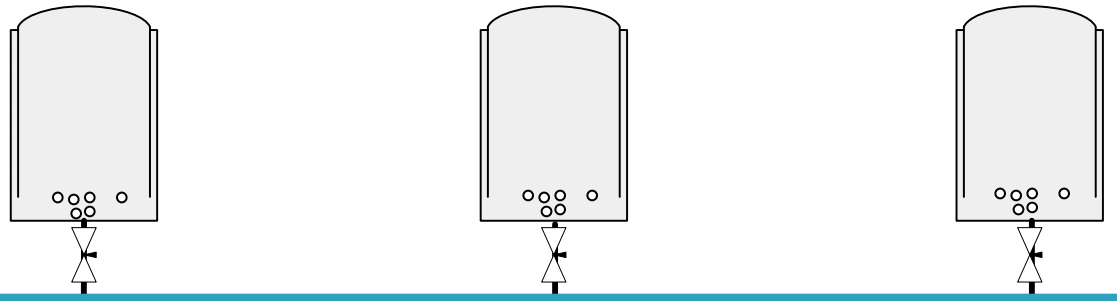
- **Pretreatment**
  - Ultrasonic
  - Chemical (New)
  - Chemical-ultrasonic (New)
- **Ultrasonic pretreatment**
  - Horn : 3.8 cm
  - Horn immersion depth: 2cm
- **Anaerobic Digestion (part stream)**
  - Control
  - Ultrasonic
  - Chemical-ultrasonic
  - SRT : 10, 15 and 25 days

RESEARCH METHODOLOGY



Final Exam

EXPERIMENTAL SET-UP



SRT (day)	Control (D_0)	Ultrasonic (D_u)		Chemical-ultrasonic (D_{cu})		SLR
	Fresh WAS mL/day	50% Sonicated mL/day	50% Fresh WAS mL/day	50% Che-ult mL/day	50% Fresh WAS mL/day	Kg TS/ m^3 days
25	120	60	60	60	60	1.2
15	200	100	100	100	100	2
10	300	150	150	150	150	3
Digester Working Volume: 3L						

Samp
(3L)

Final Exam

Pictorial View of Experimental Set-up

Anaerobic Digester

Ultrasonic Unit

5. Converter
6. Booster
7. Horn
8. Sonication chamber
9. Ultrasonic power supply

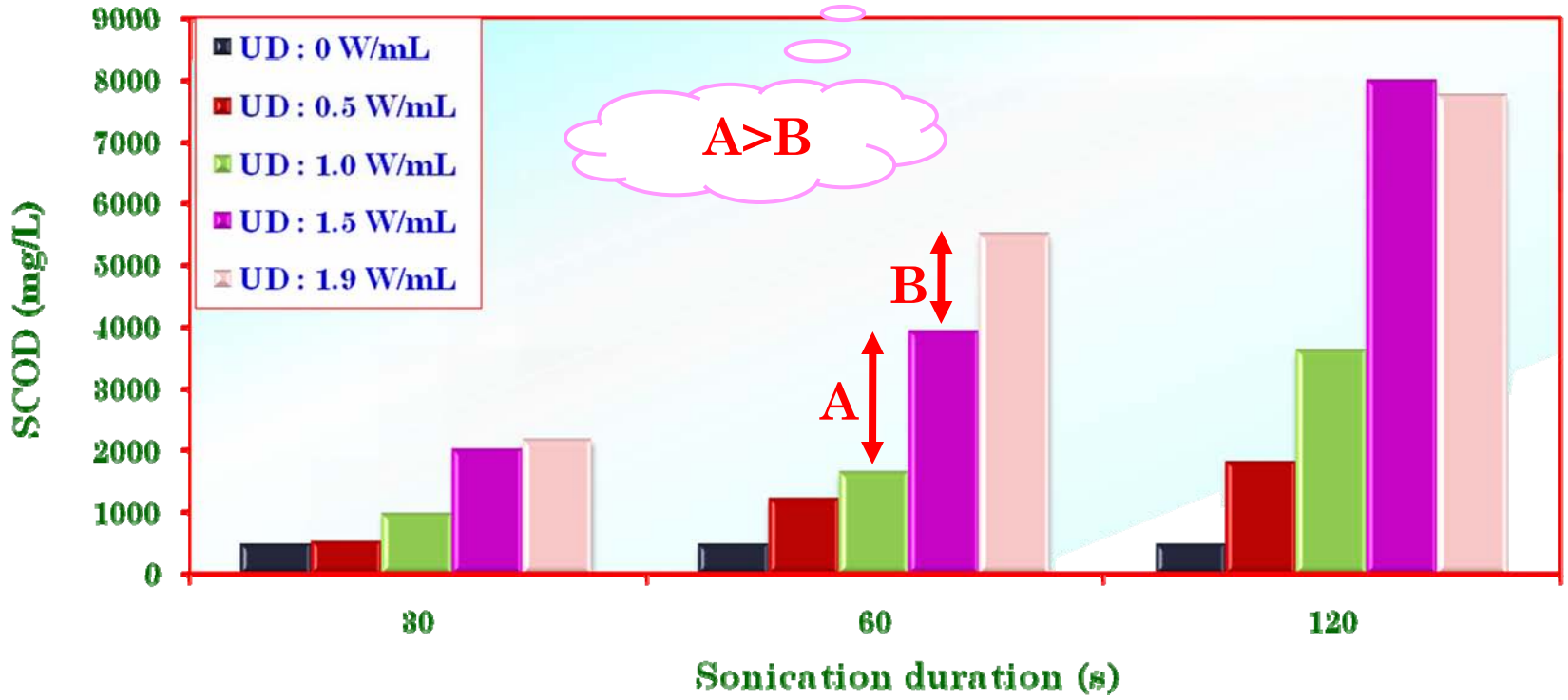
RESULTS AND DISCUSSIONS

Sludge Characteristics		
Parameters	**Raw Sludge**	**Feed Sludge**
TS (%)	1.01±0.08	3 ± 0.1
VS (%)	0.84±0.07	2.6 ± 0.01
TCOD (mg/L)	11719±333	42196±875
SCOD (mg/L)	77±14	458±35
TKN (mg/L)	814±104	3136±150
NH_3 (mg/L)	26±3	245±1
CST (s)	55±1	992±23
pH	6.67±0.2	6.97±0.24

ULTRASONIC OPTIMIZATION

Effect of UD on SCOD Release

UD = 1.5 W/mL was considered to be the optimum ultrasonic density for effective SCOD release



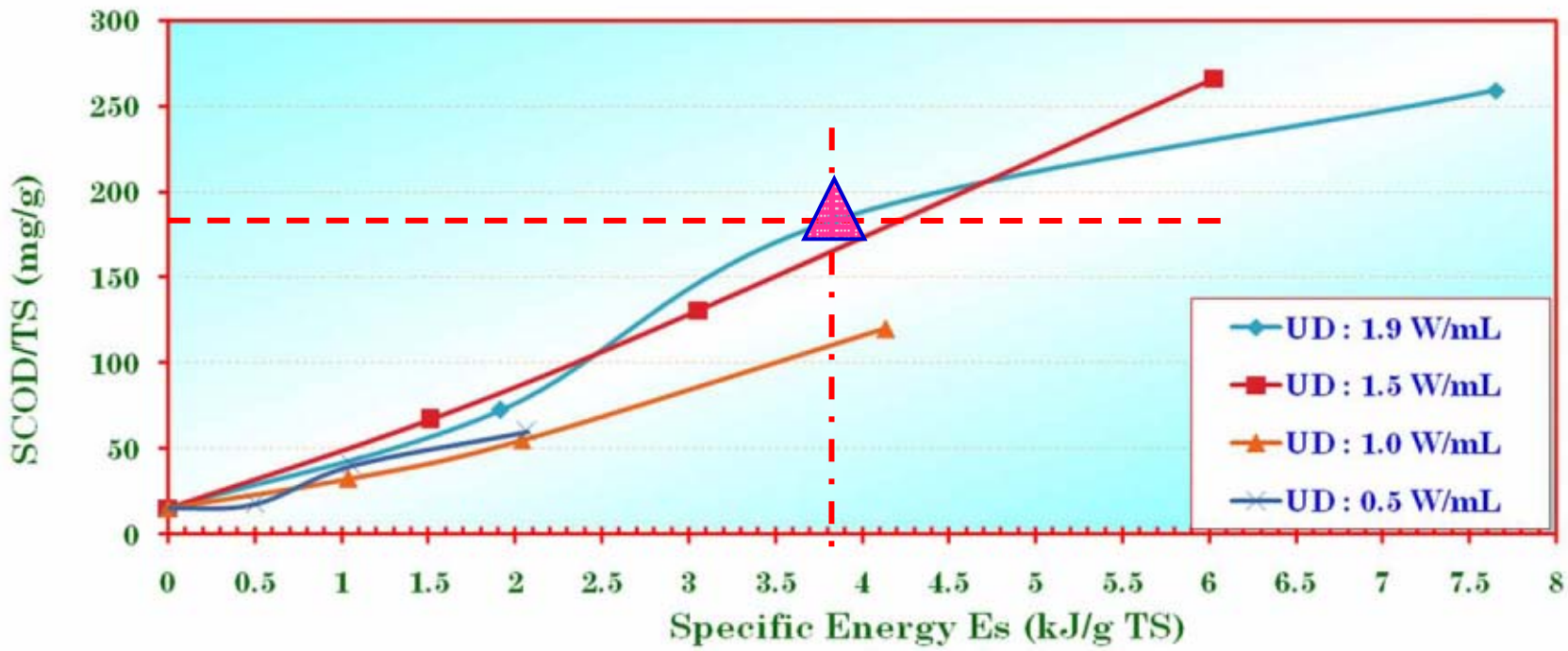
UD: Ultrasonic Density

ULTRASONIC OPTIMIZATION (CONT.)

Effect of Es on SCOD Release

$$E_s = \frac{}{V}$$

3.8 kJ/g TS with 1.9 W/mL was the effective specific energy input on sludge disintegration

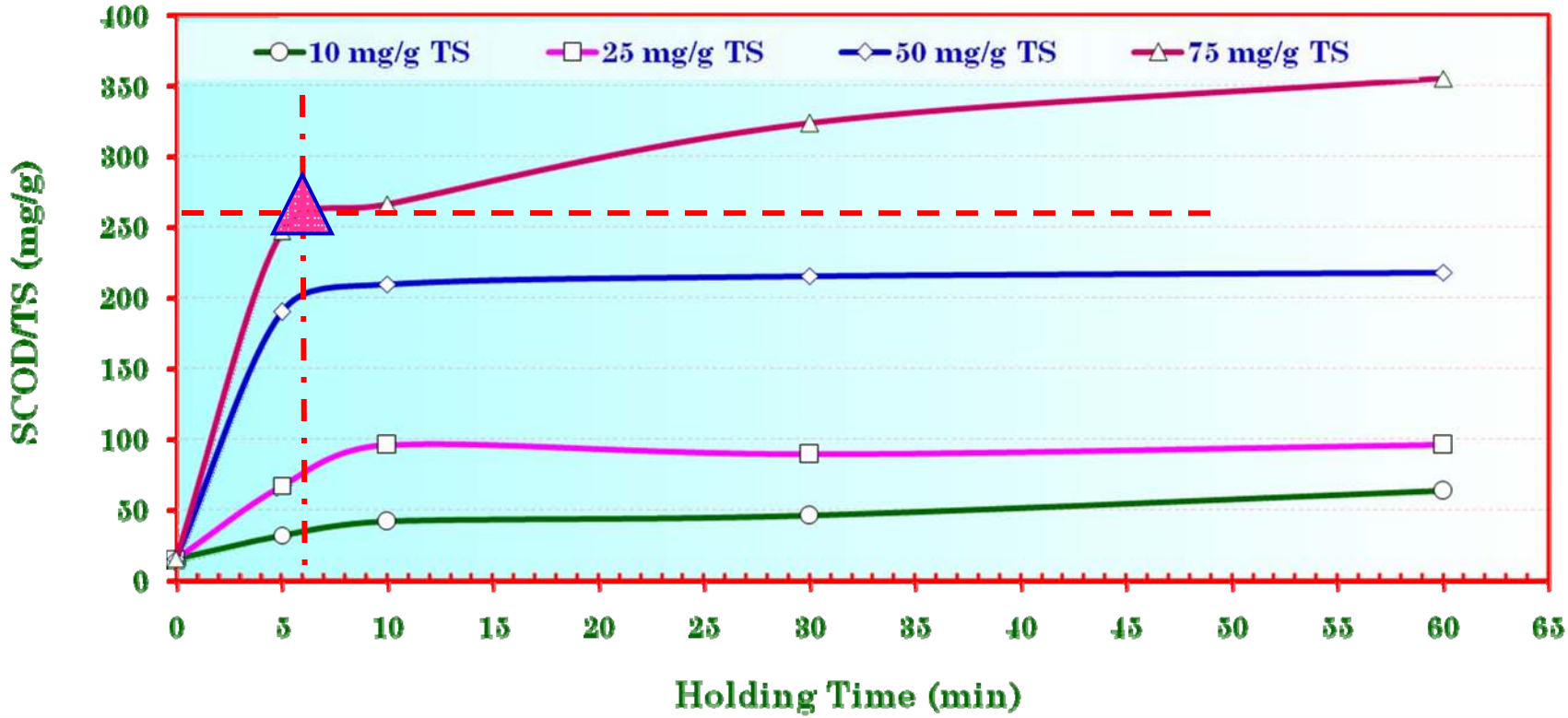


E_s : Specific Energy Input

CHEMICAL OPTIMIZATION

Effect of Holding Time on SCOD Release

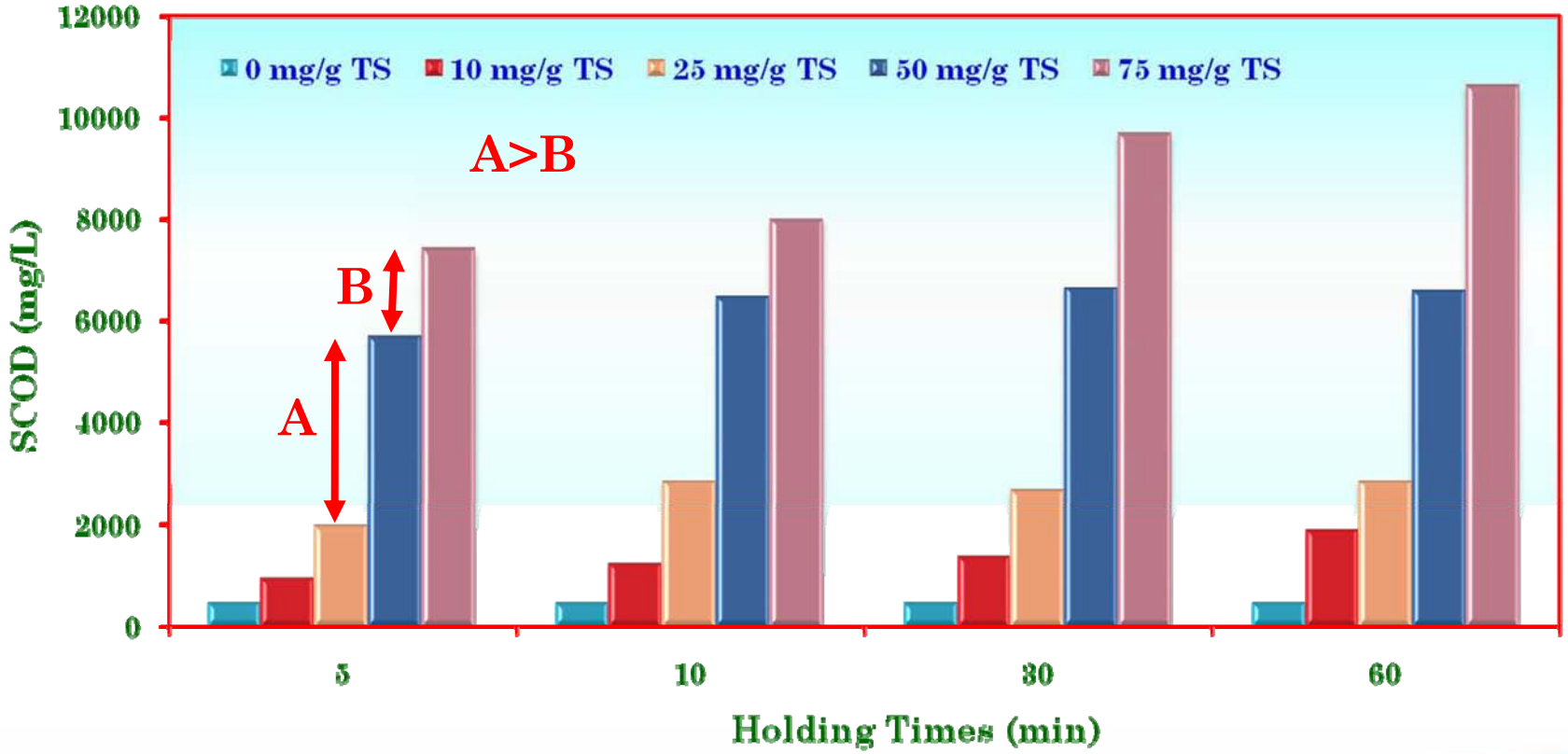
6 min was considered to be the effective chemical treatment duration on sludge disintegration.



CHEMICAL OPTIMIZATION

Effect of Chemical Dose on SCOD Release

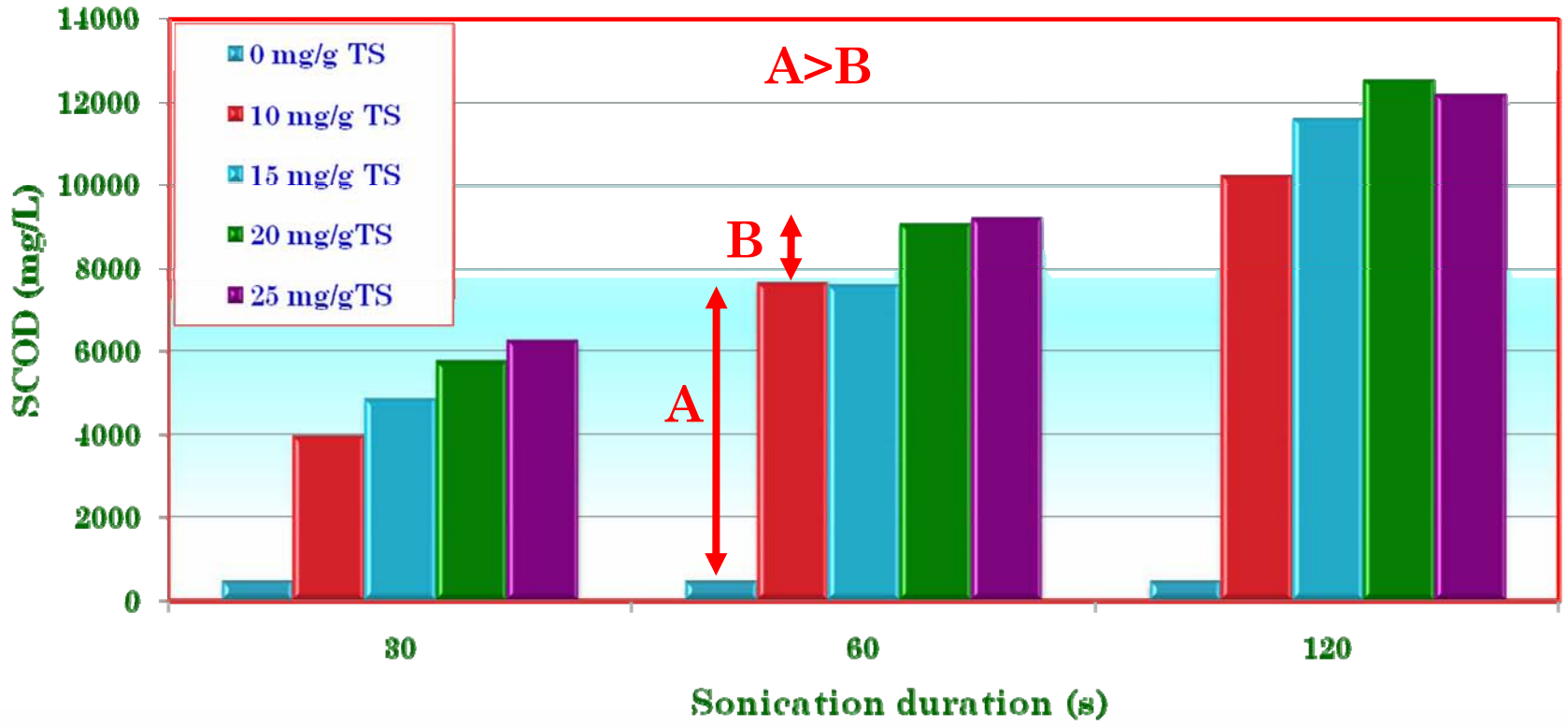
50 mg/g TS was the optimum chemical dose on sludge disintegration.



CHEMICAL-ULTRASONIC OPTIMIZATION

Effect of Chemical Dose on SCOD Release

10 mg/g TS was the optimum chemical dose for chemical-ultrasonic treatment

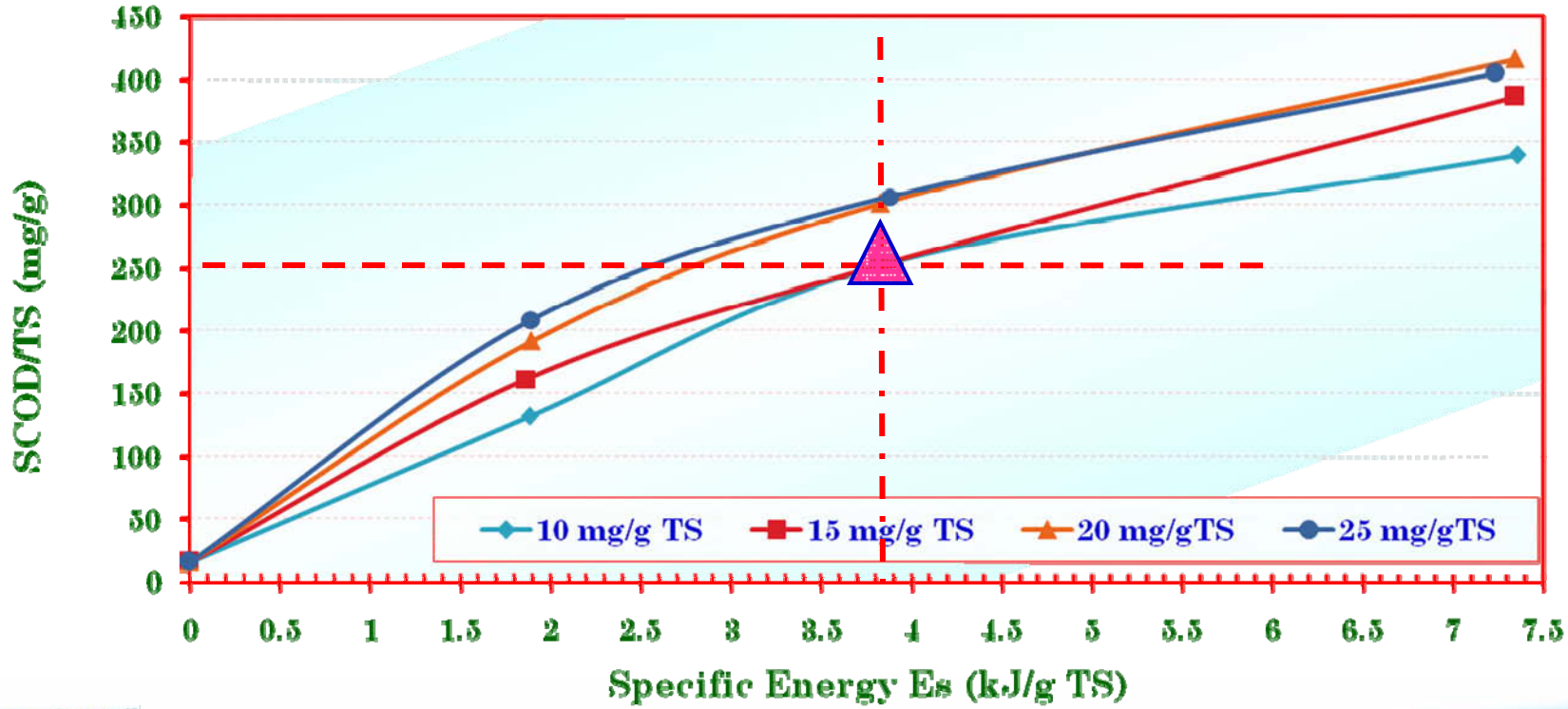


CHEMICAL-ULTRASONIC OPTIMIZATION

Effect of Es on SCOD Release

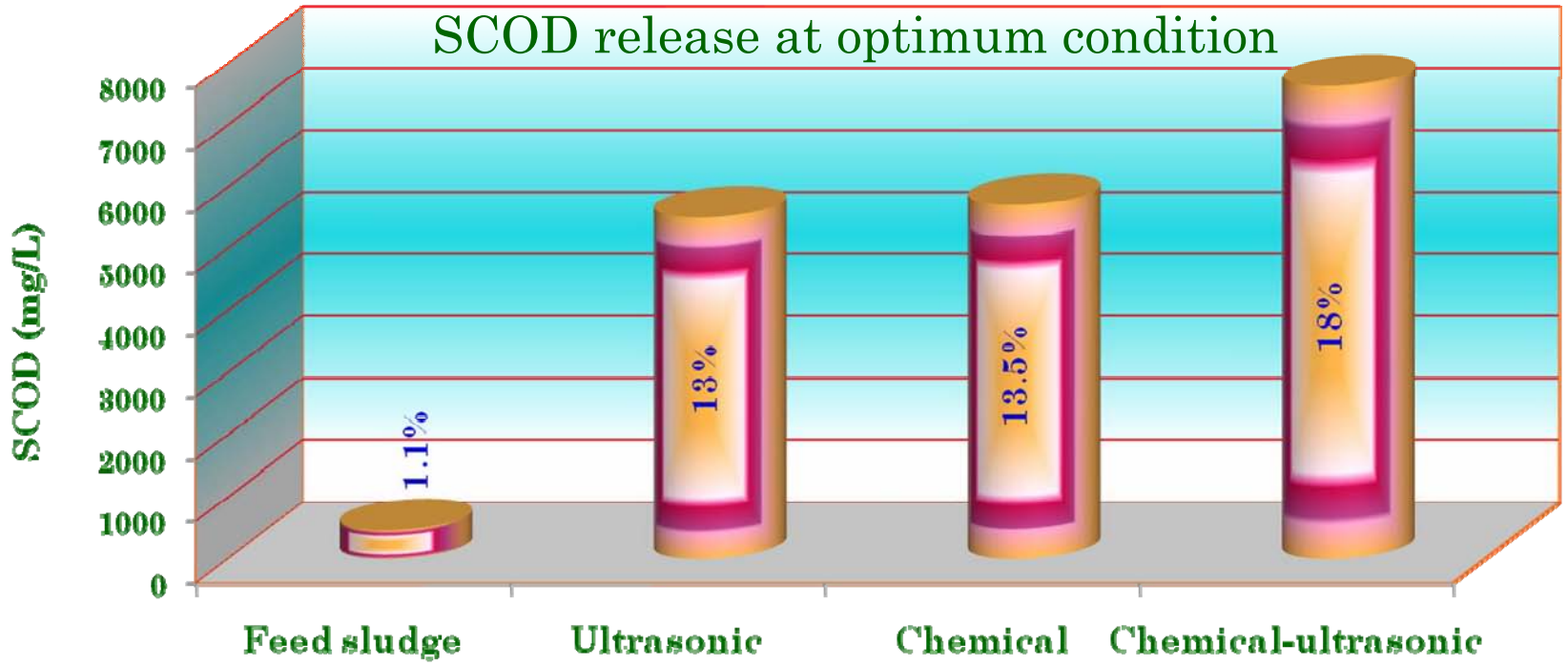
E_s

3.8 kJ/g TS was considered to be the effective specific energy input for chemical-ultrasonic treatment



EVALUATION OF SLUDGE DISINTEGRATION

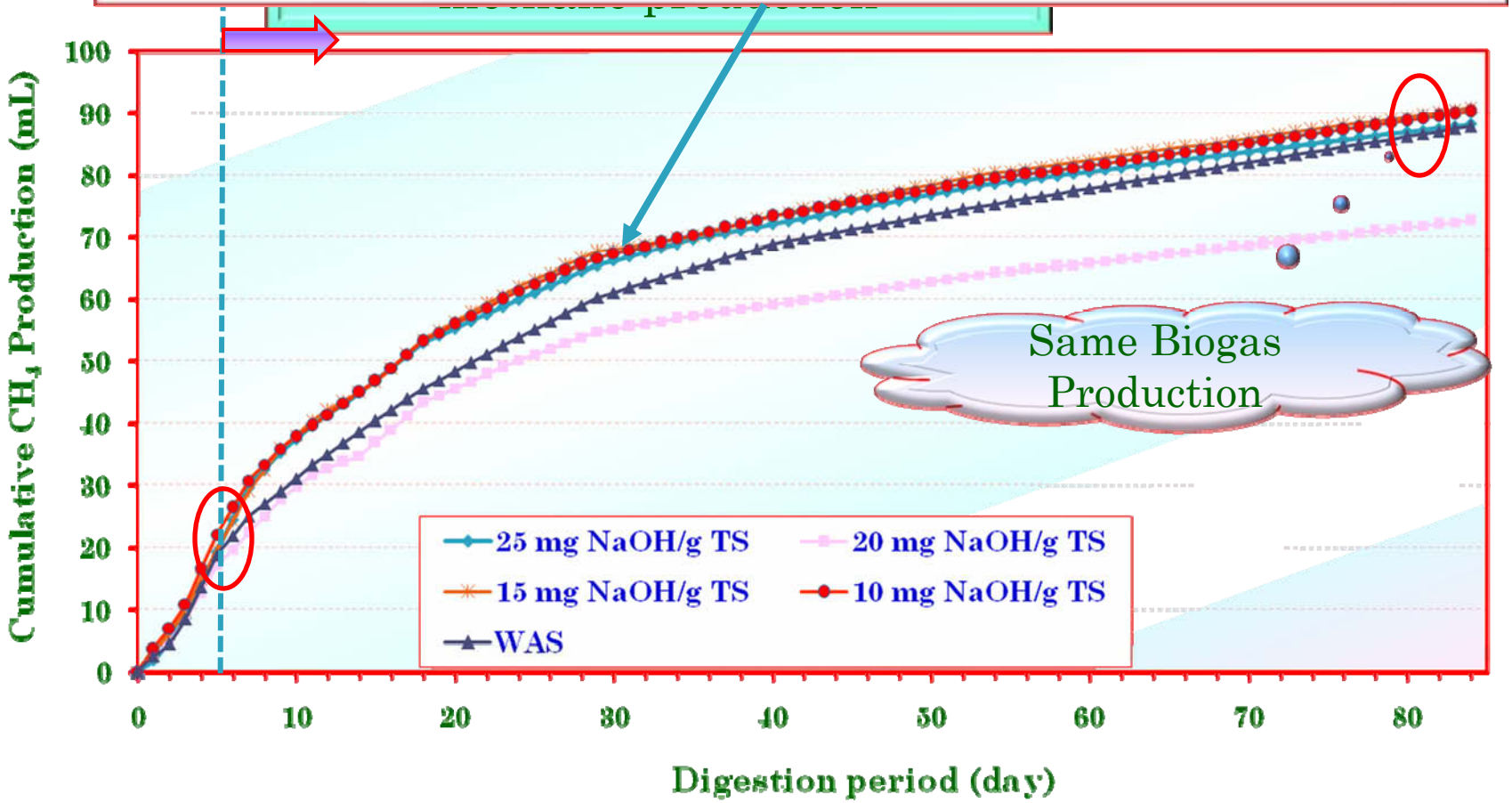
$$\%\ SCOD_{soluble} = \frac{SCOD_{after\ pre.}}{TCOD} x\ 100$$



Chemical-ultrasonic gives better sludge disintegration

BMP OF CHEMICAL-ULTRASONICATED SLUDGE

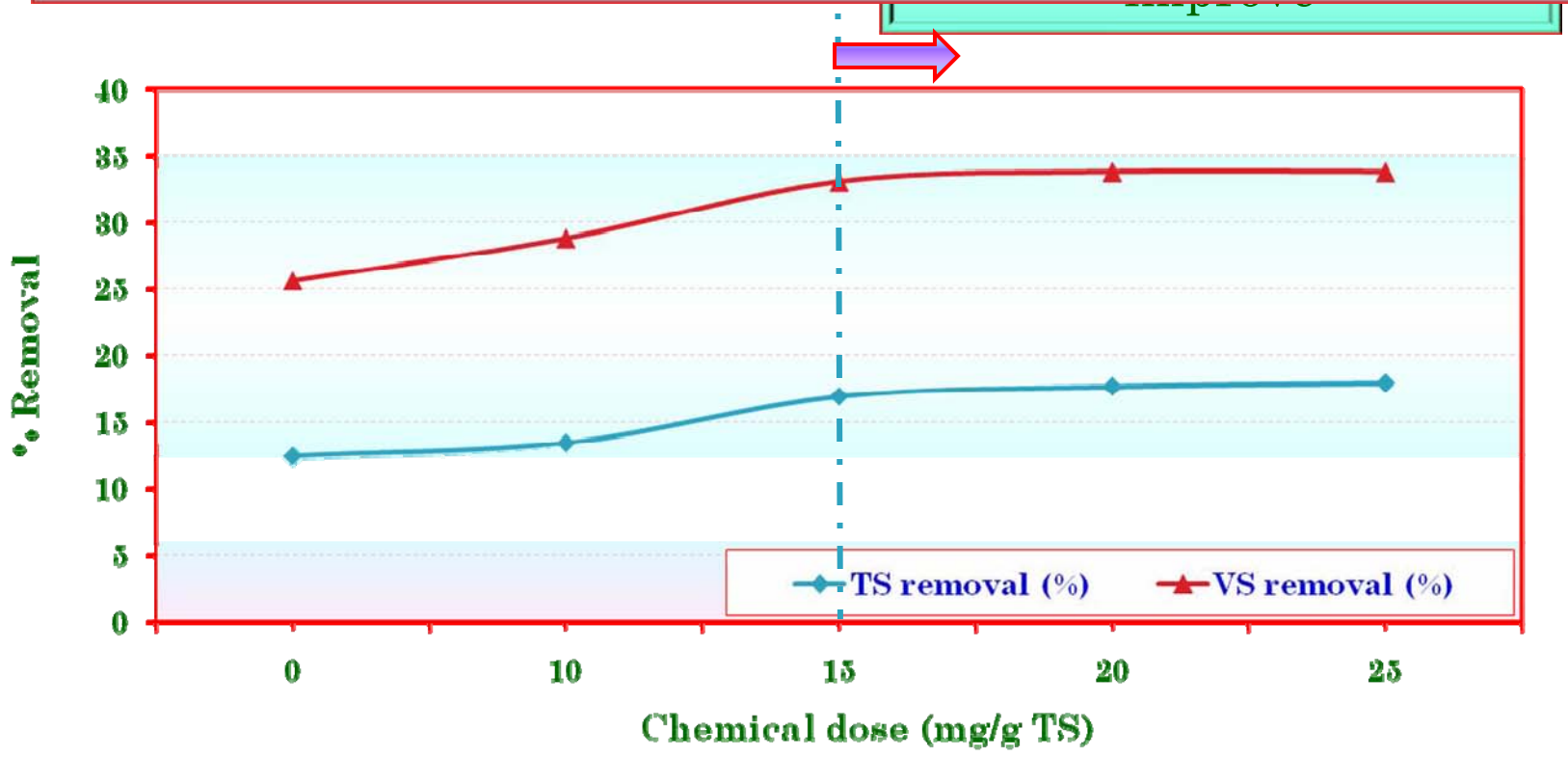
10 mg/g TS was the effective chemical dose for anaerobic digester in term of biogas production



BMP: Biochemical Methane Potential

TS AND VS REMOVAL FROM BMP TEST

- 15 mg/g TS was the optimum chemical dose in term of TS and VS removal.
- 10 mg/g TS was selected for semi-continuous anaerobic digester as it gives higher methane

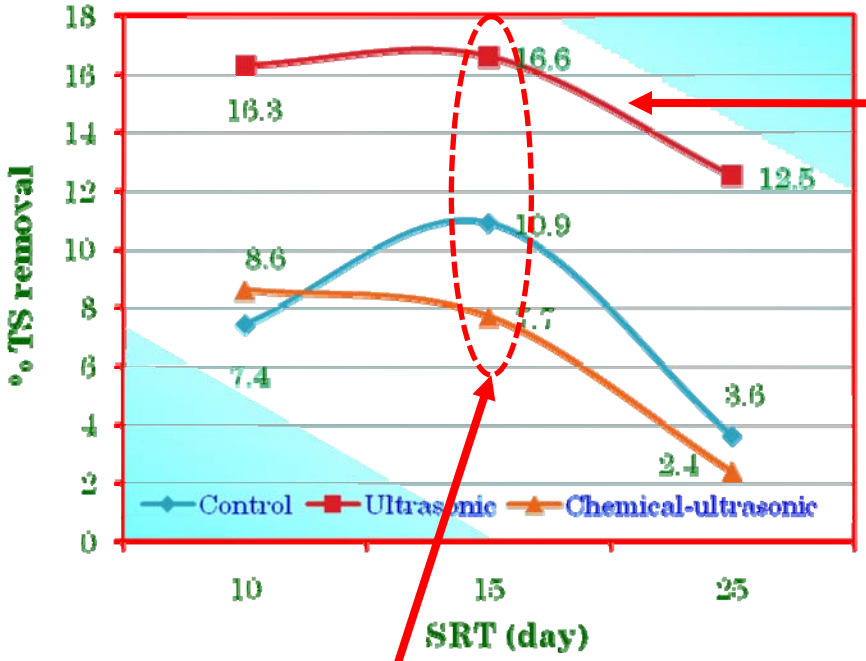


DIGESTER PERFORMANCE AT STEADY STATE

Parameters	Control			Ultrasonic			Chemical-ultrasonic		
SRT (day)	25	15	10	25	15	10	25	15	10
pH	6.96	6.92	6.82	7.04	7.02	7	7.06	7.06	7
Alkalinity (mg/L)	3350	3075	2756	3753	3500	3169	4009	3941	3528
TS removal (%)	3.6	10.9	7.4	12.5	16.6	16.3	2.4	7.7	8.6
VS removal (%)	19.6	17.8	17.4	22.2	21.5	19.6	24.8	24.8	22.2
Biogas production (mL/day)	618	816	835	706	1016	1073	722	1078	1167
CH_4 production (mL/day)	358	478	485	404	589	636	420	626	688
Specific Methane Yield (L/g $VS_{removed}$)	0.56	0.52	0.34	0.56	0.54	0.4	0.52	0.49	0.38
CST (s)	1369	1283	635	2172	2042	1404	2065	1964	1345
Methane content (%)	58	58	58	57	58	59	58	58	59

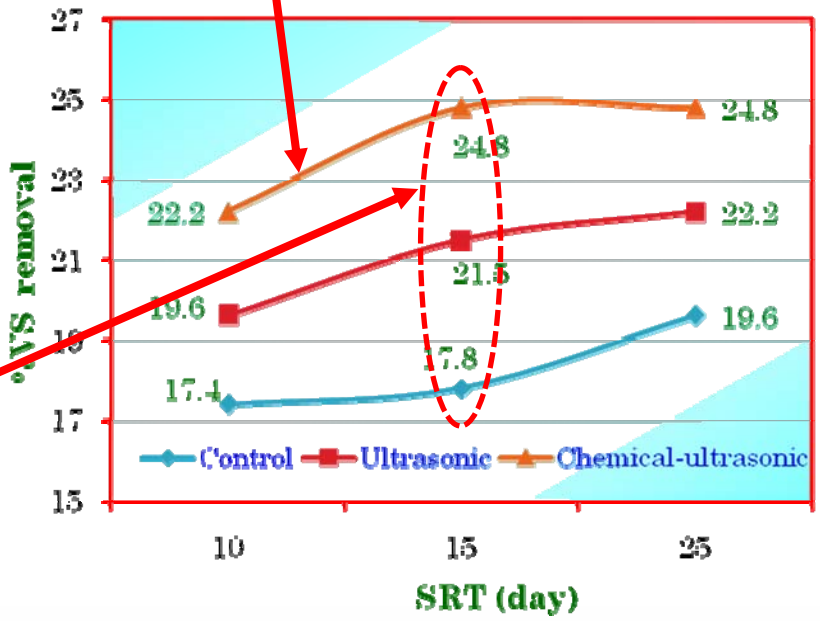
TS AND VS REMOVAL EFFICIENCY

%TS removal



Chemical-ultrasonicated sludge gave higher VS removal efficiency

%VS removal



15 days SRT was the optimum digestion time

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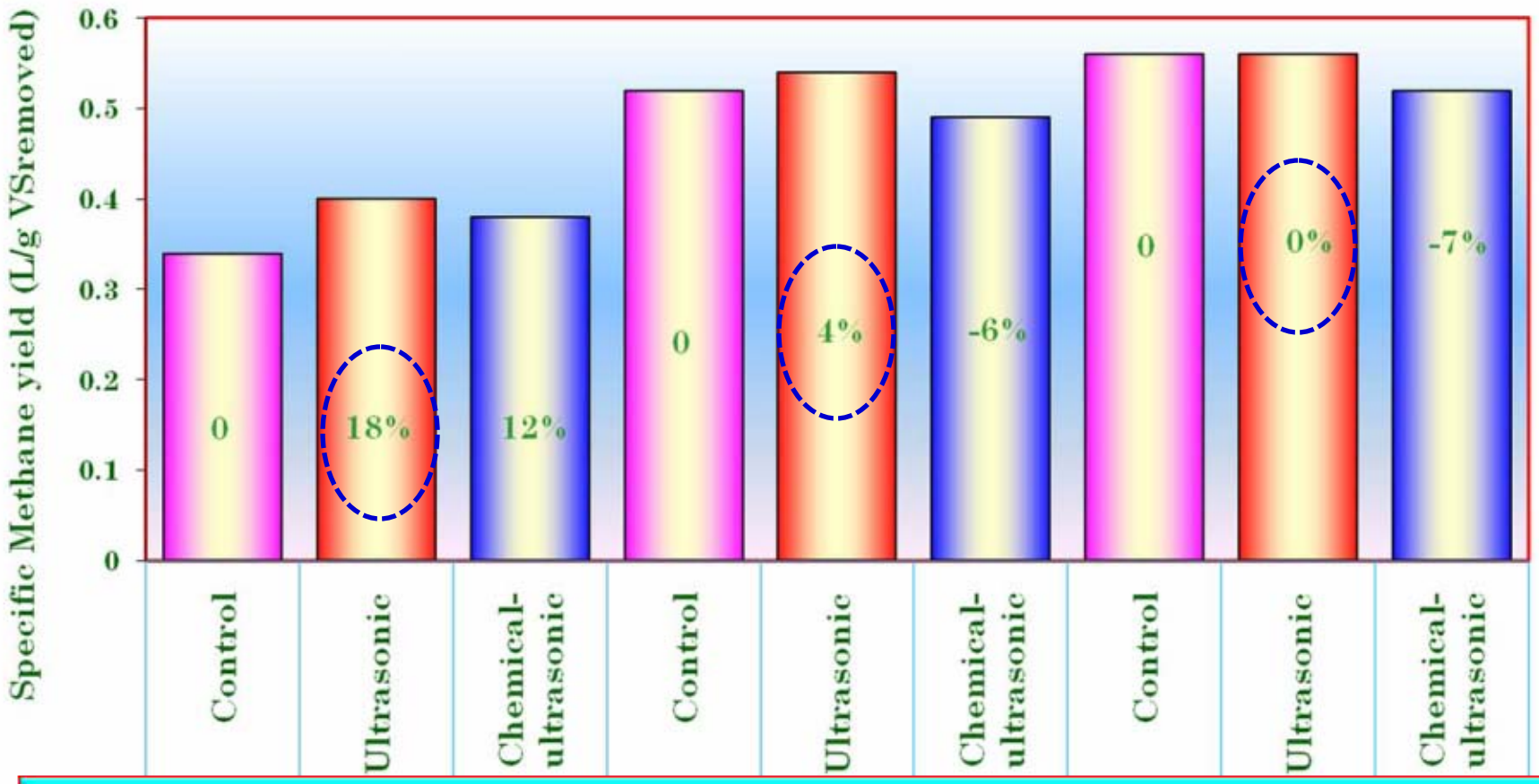
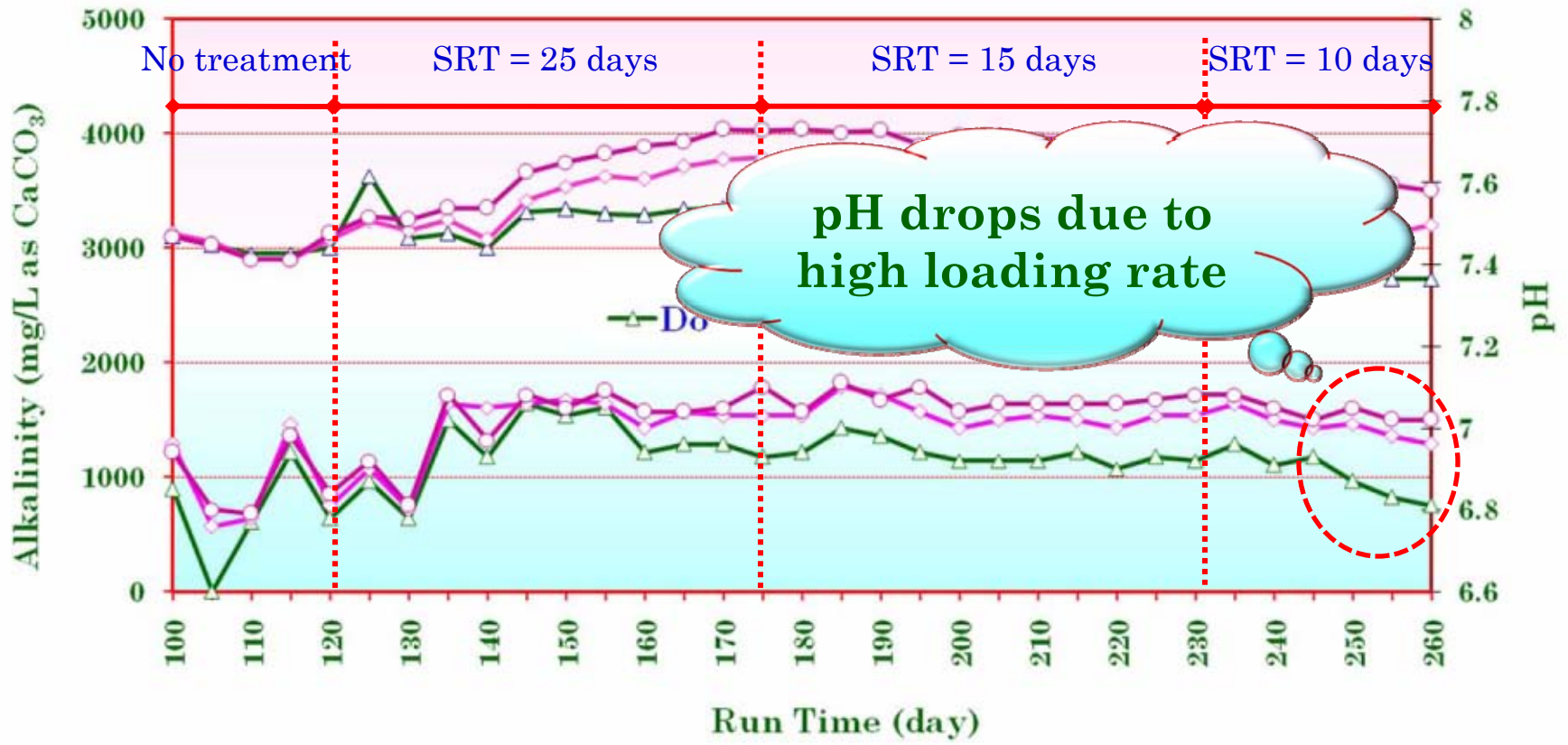
METHANE PRODUCTION RATE



Ultrasonic pretreatment gave higher improvement in term of Specific methane yield due to no inhibition of Na^+

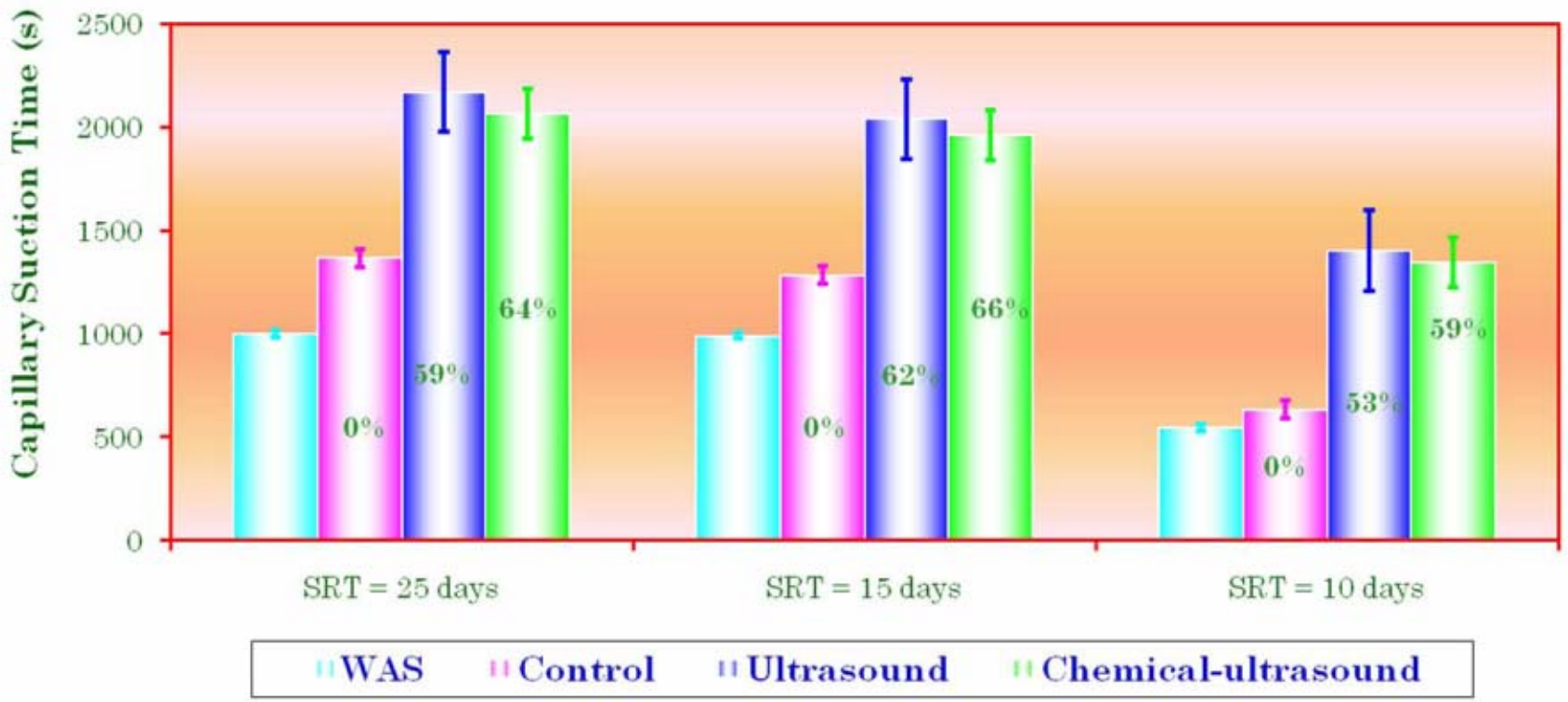
pH AND ALKALINITY



pH and Alkalinity of Du and Dcu are more stable than Do

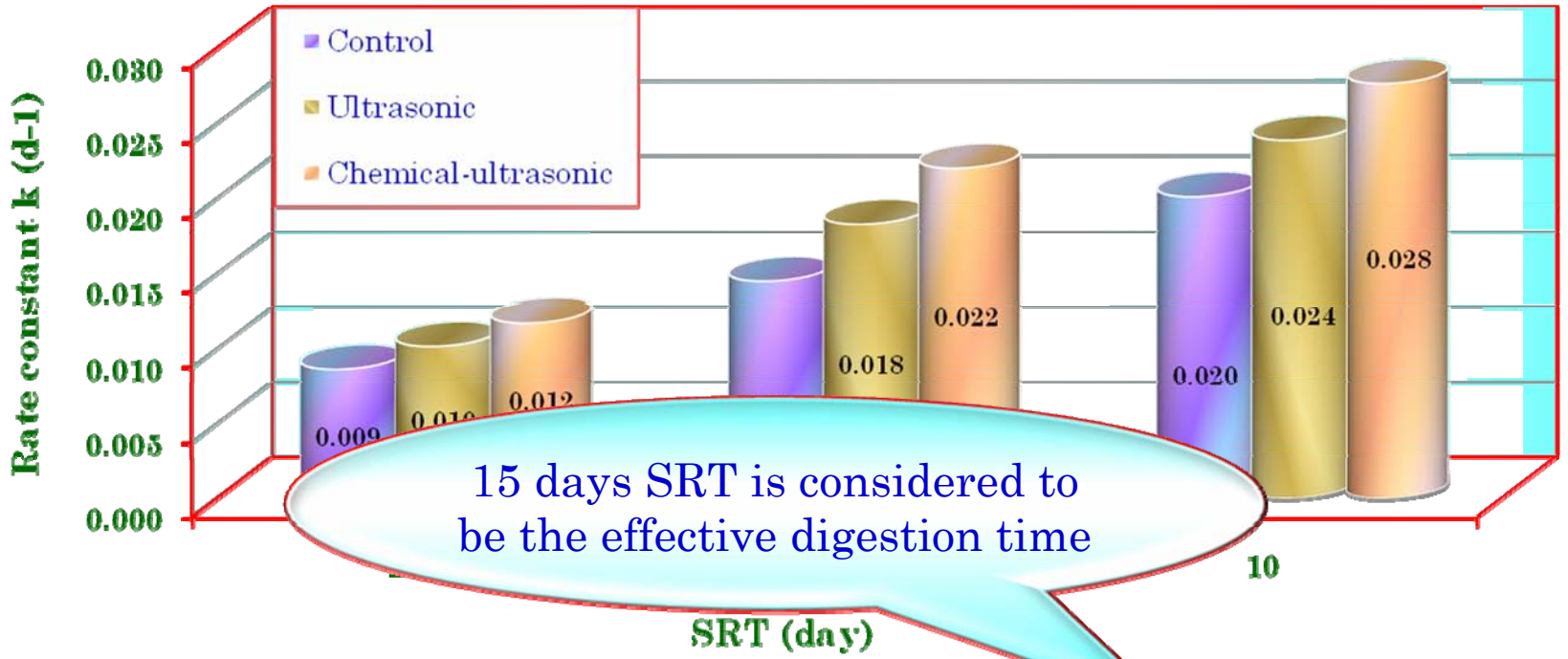
Final Exam

DEWATERABILITY



Chemical-ultrasonic ts red Ultrasonic wa era Non-pretreatment

Rate Constant of the Hydrolysis Step



15 days SRT is considered to be the effective digestion time

Compared to control

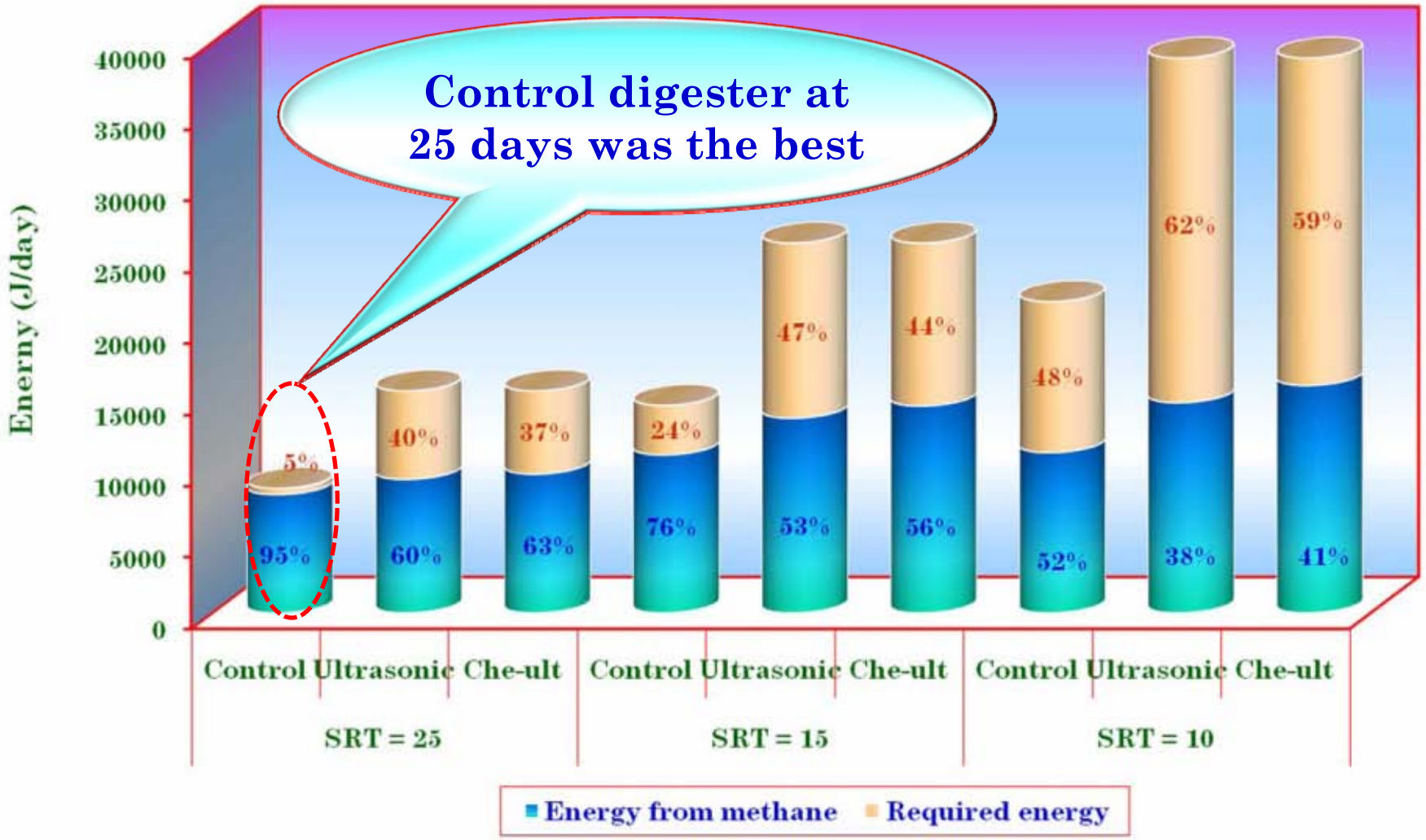
SRT (day)	25	15	10
Ultrasonic	↑18%	↑26%	↑19%
Chemical-ultrasonic	↑36%	↑52%	↑38%

Final Exam

ENERGY BALANCE

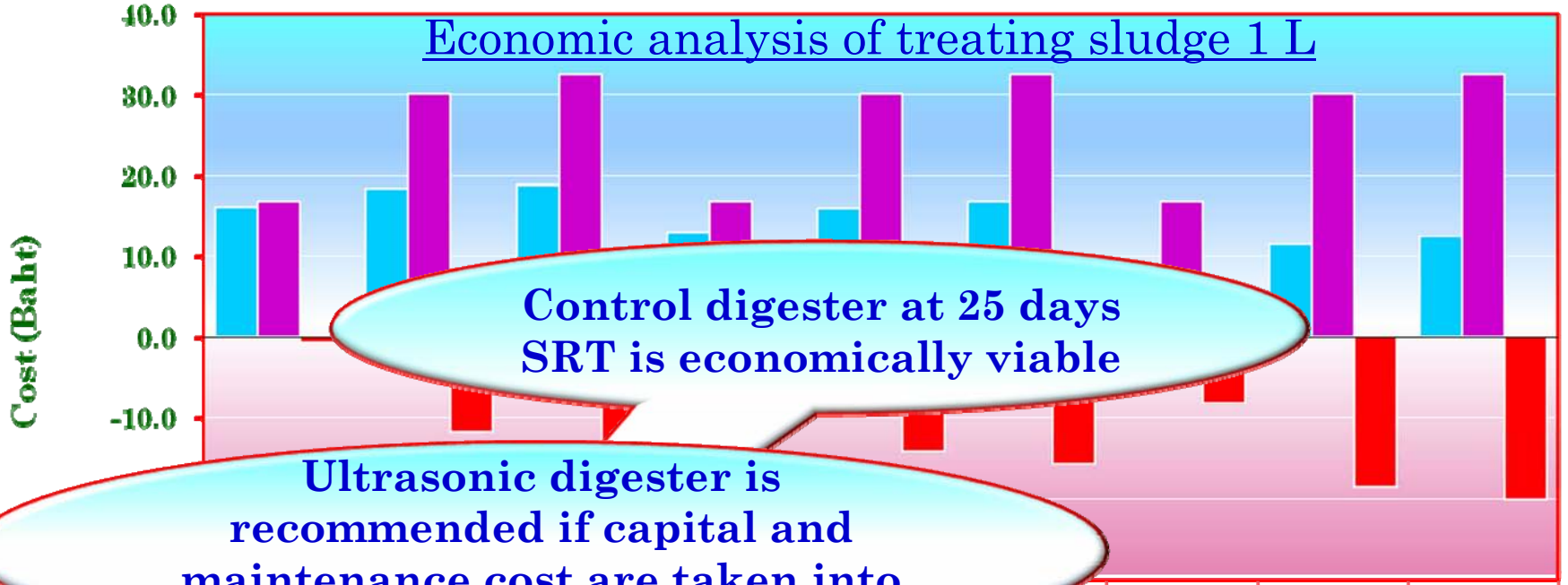
Operating condition		Mixer (J/day)	Heat loss (J/day)	Heat requirement for sludge (J/day)	Ultrasound Unit (J/day)	Total Energy requirement (J/day)	Energy gained from CH_4 (J/day)
Digesters	(day)	b	c	a	d	e = a+b+c	f
Control	25	777600	174485	8739	-	960824	8310
	15	777600	174485	14566	-	966650	11095
	10	777600	174485	21848	-	973933	11258
Ultrasonic	25	777600	174485	8739	6840	960824	9378
	15	777600	174485	14566	11400	966650	13672
	10	777600	174485	21848	17100	973933	14763
Chemical-ultrasonic	25	777600	174485	8739	6840	960824	9749
	15	777600	174485	14566	11400	966650	14531
	10	777600	174485	21848	17100	973933	15970

ENERGY BALANCE (CONT.)



Economic Analysis

- Income = Cost from bioenergy recovery + Landfill cost reduction
- Expense = Energy cost for heating sludge + ultrasonic + chemical cost
- Benefit = Income - Expense



					SRT = 15		control	ultrasonic	che-ult
							SRT = 10		
Income	16.1	18.3	18.9		16.0	16.9	8.7	11.5	12.4
Expense	16.9	30.1	32.6	16.8	30.1	32.6	16.9	30.1	32.6
Benefit	-0.8	-11.7	-13.7	-8.9	-14.0	-15.6	-8.1	-18.5	-20.2

CONCLUSIONS

- 190 W and 60 s corresponds to 3.8 kJ/g TS was considered to be the optimum condition of ultrasonic pretreatment.
- 6 min and 50 mg/g TS corresponds to pH of 11 was considered to be the effective condition for chemical pretreatment.
- 10 mg/g TS and 3.8 kJ/g TS was considered to be the effective chemical dose and Es for chemical-ultrasonic pretreatment.
- Amongst three pretreatments, chemical-ultrasonic gave higher efficiency than others.

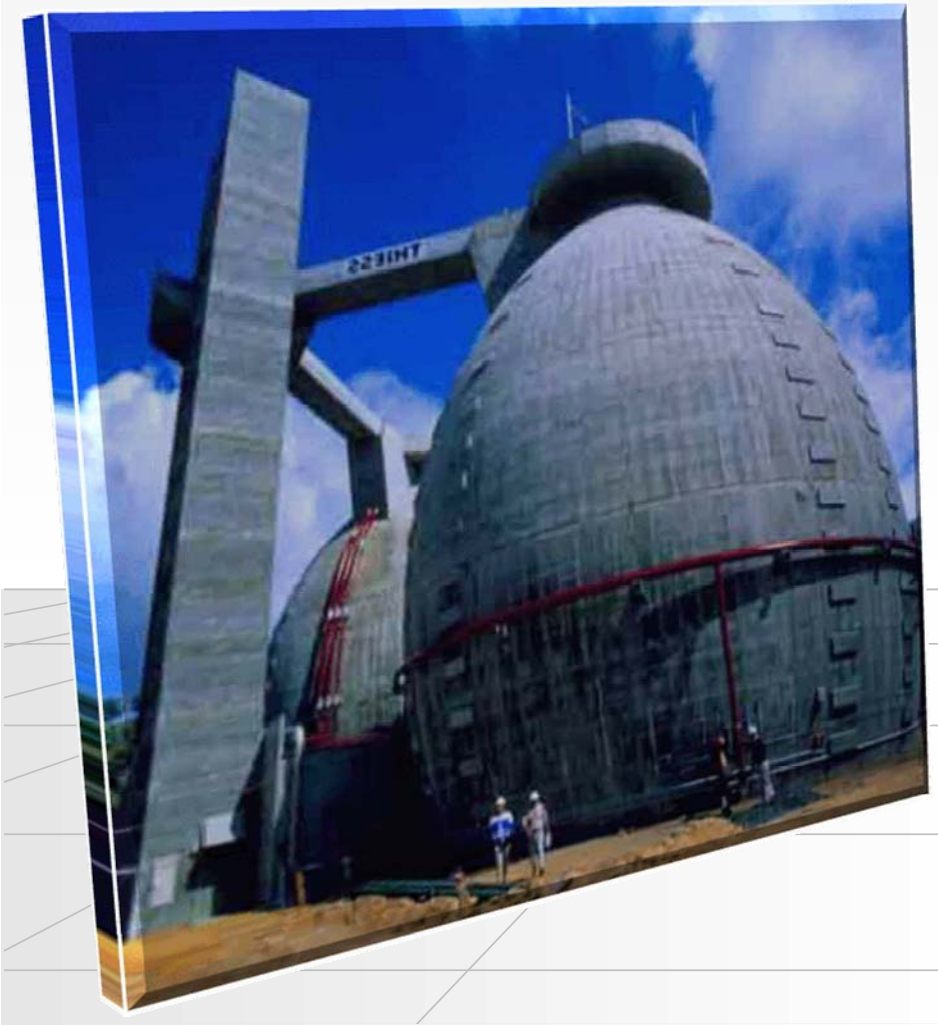
CONCLUSIONS

- 15 days SRT was found to be the effective digestion time.
- There was no methane content improvement.
- Degradation rate of Che-ult. pretreated sludge was faster than ultrasonic and non-pretreated sludge.
- Dewaterability of digested sludge was improved compared to feed sludge.
- Control digester at 25 days SRT was economically viable
- 15 days SRT with ultrasonic pretreatment was recommended.

RECOMMENDATIONS

- TS of WAS should be thoroughly investigated for both pretreatment and anaerobic digestion.
- Mathematical modeling by combining all affecting parameters should be studied.
- Chemical dose and power input of che-ult. Pretreatment should be more focused particularly simultaneous operation.
- Kinetic study should be further studied in a deep detail of the biodegradable organic fraction of WAS during AD.
- Pilot scale study should be implemented in order to get a reliable cost benefit analysis.

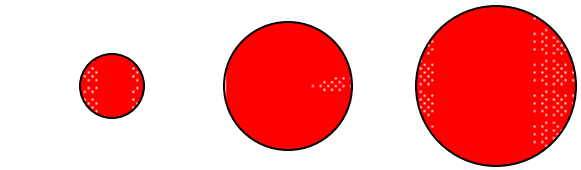
Thank you!



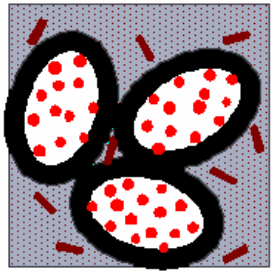
SCOPE OF THE STUDY

- WAS was collected from Future Park Rangsit WWTP
- TS content was increased to 3%
- Semi-continuous feeding (2 times/day).
- SRT: 10, 15, and 25 days.
- The sonics ultrasound unit
  - VC750 model, Newtown, CT, USA
  - Frequency of 20 kHz
  - Power input 750W

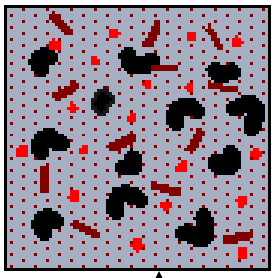
ULTRASONIC



Bubbles development



Sludge floc



Electrical energy → Pressure wave → Cavitation bubbles → Sludge disintegration

Factors affecting ultrasonication

Sludge characteristic

- TS content
- Type of sludge

Sonication conditions

- Frequency
- Power input
- Sonication time

Ultrasonic component

- Design of horn

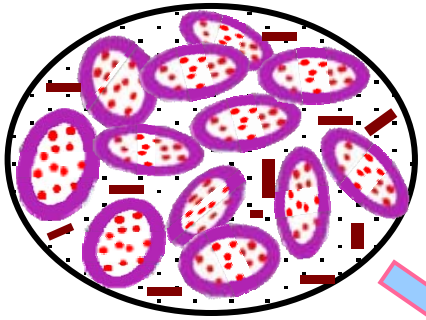
Temperature : 5000 K

IPa

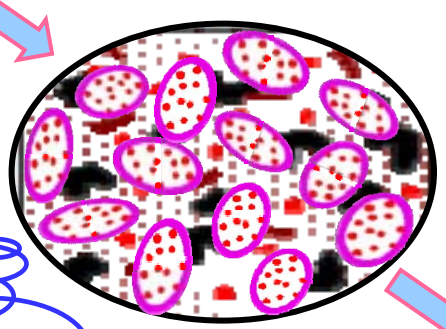
Reactive radicals : H, HO_2, OH, and H_2O_2

CHEMICAL AND CHEMICAL-ULTRASONIC

Raw Sludge

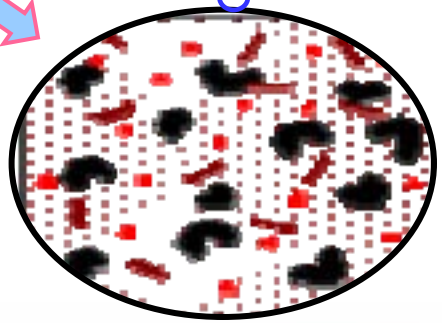


Chemical addition (NaOH)



1. Cell membranes are broken down
2. High SCOD

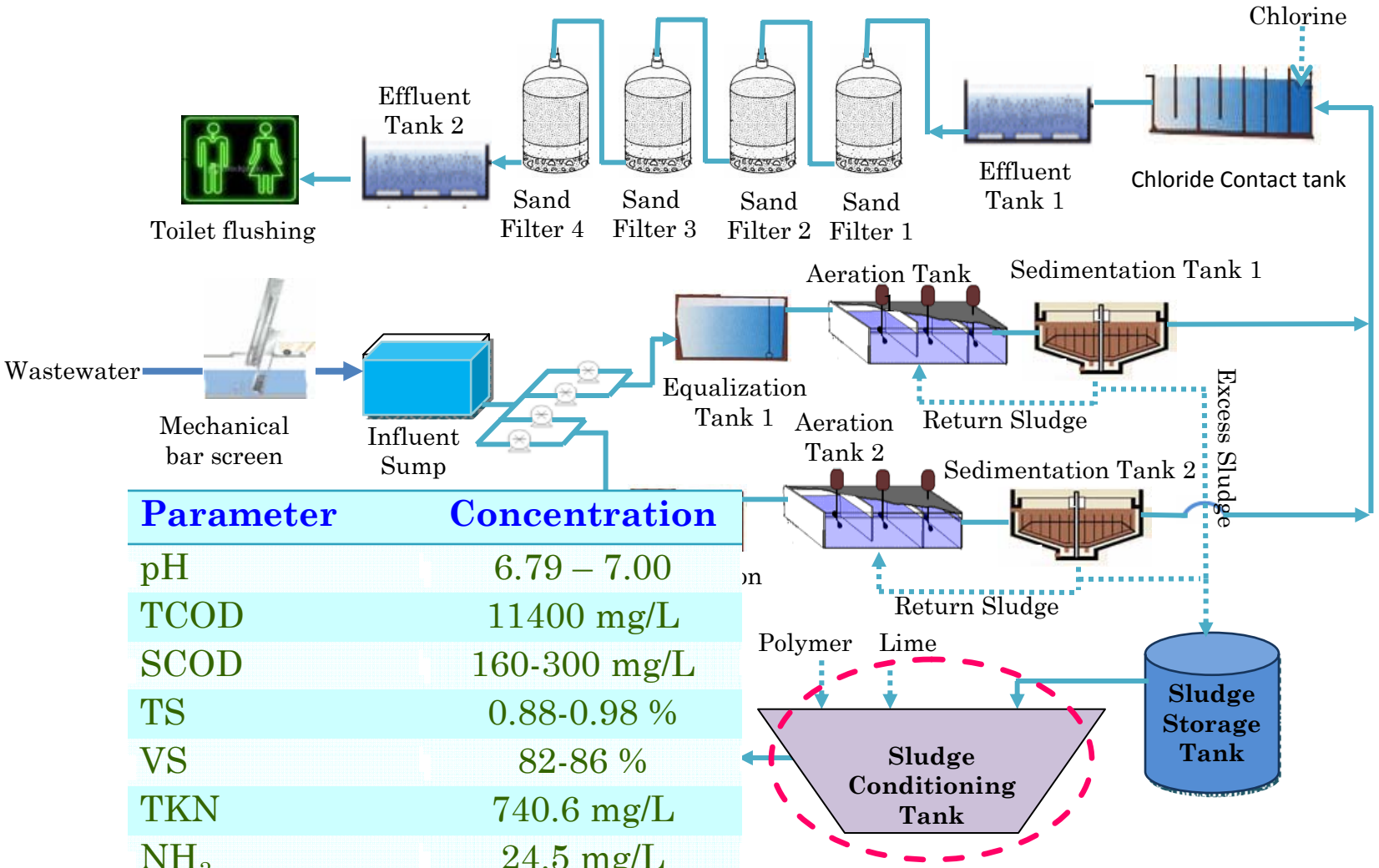
Sonication



1. Less effect to cell membrane break down
2. Make cell weak (less resistance)

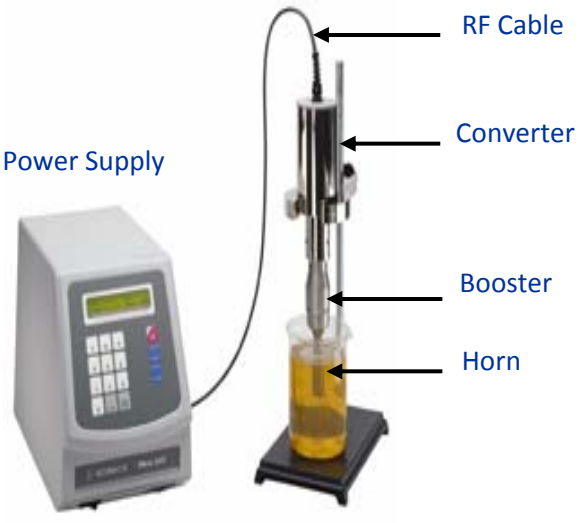
SAMPLE COLLECTION

Future Park Rangsit WWTP



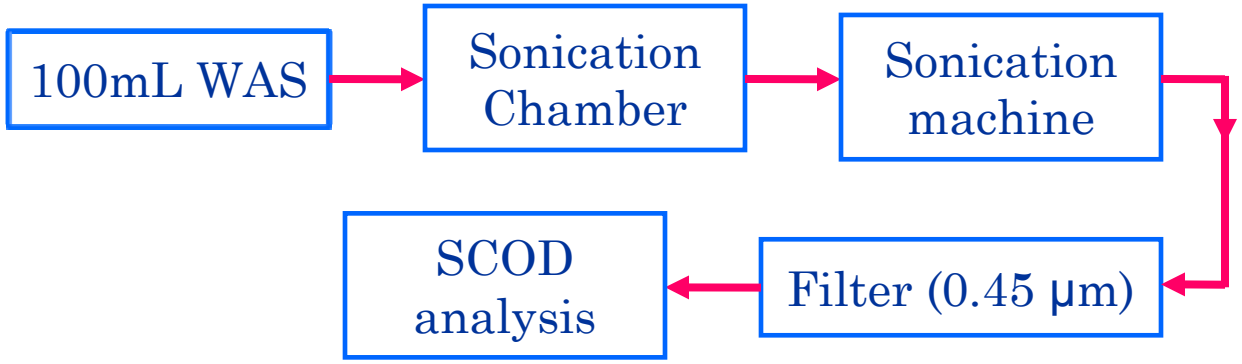
Parameter	Concentration
pH	6.79 – 7.00
TCOD	11400 mg/L
SCOD	160-300 mg/L
TS	0.88-0.98 %
VS	82-86 %
TKN	740.6 mg/L
NH_3	24.5 mg/L

SONICATION PRETREATMENT

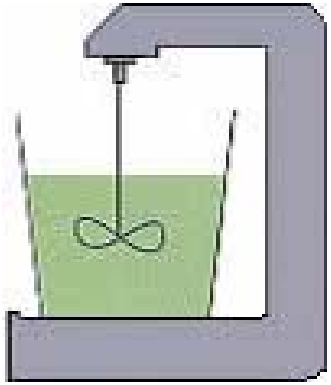


Parameters	Ultrasonic	Remarks
Sludge volume	100 ml	Fixed
Sonication times	30, 60, 120, 240 and 480 s	Variable
Power input	50, 100, 150 and 190 W	Variable
Frequency	20 kHz	Fixed
Probe immersed	2 cm	Fixed
Horn size	3.8 cm	Fixed

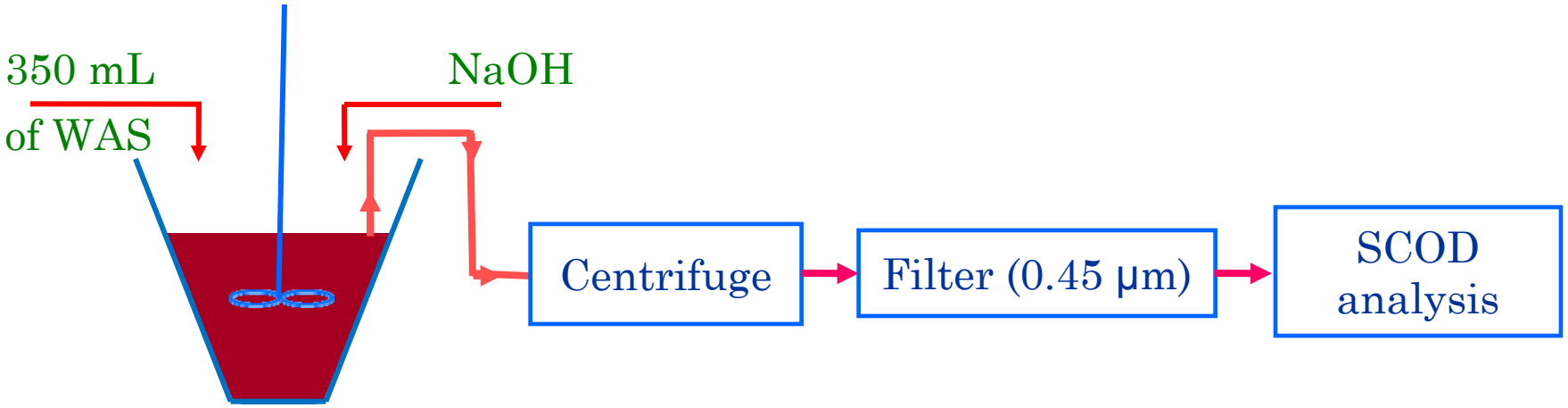
Sonication Chamber



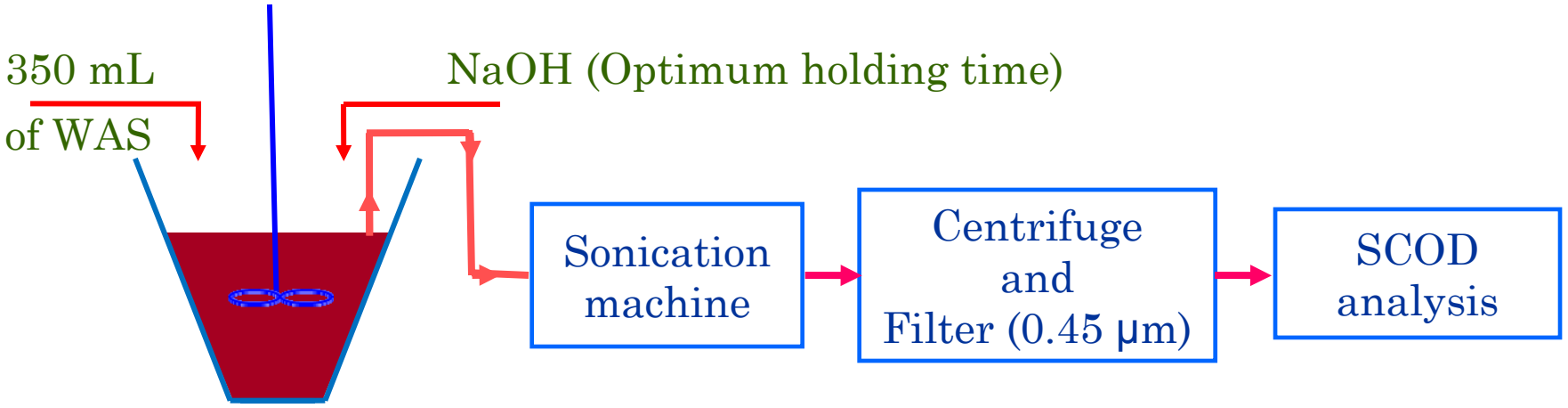
Chemical Pretreatment



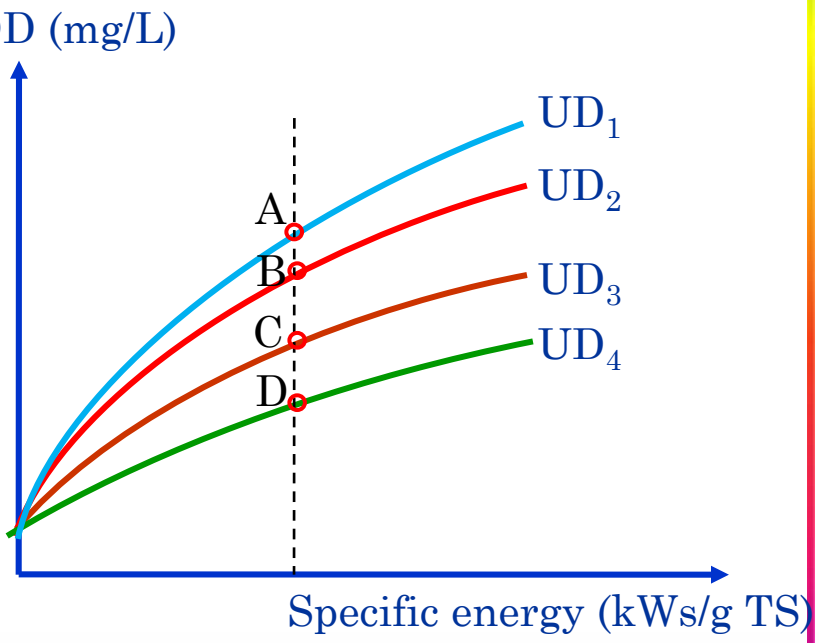
Parameters	Chemical	Remarks
Sludge volume	350 ml	Fixed
Contact time	10, 30, and 60 min	Variable
NaOH dose	25, 50 and 75 mg NaOH/g TS	Variable
Mixing	100 rpm	Fixed



CHEMICAL-ULTRASONIC PRETREATMENT



- ✓ **From the graph, A will be selected as optimum condition (High SCOD and Low SE)**
- ✓ **Optimum sonication time will be calculated by applying the equation (2).**



INHIBITORY COMPOUND IN AD

Ammonia Nitrogen

NH_3-N concentration (mg/L)	Effect
50-200	Beneficial
200-1000	No adverse effects
1500-3000	Inhibitory at pH over 7.4-7.6
Above 3000	Toxic

Total concentration of individual metals required to severed inhibit anaerobic digestion

Metal	Percent dry solids	M mole metal/kg dry solids	Soluble metal (mg/L)
Copper	0.93	150	0.5
Cadmium	1.08	100	-
Zinc	0.97	150	1.0
Iron	9.56	1710	-
Chromium			
+6	2.20	420	3.0
+3	2.60	500	-
Nickel	-	-	2.0

Stimulating and inhibitory concentrations of light metal cations

Cation	Stimulatory	Moderately inhibitory	Strongly inhibitory
Calcium	100-200	2500-4500	8 000
Magnesium	75-150	1000-1500	3 000
Potassium	200-400	2500-4500	12 000
Sodium	100-200	3500-5500	8 000

VFA AND ALKALINITY

Individual VFA	Healthy digestion	Failure of digestion
Acetic acid	< 8000 mg/L	> 8000 mg/L
Iso-butyric	< 5 mg/L	> 15 mg/L
Iso-valeric	< 5 mg/L	> 15 mg/L
Propionic/Acetic ratio	< 1.4	> 1.4

- **Alkalinity should be maintained in the range of 2500 to 5000 mg/L**

BMP Test Bottle

120 mL serum bottle

HEAT PREVENTING TECHNIQUE

HEAT PREVENTING TECHNIQUE