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THE EFFECT OF ULTRASONIC SLUDGE PRE-TREATMENT ON ANAEROBIC DIGESTION

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ABSTRACT

As sewage sludge dumping at sea is banned from 31st December 1998, water companies are having to find alternative outlets for large volumes of sludge. This project looks at a method of improving the conventional sludge treatment process of anaerobic sludge digestion, by pre-treating sludge with ultrasound. Ultrasound produces cavitation, which breaks up sludge solids, making it easier for bacteria to utilise them. In theory, this leads to a greater reduction in volatile solids and an increase in the volume of methane produced.

Sludge was thickened in a drum thickener and passed through a Nearfield Acoustical Processor (NAP-3606-HP-TC) ultrasound generator. Two different intensities were used, 5 amps and 16 amps. Sludge was then placed into the respective holding tanks of three 100 litre (l) capacity pilot scale digesters. Sludge treated at 5 amps was supplied to the first digester, sludge treated at 16 amps was supplied to the second digester, and the third digester received unsonicated sludge. Batch digestion tests, soluble chemical oxygen demand (sCOD) tests and capillary suction time (CST) tests were also carried out.

No major differences in gas production or volatile solids reduction were found between the three pilot scale digesters, possibly because the effects of sonication were negated during the sludge storage stage, due to shearing by the mixing device. However, the batch tests, soluble COD tests and CST tests carried out all showed that sonication had a marked effect. The CST tests showed that sonication made sludge harder to dewater, before and after subsequent digestion.

Future work should include a more detailed investigation into the effects of sonication on sludge dewaterability. This is because a pre-treatment process that produces a sludge that cannot be dewatered may not be economically viable, despite any advantages of the pre-treatment process.

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NOMENCLATURE

δ	Boundary layer thickness
η	Kinematic viscosity of a medium
ρ	Density of a medium
τ	Hydrodynamic stress
ξ_0	Displacement amplitude
μm	Micrometers
a	Radius of wire as an ultrasonic source
A	Amperes (amps); unit of frequency
BOD	Biological oxygen demand
c	Velocity of sound in a specific medium
CH_4	Methane
CO_2	Carbon dioxide
COD	Chemical oxygen demand
CST	Capillary suction time
$E_{(crit)}$	Critical shear stress
d_{50}	Equivalent diameter of particles at 50 % cumulative frequency
DoE	Department of the Environment
DWF	Dry weather flow
f	Frequency
H_2	Hydrogen Gas
H^+	Proton
H_2O	Water
H_2O_2	Hydrogen peroxide
HRT	Hydraulic retention time
H_2S	Hydrogen sulphide
KHz	Kilo hertz; unit of frequency
MHz	Mega hertz; unit of frequency
NAP	Nearfield acoustical processor

nm	Nanometer
NaOH	Sodium Hydroxide
OH^-	Hydroxyl radical
P	Energy in W.min
Pa	Pascals; measure of pressure
P_A	Maximal acoustical pressure of a sound wave
ppm	Parts per million
sCOD	Soluble chemical oxygen demand
SS	Suspended solids
SVI	Sludge volume index
TDS	Total dry solids
UK	United Kingdom
US	Ultrasound
UWWT	Urban waste water regulations
VFA	Volatile fatty acid
VS	Volatile solids
W	Watt; unit of power
$W.cm^{-2}$	Watts per square centimetre
$W.cm^{-3}$	Watts per cubic centimetre
W.min	Watts per minute
WWTW	Waste water treatment works

EXECUTIVE SUMMARY

E.1 INTRODUCTION

The EC Council Directive on Urban Wastewater Treatment (21st May 1991) stated that "member states shall ensure that by 31st December 1998 the disposal of sludge to surface waters by dumping from ships, by discharge from pipelines or by other means is phased out". This statement has been introduced into UK legislation under the Urban Waste Water Treatment (UWWT) Regulations 1994. UK water companies will therefore be required to find alternative sewage sludge disposal outlets for large volumes of sewage sludge.

Anaerobic digestion is a commonly used sludge treatment process, which stabilises sludge, reduces its volume and produces methane, a valuable fuel. This project investigates the use of ultrasound as a method of sludge pre-treatment to improve the anaerobic digestion process in terms of possible increases in volatile solids reduction and gas yields. Such improvements would result in a reduced volume of sludge that subsequently needs to be dewatered, transported and disposed of.

The effect of the ultrasound is to induce cavitation, which promotes conditions of intense localised heat, pressure and shear forces, which break up sludge solids. This releases previously unavailable volatile solids into the liquid, where they can be rapidly utilised by bacteria in the digester. The overall effect is a greater reduction in volatile solids content, and a consequent increase in the volume of gas produced. The effect of ultrasound on the dewaterability of the final digested sludge is also investigated.

E.2 LITERATURE SURVEY

A literature survey found that a number of studies have looked at various applications of ultrasound in sewage sludge treatment. Some studies have been carried out into the effect of ultrasound on the size of sludge particles, and found that it resulted in smaller

sized particles, which improved digestion efficiency. Another consequence of the reduction in particle size caused by ultrasonic treatment is that CST values increase so that dewaterability becomes more difficult. Conversely, another study reported that ultrasonic treatment of sludge improved dewaterability.

The effect of ultrasound on the enhancement of sludge sedimentation has also been investigated, with improvements in sedimentation rates reported.

Work has been carried out into the effect of ultrasound on sludge supernatant soluble COD and found that it increased following sonication.

A small number of studies have been carried out that look specifically at the effects of ultrasonic pre-treatment of sludge on the anaerobic digestion. The main findings of this work were an increase in volatile solids reduction and an increase in the volume of gas produced in digesters fed with sludge that had been sonically treated.

E.3 OBJECTIVES

The aim of the project is to investigate the effects that the ultrasonic pre-treatment of sewage sludge has on:

- The time necessary for satisfactory digestion to occur.
- The amount of volatile solids destruction and the resultant amount of methane produced during the anaerobic digestion process.
- The structure and consequent dewaterability of the sludge, following digestion.

E.4 MATERIALS AND METHODS

The practical work involved in this project was all carried out at Southern Water's Millbrook WWTW in Southampton. The works receive wastewater from Southampton docks and the residential areas of Millbrook and Shirley, at an average dry weather flow (DWF) of 350 l/s and a maximum flow of 850 l/s.

Sludge from the WWTW primary sedimentation tanks and waste activated sludge enter a sludge main. Sludge can be taken from the main when required, for use in pilot scale digester trials. In this trial, sludge was taken from the main and thickened using a drum screen thickener. It was then pumped through a Nearfield Acoustical Processor (NAP), with thermal control feature: model number NAP-3606-HP-TC (manufactured by Advanced Sonic Processing Systems, Connecticut, USA). Following sonication, sludge was placed into the respective holding tanks of three 100 l capacity pilot scale anaerobic digesters.

The first part of the practical work involved a scoping study to find out at what intensity the NAP would be run at and at what speed the sludge would be pumped through the NAP. The results of this work led to the decision to run the NAP at an intensity of 5 amps for sludge received by the first digester and at 16 amps for sludge received by the second digester . The remaining digester was fed with sludge that was pumped along the same route, through the sonicator, but was not sonicated, so acted as a control. The scoping study also helped to set the rate at which sludge was pumped through the ultrasound unit to 0.14 l/s.

The first stage of the main experiment involved setting up the digesters and running them until they were performing similarly, at this stage none of the digesters were fed with sonicated sludge. In the second stage, sludge sonicated at the low intensity of 5 amps was fed to the first digester, sludge sonicated at the high intensity of 16 amps was added to the second digester and the third digester continued to be fed unsonicated

sludge. The trial started on May 7th 1998, and lasted for 90 days. This time period was subdivided into six retention times, each of 15 days.

Throughout the trial, the daily volume of gas produced by each digester was recorded. Samples of feed and digested sludge were taken from each digester and tested for total dry solids (TDS), volatile solids (VS) and pH.

To compliment the pilot scale digester trial, batch digestion trials using 500 ml bottles were set up to try to find out more about what was happening inside the pilot scale digesters over time. Samples from this trial were tested for total COD, ammoniacal nitrogen content and volatile fatty acid (VFA) content at Southern Water's laboratories in Otterbourne.

Other work included testing the soluble COD of the 3 differently pre-treated sludges and their CST before and after digestion.

E.5 RESULTS

No marked differences in the volume of gas produced by the three digesters was found. Neither were any differences found in the percentage methane composition of the gas produced by the three digesters.

The percentage volatile solids reduction in all three digesters was also very similar, with no marked differences, as is shown in table E.1.

DIGESTER	5 amp ultrasound (US)	16 amp ultrasound (US)	Control
Average VS reduction (%) over 90 days	47.38	44.53	44.82

Table E.1 Average volatile solids (VS) reduction in pilot scale digesters

In the batch digestion tests, the bottles containing sonicated sludge produced more gas on average than those containing unsonicated sludge, with the bottles containing sludge sonicated at the higher intensity of 16 amps producing the greatest total volume of gas. Figure E.1 shows the total volatile fatty acids results of the first batch digestion test. The 16 amp ultrasound treated sludge shows a high peak compared to the 5 amp ultrasound treated sludge, with the lowest total level of VFAs found in the control sludge samples. Batch test 2 showed similar results.

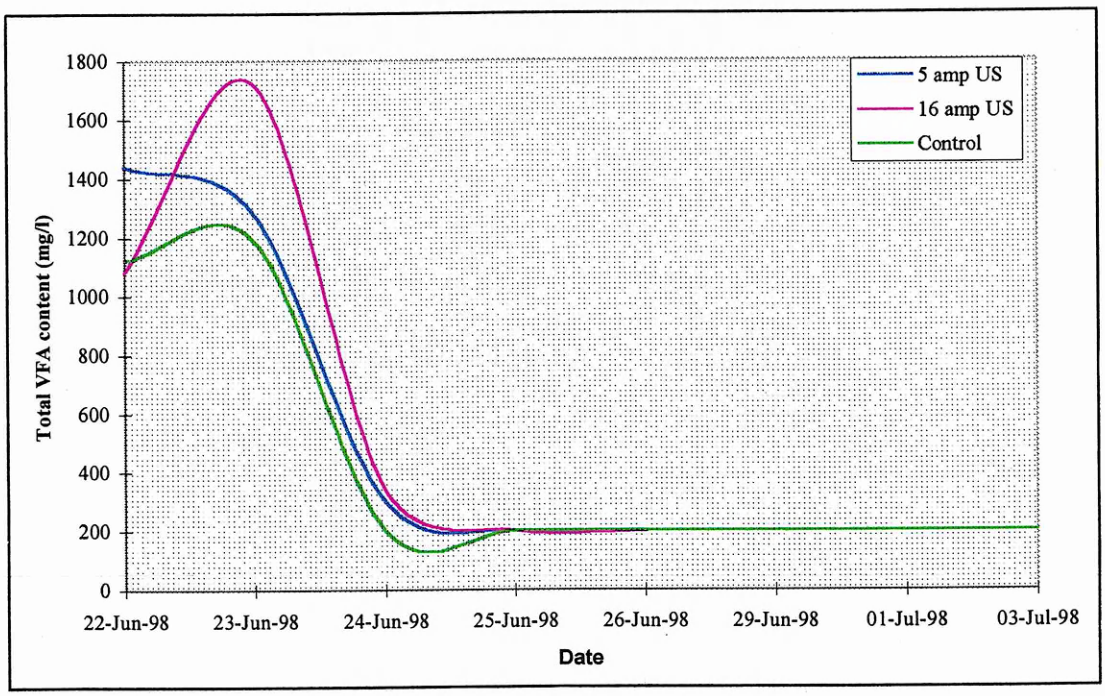

Figure E.1 Total volatile fatty acids results of the first batch digestion test

In total, five soluble COD tests were carried out on the three differently pre-treated sludges, prior to digestion. The highest average value found was from the 16 amp ultrasound treated sludge, at 1684 mg/l, the second highest was the 5 amp ultrasound treated sludge at 1571 mg/l. The control sludge samples had the lowest average soluble COD value of 1435 mg/l.

In all of the CST tests that were carried out, the results followed the same trend, with the high intensity sonicated sludge having the longest CST, followed by the low intensity

sonicated sludge, with the control sludge having the shortest CST. This trend was apparent in all of the samples, including those taken after digestion.

E.6 DISCUSSION

No significant difference was found in the gas production and volatile solids reduction of the three pilot scale digesters. This contradicts work reported by other authors that found more gas was produced by digesters that were fed sonicated sludge, the methane content of the gas produced was higher and that the volatile solids reduction was greater.

In the batch tests however, the findings were similar to previous work, with more gas produced by the sonicated sludges and a higher VFA production. The sharp increases in the total VFA levels of the samples sonicated with high intensity ultrasound are what might have been expected. The ultrasound causes cavitation, which breaks up the solids in the sludge, which makes more VFA's available for bacteria to rapidly utilise, which then leads to the sharp drop in VFA levels as they are used up.

Although there were major differences in the operation of the continuous fed and batch fed digesters, and the latter was of a much smaller scale, their performance was expected to follow similar trends. The most likely explanation for this is the use of the sludge holding tank in the main experiment. Sludge was held in a holding tank for up to four days before being fed into the pilot scale digesters. The holding tanks were continually mixed, which produces shear effects and so breaks up the sludge, possibly negating the effects of sonication.

The soluble COD tests that were carried out clearly show that sonication was causing the sludge to break up, releasing volatile compounds into the liquid portion of the sludge. The CST tests also show that sonication has marked effects on sludge structure as the CST values for sonicated sludge were much higher. The ultrasound made the particles of sludge smaller, which means the sludge gives up water less easily and so has

a higher CST. The fact that sonication makes sludge harder to dewater may have serious economic implications on the use of this technology.

E.7 CONCLUSIONS

- The batch tests and soluble COD tests both showed that sonication had a marked effect on the sludge, particularly when a high intensity was used.

- The pilot scale digesters did not perform as expected, possibly due to the effect of the sludge holding tanks, where the sludge was stored and subjected to shear forces, breaking it up. Once all the sludge had been broken up in this way, any effects of sonication would no longer be evident.

- The CST tests clearly show that treating sludge with ultrasound makes it harder to dewater, even after subsequent digestion. This is very important as there is no point using a pre-treatment process that leaves a sludge that cannot be dewatered, despite any advantages the process may have.

E.8 FUTURE WORK

Future work could look further into the effect of the holding tanks, but this is not a priority as on a large scale system, sonicated sludge would not be stored, it would be sonicated directly before entry into the digester.

Ideally, future work would involve a similar trial, but without the sludge holding stage.

The most important future work is to look further into the effect of ultrasound on the dewaterability of sludge once it has been digested. This is because if sonicated sludge does not dewater well enough after digestion, and no cheap methods of improvement can be found, ultrasonic pre-treatment of sludge prior to digestion is not viable.

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1.0 INTRODUCTION

The Council of the European Communities Directive on Urban Wastewater Treatment (21 May 1991) stated that "member states shall ensure that by 31 December 1998 the disposal of sludge to surface water by dumping from ships, by discharge from pipelines or by other means is phased out". This was introduced into UK legislation in the Urban Waste Water Treatment (UWWT) Regulations 1994.

In 1996, 30% of the UK's sewage sludge was disposed of at sea (Smith, 1996), which means that the UK water companies that rely on this outlet are now having to find alternative methods to dispose of large volumes of sludge. The other options that are available include application to agricultural land, incineration, land reclamation, landfill, forestry and dedicated sacrificial land.

Southern Water currently discharge sludge to sea from ships or by pipeline. At Millbrook Wastewater Treatment Works (WWTW) for example, sludge is stored in tanks and then pumped to a ship twice a week. From there, it is taken to be dumped off the Isle of Wight where it is dispersed by strong currents.

As a result of the ban on sea dumping from the end of 1998, the Southern Water Sludge Strategy has been developed. Sixteen sludge treatment centres will become the collection points for liquid sludge with solids content of approximately 3-4% from across the region. The sludge will be anaerobically treated and dewatered to form a treated sludge cake of approximately 25-30% solids. The sludge cake will then be disposed of to agricultural land as a soil conditioner.

The costs associated with sludge treatment are high, even when a useful agricultural biosolid and a fuel (methane gas) are produced. Southern Water's aim is therefore to reduce sludge volume to a minimum in as cost effective way as possible.

This project is a study into the effect of sludge pre-treatment using ultrasonics as a means to increase volatile solid destruction and therefore increase gas yield in the anaerobic digester. This in turn would reduce the volume of sludge to be dewatered and transported and hence reduce costs. An increase in the production of methane would also be economically advantageous, as would any possible improvements in sludge dewaterability of the final digested sludge.

This work will be carried out on a pilot plant at Millbrook Wastewater Treatment Works in Southampton. This treatment works receives the predominantly municipal wastewater from a population equivalent of 120,000 people. This site is suitable as the host of the project due to the availability of on-site analysis and the pilot equipment that is already established there as a result of earlier work carried out in this area of investigation. The work that was carried out previously was constrained by time and various other problems that led to inconclusive results. This new phase of work will seek to overcome these problems.

2.0 LITERATURE REVIEW

2.1 Anaerobic Digestion

Anaerobic digestion is used in the Water Industry to stabilise organic material and biological solids contained in sewage sludge. The resultant sludge is then suitable for application to agricultural land according to the EC *Directive on the Protection of the Environment, and in Particular of the Soil, when Sewage Sludge is Used in Agriculture* (86/278/EEC) and the UK legislation: *Code of Practice for Agricultural Use of Sewage Sludge* (Department of the Environment 1989).

2.1.1 Process Description

Anaerobic digestion is one of the oldest processes used for the stabilisation of biological wastewater sludges. It involves the decomposition of organic and inorganic matter in the absence of oxygen by microbial action. The specific objectives of anaerobic digestion according to the American society of civil engineers (1977) are to:

(a) reduce the mass and volume of sludges,

(b) obtain useful by-products, and

(c) destroy or control pathogenic organisms.

Anaerobic bacteria break-down the substrate feed sludge, in various stages, producing a number of by-products, the most significant of which is methane, which has economic value as a fuel. Other products include carbon dioxide, hydrogen sulphide and the remaining sludge, which is stable i.e. has a much lower biological oxygen demand (BOD) and greatly reduced pathogen content.

There are various methods of carrying out anaerobic digestion the most common being standard and high rate digestion processes. In the standard rate process, the hydraulic retention period is usually between 30 and 60 days. The digesters used in this process are generally not mixed or heated. High rate digesters have a shorter hydraulic retention time (HRT) of 15 days or less, as the reactors are heated and mixed. Mixing in anaerobic digesters is achieved through gas injection, mechanical stirring or mechanical pumping.

2.1.2 Process Microbiology

The biological conversion of organic matter during anaerobic digestion is thought to occur in three stages. The first stage is hydrolysis, where molecules of high molecular mass (such as proteins, lipids and polysaccharides) are broken down into compounds suitable for use as a source of energy and cell carbon, such as glucose.

The second step, acidogenesis, involves the bacterial conversion of the simple compounds produced during hydrolysis into simple organic acids, the most common of which in an anaerobic digester is acetic acid. The micro-organisms involved in this stage include facultative and obligate anaerobic bacteria, and are collectively known as "acidogens". Bacterial species from this group that have been isolated in anaerobic digesters include *Clostridium spp., Peptococcus anaerobus, Bifidobacterium spp., Desulphovibrio spp., Corynebacterium spp., Lactobacillus, Actinomyces, Staphylococcus* and *Escherichia coli.*

A third group of micro-organisms convert hydrogen and acetic acid formed by the acidogens to methane gas and carbon dioxide. The bacteria responsible for this conversion are strict anaerobes and all produce methane, and are hence referred to as "methanogens". The principle genera of micro-organisms that have been identified include rods such as *Methanobacterium* and *Methanobacillus*, and spheres, that include *Methanococcus* and *Methanosarcina*. The methanogens have very slow growth rates,

and as a result, their metabolism is usually considered rate-limiting in the anaerobic treatment of an organic waste.

2.1.3 Process Analysis

The disadvantages and advantages of the anaerobic digestion process compared to aerobic treatment, stem from the slow growth rate of methanogenic bacteria. Slow growth rates require a relatively long detention time in the digester for adequate waste stabilisation to occur. However, the low growth yield signifies that only a small portion of degradable organic waste is being synthesised into new cells. In fact, only 3-5 % of the infeed carbon is converted into bacterial growth and approximately 95 % of the carbon removed from the sludge is converted into biogas (typically 70 % methane), as compared to aerobic digestion, in which 50 % of the infeed carbon is converted into bacterial mass.

The methane produced is combustible and is therefore a useful end product. If sufficient quantities are produced, the methane can be used to operate dual-fuel engines to produce electricity and to provide building heat.

The high temperatures necessary to achieve adequate treatment are often listed as disadvantages of the anaerobic treatment process; however, high temperatures are necessary only when sufficiently long mean cell-residence time cannot be obtained at nominal temperatures. As the operation temperature is increased, the minimum mean cell-residence time is reduced significantly. Thus, heating of the reactor contents lowers not only the mean cell-residence time necessary to achieve adequate treatment but also the hydraulic retention time, so a smaller reactor volume can be used.

2.1.4 Feed Sludge Thickening

It is usual practice to thicken feed sludges prior to digestion. Thickening is a procedure used to increase the solids content of sludge by removing a portion of the liquid

fraction, which reduces the volume of sludge to be digested. Waste activated sludge typically has a solids content of approximately 1%, which can then be thickened in various ways to produce a sludge of 5-6% solids.

There are a number of process advantages that result from the pre-thickening of sludge:

- Thickening avoids the problem of reduced digester heating efficiency that is found with sludges that have a solids content of less than 4.8 % (Bruce *et al.* 1987).

- The volume of sludge to be treated is reduced, resulting in a reduction in digester size and heating costs, and an increase in mixing efficiency.

- Better control of the process is possible, due to greater stability.

- More concentrated sludge produces greater microbial metabolic efficiency.

2.1.5 Optimisation of the Anaerobic Digestion Process

2.1.5.1 Feed particle size

Limited literature has been published on the effect of feed sludge particle size on the anaerobic digestion process. It is reasonable to assume however that the smaller the particle size, the greater the surface area to volume ratio available for conversion to methane by micro-organisms.

Lawler *et al.* (1986) found that the rate of hydrolysis is directly related to the surface area of particles available. Kayhanian and Hardy (1994) showed that the rate of methane gas production was inversely proportional to the average particle size of office paper fed into an anaerobic digester. This work therefore suggests that pre-treatment of sludge by a process that decreases particle size would result in more effective, faster digestion with a consequent increase in methane production.

2.1.5.2 Mechanical Disintegration

Mechanical disintegration is a well known process to obtain intracellular products such as proteins or enzymes in biotechnological applications. Kopp *et al.* (1997) looked at anaerobic digestion and dewatering characteristics of mechanically disintegrated excess sludge. The best results were found using a stirred ball mill and a high-pressure homogeniser. It was shown that the mechanical disintegration resulted in a disruption of particle structure and a decrease in digestion time. Similar work was also carried out by Baier and Schmidheiny (1997).

2.1.5.3 Chemical Pre-treatment

Chemical pre-treatment is a process that can be used to hydrolyse and decompose lipids, hydrocarbons and protein into smaller soluble substances such as aliphatic acids, polysaccharides, and amino acids. This can be achieved through the addition of enzymes (Knapp and Howell, 1978), ozonation (Yasui and Shibata, 1994) acidification (Gaudy *et al.*, 1971) or through alkaline hydrolysis. Chiu *et al.* (1997) compared the soluble chemical oxygen demand (sCOD) of sludge following alkaline (NaOH) treatment alone, simultaneous alkaline and ultrasound treatment, and alkaline treatment followed by ultrasound treatment. The alkaline and simultaneous ultrasound treatment was found to have the highest hydrolysis rate.

2.1.5.4 Temperature

Hatziconstantinou *et al.* (1996) found that by controlling the temperature and retention time of an anaerobic digester, it is possible to optimise hydrolysis, thus maximising the rate at which soluble organics become available. This was shown in terms of a significant increase in the soluble chemical oxygen demand (sCOD) of the feed sludge.

Heat pre-treatment of activated sludge to 120° C has been shown to release large amounts of organic matter from the insoluble fraction into the filtrate (Paulson, 1965),

and thus improves subsequent biological degradation processes. The author compared the effects of heat pre-treatment with the effects of ultrasonic pre-treatment of the sludge, and gained comparable results. This suggests that it would be possible to pre-treat primary sludge with ultrasound to improve digestion through an increase in volatile solids reduction and a consequent increase in methane yield.

2.2 Ultrasonics

Ultrasound is sound pitched above human hearing, at a frequency between 16 kHz and 10 MHz. Two distinct ranges of ultrasound have been identified: diagnostic and destructive. Diagnostic ultrasound (frequency 2-10 MHz, power input 1 $mW.cm^{-2}$) has physical measurement applications. Examples of such applications include SONAR measurements, foetal scanning and metal fault detection. Destructive ultrasound (frequency 16-100 kHz, power input 1 to 10^3 $W.cm^{-2}$) has the ability to influence physical properties and chemical reactivity. It is destructive ultrasound that is of interest to this project as it produces irreversible physical and/or chemical changes in a media. The destructive effects of ultrasound have been utilised effectively by chemists to aid chemical reactions, such as polymerisation, polymer destruction and the destruction of organic chemical contaminants in water (Hoffman *et al.*, 1996; Drijvers *et al.*, 1996; Francony and Petrier, 1996; Toy *et al.*, 1993; Kotronatou *et al.*, 1992). Such use of ultrasound in chemistry, "sonochemistry" has recently received a great deal of attention. An example of a physical effect of ultrasound is the mechanical disruption of settling particles to reduce fouling in cross-flow microfiltration (Tarleton, 1992).

Ultrasonics is also commonly used in industry as a method of homogenisation and emulsification, particularly in the food industry (Mason and Lorimer, 1988). Another application of high-intensity ultrasound is in medicinal research, where it has been used to disrupt cells in order to extract active antigens for making vaccines and also in the study of lipids, enzymes and viruses (Shoh, 1988).

2.2.1 Ultrasonically Enhanced Sedimentation

Kowalska *et al.* (1979) and Bien *et al.* (1979) discussed the use of ultrasound to supplement the addition of flocculating agents to sludges from sewage treatment plants. Ultrasound of frequency 20 kHz produced beneficial effects, but used in conjunction with the addition of polyelectrolyte flocculating agents, ultrasound was found to be effective in increasing the rate of sedimentation and the strength and density of the flocs, reducing the final volume of the resultant sludge by 20 %.

King and Forster (1990) subjected samples of activated sludge from domestic sewage to ultrasound of 20 kHz frequency. Their conclusions were that high ultrasonic intensity levels caused an increase in settling rate but at the expense of supernatant clarity. Sonication of the sludge in the range of 7.5 - 75 W.min caused shearing of the sludge flocs and a significant reduction in ease of filtration of the sludge due to a reduction of the mean particle size in both the sludge and the supernatant. The authors outlined the need for further detailed studies in order to explain the contradiction between these results and those of Bien *et al* (1979).

Bien (1988) discussed the use of low frequency ultrasound (20 kHz) in the preparation of mineral and organic sludges to improve the levels of dewatering achieved by filtration. Sonication was found to reduce final hydration by around 10 %. As a result of the joint action of ultrasound and polyelectrolytes it was possible to reduce the dose of the polyelectrolyte added by 50 %.

It is evident that despite the diverse research that has been carried out, there is little evidence to suggest that large-scale sonically-enhanced processes are widespread in industry. This is possibly due to the high capital cost of installing industrial scale acoustic and ultrasonic equipment, the relative lack of process scale data and lack of agreement as to the mechanisms and effects of sonication.

2.2.2 Cavitation

Ultrasonic waves, like all sound waves, consist of alternating cycles of compression and expansion. Compression cycles exert a positive pressure on the liquid, pushing the molecules together; expansion cycles exert a negative pressure, pulling the molecules away from one another. During the expansion cycle a sound wave of sufficient intensity can generate cavities (bubbles). A liquid is held together by attractive forces, which determine it's tensile strength. In order for a cavity to form, a large negative pressure associated with the expansion cycle of the sound wave is needed to overcome the liquid's tensile strength.

The amount of negative pressure needed depends on the type and purity of the liquid. For pure liquids, tensile strength is so great that the ultrasound generators available cannot produce enough negative pressure. Most liquids however are sufficiently contaminated by small particles to initiate cavitation.

Once formed, depending on their size the cavities grow and contract in phase with the alternating compression and expansion cycles of the sound wave, striking a dynamic balance between the vapour inside the cavity and the liquid outside. If the ultrasound is of a high intensity, the cavity can expand so rapidly during the negative-pressure cycle that the cavity never has a chance to shrink during the positive-pressure cycle. In this process, therefore, cavities can grow rapidly in the course of a single cycle of sound.

For low-intensity ultrasound the size of the cavity oscillates in phase with the expansion and compression cycles. The surface area of a cavity produced by low-intensity ultrasound is slightly greater during expansion cycles than during compression cycles. Since the amount of gas that diffuses in or out of the cavity depends on the surface area, diffusion into the cavity during expansion cycles will be slightly greater than diffusion out during compression cycles. For each cycle of sound, the cavity expands a little more than it shrinks so that over many cycles the cavities will grow.

The growing cavity can eventually reach a critical size where it will most efficiently absorb energy from the ultrasound. The critical size depends on the frequency of the ultrasound wave. Once a cavity has experienced a very rapid growth caused by either low- or high-intensity ultrasound, it can no longer absorb energy as efficiently from the sound waves. Without this energy input the cavity can no longer sustain itself. The liquid rushes in and the cavity implodes.

When gases are compressed, they heat up. As bubble cavities implode in a liquid irradiated with ultrasound, this compression happens so fast that little heat can transfer from the bubble to the liquid. Extremely high temperatures, equivalent to the range of temperatures that are found on the Sun's surface (5000 °C), are generated nearly instantaneously, in a localised "hot spot" (Suslick and Doktycz, 1990). In addition, the collapsing liquid wall surrounding the bubble will compress the contents to pressures of hundreds of atmospheres (Suslick and Doktycz, 1990). This all happens in less than a microsecond without heating the bulk of the liquid

As the cavity collapses, the heat produced sends shock waves into the surrounding liquid, travelling at or greater than the speed of sound. These shock waves have been found to induce pressures up to 3000 atmospheres with lifetimes of approximately 40 nanoseconds (Suslick and Doktycz, 1990). The shock waves can also cause small particles to be accelerated to speeds as high as 500 kilometres per second. These fast-moving particles go on to collide with other particles causing large amounts of structural and chemical alterations to the solids in the liquid.

It is universally accepted that the immense temperatures and pressures developed in "hot spots" of imploding cavities are the main cause of the sonochemical effects of ultrasound, and that the extreme conditions provide a unique catalytic environment. In water the effect of cavitation is to decompose the H_2O molecule forming extremely reactive protons (H^+) and hydroxyl radicals (OH^-). These radicals can recombine, as the local hot spot is instantly cooled, to form hydrogen peroxide (H_2O_2) and molecular

hydrogen (H_2). Evidently when other chemicals are added to the water, cavitation can support a wide range of reactions.

2.2.3 Factors Influencing Cavitation

There are a number of factors that influence the extent of cavitation, and so can be used to control it, these include; properties of the solvent, temperature, ultrasonic frequency, dissolved gas content, external pressure and ultrasonic intensity.

2.2.3.1 Properties of the solvent

A viscous solvent will require waves with greater amplitude and therefore greater intensity in order to break the cohesive forces of the solvent to form cavities. However, once a cavity is formed, the effects of vapour pressure on temperature and pressure will control the violence of the collapsing cavity.

2.2.3.2 Temperature

Connolly and Fox (1954) examined the thresholds of ultrasonic cavitation in water in relation to a number of external factors including the temperature of the liquid. The dependence of the cavitation sound pressure threshold on temperature was found to be non-linear and increased by a factor of 2.5, suggesting that for cavitation to occur at lower temperatures, a greater intensity of ultrasound is required.

2.2.3.3 Ultrasonic Frequency

The sound pressure threshold for the onset of cavitation increases with increasing ultrasonic frequency. At very high frequencies the cavities have insufficient time for growth during the expansion phase of the wave before being influenced by the compression phase. This frequency relationship is further complicated by the dependency of the threshold on the size of the cavities already present in the liquid as

there is an optimum cavity size that corresponds to a resonant frequency. As the frequency increases the optimum cavity radius for cavitation decreases (Lewin & Bjomo, 1981). Cavitation is unlikely to occur at frequencies greater than 3 MHz, even at very high intensities.

2.2.3.4 Dissolved Gas Content

The presence of dissolved gas in a sonicated liquid greatly increases the risk of cavitation. Galloway (1954) found that the sound pressure required to initiate cavitation in de-aerated water at 20-40 kHz was in the order of 2×10^7 Pa and fell to 1×10^5 Pa at 100 % saturation. Therefore, the less gas present in a liquid, the more sound pressure is required to produce cavitation.

2.2.3.5 External pressure

The cavitation threshold depends on the expansion phase of the sound wave generating negative pressures that overcome the ambient pressure on the system. Therefore, increasing the external pressure increases the cavitation threshold for the system.

2.2.3.6 Ultrasonic Intensity

Intensity is defined as the energy flow per unit area per unit time, and is given by the following equation (Pestman, 1994):

$$I = \frac{P^2_A}{(2.\rho.c)}$$

where P_A is the maximum acoustic pressure of the wave, ρ is the density of the medium and c is the velocity of sound in that medium. As the intensity is proportional to P_A, it therefore follows that increasing the intensity will increase the sonicating effect of the wave.

2.2.4 Effect of Ultrasound on Particle Size

Williams *et al.* (1970) and Banks and Walker (1977) reported the use of ultrasound to break up activated sludge flocs in order to collect viable bacterial cells for analysis. Williams *et al.* (1970) describe previous attempts that had successfully broken down flocs to provide discrete viable cells, but also caused some bacterial disruption leading to artificially low counts. By using a novel ultrasonic device which generated acoustic microstreaming around a thin wire, a shear force could be chosen which would disperse flocs but would not destroy the bacteria. In fact, cell counts were found to increase 20-fold after 30 minutes sonication at a frequency of 20 kHz. Banks and Walker (1977) established that the shearing effect was dependent on the intensity of sonication and not the duration of the treatment, with an input of 26 W providing optimum recovery of viable cells. Intensities above this optimum were found to cause cell destruction. King and Forster (1990) found that increased levels of sonication led to an increased number of smaller particles (1-4 μm) in activated sludge. This was due to the corresponding release of free micro-organisms smaller than 4 μm from the flocs which had a mean particle size of 8 μm.

The approximate level of hydrodynamic stress (τ) can be calculated to establish the forces exerted on a floc particle during sonication (Nyborg, 1965).

$$\tau = \frac{\eta.2.\pi.f.\xi_0^2}{a.\delta}$$

where η= kinematic viscosity of suspending medium; f = frequency; ξ_0 = displacement amplitude (cm); a = radius of wire (cm); δ = boundary layer thickness (cm) (4 μm for water).

Williams and Nyborg (1970) found that any particle denser than the surrounding media would be subjected to radiation pressure forces that would attract it to regions of high acoustic pressure amplitude. Large flocs are therefore disrupted quickly whilst smaller particles will only do so at a constant rate depending on the volume of media being treated per unit of time.

Ultrasonics has been used to apply shear forces to activated sludges (Hall, 1981; King and Forster., 1990). King and Forster (1990) were able to produce an expression relating the amount of energy applied to activated sludge by sonication with the particle sizes produced. This was confirmed by Morgan and Forster (1992). The expression is defined as:

$$d_{50} = 6.31\ (P + 0.1)^{-0.099}$$

where d_{50} = equivalent diameter at 50 % cumulative frequency and P = energy (W.min) (King and Forster, 1990) or specific energy (kJg^{-1}) (Morgan and Forster, 1992). Morgan and Forster (1992) produced graphs to find the critical shear stress (E_{crit}) required for complete disruption of sludges. This was achieved using data collected based on the turbidity of the supernatant and d-50 values following sonication. The E_{crit} can be used as a measure of floc stability which is dependent on the amount of filamentous microbes and the presence of extracellular polymers produced in aerobic and anaerobic sludges. It has been found that when energy is introduced to biological sludges, these polymers are released into solution (King and Forster, 1990; Forster, 1988). King and Forster (1990) found that treatment of activated sludge with ultrasound could lead to a 200 % increase in soluble carbohydrate concentration. Mustapha and Forster (1985) found the nature of the release of polymers to be dependent on whether the sludge is filamentous or not. Brown and Lester (1980) however, found that the extraction of extracellular

polymer from activated sludge by sonication (10 minutes at 18 W) and centrifugation (low and high speed) was unsuccessful. However it was suggested that, if used in conjunction with another method, then polymer extraction from flocs may be possible.

Ultrasound can also be used to increase the size of particles, through agglomeration. Agglomeration is possible if the intensity of the ultrasound used is kept below the critical intensity required for cavitation (Muralidhara *et al.*, 1987) and at high frequencies (Pankou and Jekel, 1996). As particles undergo sonication, they move in response to the sound waves. Small particles undergo relatively large movements whilst larger particles are more able to resist the movement. The sound induced movement of the small particles causes them to collide more frequently with other particles. If particles stick following collision, then the mass and surface area will increase. Agglomeration also leads to the release of interstitial and surface water as free water from the particles due to the increase in surface area.

2.2.5 Effect of Ultrasound on Sludge Characteristics

2.2.5.1 Dewaterability and Capillary Suction Times

The potential dewaterability of a sludge is commonly measured using the capillary suction time (CST). CST is a measurement of the time required for a small volume of filtrate to be withdrawn from a sludge when subjected to the capillary suction pressure of dry filter paper. The CST can then be used as an indication of the free water available in a sludge and its resistance to removal.

Particle size can be an indication of a sludges dewaterability (Lawler *et al.*, 1986). It was found that the surface area of sludge is particularly important as it provides a surface for water to attach, and also provides frictional resistance to the withdrawal of water. Karr and Keinath (1978) showed that a sludge with a "supracolloidal" fraction (approximately 1 to 100 μm) had the most significant detrimental effect on dewaterability.

Several studies have used CST as a method for determining the break up of sludges due to sonication. Hall (1981) subjected activated sludge to low sonication levels of between 3.25 and 32.5 W.min and found there were large increases in CST values due to shearing of flocs and thus a release of water bound up in them. It should be noted that the range of sludges used showed differing CST values of up to 300 seconds, indicating the variable characteristics of activated sludges. FitzGerald *et al.* (1993) found that sonication had a considerable effect on the CST of wastewater sludges that had been sonicated with different intensities for various lengths of time. However, Hall (1981), found that a higher intensity range of 7.5-75 W.min was required to cause sufficient disruptive shear of activated sludge flocs.

Kowalaska *et al.* (1979) studied the effect of a short one or two minute burst of ultrasound on the CST values of various sludges containing different polyelectrolytes. Depending on the polyelectrolyte used, it was found that there were multiple increases in CST values. These sludges were found to have shorter CST values than sludge containing no polyelectrolyte. FitzGerald *et al.* found a stronger link between total dry solids (TDS) and CST than suspended solids (SS) which implies that solutes may contribute to CST values.

It is important to consider temperature when studying the effect of ultrasound on CST values, as sonication causes sludge to increase in temperature. FitzGerald *et al.* (1993) reported that CST values of sludges decreased as temperature increased.

2.2.5.2 Sludge Settleability

King and Forster (1990), Kowalska *et al.* (1979) and Lyon (1951) found that settleability of activated sludges improved following sonication. This improvement of settleability was measured as a ratio of sediment height to total volume after a certain settlement period (Lyon, 1951) or as a percentage of the initial sludge volume index (SVI) (King and Forster, 1990). The SVI is a method of defining a sludge's

characteristics and involves taking 1 dm^3 of sludge and allowing it to settle for 30 minutes.

According to Kowalska *et al.* (1979) when flocs are broken down by sonication, the settled sludge will be more dense and compact easily, due to the fact that the porosity of a sediment is proportionally dependent on the particle size. King and Forster (1990) however, found that this phenomenon was at the expense of supernatant suspended solids, which increased with sonication power. Conversely, Paulson (1965) stated that the ultrasonic treatment of activated sludge led to a reduced suspended solids concentration and impaired settleability.

Kowalska *et al.* (1979) and Bien *et al.* (1980) found that the type of polyelectrolyte used for thickening the sludge following sonication, had a direct effect on the settleability of the sludge under gravity. Polyelectrolytes designed for vacuum filtration and centrifugation increased settled sludge volume when compared to unprepared and non-sound irradiated sludges. Polyelectrolytes designed for gravity thickening, however, reduced settled sewage sludge volume, a property enhanced by sonication. Bien *et al.* (1979) found, however, that these gravity thickening polyelectrolytes were unsuccessful in reducing sludges from cellulose and paper industries. Bien (1988) also found that hydrated sludge, when treated with polyelectrolyte and ultrasound, had an improved percentage hydration, and required nearly 50 % less polyelectrolyte dosage than if dosed and not sonicated.

2.2.6 Effect of Ultrasound on Anaerobic Digester Performance

Clark and Nujjoo (1998) looked at the effects on digestion of sludge pre-treatment with ultrasound. It was found that sludge sonication could increase gas yields by up to 60 %. It was also found that the methane content of digester gas increased by 5-10 %. The digesters used were very small (10 litres) and were fed daily.

Tiehm et al (1997) investigated the effect of ultrasound on sludge supernatant COD and found that ultrasonic treatment caused an increase in the soluble COD of sludge supernatant. Tiehm et al (1997) also looked at the volatile solids of sonicated and unsonicated sludges following digestion. It was found that the sonicated sludge gave rise to an extra 4.5 % reduction in volatile solids compared to unsonicated sludge.

3.0 OBJECTIVES

The aim of the project is to investigate the effects that the ultrasonic pre-treatment of sewage sludge has on:

- the time necessary for satisfactory anaerobic digestion to occur,
- the amount of volatile solids destruction and the resultant amount of methane produced during the anaerobic digestion process and,
- the structure and consequent dewaterability of the sludge, following digestion.

4.0 MATERIALS AND METHODS

4.1 Overview

The practical work involved in this project was all carried out at Southern Water's Millbrook WWTW in Southampton. The works receive wastewater from the docks, and the residential areas of Millbrook and Shirley, at an average dry weather flow (DWF) of 350 l/s and a maximum flow of 850 l/s. A process flow diagram for the works is shown in figure 4.1.

Wastewater entering the works passes through a set of mechanically raked 12 mm bar screens, followed by a grit removal chamber, before being split into two streams to feed the original works and new section respectively. At this point, 5 % of the flow is redirected through a pilot scale lamellae settler. The settled sewage from the settler is returned to the head of the works, whilst the sludge generated is pumped to a holding tank containing a mixing pump. Overflow from this tank is returned to the head of the works.

Sludge from the holding tank can be pumped through a drum screen thickener, following the addition of Zetag 78 polyelectrolyte, and thickened from approximately 1 % dry solids up to 6-7 % dry solids. The thickening process is controlled by means of a variable speed sludge feed pump and a variable speed polyelectrolyte pump, so that the dosage of both can be adjusted. Thickened sludge is pumped into a small holding tank, from where it is pumped through a variable speed progressive cavity monopump, through a Nearfield Acoustical Processor (NAP), into 25 litre containers. From here, the sludge is taken to where three pilot scale digesters are located and is put into the three respective sludge holding tanks. The sludge treatment system is shown in a diagram in figure 4.2. The NAP and the pilot scale digesters are discussed in more detail in sections 4.2.1 and 4.3.1 respectively.

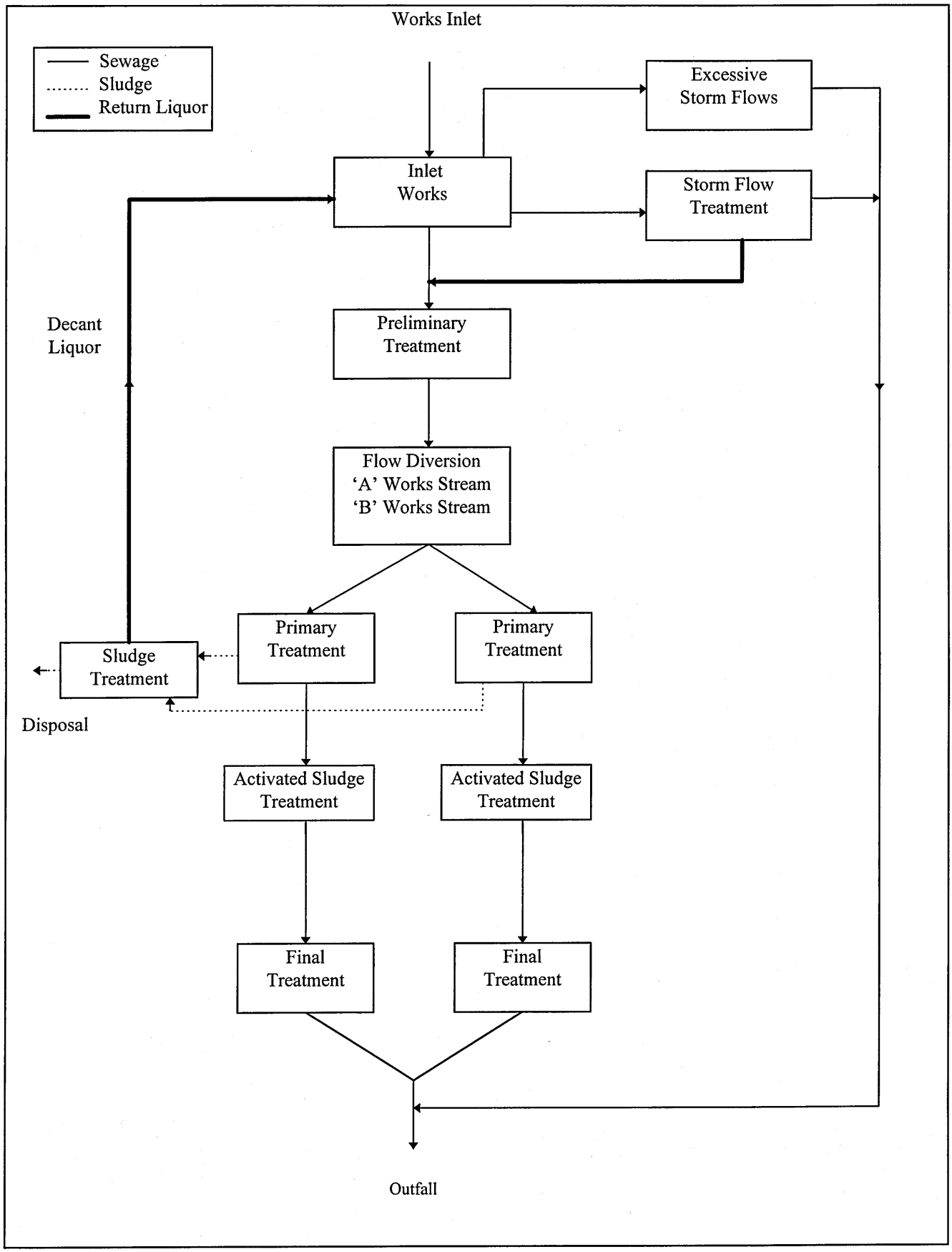


Figure 4.1 Process Flow Diagram of Millbrook WWTW

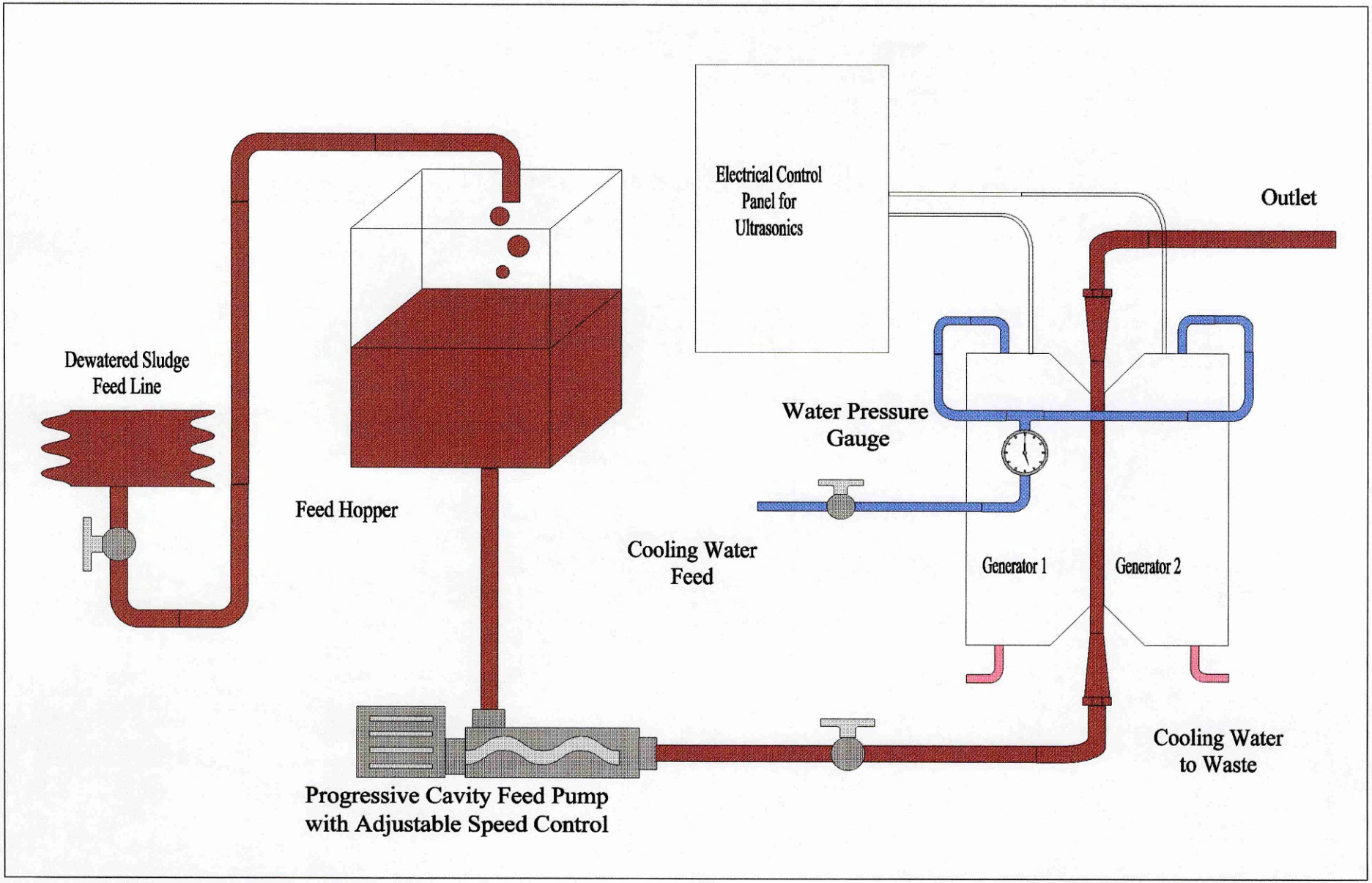


Figure 4.2 Sludge treatment process diagram

4.2 Scoping Study

A scoping study was carried out to gain the information needed to begin the main experimental section of the project. The main work in this study was determining what settings to use on the NAP ultrasonic generator.

4.2.1 Nearfield Acoustical Processor (NAP) Description

The Nearfield Acoustical Processor, with Thermal Control Feature: model number NAP-3606-HP-TC was manufactured by Advanced Sonic Processing Systems, Connecticut, USA. A photograph and diagram of the unit are shown in plate 4.1 and figure 4.3 respectively.

The unit consists of two parallel plates, separated by a spacer, which are vibrated at two different frequencies (16kHz and 20 kHz) by transducers set onto each plate, producing potential intensities of 100 $W.cm^{-3}$. Two different frequencies are required to prevent the formation of standing waves, which would produce less effective compression and expansion sites, and consequently, less intense cavitation and mixing.

The NAP unit can be set with the power on filtered or unfiltered mode, producing either stable or transient cavitational energy. Filtering the power supply produces a constant power amplitude and results in a stable and predictable cavitational effect. By not filtering the power supply, a chaotic wave pattern is produced which could result in increased cavitational effects, depending on the substance that is being sonicated.

The NAP unit has a thermal control feature by means of a water cooling system, which passes cooling water through the transducers. In this case, this system was not deemed necessary as the sludge temperature only increased by a maximum of 5 °C during sonication.

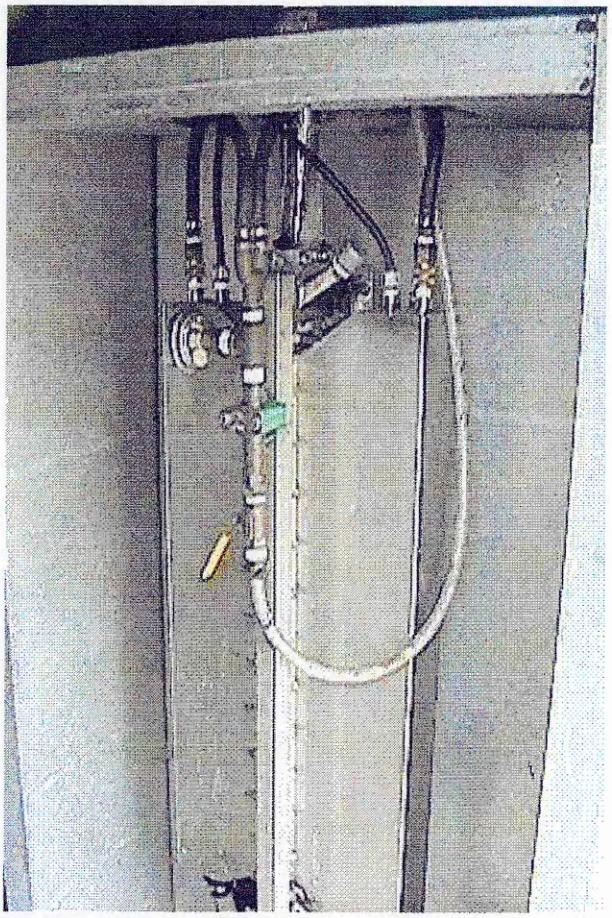


Plate 4.1 Nearfield Acoustical Processor

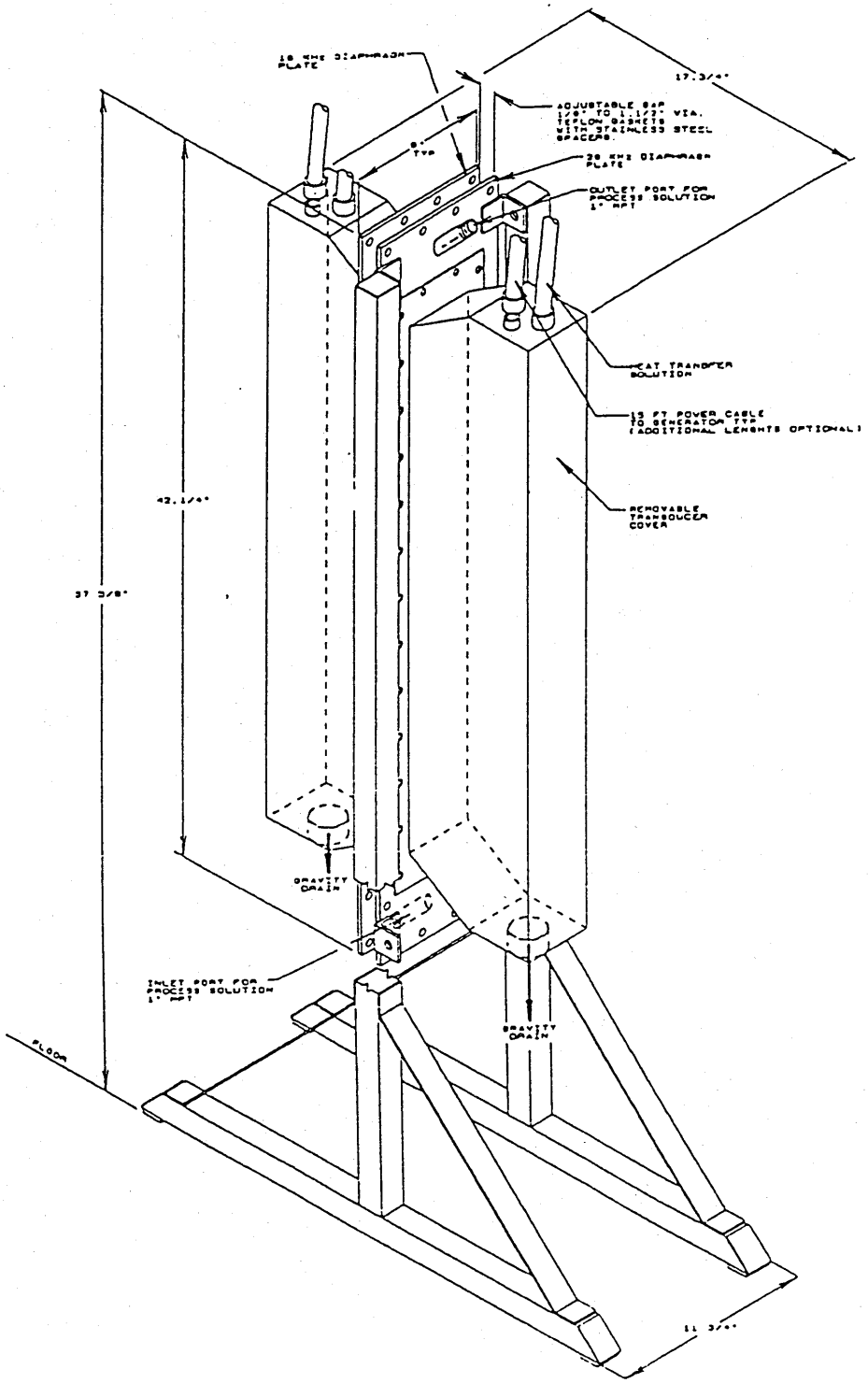


Figure 4.3 Diagram of Nearfield Acoustical Processor

Sludge enters the unit at the base, and is pumped up between the plates and leaves through an outlet pipe at the top, which allows any gas that is released from solution to be removed from the unit, with the sludge.

4.2.2 NAP Control Methods

Using the NAP unit, the sonication of sludge can be controlled in three different ways:

1. Flow rate of sludge through the unit
2. Intensity of ultrasound used
3. Width of spacer separating the two plates

4.2.2.1 Flow Rate

It is possible to control the flow rate of the sludge by altering the speed of the sludge feed pump. The slower the pump speed, the longer the sludge residence time inside the unit. Hence at slow pump speeds sludge is exposed to the ultrasonic field for a longer period of time.

4.2.2.2 Intensity

It is possible to alter the intensity of the ultrasound produced by the NAP unit by adjusting the amperage of the current supplied to each plate, from a minimum of 5 amps up to a maximum of 16 amps (A).

The total power supplied to each plate was calculated using the equation:

$$\frac{\text{maximum total power input}}{\text{maximum current output}} \times \text{current input}$$

The maximum power input of the NAP unit is 2 kW and the maximum current output is 16 A. The current input is displayed on the control panel.

To calculate the ultrasonic intensity being applied, the power supply per unit area or volume is used. The active internal volume of the unit is 2000 cm^3. At the lower intensity used (5 A) the power required was approximately 0.5 $W.cm^{-3}$, and at the higher intensity of 16 A, the power required was approximately 1.6 W/cm^{-3}.

4.2.2.3 Spacer Distance

A variety of separators were supplied with the unit, which can be used to alter the distance between the plates to change the internal volume of the unit and so change the residence time of the sludge inside the sonicator. The spacers provided have thickness of: 1", ½", ¼", 1/8" and 1/16" . As the distance between the plates increases, the relative intensity per unit volume of sludge being processed decreases.

4.2.3 Operation

It was decided that ultrasonic intensity would be the variable factor and that residence time within the unit and the frequency of the ultrasound would be left constant. Three pilot scale digesters were to be used, one as a control with unsonicated sludge, and two using sludge sonicated at different intensities. The scoping study involved finding the optimum intensity (the intensity causing most cavitation), which was to be used to treat the feed sludge for one digester. The remaining digester was to be fed with sludge treated at a lower intensity approximately one third of the optimum intensity, to determine whether the same effects of ultrasound could be achieved using less energy and hence at a lower cost.

At this stage it was also necessary to determine at what speed to pump the feed sludge through the sonicator unit, as this was to be kept constant throughout the main experiment.

These factors were determined by means of soluble COD (chemical oxygen demand) testing (see section 4.6.6 for methodology). The soluble COD was used as an indicator of effective cavitation as it is known that cavitation has the effect of lysing cells, releasing the cell contents, which contain soluble COD. Therefore, the greater the soluble COD of a sludge following sonication, the greater the effect of cavitation. In a series of tests, samples were taken from sludge that had been through the ultrasound unit at different pump speeds under a range of ultrasonic intensities. The intensities used were 16 amps (maximum intensity for this unit), a lower value of 14 amps and then a setting found using a mechanical test suggested in the manual of 16 amps on one generator and 14 amps from the second generator. The pump speed chosen for the main experiment was setting 6 (0.14 l/s) and intensities used in the main experiment were 16 amps and 5 amps (for details of these results see section 5.1).

4.3 Main Experiment

4.3.1 Pilot Scale Digesters

Three identical self-contained pilot scale digesters were available for this project. A photograph and diagram of one such digester is shown in plate 4.2 and figure 4.4 respectively. The temperature of each digester is maintained at 35 °C by the computer system, which is connected to a temperature probe inside the digester and two external electric heaters, one on the base and one on the side walls. The digesters are also mechanically stirred to ensure thorough mixing. Feed sludge is held in a small tank above each digester, these tanks are also stirred. The holding tanks are kept at approximately 7 °C by means of a refrigeration unit, which cools water and then pumps it through pipes running through the tanks. Sludge is fed into the digesters from the respective holding tank via a peristaltic pump, which runs automatically. The equipment is controlled using a computer system. The required sludge retention time in the digesters, the number of feeds required per day and the speed of the sludge flow

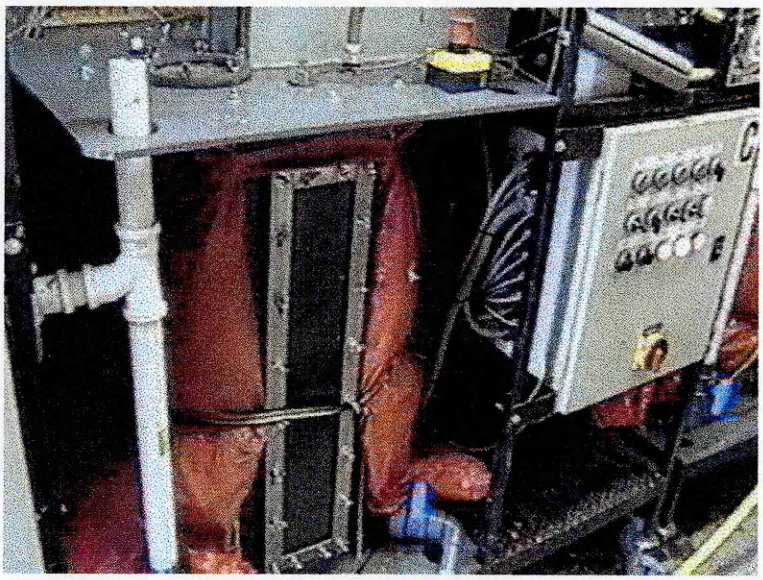


Plate 4.2 Pilot Scale Digester

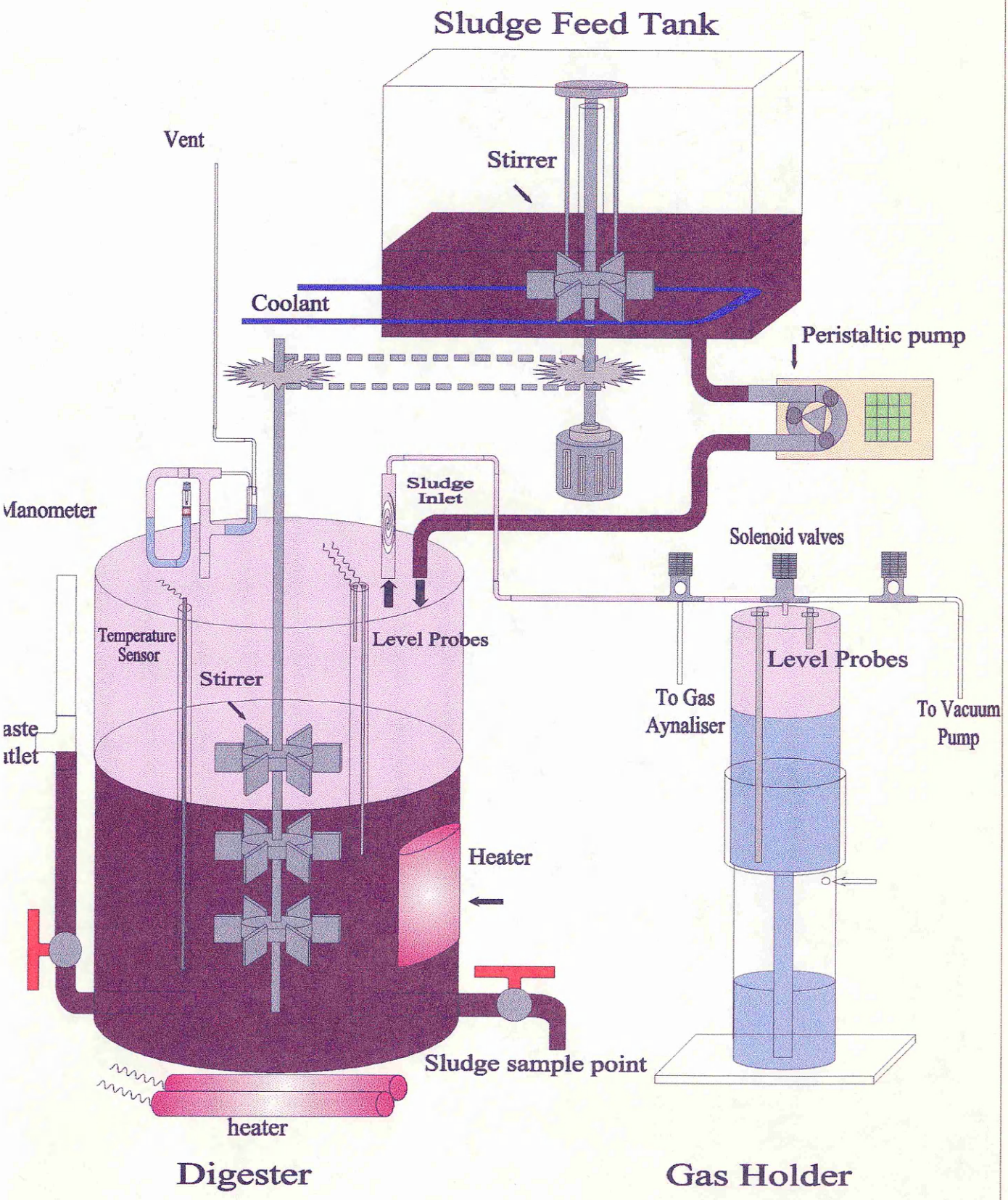


Figure 4.4 Pilot scale digester

(ml/minute) is entered into a programme. The computer then calculates at what time the digesters will be fed, how long the pumps will run for each feed and operates the pumps accordingly.

At the base of each digester there is a sample point and an outlet point. A section of pipe connects the outlet point to a drain, which goes to waste. This pipe is raised to the required height of sludge in the digester, so that the level of sludge in the digester is always correctly maintained. So, as feed sludge is fed into the top of the digester, digested sludge at the bottom is displaced and flows to waste.

4.3.1.1 Digester Gas Collection and Measurement

The pilot scale digesters are fitted with a complex system for gas collection and measurement. Gas from the digester rises up from the top of the digester into a tube that is connected to a manometer filled with water. As the gas moves into the manometer, it pushes the water in front of it. This continues until the water comes into contact with a water sensitive probe. At this point the computer responds by opening the necessary valves to let a portion of gas flow from the digester into the large water-filled gas collection jar, displacing the water into the chamber below.

The process then starts again, so that portions of gas from the digester are continually allowed to displace water in the large gas collection jar, until it is empty. Water sensitive probes detect when the jar is empty of water, and then the computer switches on the vacuum pump, which sucks the water from the chamber back up into the gas jar.

The computer system calculates the rate of gas production in l/day, using the time elapsing between gas jar refills. This information is of limited use and can be unreliable as the complexity of the system makes it fragile. For this reason, a data logger was used in conjunction with the existing system. A squirrel series 1000 logger was used to count the number of times each gas jar was filled with gas. This

information, along with the gas jar volume, can be used to find the actual volume of gas produced by each digester per day.

4.3.2 Operation

The main experimental work consisted of two phases. The first phase involved starting up the digesters and running them until all three were stable and performing equally well. The HRT was set at 15 days as this is typical of high-rate digesters (Metcalf and Eddy, 1991). The digesters were started up one at a time, using seed sludge from a full scale digester on another Southern Water site. The first digester to be run was set up several weeks before the others, but all three appeared to be performing equally in terms of gas production and volatile solids reduction by the start of phase two.

The second phase started on 7th May 1998 and involved adding sonicated sludge to two of the digesters and continuing to add unsonicated sludge to the third. One digester received sludge that had been sonicated with 5 amp (low) intensity ultrasound and the second digester received sludge that had been sonicated with 16 amp (high) intensity ultrasound. The retention time of the digesters was set at 15 days, and the experiment was to last for a cycle of 6 retention times totalling 90 days.

Originally, the sludge used was from the lamellae settler, but from the 11th May, sludge was taken from the main sludge main as the lamellae was put off line awaiting further trials. The sludge main contains a 1:1 mixture of sludge from the WWTW primary sedimentation tanks and waste activated sludge. The polyelectrolyte used was also changed, from Zetag 48 to Zetag 67 which was found to perform better on the sludge taken from the sludge main. This regime then continued until the end of the 90 day trial.

Throughout this period, fresh sludge was added to the digester sludge holding tanks twice a week, ensuring that the sludge age in the holding tanks was four days or less. The sludge in the holding tanks became less viscous with age due to continual mixing,

so the feed pumps were regularly calibrated to ensure that the correct amount of feed was still being added.

Every weekday, samples were taken from the feed inlet point and digested sludge outlet point of each digester. These samples were tested on site for pH, total solids and volatile solids (see sections 4.6.2, 4.6.3 and 4.6.4 for respective methods). Gas measurement readings were also taken daily, and the composition of the gas produced was analysed (see section 4.6.1 for methodology). Some samples were also taken for microscope observation and CST determination (see sections 4.6.7 and 4.6.5 for respective methodology).

4.4 Batch Digestion Tests Description

Batch digestion tests were carried out to try to find out what was happening inside the pilot scale digesters in terms of the different stages of anaerobic digestion. Small scale batch tests were carried out using 500 ml incubation bottles, connected to gas collection jars by rubber tubing. Photographs of this equipment are shown in plates 4.3 and 4.4. The bottles were filled with a mixture of "seed" sludge from a digester and fresh "feed" sludge, they were then sealed and connected to the water-filled gas collection jars. As gas is produced, water is displaced from the collection jars by the gas. The amount of gas produced per day is measured using the scale on the side of the jars. Following measurement, it is possible to re-set the jars by re-filling them with water using a vacuum pump.

The incubation bottles were placed in a water bath maintained at 35 °C. In this experiment, 39 500 ml bottles were used, and the ratio of "seed" sludge to "feed" sludge was 2:1. Into each bottle 240 ml of feed and 120 ml of seed were added, and thoroughly mixed. Unsonicated feed sludge was added to 13 bottles, low intensity

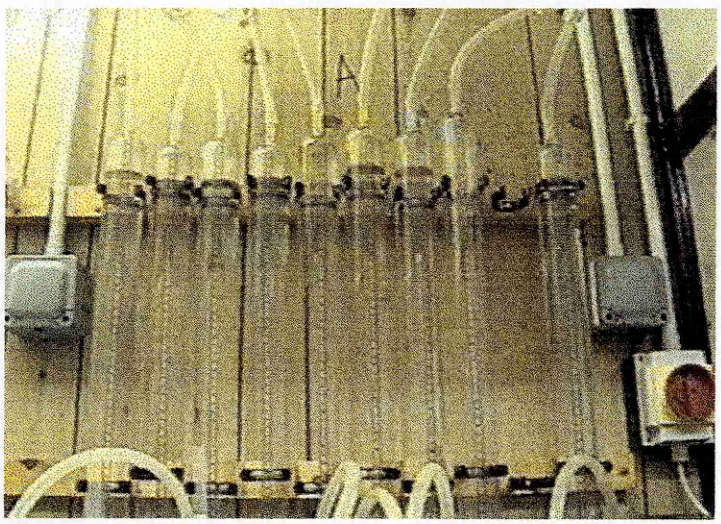
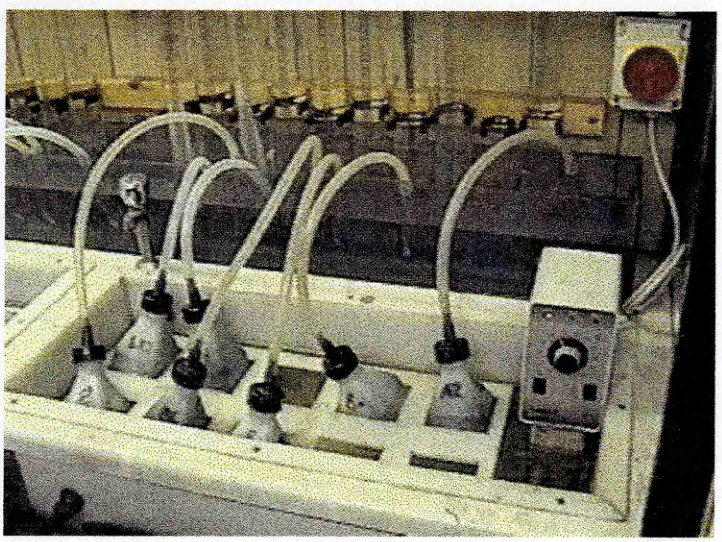


Plate 4.3 Gas Collection Jars used in Batch Tests

Plate 4.4 Digestion Bottles used in Batch Tests



ultrasound pre-treated feed sludge was added to 13 bottles and high intensity ultrasound treated feed sludge was added to a further 13 bottles.

The test was run for 14 days and bottles were removed to provide samples on days 1, 2, 3, 4, 5, and then less frequently as the digestion process slowed down as most of the feed had been utilised, on days 8, 10, and 12. The samples taken were tested on site for pH, total solids and volatile solids (see sections 4.6.2, 4.6.3 and 4.6.4 for respective methods). Further tests were carried out at Southern Water's laboratories at Otterbourne, where total COD, ammoniacal nitrogen and a wide range of volatile fatty acids (VFA's) were tested for (see section 4.6.8, 4.6.9 and 4.6.10 for respective methods).

This test was then repeated in order to back up the results of the first batch, making the end results more reliable.

4.5 Additional Experiments

Other additional work involved carrying out microscopy and soluble COD tests on sludge before and after sonication and following digestion (see section 4.6.7 and 4.6.6 for respective methodology). The aim of this was to show the effects of sonication on sludge structure.

4.6 Laboratory Analysis

4.6.1 Digester Gas Analysis

Gas composition from the pilot scale digesters was determined using a Geotechnical Instruments GA 94A infra-red gas analyser with a hydrogen sulphide (H_2S) gas pod attachment. Gas was taken from the gas analyser valve on the top of each digester, and was run for 100 seconds before a reading was taken. The percentage composition of

methane (CH_4) and carbon dioxide (CO_2) was noted and also the H_2S concentration in parts per million (ppm).

Before each successive reading, the analyser was allowed to run in fresh air until no CH_4 or H_2S was present.

4.6.2 pH Measurement

Samples taken were tested for pH as soon after sampling as possible to avoid false readings due to changes in composition. Feed sludge samples were taken from the digester feed inlet pipe following a 10 second run of the peristaltic pump. This was to ensure that the sample was from the refrigerated holding tank and not from the feed pipe itself, in which sludge could partially digest due to the higher temperatures. Digested sludge samples were taken from the sample point at the base of the digesters. A 300 ml portion of sludge was run off before sampling to ensure that the samples came from inside the digester, not from the pipe leading to the sample point.

The pH of the samples was measured using a Palintest pH meter with glass electrode. The electrode and temperature probe were rinsed with water before each test. During testing, the probes were placed in the sample and the sample was stirred. A reading was taken once a stable pH value was shown (after approximately 3 minutes) (Department of Environment (DoE) Blue Book Method, 1977).

4.6.3 Total Dry Solids

This analysis was carried out according to the DoE Blue Book Method, (1977a).
Each sample was tested in duplicate, so each sample was tested twice and then the average value was found. For each sample, a clean, dry silica crucible was weighed on a Sartorius basic four figure balance, and the weight (A) was recorded. The crucible was then filled with sludge from the sample and weight was noted (B) and the crucible was placed in an oven at 105 °C overnight. The crucibles were then removed from the

oven and placed in a dessicator to cool, after which they were weighed and the weight was noted (C). The total dry solids content as a percentage of the wet sludge could then be calculated using the following equation:

$$\frac{C - A}{B - A} \times 100\% \quad w/w$$

4.6.4 Total Volatile Solids

This analysis was carried out according to the DoE Blue Book Method, (1977b).
To measure the percentage total volatile solids of the feed and digested sludge samples, the crucibles containing dried sludge from the determination of total dry solids were placed in a furnace set at 550 °C for 30 minutes. The crucibles containing the ashed samples were then placed into a dessicator to cool, weighed and the weight was noted (D). The percentage loss of volatile solids on ignition was calculated by:

$$\frac{C - D}{C - A} \times 100\% \quad w/w$$

The total volatile solids content of the sludge samples as a percentage of the total dry solids can then be calculated:

$$\frac{\%\ \text{VS loss on ignition} \times \%\ \text{TDS}}{100}$$

4.6.5 Capillary Suction Times (CST)

The CST of digested sludge was determined using a CST machine supplied by Triton Electronics Limited, Sussex, UK. The CST apparatus automatically measures the time required for a small volume of filtrate to be withdrawn from a sludge when subjected to the capillary suction pressure of dry filter paper. The CST apparatus comprises 4 items:

(a) a metal cylindrical sludge reservoir of 18 mm diameter

(b) a rectangular piece (90 x 70 mm) of dry Whatman No.17 grade filter paper

(c) two rectangular perspex blocks, the upper one of which has a central hole for locating the sludge reservoir. Embedded into the upper block are three electrical probes, two of which are positioned on a common radial distance from the centre of the block and the third at a greater radial distance from the centre. These probes rest on the filter paper and are used as conductivity sensor to start and stop a timing mechanism.

(d) a timing mechanism which displays the time in seconds (the CST) taken for the filtrate interface to move radially through the filter paper from the inner probes to the outer probe.

To use the apparatus, the piece of dry filter paper is placed between the two perspex blocks. The sludge reservoir is placed into the central hole of the upper block so that it rests on the filter paper and is then filled with sludge. A flow of filtrate radiates out from the reservoir and when it reaches the first two probes the timer starts. The timer stops when the filtrate reaches the third probe - the time interval being the capillary suction time, which is displayed on a screen on the timing mechanism.

This procedure was repeated three times for each sample and the average was calculated.

4.6.6 Soluble Chemical Oxygen Demand

COD was determined using a palintest 5000 series photometer and the methodology provided by the manufacturer was followed. Using this method a sample is added to a tube containing a reagent and time is allowed for the ensuing reaction to proceed. The percentage transmittance of 570 nanometer (nm) wavelength light is then measured compared to a blank sample and is proportional to the COD concentration of the sample.

This test was to find the soluble COD of sludge samples, so firstly they were filtered to obtain only the liquid portion of the sludge. One ml of filtrate and 1 ml of distilled water was added to a test tube containing a precisely measured amount of reagent composed of sulphuric acid and potassium dichromate with a silver sulphate catalyst. The tube was then sealed, shaken and placed in a heating block at 150 °C for two hours. During this period, the reagents oxidise the sample leading to a reduction in dichromate which causes a colour change that is proportional to the COD.

After two hours the tube is cooled and then its transmittance is measured against a blank. To compensate for the light absorbed by the reagents, the photometer is set to 100 % transmittance using the blank (which only contains distilled water and reagents). The percentage transmittance of the sample is then measured relative to the blank. The percentage transmittance is converted into the COD value (expressed as milligrams of oxygen consumed per litre of sample (mg/l)) by means of a calibration chart supplied with the equipment.

4.6.7 Microscopy

A small amount of sludge was placed onto a microscope slide, covered with a cover slip and observed under a light microscope with a maximum magnification of 40 x. If the sludge being studied was too thick, a dilution was made prior to observation.

4.6.8 Total COD

This analysis was carried out at Southern Water's laboratories. The procedure employed spectrophotometry and was based on the DoE Blue Book Method (1977).

4.6.9 Ammoniacal Nitrogen

This analysis was carried out at Southern Water's laboratories. The procedure involved colormetric discrete analysis using sodium salicylete and was based on the DoE Blue Book Method (1977).

4.6.10 Volatile Fatty Acids

This analysis was carried out at Southern Water's laboratories. The procedure employed the use of gas chromatography, comparing the sample to a known standard.

5.0 RESULTS

5.1 Scoping Study Results

The table below shows the soluble COD values (mg/l) found from the first scoping study COD trial, where two high intensities were used, to find out which one to use:

	SOLUBLE COD (mg/l)		
PUMP SPEED (l/s)	one generator @ **l6** amps and one @ **14** amps	both generators @ **16** amps (maximum)	both generators @ **14** amps
0.05	475	590	475
0.09	610	475	475
0.14	550	570	570
0.20	535	550	535
	No sonication	**No sonication**	
0.05	390	340	

Table 5.1 Scoping soluble COD test (1) results

Table 5.2 shows the soluble COD (mg/l) values found from the second scoping study COD trial:

	SOLUBLE COD (mg/l)		
PUMP SPEED (l/s)	one generator @ **16** amps and one @**14** amps	both generators @ **16** amps (maximum)	both generators @ **14** amps
0.05	535	495	475
0.09	495	535	460
0.14	475	535	535
0.20	275	355	340
	no sonication	**no sonication**	
0.05	390	475	

Table 5.2 Scoping soluble COD test (2) results

Table 5.3 shows the average values of soluble COD (mg/l) found at different intensities in scoping COD tests 1 and 2:

SCOPING COD TEST 1			SCOPING COD TEST 2	
INTENSITY (amps)	**AV.SOLUBLE COD (mg/l)**		**INTENSITY (amps)**	**AV.SOLUBLE COD (mg/l)**
14/16	**543**		14/16	**445**
16	**546**		16	**480**
14	**514**		14	**453**
no sonication	**365**		no sonication	**433**

Table 5.3 Average soluble COD values at different intensities in scoping study 1 and 2

Table 5.4 shows the average values of soluble COD (mg/l) found at various pump speeds in scoping COD tests 1 and 2:

SCOPING COD TEST 1			SCOPING COD TEST 2	
PUMP SPEED (l/s)	AV.SOLUBLE COD (mg/l)		PUMP SPEED (l/s)	AV.SOLUBLE COD (mg/l)
0.05	513		0.05	502
0.09	520		0.09	497
0.14	563		0.14	515
0.20	540		0.20	323

Table 5.4 Average soluble COD values at various pump speeds in scoping study 1 and 2

In the case of both trials, the pump speed that produced the highest soluble COD on average was 0.14 l/s. The ultrasonic intensity that produced the highest soluble COD on average was also the same in both trials, at 16 amps on each generator. These results were used to decide the set up of the main experiment, in which a pump speed of 0.14 l/s was used and 16 amps was used as the optimum intensity.

5.2 Main Experiment Results

5.2.1 Daily gas production

The amount of gas produced each day was found by multiplying the volume of the gas collection jars (1.73 l) by the squirrel counts per digester per day. The results are displayed in a graph in figure 5.1 below and are shown in table form in appendix 1.

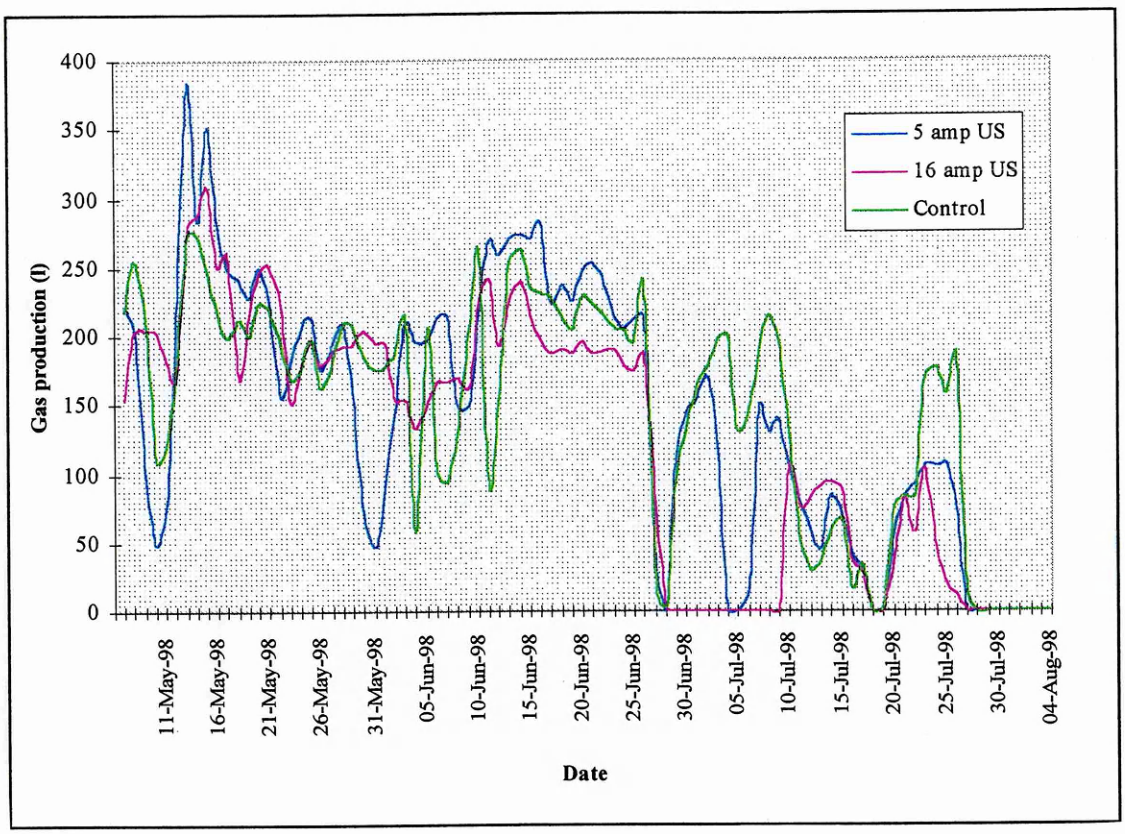


Figure 5.1 Daily gas production of pilot scale digesters over 90 day trial

The information shown on graph 5.1 is very variable, with large peaks and troughs. For the most part, the troughs correlate with incidences of mechanical and physical; problems and human error, the details of which were recorded as they occurred. The peaks are generally due to the digesters recovering from such problems.

The blue line represents the gas production of the digester fed with sludge sonicated at 5 amps. This line shows 6 major troughs representing a sharp decrease in gas production. The first such trough occurs around the 10th May and corresponds to a feed pipe blockage on the 8th May. The second large trough around the 28th June, corresponds to a feed pipe blockage that occurred over that weekend, thus preventing the digester being fed for 48 hours. Another large decrease in gas production occurred around the 25th June, the reason for this is not known. On the 6th July, the digester outlet was found to be blocked so the digester had totally filled and the gas collection system was temporarily disabled. This resulted in no gas measurements being made,

which explains the large trough around this time. The two final troughs on the 18th and 27th July both corresponded to power cuts on site, during which the digester would not have been heated, stirred or fed, resulting in less gas production. From the 27th July until the end of the trial on 4th August, no gas production was recorded. This was due to the production of a large volume of foam inside the digester, which entered the gas collection/measurement apparatus, temporarily disabling it.

The pink line represents the digester that was fed with sludge sonicated at the higher intensity of 16 amps. There are 4 major decreases in gas production by this digester shown on the graph, the first of which occurred around the 13th May and corresponded to a feed pipe blockage on the 12th May. The second major decrease was from 26th June to the 10th July, during which time major mechanical problems with the gas collection/measuring system were experienced. The final two troughs around the 18th and 27th July both corresponded to power cuts on site, during which the digester would not have been heated, stirred or fed, resulting in less gas production. From the 27th July until the end of the trial on 4th August, no gas production was recorded. This was due to the production of a large volume of foam inside the digester, which entered the gas collection/measurement apparatus, temporarily disabling it.

The green line represents the control digester that received unsonicated sludge. The first 3 large troughs on the 8th May, 4th June and 5th June correspond to feed pipe blockages. The reason for the next large fall in gas production around the 10th of June is not known. Another large trough around the 28th June, corresponds to a feed pipe blockage that occurred over that weekend, thus preventing the digester being fed for 48 hours. The final two depressions around the 18th and 27th July both corresponded to power cuts on site, during which the digester would not have been heated, stirred or fed, resulting in less gas production. From the 27th July until the end of the trial on 4th August, no gas production was recorded. This was due to the production of a large volume of foam inside the digester, which entered the gas collection/measurement apparatus, temporarily disabling it.

5.2.2 Total Gas Production

The information shown in figure 5.1 is shown in a cumulative form in figure 5.2. This then shows the total gas production by each digester over time.

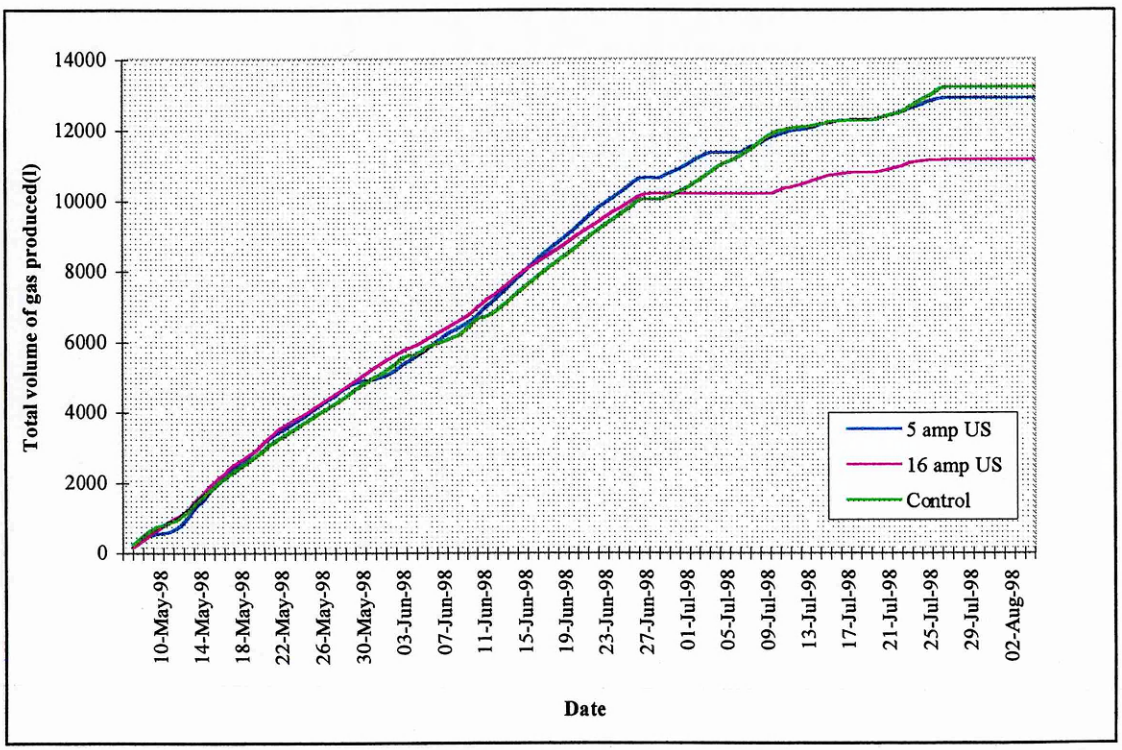


Figure 5.2 Total gas production of pilot scale digesters over 90 day trial

In figure 5.2, points at which gas production was very low are shown where the lines are in the form of flat plateaus. These plateaus correspond with the negative peaks in figure 5.1.

The three lines all show a very similar course until 26th June, when the 16 amp US digester fell behind in total gas production due to the mechanical problems that were experienced with the gas collection equipment. From the 27th July until the end of the trial on 4th August, no gas production was recorded. This was due to the production of a large volume of foam inside the digesters, which entered the gas collection/measurement apparatus, temporarily disabling them.

5.2.3 Volatile solids reduction

Volatile solids reduction is the amount of volatile solids removed during the digestion process and was found using the average of the previous fifteen days feed sludge volatile solids content. The volatile solids reduction results are shown in figure 5.3 and in more detail in appendix 2.

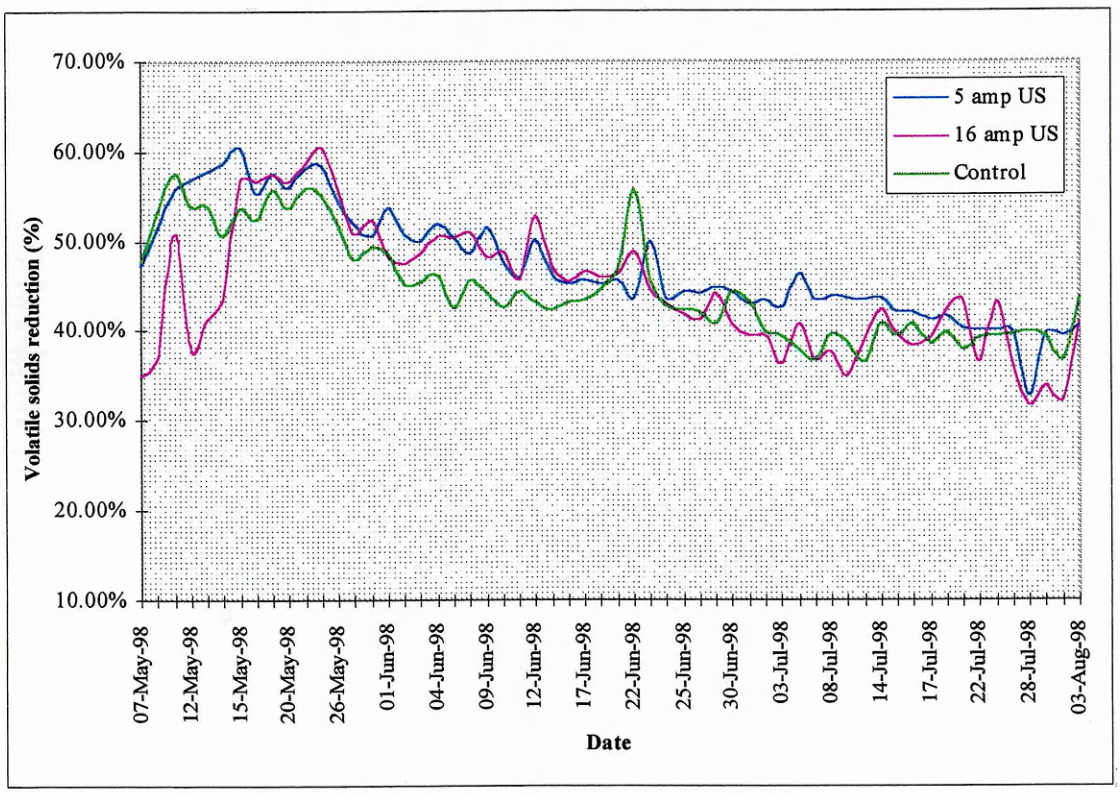


Figure 5.3 Volatile Solids Reduction in pilot scale digesters

DIGESTER	5 amp US	16 amp US	Control
Average VS reduction (%)	47.38	44.53	44.82

Table 5.5 Average volatile solids reduction in pilot digesters over 90 day trial

Many of the trends shown in figure 5.3 can again be related to the factors mentioned in section 5.2.1. Up until the 26th June, the control digester had a markedly lower VS

reduction than the digesters containing sonicated sludge, which performed similarly up to this point. After the 26th June the control digester and the 16 amp US digester both performed less well than the 5 amp US digester.

The overall trend for the lines representing all three digesters is a gradual decrease in volatile solids reduction

5.2.4 Digester Gas analysis

Digester gas was not analysed every day and the analyser was unavailable for two long periods for which data is therefore unavailable. The analysis data for the methane content of the digester gas is shown graphically in figure 5.4, gas analysis data is shown in full detail in appendix 3.

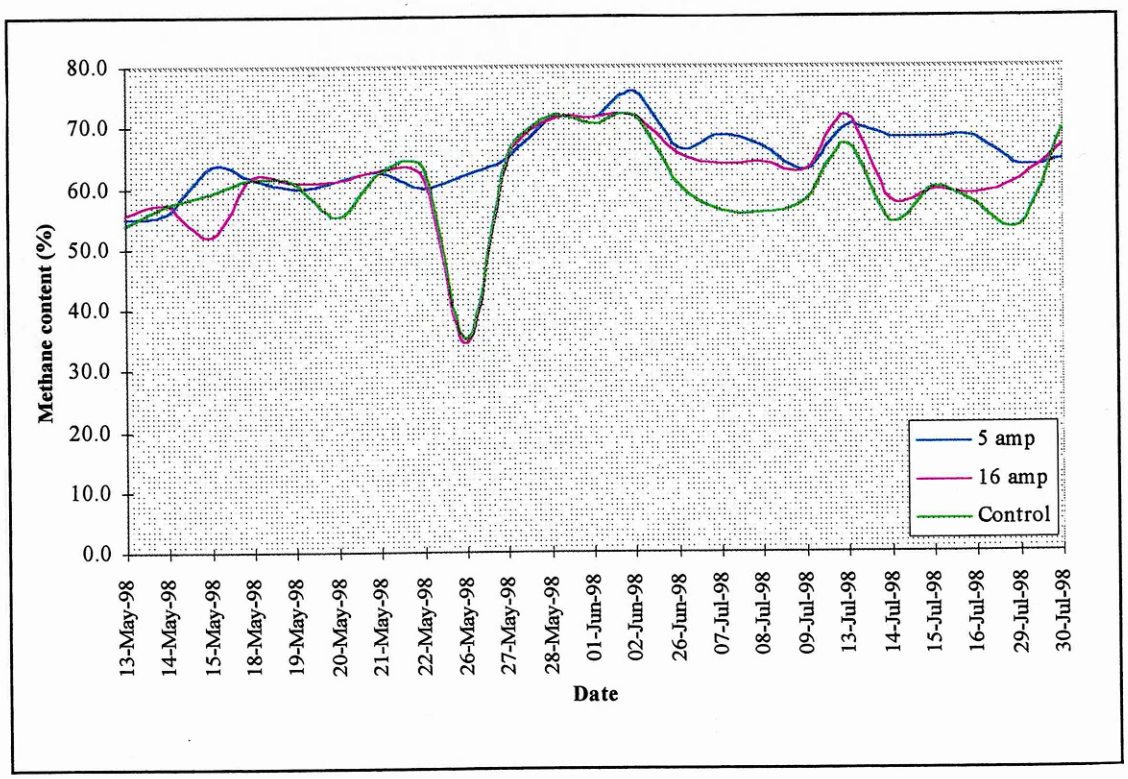


Figure 5.4 Methane content of gas produced by pilot scale digesters

As with other characteristics mentioned previously, the methane content of the gas produced can be affected by mechanical and physical problems such as lack of feed. The trends for each digester are similar although from the 2nd June the control digester produced gas containing a lower percentage of methane than the digesters containing sonicated sludge.

5.3 Additional experimentation

5.3.1 Soluble COD of sludge prior to digestion

The soluble COD of the cosettled sludge following sonication used was determined. The results of these tests are shown in table 5.6

DATE	5 AMP U.S Soluble COD (mg/l)	16 AMP U.S Soluble COD (mg/l)	CONTROL Soluble COD (mg/l)
9-JUNE-98	1730	1730	1535
	1585	1790	1490
16-JUNE-98	1635	1680	1370
	1370	1635	1370
	1535	1585	1410
AVERAGE	**1571**	**1684**	**1435**

Table 5.6 Soluble COD of sludge following sonication

The sludge samples found to have the lowest average soluble COD value of 1435 mg/l were the control samples that had not been sonicated. The sludge that was treated by low intensity ultrasound at 5 amps had an average soluble COD of 1571 mg/l. The

highest average soluble COD value was found to be from sludge samples that had been sonicated by 16 amp intensity ultrasound.

5.3.2 CST values of sludge directly following sonication

Table 5.7 shows the results of CST tests carried out on sludge samples directly after sonication.

	CST VALUE (s)			
DATE	**5 AMP U.S**	**16 AMP U.S**	**CONTROL***	**RAW****
16/7/98	490.3	605.6	366.1	381.2
	459.1	466.8	372.9	352.4
AVERAGE	**474.7**	**535.9**	**369.5**	**366.8**

* Not sonicated but pumped through ultrasound unit

** Not sonicated and not pumped through ultrasound unit

Table 5.7 CST values of sludge following sonication

The samples with the lowest average CST value of 366.8 seconds was the unsonicated raw sludge, followed closely by the control samples at 369.5 seconds. The samples of sludge sonicated at the low intensity of 5 amps were found to have an average CST value of 474.7 seconds. The highest average CST time was from the sludge samples that had been sonicated at the higher intensity of 16 amps.

5.3.3 CST values of feed and digested sludge

The CST of sludge samples from the digester feed tanks and the digesters themselves were determined. The results are shown in table 5.8.

	CST VALUES (s)					
	FEED SAMPLES			DIGESTED SAMPLES		
DATE	5 amp US	16 amp US	Control	5 amp US	16 amp US	Control
22-Jul-98	no data	no data	no data	704.0	615.2	437.7
	no data	no data	no data	534.7	569.5	488.1
27-Jul-98	no data	no data	no data	560.2	615.5	420.3
	no data	no data	no data	545.2	633.1	398.1
	no data	no data	no data	595.6	588.7	424.1
28-Jul-98	833.1	870.8	744.2	671.6	644.2	476.3
	875.7	891.9	779.6	574.9	645.8	467.1
29-Jul-98	772.4	774.2	693.7	481.1	539.4	450.2
	746.4	746.2	723.9	514.0	532.2	463.6
30-Jul-98	815.8	870.7	740.8	590.9	563.6	337.2
	748.1	891.4	662.1	576.8	561.6	387.3
AVERAGE	**798.6**	**840.9**	**724.1**	**579.6**	**591.4**	**432.2**

Table 5.8 CST values of feed and digested sludge

The feed sludge samples with the lowest average CST value were the unsonicated controls, at 724.1 seconds. The samples sonicated at the low intensity of 5 amps had an average CST value of 798.6 seconds. The feed sludge with the longest average CST value of 840.9 seconds was that of the samples sonicated at 16 amps.

The digested sludge samples with the lowest average CST values were again the unsonicated controls, at 432.2 seconds. The samples sonicated at the low intensity of 5 amps had an average CST value of 579.6 seconds. The digested sludge with the longest average CST value of 591.4 seconds was that of the samples sonicated at 16 amps.

5.3.4 Batch Digestion Test 1

The results of this trial are outlined in the following sections and are shown in full in appendix 4.

5.3.4.1 Gas Production

The amount of gas produced by each bottle was recorded and averages were taken, giving one number per day per condition. For batch digestion test 1, the daily gas production is displayed graphically in figure 5.5 and the same information is shown cumulatively in figure 5.6.

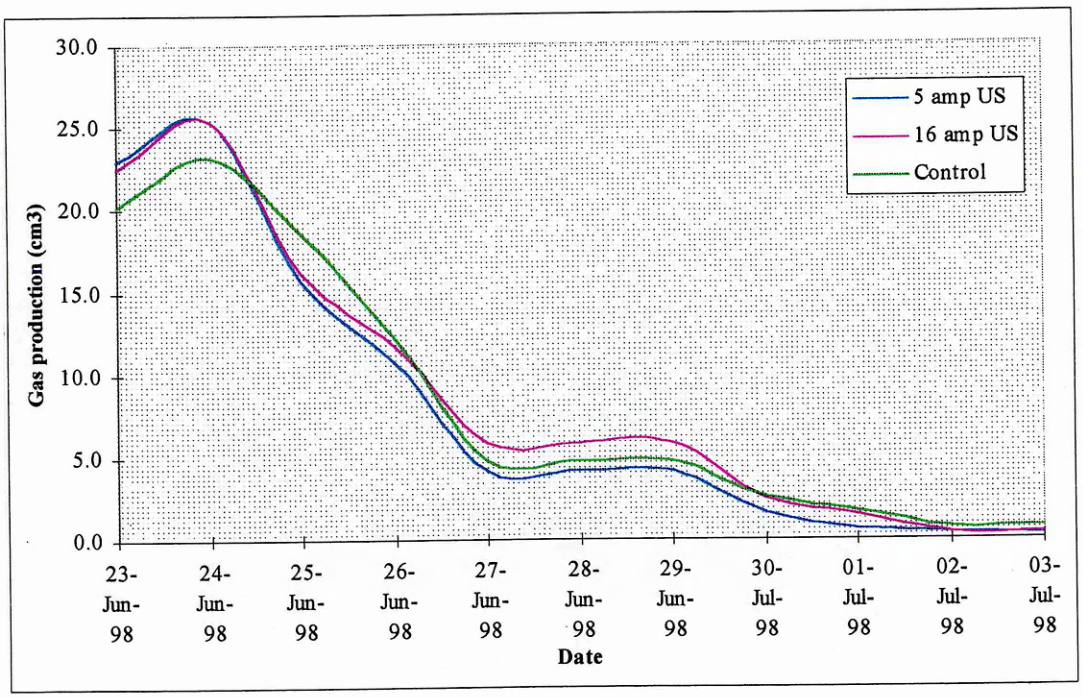


Figure 5.5 Batch digestion test 1 average daily gas production per bottle

For the first four days of the trial, the average gas production by the two sonicated sludge samples was very similar, at approximately 23.0 cm^3 on 23rd June, rising to over 25 cm^3 by the second day. The average amount of gas produced by these sludges then

rapidly decreased down to around 10 cm^3 by 26th June. After this date, both sets of samples carried on producing less gas every day, but the samples sonicated at 5 amps produced notably less gas on average than those sonicated at 16 amps. The control samples produced a maximum average of only 22.5 cm^3 of gas. However, the decrease following this peak was more gradual than with the sonicated sludges. From the 26th June, the control samples had an average gas production slightly greater than the samples sonicated at 5 amps.

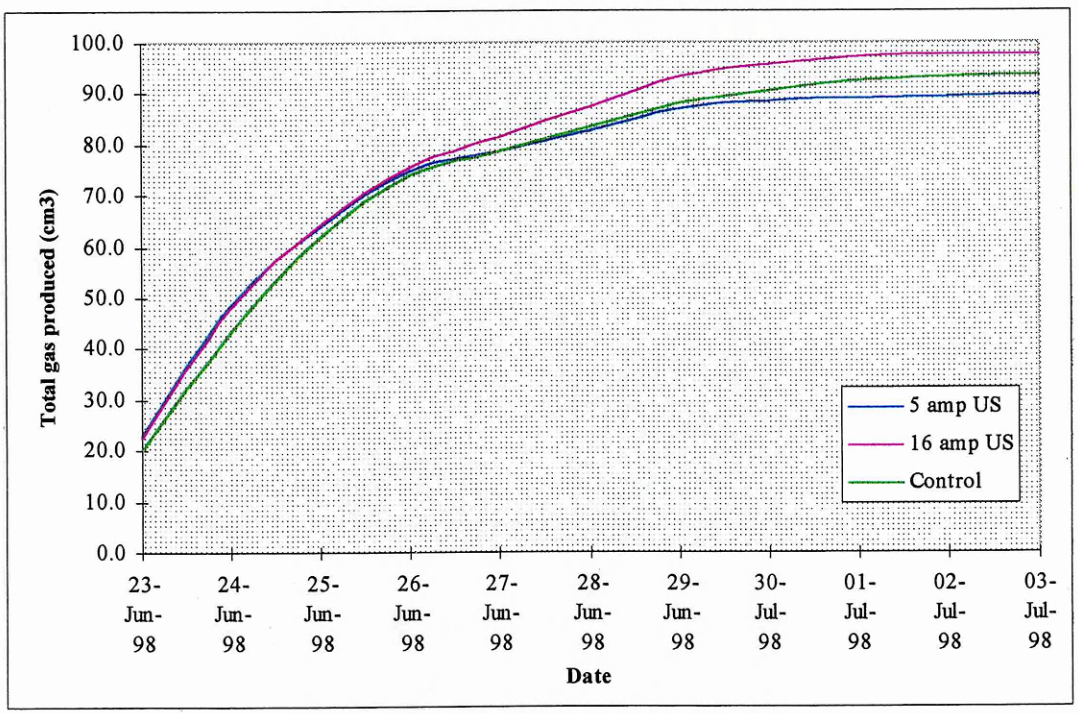


Figure 5.6 Batch digestion test 1 average total gas production per bottle

Figure 5.6 clearly shows that up until 26th June the sonicated samples produced more gas on average than the control sludges. After this point, the samples sonicated at 16 amps clearly produced more gas, followed by the control, with the samples sonicated at 5 amps producing the least gas.

5.3.4.2 Volatile Fatty Acid Content

The samples were analysed for 8 different VFA's, the concentrations of which were added together, giving a total VFA content for each sample. The total VFA content of the samples from the first batch test are shown in figure 5.7.

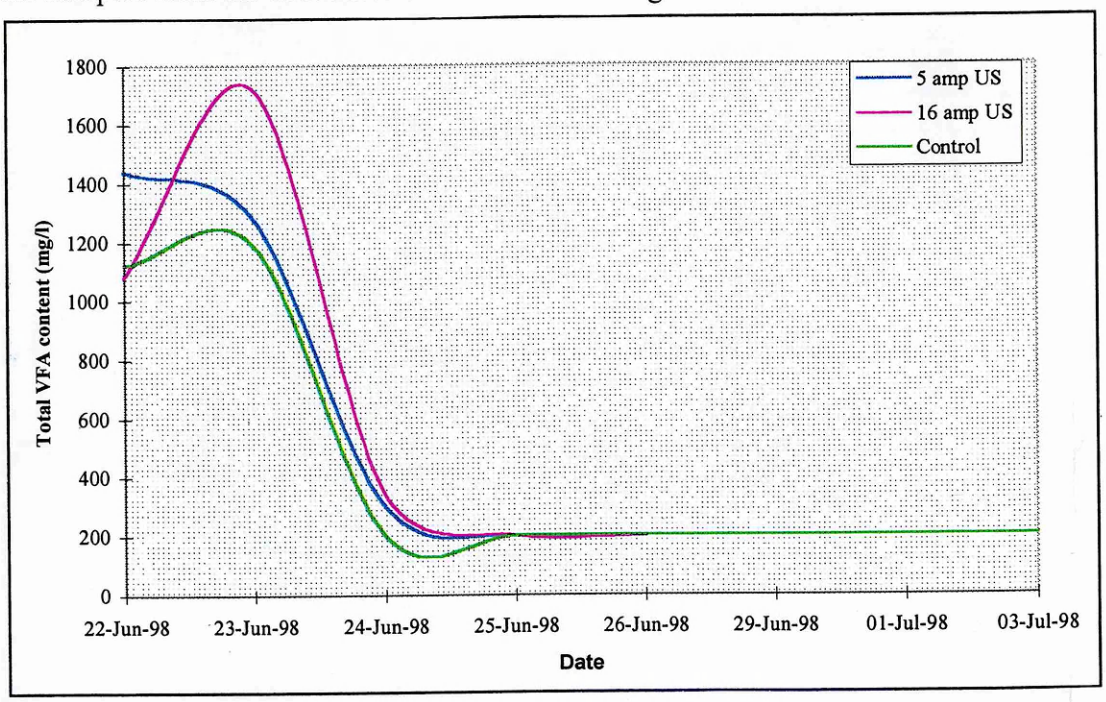


Figure 5.7 Batch digestion test 1 total VFA content of samples taken

The samples sonicated at 5 amps had an initial total VFA content of just over 1400 mg/l, which then decreased slightly to approximately 1300 mg/l and then decreased rapidly to 200 mg/l (the minimum total VFA content detectable). The samples sonicated at 16 amps had the lowest initial total VFA content of just less than 1100 mg/l, which then increased dramatically by 23rd June, up to just over 1700 mg/l, after which the total VFA content decreased rapidly down to 200 mg/l. The control samples were found to have the lowest total VFA content all through the trial, starting at approximately 1100 mg/l, increasing to 1250 mg/l and then decreased rapidly down to 200 mg/l.

Propionic acid is one of the 8 individual VFA's that was tested for. The results of this analysis are shown in figure 5.8. The results of the analysis for the remaining VFA's tested for are displayed in appendix 4.

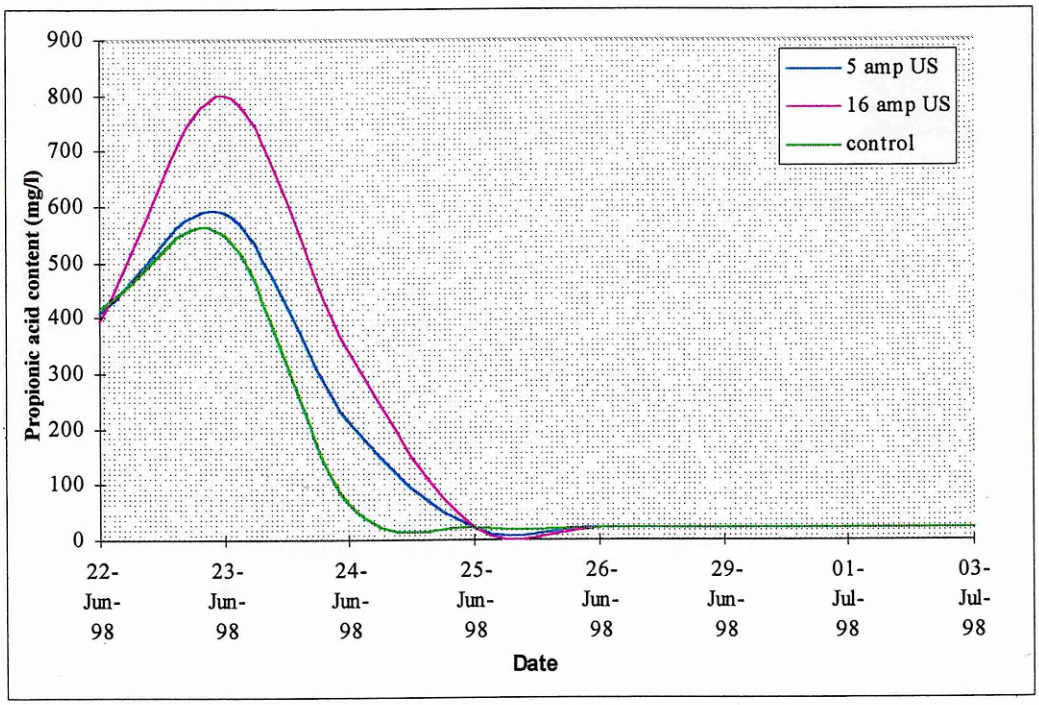


Figure 5.8 Batch digestion test 1 propionic acid content of samples taken

All three sludge types had an initial propionic acid content of approximately 425 mg/l. In the case of the control samples, this then increased up to 460 mg/l and then fell rapidly to 25 mg/l (the minimum detectable). The samples sonicated at 5 amps peaked at 600 mg/l and then fell to 25 mg/l. The highest peak was from the sludge sonicated at 16 amps, which rose to 800 mg/l, before falling to 25 mg/l.

5.3.5 Batch Digestion Test 2

5.3.5.1 Gas Production

The amount of gas produced by each bottle was recorded and averages were taken, giving one number per day per condition. For batch digestion test 2, the daily gas

production is displayed graphically in figure 5.9 and the same information is shown cumulatively in figure 5.10.

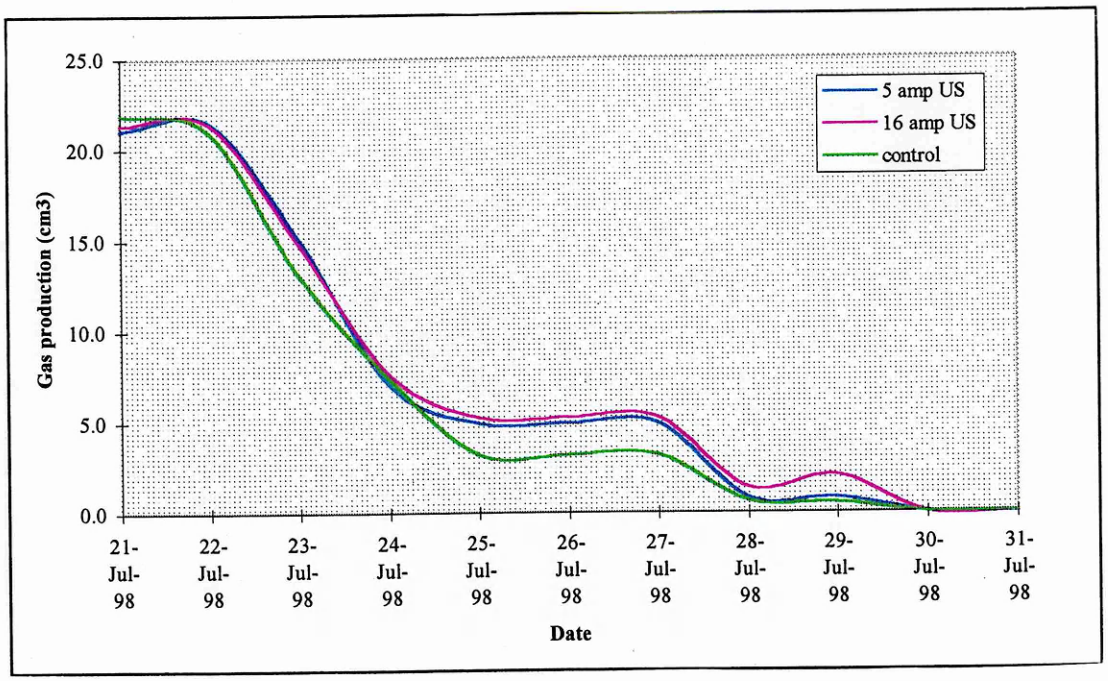


Figure 5.9 Batch digestion test 2 average daily gas production per bottle

For the first four days of the trial, the average gas production by all three sludges samples was very similar, at approximately 21.0 cm^3 on 21st July, rising slightly and then decreasing to 6.0 cm^3 by 24th July. After this date, all three sample types carried on producing less gas every day, but the unsonicated control samples produced notably less gas on average than the sonicated samples. Throughout the trial it was the sample sonicated at 16 amps that produced the most gas.

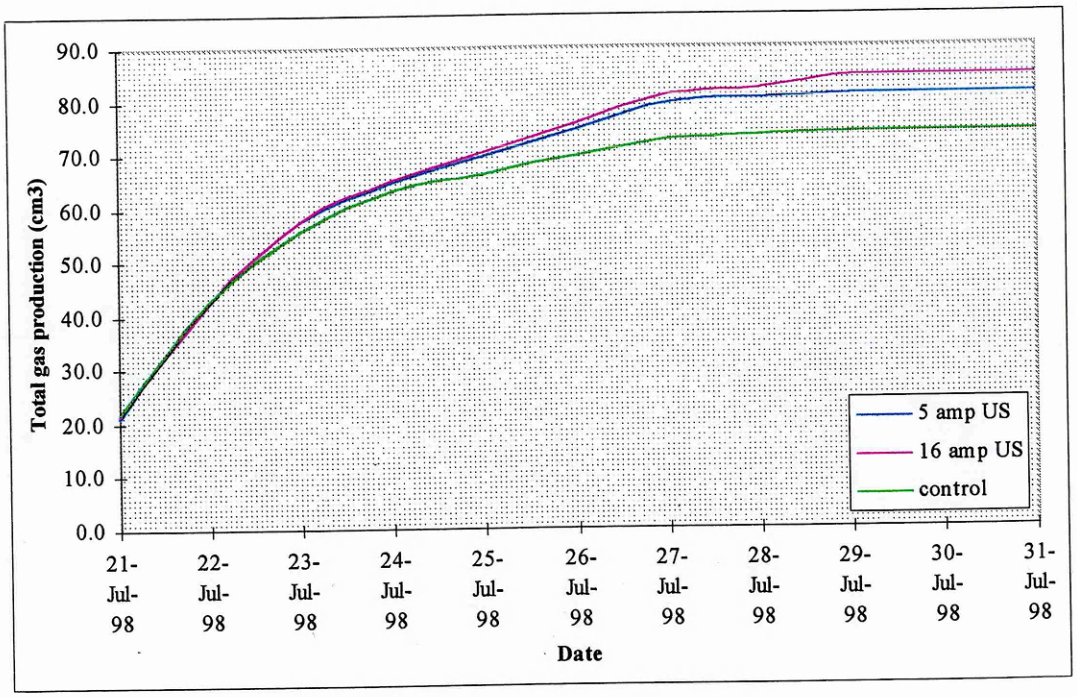


Figure 5.10 Batch digestion test 2 total average gas production per bottle

Figure 5.10 clearly shows that the samples all produced a similar amount of gas in the first 3 days, after which the control samples fell markedly behind the sonicated samples in gas production. All through the trial, the samples producing the greatest amount of gas on average were those sonicated at 16 amps.

5.3.5.2 Volatile Fatty Acid Content

The samples were analysed for 8 different VFA's, the concentrations of which were added together, giving a total VFA content for each sample. The total VFA content of the samples from the first batch test are shown in figure 5.11.

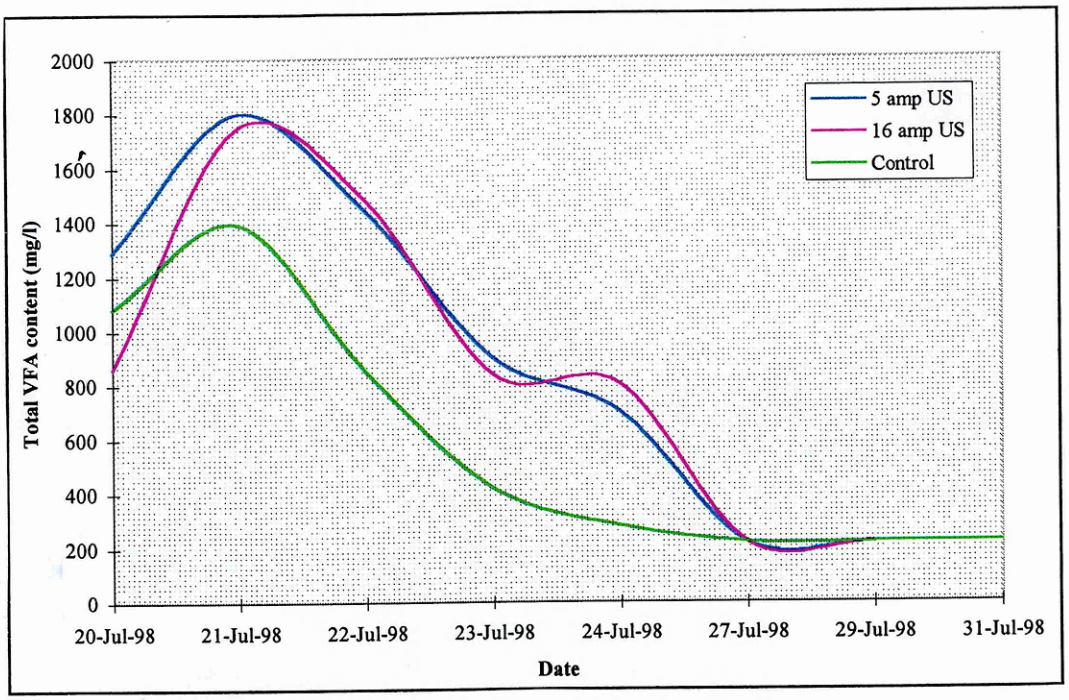


Figure 5.11 Batch digestion test 2 total VFA content of samples taken

The control sample has an initial total VFA content of approximately 1100 mg/l, which peaked at 1400 mg/l on 21st July, before gradually decreasing down to 86 mg/l. The samples sonicated at 5 amps had an initial total VFA content of 1300 mg/l, which increased to a maximum of 1800 mg/l after which the total VFA content fell to 200 mg/l. The sludge sonicated at 16 amps had the lowest initial total VFA content of 900 mg/l, which then rose dramatically by 21st July, to 1800 mg/l and then steadily decreased down to 170 mg/l.

Propionic acid is one of the 8 individual VFA's that was tested for. The results of this analysis are shown in figure 5.12. The results of the analysis for the remaining VFA's tested for are displayed in appendix 4.

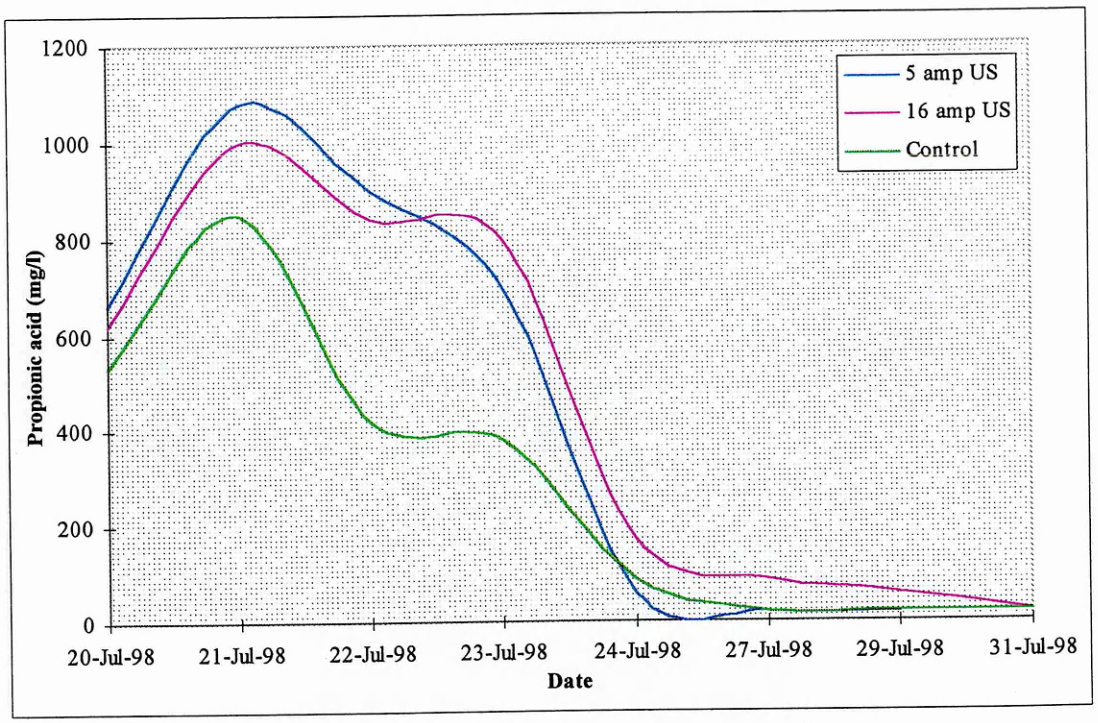


Figure 5.12 Batch digestion test 2 propionic acid content of samples taken

The control samples were found to have the lowest initial propionic acid content of 550 mg/l which then increased to a maximum of 850 mg/l on 21st July, before falling to 400 mg/l by 22nd July and then down to 20 mg/l by 27th July. The samples sonicated at 5 amps had the highest initial propionic acid content of 675 mg/l, which rose to a maximum of 1100 mg/l, before falling to 20 mg/l by 24th July. The samples sonicated at 16 amps had an initial propionic acid content of 620 mg/l, which increased to a maximum of 1000 mg/l by 21st July, before decreasing to 86 mg/l by the 27th July.

5.3.6 Microscopy

Sludge samples from before and after 5 and 16 amp sonication were observed under the microscope. Unfortunately the equipment necessary to take photographs from the microscope was not available.

The visible structure of the sludge was very similar before and after sonication, with no obvious differences. Both sets of samples contained large floc-like particles of fibres and cells.

The structure of sludges that had been in the digester feed holding tanks for 4 days were also observed under the microscope. Although no differences were noted between the different feed sludges i.e. control, 5 amp US and 16 amp US, when these were compared to freshly sonicated sludge, the structure was notably different. The sludges that had been in the feed holding tanks were much finer in structure, with a relatively uniform particle size, compared to the fresh sludge structure of large particles and flocs.

6.0 DISCUSSION

The scoping study found that the maximum intensity of 16 amps and a pump speed of 0.14 l/s produced the highest sludge supernatant soluble COD values. It is logical that the highest intensity would produce the highest supernatant soluble COD, as increasing the intensity increases the cavitational effect (Pestman, 1994). This increase in cavitation would cause more cell damage, thus releasing more cell contents into the liquid portion of the sludge, increasing its soluble COD value.

It could be assumed that the slowest pump speed through the sonication unit would produce the highest sludge supernatant COD, as the slowest speed means the longest residence time, so that maximum exposure to the ultrasound would be achieved. According to the NAP unit manual this is not the case. It states that the residence time inside the unit is not very important as cavitation and its associated effects occur instantaneously. Experimentally, it was found that a mid-range pump speed (0.14 l/s in the case of this pump) produced the highest sludge supernatant soluble COD values, and consequently that was the speed used in the main experiment.

In economic terms, a rapid flow through the sonication unit would be preferable, as a shorter residence time in the sonicator increases the volume of sludge that can be sonicated per unit of power. This of course is assuming that any possible increase in pumping costs would be relatively small.

It is important to note that the COD testing that took place involved very basic equipment, and involved dilution, both of which could introduce inaccuracies into the procedure .

Throughout the 90 day trial no major differences were found between the volumes of gas produced by each digester. There was also no notable difference in the methane content of the gas produced by each digester. In previous work the sonication of sludge before digestion has been shown to result in a greater volume of gas being

produced and the gas produced has been found to have a higher methane content. For example, Clark and Nujjoo (1998) found up to 60% more gas was produced by digesters fed with sonicated sludge and the methane content of the gas produced increased by 5-10%.

The increases in methane production that have been found in other trials are due to the digestion of a greater proportion of the feed sludge volatile solids. This was shown by Thiem *et al* (1997) who found that digesters fed with sludge treated with high intensity ultrasound showed a volatile solids reduction of 50.3% after 22 days whilst digesters fed with unsonicated sludge showed a volatile solids reduction of 45.8% over the same period. In this trial however, this effect of sludge sonication on digestion was not shown as there was very little difference in the average volatile solids reduction between digesters.

Some tests were carried out to determine the soluble COD of sludge supernatant directly after sonication and the results showed definite trends. The control samples supernatant had the lowest average soluble COD, followed by the 5 amp intensity sonicated sludges. The sludges found to have the highest supernatant soluble COD were those treated with 16 amp intensity ultrasound. These results are what might have been expected, with the samples exposed to the highest intensity ultrasound releasing more soluble COD into the liquid portion of the sludge. Tiehm *et al* (1997) investigated the effect of ultrasound on sludge supernatant soluble COD and reported similar results to those found in this trial. Tiehm *et al* (1997) concluded that the ultrasound caused sludge disintegration, causing organic compounds to be transferred from the sludge solids into the aqueous phase resulting in an enhanced biodegradability.

In previous studies, sonication has been found to increase sludge CST values, as cavitation decreases the overall particle size, meaning that water has a greater surface area to bind to and so is released less easily (Lawler *et al.*, 1986). Various CST tests were carried out during this trial, the first of which looked at sludge directly following

the various pre-treatment methods. Of these samples the shortest CST value was found to be from the sludge subjected to the least amount of stress - sludge taken straight from the dewatering machine. The control sludge that was pumped through the sonicator unit and was therefore subjected to some shear stress had a slightly higher CST value. The two sonicated samples were found to have much higher CST values, with the sample sonicated at the higher intensity of 16 amps having the highest CST value. All of these findings are what might have been expected based on previous research and show that sonicating sludge results in it being more difficult to dewater, which increases the cost of sludge treatment.

Other CST tests were carried out on sludge from the digester feed holding tanks and digested sludge. Of the feed sludges, the control samples had the shortest CST times, followed by the low intensity sonicated samples, with the 16 amp intensity sonicated sludge feed producing the longest CST times. The digested samples had much shorter CST times than the feed sludges, but followed the same pattern. A high CST time before digestion is not necessarily a problem as sludge is dewatered before sonication, so an increase in the amount of polyelectrolyte dosed is not needed. However the further dewatering that follows digestion would be affected. The new sludge treatment centre at Millbrook WWTW will involve sludge digestion, dewatering by filter belt presses and then drying. If ultrasonic pre-treatment of sludge was to be carried out, this investigation has shown that dewatering sludge from the digesters would be much more difficult. Before any more work is carried out into the effects of ultrasonic sludge pre-treatment on digestion, this issue needs to be looked into further. No matter what improvements ultrasonic pre-treatment may produce in volatile solids reduction and volume of methane production, there is no point in including a sludge treatment process which results in a final sludge that is impossible or extremely difficult to dewater. This is because it would greatly increase costs and could mean that down-stream processes would become unsuitable, such as the drying process to be used at Millbrook WWTW, which is designed to accept dewatered sludge.

The two batch digestion trials that were carried out were used to try and find out more about what was happening to the three different sludges during the digestion process. The amount of gas produced by each bottle was recorded and averages were calculated. On average, in both batch tests the 16 amp sonicated samples produced the most gas, followed by the 5 amp sonicated samples, with the control samples producing the smallest volume of gas. These results complement work carried out by Clark and Nujjoo (1998) who found increased gas yields following sonication, an effect which was shown in these batch trials but was not evident in the main experiment.

The batch trials were also used to look at the VFA content of the different sludges throughout the digestion process. In the first stage of the digestion process, high molecular compounds are broken down into smaller molecules, which are then converted into VFA's during the second stage. Sonication has the same effect as the first bacterial stage, so that small molecules are made readily available for conversion to VFA's. In theory, this means that when sonicated sludge is added to a seeded batch digester (as in these batch trials), the acidogenic bacteria can immediately utilise it to produce VFA's resulting in a sharp increase in total VFA content. The VFA's present will then be converted into methane, carbon dioxide and water by the methanogen bacteria population present, resulting in a dramatic decrease in the level of VFA's present. In the batch digestion trials carried out, this was found to happen, with the sonicated samples - particularly the high intensity sonicated samples - showing a higher total VFA yield than the control samples, and a greater increase in total VFA content at the start of the trial than the control samples. The same trend was shown clearly by some of the individual VFA's, with propionic acid being the example outlined in results sections 5.3.4.2 and 5.3.5.2. These results suggest that pre-treatment with ultrasound has a profound effect on the digestion process.

The batch digestion tests that were carried out suggests that pre-treatment of sludge with ultrasound does have a marked effect on the digestion process. The main experimental work however conflicts with these findings as no difference in the gas

volumes produced, digester gas methane content or volatile solids reduction were found.

One possible explanation for this could be in the hydraulic retention time (HRT) of the pilot scale digesters, which was set at 15 days as this is typical of high-rate digesters (Metcalf and Eddy, 1991). The digesters used had a capacity of 100 litres, meaning that feed sludge and the heat supplied would probably have been very well dispersed throughout the vessels, possibly making them more efficient than full-scale digesters. If this is the case then the HRT of 15 days that was used may have been unnecessarily long. It is possible that differences in gas production and volatile solids reduction between the control and sonicated sludge fed digesters would have been evident had the HRT been shorter. Conversely, it is also reasonable to suggest that the 15 day HRT chosen may not have been long enough and that more differences between the digesters would have been found had it been longer. Thiem *et al* (1997) for example used a 22 day HRT when looking at the effect of ultrasonic sludge pre-treatment on volatile solids reduction following digestion.

The batch digestion tests that were carried out differed from the main experiment in a number of ways, which could help to explain why the results from the 2 different types of trial were so different. For example, the pilot-scale digesters used are 200 times bigger than the bottles used in the batch trials. Another difference is that the pilot digesters were fed continuously, which could have meant that VS reduction was obscured by new feed, whereas the batch bottles were filled and sealed and not fed again throughout the trial. Also, the pilot-scale digesters were continually mixed and the batch bottles were not. Basically, a large continuously fed system is very different to a small batch fed system, making comparisons between them difficult and possibly unwise.

Despite the obvious differences between the two systems (batch and continuous), it would be expected that sonicated sludge added to either system would result in an increase in gas production, which did not occur in the pilot-scale digesters. The

experiments carried out by Clark and Nujjoo (1998) used digester vessels that were fed continuously and in those trials a large increase in gas production was found. However, these vessels were quite small at only 10 litres. Perhaps it is possible therefore, that this increase in gas production reduces as the scale of digesters increase.

A major difference between the batch trial and the pilot-scale digester work is that the batch tests did not involve a holding tank - the sonicated sludge went straight into the digestion vessel - whereas sludge entering the pilot-scale digesters had been held in a holding tank for up to four days. It is possible that the effects of sludge sonication that have been clearly shown by the various soluble COD tests, CST tests and batch digestion tests, are somehow negated during the sludge holding period. The sludge holding tanks are refrigerated, which prevents any bacterial degradation of the sludge, but they are mechanically stirred, which subjects the sludge to shear for up to four days. This shear effect may result in all the sludges being broken down to a similar structure, despite the original differences in structure due to sonication. Certainly after four days in the feed holding tanks, the sludges all looked very similar under the microscope, although no scientific evidence of this was obtained.

It is also feasible that whilst in the holding tanks, soluble compounds that were released from the solid portion of the sludge by the effects of sonication, were adsorbed onto the surfaces of sludge particles, making them less available and so negating the effect of ultrasound pre-treatment.

The possible reasons why the sludge holding stage may have negated the effects of sonication are of relatively little importance. This is because in a full-scale sludge treatment system, a holding stage following sonication would not be necessary. In a full scale system, The ultrasound unit would be situated on the sludge line just before the digester, so that sludge would be sonicated seconds before entering a digester, with the sonicator being switched on whenever sludge was moving through the line.

7.0 CONCLUSIONS

- One of the findings of this investigation was that sludge CST values were longer after sonication. This means that sludge dewatering becomes more difficult following sonication. This issue needs to be looked into further as it may mean that ultrasonic pre-treatment is not economically viable despite any possible advantages of the process.

- Soluble COD tests and the batch digestion trials show that sludge pre-treatment with ultrasound does have an effect on sludge structure and anaerobic digestion. Sludge treated at the higher intensity had a higher soluble COD, which resulted in a greater volume of gas production due to more efficient digestion. This shows that ultrasonic pre-treatment of sludge can be shown to have definite advantages.

- The main experiment used larger scale digesters than the batch digestion trials and added a sludge storage stage prior to digestion. The results showed no differences in volatile solids reduction or in the volume of digester gas produced. There were a number of differences between the operation of the batch digestion trials and the main experiment, such as the sludge holding stage. However, similar patterns of results, for example gas production being higher for the sludge treated with the higher intensity ultrasound, were expected. The presence of the sludge holding stage is likely to be primarily responsible for the fact that similar patterns of results were not found.

- The possible reasons why the sludge holding stage may have negated the effects of sonication have already been discussed, but are of little importance. This is because in a full-scale sludge treatment system, a holding stage following sonication would not be necessary.

- Overall, the work carried out has shown the potential of ultrasonic pre-treatment of sludge prior to digestion, but has also highlighted potential problems in this use of the technology, which can now be avoided in future work of this kind.

8.0 FUTURE WORK

One possibility for future work is to look into why the holding tanks negated the effect of ultrasonic treatment, although this is of interest, it is not seen as a priority as on a large scale system, sonicated sludge would not be stored, as it would be pumped straight into a digester.

Ideally, future work would involve a similar trial to that carried out, but using a different set-up, with no sludge holding stage. With the present system this would be impractical as it would be very labour intensive to sonicate sludge prior to every digester feed. Possibly the most practical method of carrying out this trial would be to use an established full scale digester, with a sonication unit attached to the digester sludge feed line, that would be switched on automatically whenever sludge was pumped through. However, a trial on this scale may be undesirable until more small scale background work has been carried out.

The most urgent follow-up work to be done is to carry out more investigations into the effect of ultrasound on the dewaterability of sludge once it has been digested. This work would involve conventional testing using CST apparatus, but would also have to include the use of the intended dewatering method, which in this case is a belt filter press. This work is important and should be carried out before any more detailed investigations into the effects of ultrasound on the anaerobic digestion process, because if sonicated sludge does not dewater well enough after digestion, and no cheap methods of improvement can be found, ultrasonic pre-treatment of sludge prior to digestion is not viable.

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10.0 APPENDICES

10.1 Daily Gas Production

	Volume gas	Produced	(litres)		Volume gas	Produced	(litres)
DATE	5 amp US	16 amp US	Control	DATE	5 amp US	16 amp US	Control
07-May-98	219.71	153.97	217.98	21-Jun-98	252.58	186.84	223.17
08-May-98	205.87	202.41	254.31	22-Jun-98	243.93	188.57	216.25
09-May-98	95.15	204.14	205.87	23-Jun-98	212.79	190.30	205.87
10-May-98	48.44	202.41	108.99	24-Jun-98	204.14	178.19	204.14
11-May-98	74.39	183.38	117.64	25-Jun-98	211.06	173.00	195.49
12-May-98	205.87	167.81	176.46	26-Jun-98	214.52	185.11	237.01
13-May-98	382.33	280.26	275.07	27-Jun-98	29.41	72.66	13.84
14-May-98	283.72	287.18	273.34	28-Jun-98	1.73	1.73	3.46
15-May-98	351.19	307.94	247.39	29-Jun-98	110.72	0.00	96.88
16-May-98	280.26	249.12	216.25	30-Jun-98	141.86	0.00	129.75
17-May-98	249.12	259.50	198.95	01-Jul-98	153.97	0.00	160.89
18-May-98	240.47	169.54	211.06	02-Jul-98	169.54	0.00	178.19
19-May-98	228.36	214.52	200.68	03-Jul-98	119.37	0.00	197.22
20-May-98	249.12	245.66	223.17	04-Jul-98	0.00	0.00	200.68
21-May-98	226.63	250.85	219.71	05-Jul-98	0.00	0.00	131.48
22-May-98	155.70	221.44	192.03	06-Jul-98	17.30	0.00	147.05
23-May-98	181.65	152.24	167.81	07-Jul-98	148.78	0.00	190.30
24-May-98	204.14	176.46	176.46	08-Jul-98	129.75	0.00	214.52
25-May-98	212.79	195.49	197.22	09-Jul-98	138.40	0.00	195.49
26-May-98	174.73	178.19	162.62	10-Jul-98	105.53	105.53	129.75
27-May-98	195.49	190.30	183.38	11-Jul-98	77.85	76.12	50.17
28-May-98	207.60	192.03	209.33	12-Jul-98	58.82	83.04	29.41
29-May-98	155.70	193.76	205.87	13-Jul-98	44.98	89.96	36.33
30-May-98	72.66	204.14	185.11	14-Jul-98	83.04	93.42	57.09
31-May-98	46.71	193.76	174.73	15-Jul-98	69.20	88.23	65.74
01-Jun-98	93.42	193.76	178.19	16-Jul-98	41.52	36.33	15.57
02-Jun-98	145.32	153.97	188.57	17-Jul-98	31.14	29.41	32.87
03-Jun-98	209.33	153.97	212.79	18-Jul-98	0.00	0.00	0.00
04-Jun-98	195.49	133.21	57.09	19-Jul-98	0.00	0.00	1.73
05-Jun-98	197.22	152.24	205.87	20-Jul-98	57.09	39.79	76.12
06-Jun-98	212.79	166.08	100.34	21-Jul-98	83.04	81.31	83.04
07-Jun-98	214.52	166.08	93.42	22-Jul-98	91.69	57.09	83.04
08-Jun-98	150.51	169.54	128.02	23-Jul-98	105.53	103.80	167.81
09-Jun-98	148.78	162.62	204.14	24-Jul-98	105.53	44.98	176.46
10-Jun-98	216.25	223.17	259.50	25-Jul-98	107.26	19.03	157.43
11-Jun-98	268.15	240.47	89.96	26-Jul-98	74.39	12.11	185.11
12-Jun-98	257.77	192.03	152.24	27-Jul-98	0.00	0.00	13.84
13-Jun-98	269.88	223.17	252.58	28-Jul-98	0.00	0.00	0.00
14-Jun-98	273.34	238.74	262.96	29-Jul-98	0.00	0.00	0.00
15-Jun-98	269.88	214.52	235.28	30-Jul-98	0.00	0.00	0.00
16-Jun-98	281.99	197.22	230.09	31-Jul-98	0.00	0.00	0.00
17-Jun-98	224.90	186.84	228.36	01-Aug-98	0.00	0.00	0.00
18-Jun-98	237.01	190.30	212.79	02-Aug-98	0.00	0.00	0.00
19-Jun-98	224.90	186.84	204.14	03-Aug-98	0.00	0.00	0.00
20-Jun-98	247.39	195.49	228.36	04-Aug-98	0.00	0.00	0.00

10.2 Volatile Solids Reduction

Date	B	C	D	Date	B	C	D
07-May-98	47.24%	34.88%	47.64%	**19-Jun-98**	45.68%	46.34%	46.97%
08-May-98	51.28%	36.70%	52.95%	**22-Jun-98**	43.67%	48.83%	55.78%
11-May-98	55.83%	50.72%	57.46%	**23-Jun-98**	49.84%	44.54%	45.74%
12-May-98	56.81%	37.93%	54.05%	**24-Jun-98**	43.59%	43.11%	42.85%
13-May-98	57.76%	41.12%	54.04%	**25-Jun-98**	44.44%	41.85%	42.25%
14-May-98	58.65%	43.37%	50.61%	**26-Jun-98**	44.19%	41.25%	42.23%
15-May-98	60.34%	56.84%	53.82%	**29-Jun-98**	44.80%	44.05%	40.89%
18-May-98	55.18%	56.57%	52.40%	**30-Jun-98**	44.39%	40.75%	44.39%
19-May-98	57.42%	57.61%	55.66%	**01-Jul-98**	43.06%	39.35%	43.23%
20-May-98	55.92%	56.67%	53.68%	**02-Jul-98**	43.44%	39.41%	39.96%
21-May-98	58.23%	58.65%	56.01%	**03-Jul-98**	42.55%	36.38%	39.43%
22-May-98	58.47%	60.40%	55.11%	**06-Jul-98**	46.35%	40.67%	37.91%
26-May-98	54.59%	55.96%	51.91%	**07-Jul-98**	43.48%	36.80%	36.78%
27-May-98	52.03%	50.72%	48.03%	**08-Jul-98**	43.87%	37.74%	39.63%
28-May-98	50.56%	52.33%	49.53%	**09-Jul-98**	43.69%	35.06%	38.72%
01-Jun-98	53.65%	48.40%	48.52%	**13-Jul-98**	43.55%	38.91%	36.60%
02-Jun-98	50.87%	47.39%	45.34%	**14-Jul-98**	43.77%	42.37%	40.80%
03-Jun-98	50.00%	48.48%	45.36%	**15-Jul-98**	42.14%	39.59%	39.41%
04-Jun-98	51.97%	50.69%	46.08%	**16-Jul-98**	42.15%	38.36%	40.87%
05-Jun-98	50.45%	50.47%	42.50%	**17-Jul-98**	41.29%	39.13%	38.64%
08-Jun-98	48.58%	51.10%	45.61%	**20-Jul-98**	41.57%	42.33%	39.93%
09-Jun-98	51.59%	48.06%	44.21%	**21-Jul-98**	40.30%	43.50%	37.89%
10-Jun-98	47.76%	48.77%	42.59%	**22-Jul-98**	40.22%	36.52%	39.27%
11-Jun-98	46.01%	45.73%	44.46%	**23-Jul-98**	40.11%	43.28%	39.38%
12-Jun-98	50.06%	52.82%	43.16%	**27-Jul-98**	39.82%	36.13%	39.57%
15-Jun-98	46.16%	47.13%	42.37%	**28-Jul-98**	32.74%	36.13%	39.80%
16-Jun-98	45.31%	45.36%	43.18%	**29-Jul-98**	34.56%	35.67%	38.87%
17-Jun-98	45.64%	46.61%	43.54%	**30-Jul-98**	35.67%	35.97%	36.45%
18-Jun-98	45.24%	45.87%	44.46%	**03-Aug-98**	34.56%	36.34%	37.43%

10.3 Gas Analysis Data

Date	5 amp US		16 amp US		Control	
	CH4(%)	CO2(%)	CH4(%)	CO2(%)	CH4(%)	CO2(%)
13-May-98	55	43	55.5	43	54	44.5
14-May-98	56	40	57.3	39.3	57.3	40.1
15-May-98	63.5	25	52	31.1	59.3	39.6
18-May-98	61.4	37.5	61.9	37.8	61.5	37.2
19-May-98	59.9	37.7	60.7	37.8	60.7	38.1
20-May-98	61.2	38.1	61.2	33.3	55.2	22.4
21-May-98	62.4	36.6	62.8	35.9	62.5	37.1
22-May-98	60	37.4	61.4	36.9	63	36.3
26-May-98	62.2	35.5	34.5	35.5	34.9	34.5
27-May-98	64.9	34.5	64.9	34.8	65.2	34.4
28-May-98	71.3	34.8	71	35.7	71.6	34.8
01-Jun-98	71.5	34.2	71.4	34	70.4	34.7
02-Jun-98	75.6	31.5	71.9	34.3	71.6	34.5
26-Jun-98	66.3	32.3	65.3	33.5	60.5	28
07-Jul-98	68.5	30.2	63.8	35.9	56.1	26
08-Jul-98	66.7	33	64	36.1	56	21.9
09-Jul-98	62.8	37.2	62.8	21.8	57.7	24.5
13-Jul-98	70.1	28.5	71.8	27.2	67.1	32.4
14-Jul-98	68	32	58	17.6	54.2	14.3
15-Jul-98	68	30.7	59.6	22.4	59.8	21.6
16-Jul-98	68	31.2	58.9	19.4	57.2	20
29-Jul-98	63.5	35.4	61.2	27.6	53.7	22.3
30-Jul-98	64.3	35.2	66.7	31.6	69.4	30.2

10.4 Batch Test 1 Data

CONTROL

Date	Sample pH	Total Dry Solids	Volatile solids	VS as % of DS
22-Jun-98	7.66	6.08%	4.62%	75.96%
23-Jun-98	7.5	6.10%	4.57%	74.96%
24-Jun-98	7.56	5.80%	4.33%	74.62%
25-Jun-98	7.82	5.64%	4.16%	73.79%
26-Jun-98	7.83	5.46%	3.98%	72.94%
29-Jun-98	7.95	5.40%	3.84%	71.14%
01-Jul-98	8.21	6.04%	4.26%	70.54%
03-Jul-98	7.94	5.58%	3.98%	71.26%

5 amp US

Date	Sample pH	Total Dry Solids	Volatile solids	VS as % of DS
22-Jun-98	7.68	6.45%	4.96%	77.00%
23-Jun-98	7.57	5.92%	4.48%	75.67%
24-Jun-98	7.73	6.10%	4.44%	72.67%
25-Jun-98	7.88	5.48%	3.92%	71.51%
26-Jun-98	7.99	5.66%	4.12%	72.89%
29-Jun-98	8.03	5.94%	3.95%	66.51%
01-Jul-98	8.32	5.57%	3.65%	65.46%
03-Jul-98	7.95	5.29%	3.77%	71.31%

16 amp US

Date	Sample pH	Total Dry Solids	Volatile solids	VS as % of DS
22-Jun-98	7.65	6.26%	4.78%	76.39%
23-Jun-98	7.57	6.56%	4.93%	75.24%
24-Jun-98	7.86	6.04%	4.49%	74.32%
25-Jun-98	7.93	5.54%	4.08%	73.58%
26-Jun-98	7.99	5.70%	4.18%	73.33%
29-Jun-98	8.04	5.65%	3.62%	64.09%
01-Jul-98	8.26	5.38%	3.47%	64.38%
03-Jul-98	7.97	5.25%	3.69%	70.21%

CONTROL	COD	acetic A	ropionic	n-butyric A	so-butyric	n-valeric A
22-Jun-98	59500	445	415	46	62	56
23-Jun-98	66500	540	550	16	53	37
24-Jun-98	44200	35	64	16	35	37
25-Jun-98	52100	35	20	16	35	37
26-Jun-98	66800	35	20	16	35	37
29-Jun-98	68400	35	20	16	35	37
01-Jul-98	45000	35	20	16	35	37
03-Jul-98	33900	35	20	16	35	37

	iso-valeric A	n-caproic	o-caproic	VFA total
22-Jun-98	97	32	15	1120
23-Jun-98	42	32	15	1190
24-Jun-98	30	32	15	< 220
25-Jun-98	30	32	15	< 220
26-Jun-98	30	32	15	< 220
29-Jun-98	30	30	32	< 220
01-Jul-98	30	32	15	< 220
03-Jul-98	30	32	15	< 220

5 AMP US	COD	acetic A	ropionic	n-butyric A	so-butyric	n-valeric A
22-Jun-98	26200	760	410	54	59	58
23-Jun-98	56800	530	590	16	77	37
24-Jun-98	68800	84	215	16	35	37
25-Jun-98	59500	35	20	16	35	37
26-Jun-98	64700	35	20	16	35	37
29-Jun-98	50500	35	20	16	35	37
01-Jul-98	49400	35	20	16	35	37
03-Jul-98	36200	35	20	16	35	37

	iso-valeric A	n-caproic	o-caproic	VFA total
22-Jun-98	96	32	15	1440
23-Jun-98	78	32	15	1280
24-Jun-98	30	32	15	300
25-Jun-98	30	32	15	< 220
26-Jun-98	30	32	15	< 220
29-Jun-98	30	32	15	< 220
01-Jul-98	30	32	15	< 220
03-Jul-98	30	32	15	< 220

16 AMP US	COD	acetic A	ropionic	n-butyric A	so-butyric	n-valeric A
22-Jun-98	43400	430	395	45	57	55
23-Jun-98	67700	720	800	16	90	37
24-Jun-98	37600	35	340	16	35	37
25-Jun-98	64000	35	20	16	35	37
26-Jun-98	55800	35	20	16	35	37
29-Jun-98	76000	35	20	16	35	37
01-Jul-98	44200	35	20	16	35	37
03-Jul-98	40500	35	20	16	35	37

	iso-valeric A	n-caproic	o-caproic	VFA total
22-Jun-98	93	32	15	1080
23-Jun-98	105	32	15	1720
24-Jun-98	30	32	15	340
25-Jun-98	30	32	15	< 220
26-Jun-98	30	32	15	< 220
29-Jun-98	30	32	15	< 220
01-Jul-98	30	32	15	< 220
03-Jul-98	30	32	15	< 220

Daily gas production (litres)			
Date	5 amp US	16 amp US	Control
23-Jun-98	22.9	22.5	20.1
24-Jun-98	25.4	25.4	23.2
25-Jun-98	15.7	16.1	18.6
26-Jun-98	10.7	11.7	12.2
27-Jun-98	4.1	5.8	4.7
28-Jun-98	4.1	5.8	4.7
29-Jun-98	4.1	5.8	4.7
30-Jul-98	1.6	2.5	2.5
01-Jul-98	0.6	1.5	1.6
02-Jul-98	0.3	0.4	0.8
03-Jul-98	0.3	0.3	0.7

10.5 Batch Test 2 Data

CONTROL

Date	Sample Point	Sample pH	otal Dr Solids	Volatile solids	VS as % of DS
20-Jul-98	Control	7.31	6.27%	4.54%	72.38%
21-Jul-98	Control	7.33	6.10%	4.35%	71.34%
22-Jul-98	Control	7.45	5.64%	6.14%	108.85%
23-Jul-98	Control	7.53	5.29%	3.72%	70.35%
24-Jul-98	Control	7.68	6.14%	4.29%	69.87%
27-Jul-98	Control	7.86	5.52%	3.78%	68.35%

5 amp US

Date	Sample Point	Sample pH	otal Dr Solids	Volatile solids	VS as % of DS
20-Jul-98	2	7.31	6.82%	5.07%	74.38%
21-Jul-98	2	7.32	6.79%	4.83%	71.15%
22-Jul-98	2	7.28	5.87%	4.12%	70.20%
23-Jul-98	2	7.58	5.62%	3.93%	69.84%
24-Jul-98	2	7.63	6.03%	4.17%	69.20%
27-Jul-98	2	7.9	5.74%	3.85%	67.13%

16 amp US

Date	Sample Point	Sample pH	otal Dr Solids	Volatile solids	VS as % of DS
20-Jul-98	8	7.43	6.05%	4.32%	71.42%
21-Jul-98	8	7.4	6.60%	4.73%	71.68%
22-Jul-98	8	7.45	5.75%	4.05%	70.46%
23-Jul-98	8	7.63	6.61%	4.49%	67.97%
24-Jul-98	8	7.58	6.44%	4.30%	66.69%
27-Jul-98	8	7.94	5.58%	3.76%	67.26%

CONTROL	COD	acetic A	ropionic	n-butyric A	so-butyric	n-valeric A
20-Jul-98	45900	580	270	66	53	39
21-Jul-98	70800	770	530	16	46	37
22-Jul-98	61500	35	850	16	35	37
23-Jul-98	48500	35	420	16	35	37
24-Jul-98	58300	35	380	16	35	37
27-Jul-98	50100	35	86	16	35	37

	iso-valeric A	Total VFA
20-Jul-98	67	1080
21-Jul-98	48	1390
22-Jul-98	30	850
23-Jul-98	30	420
24-Jul-98	30	280
27-Jul-98	30	86

5 AMP US	COD	acetic A	ropionic	n-butyric A	so-butyric	n-valeric A
20-Jul-98	72600	650	340	100	66	50
21-Jul-98	137400	1000	660	16	69	37
22-Jul-98	73800	230	1080	16	66	37
23-Jul-98	48200	35	900	16	35	37
24-Jul-98	51500	35	700	16	35	37
27-Jul-98	48900	35	20	16	35	37

	iso-valeric A	Total VFA
20-Jul-98	88	1290
21-Jul-98	78	1800
22-Jul-98	65	1440
23-Jul-98	30	900
24-Jul-98	30	700
27-Jul-98	30	220

16 AMP US	COD	acetic A	ropionic	n-butyric A	so-butyric	n-valeric A
20-Jul-98	69900	520	250	63	53	37
21-Jul-98	77100	940	620	33	73	37
22-Jul-98	69300	315	1000	16	81	37
23-Jul-98	51000	35	840	16	35	37
24-Jul-98	49900	35	800	16	35	37
27-Jul-98	47300	35	170	16	35	37

	iso-valeric A	Total VFA
20-Jul-98	72	860
21-Jul-98	86	1750
22-Jul-98	86	1480
23-Jul-98	30	840
24-Jul-98	30	800
27-Jul-98	30	170

	Daily gas production (litres)		
Date	**5 amp US**	**16 amp**	**control**
21-Jul-98	21	21.3	21.9
22-Jul-98	21.4	21.4	20.9
23-Jul-98	15.1	14.8	13.1
24-Jul-98	7.1	7.6	7.4
25-Jul-98	4.9	5.2	3.2
26-Jul-98	4.9	5.2	3.2
27-Jul-98	4.9	5.2	3.2
28-Jul-98	0.8	1.4	0.6
29-Jul-98	0.8	2.1	0.5
30-Jul-98	0	0	0
31-Jul-98	0	0	0