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Ultrasonic treatment of sewage sludge in order to increase biogas yields

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Keywords
ultrasound, waste activated sludge, anaerobic digestion, biogas, digested sewage sludge, degassing

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June 2005

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Abstract

Biogas, a mixture of methane and carbon dioxide, is produced in the anaerobic digestion of sewage sludge. After anaerobic digestion, the digested sludge is often allowed to degas for one or two days. This gas is seldom utilised, but if the degassing could be accelerated, utilisation would be easier. Ultrasound can be used as a pretreatment method for waste activated sludge. It has a disintegrating effect on the sludge and causes lysis of bacteria in the sludge. It also speeds up the hydrolysis; the limiting step of anaerobic digestion of waste activated sludge. Ultrasound can be used to degas water-based liquids. Ultrasonic degassing of sewage sludge has not been examined previously. The present study aims to investigate the effect of ultrasound on waste activated sludge as well as the potential of ultrasound to speed up the degassing of digested sludge. A semi-continuous, lab-scale digestion experiment was performed with four reactors: two receiving untreated sludge and two receiving treated sludge. The effect of the sonicator was 420 W and the treatment time was 6 min, which corresponds to an energy input of 8.4 kWh/m^3. Total solids (TS) of the waste activated sludge was $\sim$ 3.5 %. The ultrasonic treatment caused an increase in gas production of 13 %. There was no difference in methane content. The concentration of filterable chemical oxygen demand (fCOD) increased 375 %, or from 2.8 % to 11 % of total COD. In terms of energy loss/gain the increase in gas production resulted in a loss of 2.7 kWh/m^3, i.e. more energy is needed to treat the sludge than the potential energy of the increased gas production. However, if the sludge is thickened to a TS >5 %, a net energy gain should be reached. The effect of ultrasound on the degassing of digested sludge was examined in three barrels. The degassing was measured with and without circulation as well as with ultrasonic treatment. The digested sludge had a gas emission rate of 115 L (m^3 day)$^{-1}$. No direct burst of gas occurred due to ultrasonic treatment. Over two days more gas was emitted from the barrel equipped with ultrasound, probably due to an induced post-digestion. Thus, ultrasonic pretreatment of waste activated sludge increases the biogas yield. It is inconclusive, whether ultrasonic treatment of digested sludge effects the degassing or not.

Sammanfattning

Biogas, en blandning av metan och koldioxid, bildas genom anaerob nedbrytning av avloppsslam. Efter anaerob nedbrytning avgasas oftast rötslammet i ett eller två dygn. Gas från avgasningen används sällan, men om avgasningen kunde accelereras skulle omhändertagande av gasen underlättas. Ultraljud kan användas som en förbehandlingsmetod för bioslam (aktivt slam). Behandlingen sönderdelar slammet och lyserar bakterier i slammet. Behandlingen påskyndar även hydrolysen som är det begränsande steget vid anaerob nedbrytning av bioslam. Ultraljud kan användas för att avgasa vätskor. Avgasning av avloppsslam med ultraljud har ej undersökts tidigare. Denna studie har undersökt effekten av ultraljud på bioslam samt möjligheten att påskynda avgasning av avloppsslam med ultraljud. Ett semikontinuerligt rötningsförsök i laboratorieskala utfördes med fyra reaktorer: två med obehandlat slam och två med ultraljudsbehandlat slam. Sonikatoreffekten var 420 W och behandlingstiden var 6 min, vilket motsvarar en energitillförsel på 8.4 kWh/m^3. Bioslammets torrsubstans (TS) var $\sim$ 3.5 %. Gasproduktionen ökade med 13 % på grund av ultraljudsbehandlingen. Det var ingen skillnad i gasens metanhalt. Koncentrationen av filtrerbart COD ökade 375 %, motsvarande en ökning från 2.8 % till 11 % av totalt COD. Mer energi krävdes för ultraljudsbehandlingen än vad som potentiellt fås genom den ökade gasproduktionen. Om slammet förtjockas till en TS >5 % borde en energimässig nettovinst göras. Effekten av ultraljud på avgasning av rötslam undersöktes i tre tunnor. Avgasningen mättes med och utan cirkulation av rötslammet samt vid ultraljudsbehandling. Gas avgick från rötslammet med 115 L $(m^3\,dag)^{-1}$. Ingen stor, direkt avgång av gas skedde på grund av ultraljudsbehandlingen. Efter två dygn hade mer gas avgått från det ultraljudsbehandlade slammet, antagligen på grund av en ökad efterrötning. Således ökar ultraljudsbehandling av bioslam biogasutbytet. Dock kan inga slutgiltiga slutsatser dras gällande ultraljuds påverkan på avgasning av rötslam.

Preface

This final thesis concludes my Master of Science in Engineering Biology, with a profile in Environmental Science, at Linköping Institute of Technology, one of the faculties of Linköping University. The majority of the work has been carried out at Slottshagen — the sewage treatment plant in Norrköping, with additional work done at the Department of Water and Environmental Studies in Linköping.

Before you continue with the thesis, read the quote below and do not forget that sludge is more than 90 % water.

> "If there is magic on the planet, it is contained in Water."
>
> — Loren Eiseley, *The Immense Journey*, 1957

Anders Ek, May 2005
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Contents

1 Introduction

Sewage treatment plants generate sludge as the single largest residual product of the sewage treatment process. Although rich in nutrients, sewage sludge is not yet generally accepted for use as an agricultural fertilizer in Sweden. The resistance from the farming industry concerns mostly fear of heavy metals and other presumably toxic compounds. As long as no definitive solution to the sludge problem exists, means of minimising the amount of sludge are highly interesting.

Another byproduct of the sewage-treatment process is biogas; a mixture of methane and carbon dioxide. Methanogenic archaea produce biogas in anaerobic digestion of the sludge. Biogas can be utilised to produce heat and electricity or be upgraded to motor vehicle fuel. As the interest in and require of non-fossil fuels increase, ways to produce more biogas from the same amount of sludge become more attractive.

Luckily the strive for decreasing the amount of residual sludge often naturally coincides with the desire to increase the amount of biogas produced. If more matter leaves the anaerobic digestion process in the form of biogas, less is left as sludge. However, a reduced sludge mass with the same amount of toxic compounds results in raised concentrations of the toxic compounds, although the actual amount is the same.

After anaerobic digestion of sewage sludge, the digested sludge is often allowed to degas for one or two days. This gas is seldom utilised, and acts as a greenhouse gas if released into the atmosphere. But if the degassing could be accelerated, a utilisation would be easier.

The use of ultrasonic pretreatment of waste activated sludge has been investigated extensively over the last decade. Ultrasound has a disintegrating effect on the sludge and causes lysis of bacteria present in the sludge. Reports in the literature demonstrate increased concentrations of soluble organic material, increased reduction of organic material and increased gas production, after ultrasonic treatment. Another, common application, of ultrasound is the degassing of water-based liquids. Ultrasonic degassing of sewage sludge has not been examined previously.

This study aims to investigate the effect of ultrasound on waste activated sludge as well as the potential of ultrasound to speed up the degassing of digested sludge.

2 Background

2.1 Anaerobic Digestion

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

2.1.1 History of anaerobic digestion

The earliest known use of anaerobic fermentation dates back 8 000–6 000 years ago with the production of ethanol, lactic and other fatty acids for different preservation techniques (Hughes, 1980; Ecke and Lagerkvist, 2000). Throughout history, the midden of the Neolithic farmer and septic tank systems of the early cities can be seen as precursors to the anaerobic waste handling systems of today.

In the nineteenth century the microorganisms responsible for the anaerobic process were first described by Pasteur (Hughes, 1980). It was also concluded that methane forms from the biological breakdown of cellulose (Klass, 1984).

For the last hundred years anaerobic digestion systems much like the ones seen today have been used for waste disposal and stabilisation (Klass, 1984). As of today anaerobic digestion is the most commonly applied method of treatment for sewage sludge (Tiehm et al., 1997; Grönroos et al., 2005).

2.1.2 Microbiology of anaerobic digestion

Anaerobic digestion is a multi-step process carried out by a mixed culture of different groups of microorganisms. The process consists of four main steps which are carried out by at least three groups of microorganisms: acidogenic bacteria, acetogenic bacteria and methanogenic archaea (Ecke and Lagerkvist, 2000; de Mes et al., 2003). Figure 1 summarises the process.

Organic matter consists of particulate, water-insoluble polymers such as carbohydrates, lipids and proteins. Insoluble polymers cannot penetrate cellular membranes and are therefore not directly available to the microorganisms. In the first step, *hydrolysis*, acidogens excrete hydrolytic enzymes which break up the insoluble polymers to soluble mono- and oligomers. Carbohydrates are converted to sugars, lipids are broken down to long-chain fatty acids and proteins are split into amino acids. These soluble molecules are, through the *acidogenesis*, converted by acidogens to acetic acid and other longer volatile fatty acids, alcohols, carbon dioxide and hydrogen. During the *acetogenesis* the longer volatile fatty acids and alcohols are oxidised by proton-reducing acetogens to acetic acid and hydrogen. In the last step, *methanogenesis*, methanogens use acetic acid or carbon dioxide and hydrogen to produce methane and carbon dioxide.

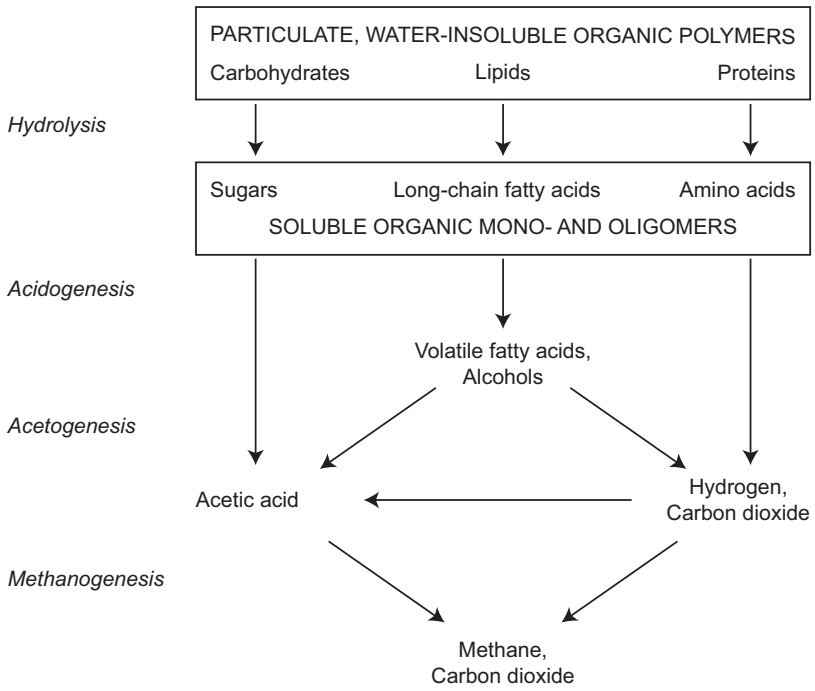

Figure 1: Summary of the anaerobic digestion chain. Modified after de Mes et al. (2003).

For the anaerobic degradation chain to work as a whole and to prevent build up of intermediate metabolites, the environmental factors has to be favourable for all organisms involved. Two of the most central environmental factors are temperature and pH.

Anaerobic digestion can occur in temperatures ranging from 0 to 97 ℃ (Bitton, 1999). Three different temperature intervals are identified: 0–20 ℃ for the psychrophilic organisms, 20–40 ℃ for the mesophilic organisms and 50–60 ℃ for the thermophilic organisms (de Mes et al., 2003). Methane production at low temperatures is slow and requires large reactor volumes and long retention times. For mesophilic bacteria the optimal methane production rate is mostly reached at 35–37 ℃. The thermophilic methanogens differ from the mesophilic ones and their maximum methanogenic activity is reached at about 55 ℃. A thermophilic digestion process can sustain a higher organic loading compared to a mesophilic one. But the thermophilic process produces a gas with a lower methane concentration (Ecke and Lagerkvist, 2000) and is more sensitive to toxicants (Bitton, 1999). Methanogens are more sensitive toward changes in

temperature than the other species, because of their slower growth rate in the reactor environment.

Of all four digestion steps only the methanogenesis is critically pH dependent (de Mes et al., 2003) in the digestion of sewage sludge. Methanogenesis occurs at neutral pH; in the range of 6.5–7.5, although optimum lies at pH 7.0–7.2 (Bitton, 1999). If, for example, a temperature shift affects the methanogens negatively there can be a build up of VFAs. This lowers the pH which further affects the methanogens in a negative way which leads to a vicious circle of negative feedback.

2.2 Utilisation of biogas

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

The European Union states that at the end of 2005 the share of biofuels in relation to all fuel used for transport shall be at least 2 % (based on energy content) in all member states (European Parliament and the Council of the European Union, 2003). The reference level for 2010 is 5.75 %. In Sweden a national goal of 3 % is set for 2005 (Ministry of Sustainable Development, 2004).

2.3 Sludges from sewage treatment

Sewage treatment generates primarily two kind of sludges: primary or raw sludge and activated or secondary sludge. They are mostly stabilised through anaerobic digestion to the end-product: digested sludge. Table 1 shows values of gas production from primary sludge and activated sludge cited by Brown et al. (2003).

Table 1: Gas production from primary sludge and activated sludge, as cited by Brown et al. (2003).

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

2.3.1 Primary sludge

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

2.3.2 Activated sludge

Activated sludge comes from the secondary treatment. The excess sludge is called waste activated sludge and is a result of overproduction of microorganisms in the active sludge process. It is light and fluffy and composed of microorganisms flocculated organic matter (Liu and Lipták, 1999). The organisms are primarily bacteria and protozoa, but also rotifers and filamentous bacteria. Activated sludge is more difficult to digest than primary sludge.

Filamentous bacteria are a normal part of the activated sludge microflora (Bitton, 1999). If the process is run suboptimally the filamentous bacteria can increase in numbers and cause foaming of the active sludge process. A high number of filamentous bacteria in the waste activated sludge can also cause foaming of anaerobic digesters. Common species are *Nocardia* spp. and *Microthrix parvicella*.

2.3.3 Digested sludge

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

2.4 Degassing of digested sludge

At sewage treatment plants the digested sludge is often allowed to degas in a storage tank before it is dewatered. Gas left in the sludge aggravates the dewatering. If the emitted gas is not collected and utilised or flared, it will escape to the atmosphere and act as a greenhouse gas.

Starberg and Welin (2004) estimates that the loss of methane from the storage tanks at the Bromma sewage treatment plant in Stockholm, Sweden corresponds to 1.75 % of the total amount of produced methane at the plant.

2.5 Pretreatment of sewage sludge

The rate-limiting step of anaerobic digestion of waste activated sludge is hydrolysis (Tiehm et al., 1997; Brown et al., 2003), e.g. break up of cell walls and disintegration of sludge flocs. A pretreatment step would render hydrolysis less difficult, thus giving a more efficient process.

Examples of pretreatment methods presented in the literature are ultrasound, thermal pretreatment, enzyme addition, ozonation, chemical solubilisation by acid or base addition and mechanical disintegration. According to Tiehm et al. (1997) full-scale operations of all pretreatment methods except ultrasound have been limited owing to high operating and capital costs.

2.6 Ultrasonic treatment of waste activated sludge

Ultrasound as a pretreatment method has been investigated on laboratory, pilot and full-scale levels. Reports in the literature tell of floc-size reduction (Chu et al., 2001, 2002), cell lysis (Tiehm et al., 1997; Chu et al., 2001; Brown et al., 2003), increased concentration of soluble chemical oxygen demand (sCOD) (Tiehm et al., 1997; Chu et al., 2001; Lafitte-Trouqué and Forster, 2002; Brown et al., 2003; Grönroos et al., 2005), increased volatile solids (VS) reduction (Tiehm et al., 1997, 2001; Brown et al., 2003; Rooksby, 2001) and increased biogas production (Tiehm et al., 2001; Chu et al., 2002; Rooksby, 2001; Brown et al., 2003; Grönroos et al., 2005). Disintegration of cellular structures is most significant at low frequencies, because the bubble radius is inversely proportional to the frequency and large bubbles mean strong shear forces (Tiehm et al., 2001). Therefore, an ultrasound frequency of 20 kHz ought to be the most appropriate. The effects of ultrasound on liquids and sludges are described in depth in section 2.8. Previous work done on ultrasonic treatment of waste activated sludge is summarised in table 2.

Table 2: Summary of previous work done on ultrasonic treatment of waste activated sludge.

Reference		sCOD	VS reduction	Biogas production	Frequency (kHz)	Power input	Treatment time	Energy input	Scale
Tiehm et al. (1997)	Control	630 mg/L[a]	45.8 %						Pilot
	Sonicated	2270 mg/L[a]	50.3 %	None[b]	31	3.6 kW	64 s	0.064 kWh	Pilot
Chu et al. (2001)	Control	0.5 %[c]							Lab
	Sonicated	20 %[c]		Increase	20	0.33 W/mL	120 min	660 kWh/m^3	Lab
Tiehm et al. (2001)	Control		21.5 %						Lab
	Sonicated		27.3 %	Increase	41		30 min		Lab
	Sonicated	Increase	33.7 %	Increase	41		150 min		Lab
Chu et al. (2002)	Sonicated			Increase	20	0.33 W/mL	20 min	110 kWh/m^3	Lab
Lafitte-Trouqué and Forster (2002)	Sonicated	+354 %	None[d]	None[d]	23	0.47 W/mL	90 s	12 kWh/m^3	Lab
Rooksby (2001)[e]	Control		46 %						Full
	Sonicated		78.7 %	+25–50 %	20	$5 \cdot 3$ kW[f]	~ 1.5 s		Full
Brown et al. (2003)[e]	Control			340 mL/g VS					Full
	Sonicated	Increase		550 mL/g VS	20	$5 \cdot 3$ kW[f]	≥ 1.5 s		Full
Grönroos et al. (2005)	Sonicated			+10–20 %[g]	27	≤ 300 W/L	≤ 30 min	≤ 150 kWh/m^3	Lab
Ola Ödmark (Ultra Sonus AB, February 2005, pers. comm.)	Sonicated	+100–400 %			20				Full

[a] Average values
[b] Only increase in gas production with reduced retention times
[c] sCOD as a percentage of total COD
[d] No statistical significance
[e] Rooksby (2002) and Brown et al. (2003) use the same kind of equipment
[f] Five ultrasonic horns each operating at 3 kW
[g] Most assays, some cases gave a slight decrease

2.7 Slottshagen sewage treatment plant

Slottshagen sewage treatment plant serves the city of Norrköping, a middle sized city on the Swedish east coast. It receives waste water from approximately 155 000 person equivalents (Per Nilsson, Norrköping Vatten AB (former Sydkraft Vatten AB), 30 March 2005, pers. comm.). Two types of sludges are produced in the treatment process. First primary sludge from the mechanical treatment and then activated sludge from the biological treatment.

Both the primary sludge and the thickened activated sludge is passed through a belt filter press to decrease the water content before being led into the digestion chambers. Two digestion chambers, with a volume of 2 000 m^3 each, produce approximately 4 500 m^3 of biogas per day. Methane content of the biogas is 62–64 %. The digestion chambers operates mesophilically at 37 ℃ with a retention time of about 18 days. The proportion of primary sludge/waste activated sludge is 70/30.

According to a report by Cenox (2003) on the gas production of primary and waste activated sludge from Slottshagen, the methane production was 350 mL CH_4/g VS (77 % CH_4) and 300 mL CH_4/g VS (83 % CH_4). Recalculated to gas production the values are 450 mL/g VS for primary sludge and 360 mL/g VS for waste activated sludge.

The digested sludge is led to a storage tank (volume 450 m^3), where degassing occurs. The retention time of the storage tank is 1–2 days and the emitted gas is not utilised. Afterwards the digested sludge is dewatered in centrifuges. An amount of 35 t (about 8 t TS) dewatered sludge is produced daily.

Slottshagen has an interest in increasing the gas production from the undigested sludge as well as decreasing the amount of digested sludge produced, because the biogas can be upgraded and sold as motor vehicle fuel and the digested sludge is a costly end product. There is also an interest in a more efficient and faster degassing of the digested sludge, because gas dissolved in the digested sludge makes the dewatering more difficult, thus yielding more sludge mass to dispose of. A faster degassing would make it easier to collect the gas and prevent it from escaping to the atmosphere.

Thus Slottshagen has incentives to investigate ultrasound as a way of increasing the biogas yield from the undigested sludge and as a way of getting a more efficient degassing of the digested sludge.

2.8 Ultrasound

The primary effect of ultrasound on a liquid (or sludge) is the formation of cavitation bubbles. The effects are either used as the destructive powers of the imploding bubbles or the degassing effect of bubbles rising to the surface.

Ultrasound has a frequency of 20 kHz and above, i.e. above the human audible range. A wave propagates in a liquid through alternating cycles of compression (high pressure) and rarefaction (low pressure). Above a certain intensity the attractive forces of the liquid can be overcome during rarefaction

and a small bubble is formed; a phenomenon called cavitation (Tiehm et al., 1997). The cavitation bubbles collapse within microseconds and give rise to strong hydromechanical shear forces (Tiehm et al., 2001). Upon collapse the local temperature and pressure rise to about 5000 K and several hundred bars. These extreme conditions lead to disintegration of material present in the liquid, e.g. cells and insoluble macromolecules.

Dissolved gas and other impurities in a liquid can act as nuclei for cavitation bubbles (Hueter and Bolt, 1962, p. 226). Such bubbles do not collapse as easily as bubbles formed without nuclei (Mason, 1990, p. 5). Instead they grow and fill with more dissolved gas from the surrounding liquid and eventually rise to the surface, finally giving rise to a degassing. Traditionally ultrasound has been used to degas a variety of water-based liquids and metal smelts. No references have been found regarding ultrasonic degassing of sludge.

3 Hypotheses and aim

3.1 Hypotheses

The two hypotheses of the thesis are:

1. Ultrasonic pretreatment of waste activated sludge increases the biogas yield from an anaerobic digestion process.
2. Ultrasonic treatment speeds up the degassing of digested sludge.

3.2 Aim

The aim is to confirm or to reject the hypotheses. To facilitate the task, the hypotheses are broken down into separate questions.

Anaerobic digestion of waste activated sludge treated with ultrasound

- Does treated waste activated sludge produce more gas than untreated?
- Is the methane content of the gas affected by the treatment?
- Does the filtrated COD (fCOD) of waste activated sludge increase after treatment?
- Are filamentous bacteria in the waste activated sludge affected by the treatment?
- Is the presence of filamentous bacteria in the reactor effluent affected by the treatment?
- Does the VS reduction increase?

Degassing of digested sludge

- How large is the degassing from digested sludge?
- Is the degassing affected by circulation of the digested sludge?
- Is the degassing affected by ultrasonic treatment?
- Does the ultrasonic treatment induce a post digestion of the digested sludge in the storage tank?

4 Materials and methods

4.1 Experimental design

To establish the effect of ultrasonic treatment on waste activated sludge a semi-continues digestion experiment was performed (scale 1:1 000 000). The main experiment was complimented with two sub-experiments, focusing on the release of filterable chemical oxygen demand (fCOD) and microscopic examination of sludge.

The effect of ultrasound on the degassing of digested sludge was examined on a larger scale (1:2 500). The degassing was measured with and without circulation as well as with ultrasonic treatment.

4.2 Digestion experiment

4.2.1 Experimental arrangement

Four reactors comprised the set-up. Each reactor had two openings: one small for feeding and withdrawal of sludge and one large plugged with a stopper. The stopper was equipped with two entrances: one for a propeller axis and one for a gas-outlet tube. On the tube there was a three-way valve for gas sampling. All four reactors were covered with black plastic to prevent light from entering the reactor and placed in a 37 ℃ water bath. Figure 2 shows the reactor design.

Two reactors were test reactors, named U1 and U2, receiving waste activated sludge treated with ultrasound. The other two were control reactors, named C1 and C2, receiving untreated sludge. Fresh sludge was taken each day prior to feeding and the waste activated sludge for the test reactors was ultrasonically treated. The digested sludge was taken alternately from digestion chamber one and two. The reactors were operated in a semi-continues mode with feeding once a day, six times per week (Monday–Saturday). On Saturdays the amount of sludge being fed to the reactors was doubled and all reactors received untreated waste activated sludge.

4.2.2 Sub-experiment 1: Filterable chemical oxygen demand (fCOD)

The concentration of filterable chemical oxygen demand (fCOD)[1] was used as a direct measurement of cell lysis. fCOD is defined as the COD of the remaining

[1] fCOD is sometimes called soluble chemical oxygen demand (sCOD). fCOD is more correct, since everything smaller than the pore width of the membrane filter is included in the measurement, i.e. soluble components and small particles alike.

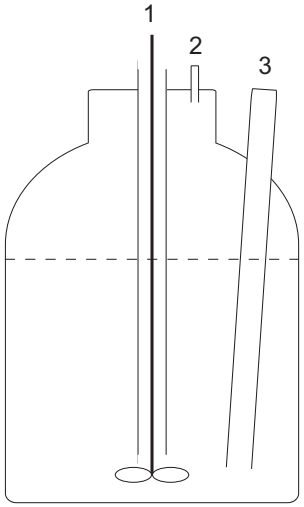

Figure 2: Illustration of a reactor used in the digestion experiment. (1) Stirrer, (2) tube leading gas to a gas meter, (3) opening used for feeding and withdrawal of sludge. The dashed line represents the height of the sludge.

filtrate after centrifugation and filtration of sludge. When cell walls are disintegrated due to ultrasonic cavitation, the material inside the cell is released into the reactor suspension. An increased fCOD after ultrasonic treatment of sludge is an indication of cell lysis.

fCOD of waste activated sludge was analysed five times, pre- and post-ultrasonic treatment. Once, double samples were used to measure the spread of the results. An analysis of total COD was made twice. Treatment lengths ranged from 45 s to 10 min. The fCOD samples were centrifuged at 1 200 rpm and the supernatant was filtered through a Dr Lange (Düsseldorf, Germany) membrane filter (LCW904) with a pore width of 1.2 µm. Two of the fCOD measurings were also accompanied by measurement of sludge temperature at different treatment lengths.

4.2.3 Sub-experiment 2: Microscopic sludge analysis

To see if filamentous bacteria were affected by ultrasonic treatment, samples of waste activated sludge with different treatment times were examined in a light microscope. Floc size and length of filaments were studied at 100 x magnification.

The reactor effluent was also studied to see if the prevalence of filamentous bacteria differed between the reactors receiving ultrasonically treated sludge and the control reactors.

Extended filament length, total filament abundance and floc firmness were studied in accordance with the active sludge microscopy manual of Dillner Westlund et al. (1996).

4.2.4 Materials and equipment

The reactors were of Duran® borosilicate glass, manufactured by Schott AG (Mainz, Germany) and had an approximative volume of 2 L. The stirrers, equipped with 4-bladed propellers, were of the model Eurostar Power Basic from IKA® (Staufen, Germany). Syringes and needles (Microlance™) used for gas and sludge sampling came from BD (Drogheda, Ireland). Rubber stoppers were used to plug the reactors.

4.2.5 Inoculum

The inoculum consisted of a mixture (1:1) of digested sludge from the two digestion chambers at Slottshagen sewage treatment plant. The sludge was taken fresh from the digestion chambers and subsequently poured into the reactors. Afterwards the reactors were immediately sealed, placed in the water bath and allowed to degas under stirring for 24 h.

4.2.6 Substrate

The substrate was partly waste activated sludge and partly digested sludge. Even though the full-scale digestion chambers at Slottshagen are run on a mixture of primary sludge and waste activated sludge, primary sludge was excluded in this experiment. Primary sludge varies heavily in both composition and quality and would have meant an unnecessary source of variation in gas production. Digested sludge as a part of the substrate ensures that the digestion process is not affected by lack of nutrients, which in the full-scale process are found in the primary sludge. It also assures the presence of an active microbial community like the one in the full-scale process. The organic matter (i.e. volatile solids (VS)) present in the digested sludge should not affect the biogas production to any considerable extent.

4.2.7 Method validation

During a start-up period of 61 days mainly two problems were dealt with: stability of the reactors and accuracy in gas-production measurements.

Stable reactors Due to foaming and occasional overflows three parameters were modified: (1) the retention time (T_R), (2) the proportion between waste activated sludge and digested sludge used for feeding and (3) the height of the propeller in the reactor. When an overflow took place, the reactor was opened and refilled with fresh digested sludge. An overview of T_R and sludge

Table 3: Retention times (T_R), proportion of waste activated sludge (WAS) and duration of sonication during the start-up period of the digestion experiment.

Day	T_R(days)[a]		WAS proportion (%)	Sonication time (mm:ss)
1	16.0	(22.9)	70.0	00:00
6	17.8	(25.4)	70.0	00:00
15	14.5	(16.0)	90.0	00:00
21	14.5	(16.0)	90.0	00:45
33	10.0	(16.0)	62.5	02:14

[a] The values in parenthesis are with regard to only the volume of the waste activated sludge. The values without parenthesis are with regard to the total amount of exchanged sludge (digested and waste activated sludge)

proportions is given in table 3. The different propeller heights are summarised in table 4

The retention time (T_R) is defined as the ratio between the total volume (V) and the volume of exchanged sludge per day (r):

$$T_R = \frac{V}{r}$$

The volume of exchanged sludge would traditionally be seen as the sum of the waste activated sludge volume and the digested sludge volume fed to the reactor. However, one could argue that only the volume of the waste activated sludge should be used. Since the digested sludge in the substrate is practically the same as the sludge withdrawn from the reactor it can be viewed only as background material being replaced. Both points of view are valid since we want to know how much sludge in fact is replaced and how much of it is waste activated sludge — the material of interest. During the first part of the start-up period retention times were looked upon from the viewpoint of the total volume. For the second part, they were seen from the viewpoint of only the waste activated sludge. Henceforth, when presenting a retention time the ditto with regard to only the waste activated sludge will be given in parenthesis.

Accuracy in gas-production measurements To increase the accuracy of measurement, two approaches were used: physical modification of the gas meters and increased gas production from increasing of the organic loading.

The first modification attempt was with pieces of polystyrene inserted into the gas meter to decrease the active volume. The second modification was made with solid plastic cylinders, which worked better than the polystyrene, and was used through out the experiment.

An alternative way to measure the gas production, as used by Tiehm et al. (2001), is gas sampling over water acidified to avoid CO_2-absorbation. This approach was tried but not used during the experiment.

Table 4: Propeller heights (measured from the bottom) in the reactors during the start-up period of the digestion experiment.

	Height (cm)		
Reactor	Day 1	Day 15	Day 19
C1	1.8	1.6	3.3[a]
C2	3.9	1.6	3.3
U1	3.8	1.6	3.3
U2	1.6	1.6	3.3

[a] At a height of 1/3 of the reactor

Start-up period The reactors were inoculated on 2004–09–22 and the first feeding took place the following day (day 1). At the beginning of the start-up period T_R was 16 (22.9) days and the proportion of waste activated sludge in the substrate was 70 %. All reactors were fed untreated sludge. On day 6 T_R was slightly increased to 17.8 (25.4) to better agree with the full-scale process. In the second week it was noted that the waste activated sludge was unusually wet (total solids (TS) $\leq$ 1 %). The sludge was in fact lacking addition of polymer; explaining the low TS. After a couple of days polymer usage was resumed in the sludge thickening process and at the beginning of the third week the sludge had a TS of $\sim$ 4 %. On day 15 T_R was decreased to 14.5 (16) days and the proportion of waste activated sludge was increased to 90 % in an effort to get a higher gas production. It was also noted that the stirring propellers were not all on the same height so they were adjusted accordingly (lowered). Day 19 all the propellers were raised to a new height of 1/3 of the sludge height. On day 21 the test reactors started receiving ultrasonically treated sludge. The test reactors were chosen by a drawing of lots. The treatment time was 45 s (after which 55 % of the sludge had been treated at least once). On day 33 the ultrasonic treatment time was increased to 2 min and 14 s (corresponding to three retention times in the ultrasonic treatment equipment, or 91 % of the sludge being treated at least once). Since there was still problems with foaming the volume of digested sludge in the substrate was increased to make sure a sufficient amount of (active) microorganisms were present. T_R was lowered to 10 (16) days and the proportion of waste activated sludge was decreased to 62.5 %. This was a suitable combination of T_R and sludge proportions, which were maintained further on. On day 61 all reactors had gas meters measuring at a sufficient resolution and the experimental period could begin. The gas meters were interchanged between the reactors to verify that they were calibrated correctly.

4.2.8 Experimental run

T_R was 10 (16) days and the proportion of waste activated sludge was 62.5 %. At the second day of the 16-day experimental period the ultrasonic treatment time was increased to 6 min, raising the possibility of getting a difference in gas production more easy to measure. The test reactors received treated sludge for twelve days. During the last three days all reactors were fed untreated sludge.

4.2.9 Sampling and analysis

Gas production was measured by gas meters from TuTech Hamburg-Harburg Technical University (Germany), which were modified as described in section 4.2.7. The Gas meters were calibrated overnight with a known gas flow, approximately 3.5 mL/min. Prior to feeding (i.e. six times a week) the gas meters were read and after feeding they were set to zero.

Methane was sampled once a week from the reactors and analysed on a Chrompack CP9001 gas chromatograph with a flame ionization detector (FID) and a Hayesep T column (80/100 mesh). Nitrogen was used as carrier gas at a flow of 30 mL/min. The injector and detector temperature was 150 ℃ and the oven temperature 125 ℃. The detector outputs were quantitated using a Perkin-Elmer LCI-100 integrator. The sample areas were compared to a standard area. Appendix B describes the analysis and following calculations in detail.

COD was analysed using a Dr Lange cuvette test (LCK114) and a Dr Lange Xion 500 spectrophotometer. Samples were heated in a Dr Lange dry thermostat LT100. fCOD samples were centrifuged at 1200 rpm and the supernatant was filtered through a Dr Lange membrane filter (LCW904) with a pore width of 1.2 µm. fCOD samples were diluted five or ten times. Samples analysed for total COD were diluted 500 times.

Temperature of the ultrasonically treated waste activated sludge was measured with a standard liquid-in-glass thermometer.

TS was analysed according to Swedish standard SS 028113-1. The reactor effluents were analysed twice a week. Collective samples of the waste activated sludge was analysed weekly.

VS was analysed according to Swedish standard SS 028113-1. The reactor effluents were analysed twice a week. Collective samples of the waste activated sludge was analysed weekly.

pH was analysed on a InoLab Level 2 pH-meter from WTW (Weilheim, Germany) according to Swedish standard SS 028122-2. pH standards from Merck KGaA (Darmstadt, Germany) was used for calibration. The reactor effluents were analysed twice a week

Organic acids (fatty acids) were analysed with a Dr Lange cuvette test (LCK365). The test gives a total measure of all organic acids expressed in mg/L acetic acid. Heating and spectrophotometric measurement were done as in the COD analysis. The reactor effluents were analysed once a week.

4.2.10 Statistic test

The Wilcoxon Signed Rank test, a non-parametric test, was used to calculate a confidence interval and an estimated median for the increase in gas production. The procedure in full is described in Appendix F. Minitab® 14.13 was used for the statistical analysis.

Figure 3: Barrel with pump, gas meter (background) and ultrasonic equipment (right) used in the degassing experiment.

4.3 Degassing experiment

4.3.1 Experimental arrangement

Three barrels were used for the experiment. One barrel without any kind of circulation at all — a control. Two barrels with circulation, of which one had the option of ultrasonic treatment. The barrel with ultrasonic equipment is shown in figure 3. The barrels were filled with 176 L digested sludge through an inlet near the bottom. Near the top a hole, at the height corresponding to a volume of 176 L, permitted outflow of gas as the barrel was filled. When

filled, the hole was plugged. The emitted gas was led from the top of the barrel through a hose to a gas meter.

The sludge was treated with ultrasound for 53 min, in intervals of 3 min with 1.5 min breaks in between, to prevent overheating of the sonicator. Thus, the effective treatment time was 36 min. The treatment began 17 min after the barrel had been filled. An effective treatment time of 36 min means that approximately 76 % of the sludge was treated at least once. The trial went on for 50 h and gas measurement data was collected during three periods. Gas emission was the only parameter measured.

4.3.2 Materials and equipment

The barrels were of plastic and were fitted with necessary valves to permit pumping and filling of sludge. The pumps were identical and were set to approximately 7 L/min. The gas meters were the same as in the digestion experiment (see section 4.2.9 for description). First they were used with the same modification as in the digestion experiment, but the modification had rendered them too sensitive for a gas flow of this magnitude. For the rest of the experiment the gas meters were returned to their original state.

4.4 The ultrasonic equipment

The ultrasonic equipment came from Ultra Sonus AB (Uppsala, Sweden). It had an effect of 420 W and treated 5 L sludge. The set-up used in the digestion experiment is depicted in figure 4.

Sludge is poured into the container on the left. It is pumped through the lower tube to the sonicator, where the ultrasonic treatment takes place, on the right. Then the sludge is returned to the container through the upper tube. The pumping speed, when the TS of the sludge was 3.3 %, was 6.7 L/min.

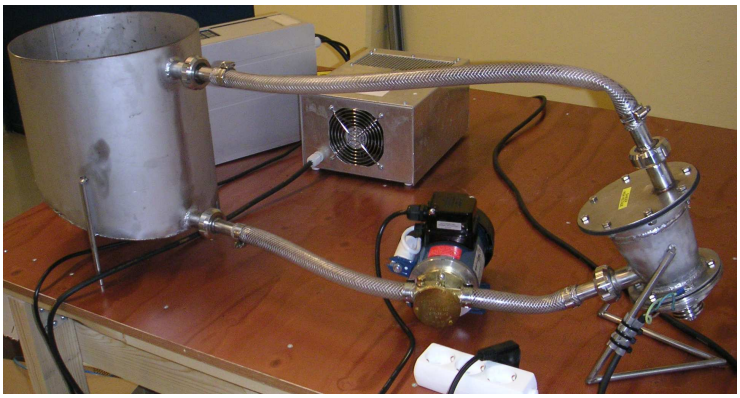

Figure 4: The ultrasonic equipment as used in the digestion experiment.

5 Results and discussion

5.1 Digestion experiment

All the raw data from the experiments are listed in Appendix A.

5.1.1 Inoculum

The digested sludge from digestion chambers one and two, comprising the inoculum, had total solids (TS), volatile solids (VS) and pH according to table 5.

Table 5: Total solids (TS), volatile solids (VS) and pH of the two sludges making up the inocula.

Digestion chamber	TS (%)	VS (%)	pH
1	3.3	62	7.4
2	3.1	63	7.5

5.1.2 Substrate

During the experimental period the TS and VS of the waste activated sludge were in the range of 2.8–3.8 % and 74–76 % with mean values of 3.5 % and 76 %, respectively. These TS and VS values gave a mean organic load of 1.7 g VS $L^{-1}d^{-1}$ for the experiment period.

TS and VS of both the start-up and experimental period are graphically represented in figure 5.

5.1.3 Method validation

Stable reactors For the first part of the start-up period foaming usually occurred several times a week and occasionally there was an overflow. The reactors overflow was due to a combination of frequent foaming and the fact that the gas-outlet tubes were too long inside the reactor. This led to that relatively thin layers of foam could plug the tube; gas could not escape the reactor; pressure was built up and sludge flowed out through the stirring hole. When an overflowing reactor was opened and refilled with fresh digested sludge the tube was shortened as to prevent future foam plugging.

The length of the pipe, where the propeller enters the reactor was also unnecessary long in two of the reactors: C1 and U2. On day 15, this led to that the propellers were adjusted to a height too low to give sufficient mixing. When discovered, due to excess foaming, the pipes were shortened on day 19 in the two

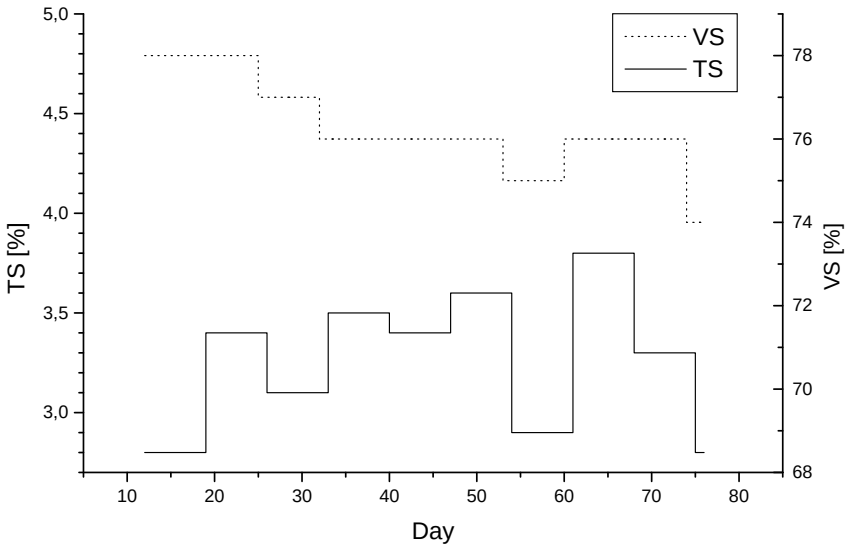

Figure 5: Total solids (TS) and volatile solids (VS) of weekly collective samples of waste activated sludge during the start-up and experimental periods. The experiment period covers day 61–76.

affected reactors and all propellers were placed at a height of 1/3 of the sludge volume. As a result the foaming tendency was abated.

Furthermore, different combinations of retention time (T_R) and proportion between waste activated sludge and digested sludge was investigated in an effort to minimise foaming. The best reactor performance was achieved with a T_R of 10 days with regard to the combined volume of waste activated sludge and digested sludge, equivalent to a T_R of 16 days with regard to only the volume of the waste activated sludge. The best proportion of waste activated sludge was found to be 63 %.

Accuracy in gas-production measurements The resolution of the measurements from the gas meters in their original design were in the range of 36–61 mL. With a daily gas production of 500–900 mL the smallest measurable unit then represents 4–12 %. The first physical modification with pieces of polystyrene to decrease the active volume was not successful. An increased resolution (15–28 mL) was achieved, but the measurements became very fluctuating. This was probably due to the porous and soft structure of polystyrene making the measuring unreliable. The second modification with solid plastic cylinders worked much better. The resolution was increased to 23–28 mL, a satisfactory improvement and the stability of the original gas-meter design was

obtained. The decreased T_R leading to an increase of organic loading and daily gas production also improved the accuracy of the gas-production measurements.

The gas-measuring construction adopted from Tiehm et al. (2001), with gas sampling over water acidified to avoid CO_2-absorbation, was not used during the experiment due to several uncertainties regarding its accuracy. First of all, it was noted that acid often was pressed into the tube connecting the reactor and the cylinder. A similar behaviour is expected just after the the pressure inside the reactor has been lowered, e.g. after feeding. It was never near of reaching the reactor but even a long time after feeding, when there should be an overpressure inside the reactor, the tube contained acid to some extent. Secondly, gas — presumably CO_2 — was dissolved in the acid despite the low pH. For example, when the cylinder was undisturbed for 24 h the surface rose the equivalent of 30 mL. After 48 h the surface had risen the equivalent of 40 mL. Thus, the gas meters modified with plastic cylinders most likely gave a result better suited for comparisons.

Two shorter treatment times (45 s and 2 min 14 s) were used during the start-up period. The effect on gas production from these treatment times are inconclusive, due to the problem with gas measurings discussed above.

5.1.4 Experimental run

Figure 6 shows the biogas yield over the experimental period. During day 1–5 there was a general increase in gas production and the increase seemed to be stronger for the two reactors receiving sludge treated with ultrasound for 6 min. For day 7–12 the difference in gas yield did not increase further. During day 14–16, when all reactors received untreated sludge, the difference in gas yield disappeared — a confirmation that the deviation in gas yield was due to the ultrasonic treatment. The missing data for reactor C2 on day 9 was because of a faulty gas meter.

The gas yield for the control reactors, a mean of 293 mL/g VS for day 7–12, was in the lower range of the reference values cited by Brown et al. (2003) and lower than the value presented in the report on Slottshagen sludge from Cenox (2003). Still, the values are in the same range, confirming that the gas measurings are correct.

The difference in gas yield for day 7–12 is statistical significant at a confidence level of 94.1 %. A Wilcoxon Signed Rank test for the difference gave a confidence interval of 28.7–48.5 mL/g VS, corresponding to an increase of 9.8–16.5 % compared to a control mean. The estimated median of the difference is 37.6 mL/g VS corresponding to an increase of 12.8 % compared to a control mean.

It should be noted that the gas production is in most likely an underestimate. This is because the gas flow used to calibrate the gas meters ($\sim$ 3.5 mL/min) is higher than the actual measured gas flow ($\sim$ 0.5 mL/min). A lower gas flow for calibration could not be attained, thus this possible source of error could not be avoided.

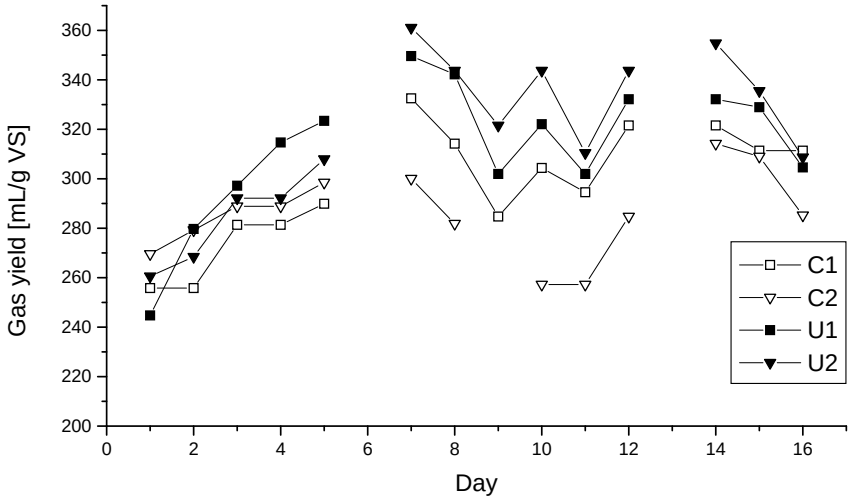

Figure 6: Biogas yield under the experimental period. Control reactors C1 and C2 have received untreated sludge. Test reactors U1 and U2 have received sludge treated with ultrasound for 6 min.

The methane content of the biogas differed only slightly between the test and the control reactors. It varied from 56.5 % to 59.7 % for the control reactors and between 57.7 % and 59.8 % for the test reactors. The small differences could depend on that the reactors are more or less difficult to feed or to plain statistical variation.

The pH was neutral through out the experiment for both test (pH of 7.3–7.7) and control (pH of 7.4–7.6) reactors. Neutral pH values correspond well with the low, <100 mg/L, concentrations of organic acids. From the neutral pH and the low concentrations of organic acids it can be concluded that the reactors were not overloaded.

TS of the reactor effluents was fairly constant (at about 2.5 %) over the experiment and equal among the reactors. There was a minor general decrease of VS in the reactor effluent, which shows that there was no build up of undegraded organic material in the reactors. However, to be able to draw further conclusions from the decrease in VS a longer experiment is required. Graphs of TS and VS of the reactor effluents are shown in figure 7.

There was no difference in VS reduction between the test reactors and the control reactors. VS reduction for C1, C2, U1 and U2 was 31, 33, 31 and 33 % respectively. The increase in gas production of 12.8 %, in this case, corresponds to approximately 0.1 g more VS being degraded per day (for calculation see

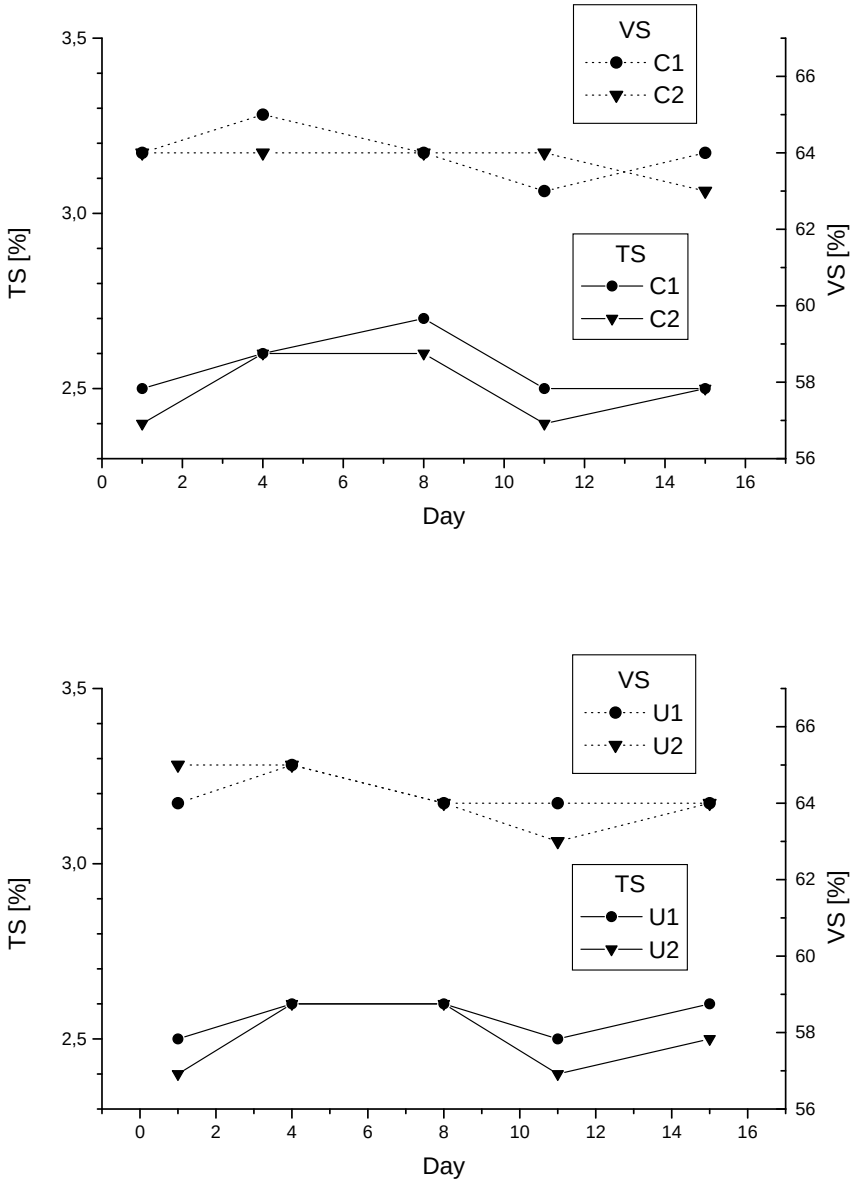

Figure 7: Total solids (TS) and volatile solids (VS) for the reactor effluents of the digestion experiment. The upper graph shows the control reactors (C1 and C2) and the lower graph shows the test reactors (U1 and U2).

Appendix C). Thus, no detectable difference in VS reduction is expected, since an increase of 0.1 g VS being degraded is rather difficult to measure. This experiment, with its high throughput of organic matter, was designed primarily for the study of gas production. For a better study of VS reduction, a longer experiment is needed, and preferable with a longer retention time.

5.1.5 Sub-experiment 1: Filterable chemical oxygen demand (fCOD)

Three out of five fCOD trials were successful. One trial probably failed owing to too short sonication times or problems with the COD analysis. The other unsuccessful trial was made with a sludge with a low TS (2.5 %). The three successful trials are shown in figure 8. For the trial where double samples were used, the sample standard deviation was low, in all cases <0.7 % of the sample mean.

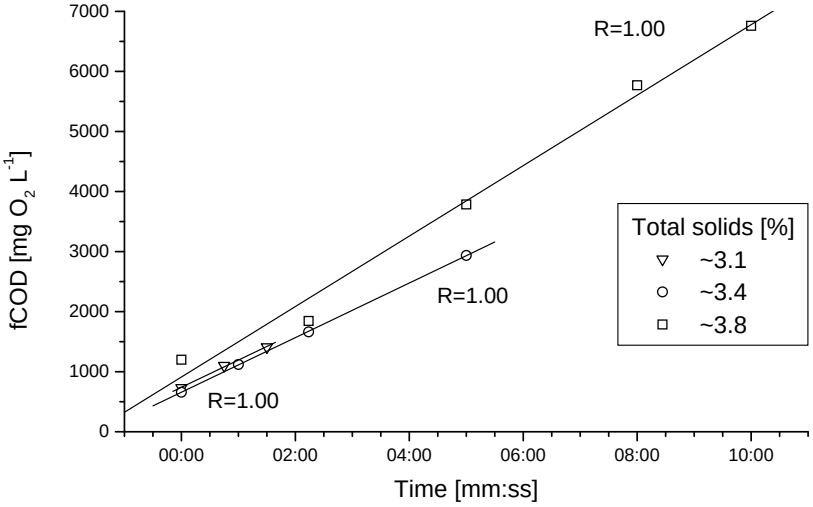

Figure 8: Filterable chemical oxygen demand (fCOD) of waste activated sludge after ultrasonic treatment.

There was a clear and linear increase of fCOD with increasing treatment time. Even when the treatment time reaches 10 min no attenuation of the increase was seen. A 6 min treatment, as used in the digestion experiment, of a sludge with an approximate TS of 3.8 % (figure 8) raised the fCOD to 375 % of the untreated sludge. If fCOD is expressed as a percentage of the total COD, a rise from 2.8 % to 10.6 % resulted from the 6 min treatment.

The enhanced fCOD values are distinctly visible to the naked eye. Filtrates from sludges with longer treatment times are much darker in colour, due the elevated concentrations of organic material. This is illustrated in figure 9.

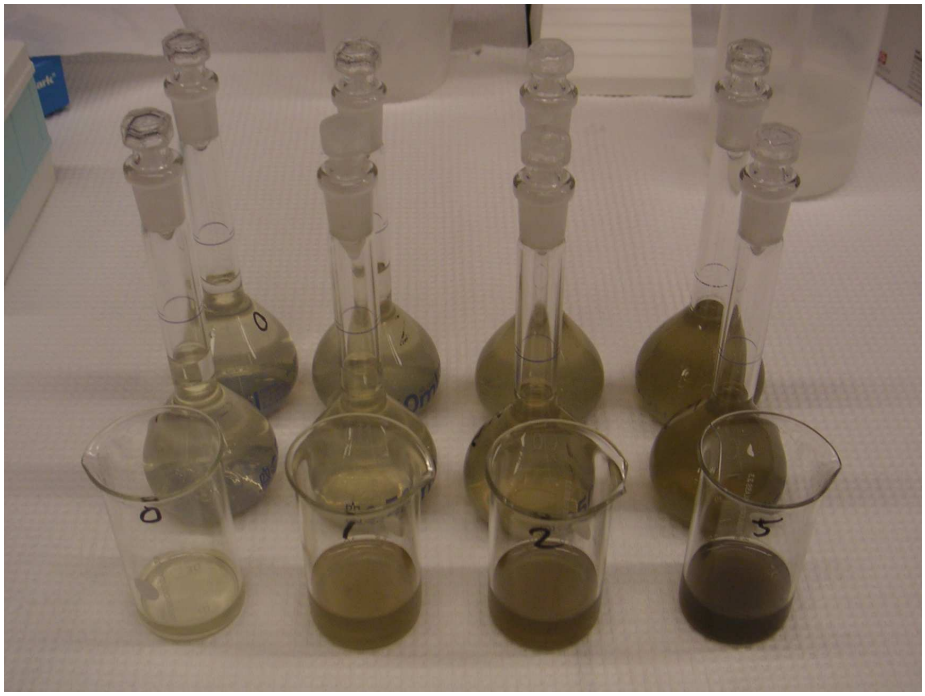

Figure 9: Filtrates of waste activated sludge for fCOD analysis. From left to right, samples are treated 0, 1, 2.34 and 5 min, respectively.

Note that cell debris larger than 1.2 µm are not included in the fCOD numbers even though they are available for methane production. Thus, more organic matter is available for anaerobic digestion than the fCOD values show. Nevertheless, fCOD is a good indicator of the sludge disintegration, especially together with a digestion experiment.

The temperature of the sludge also rose with increased treatment time. Though not as linearly, probably because heat was transfered to the cold equipment. After 2 min and 14 s the temperature had risen from 12 ℃ to 13.5 ℃, but after 10 min the temperature had reached 25.5 ℃.

5.1.6 Sub-experiment 2: Microscopic sludge analysis

The microscopic sludge examination showed that the flocs of the waste activated sludge exhibit signs of disintegration even at low sonication times like 45 s. At sonication times of 2 min the flocs were disintegrated to some extent, but the filaments showed no signs of effect. There were fewer areas with a high degree of

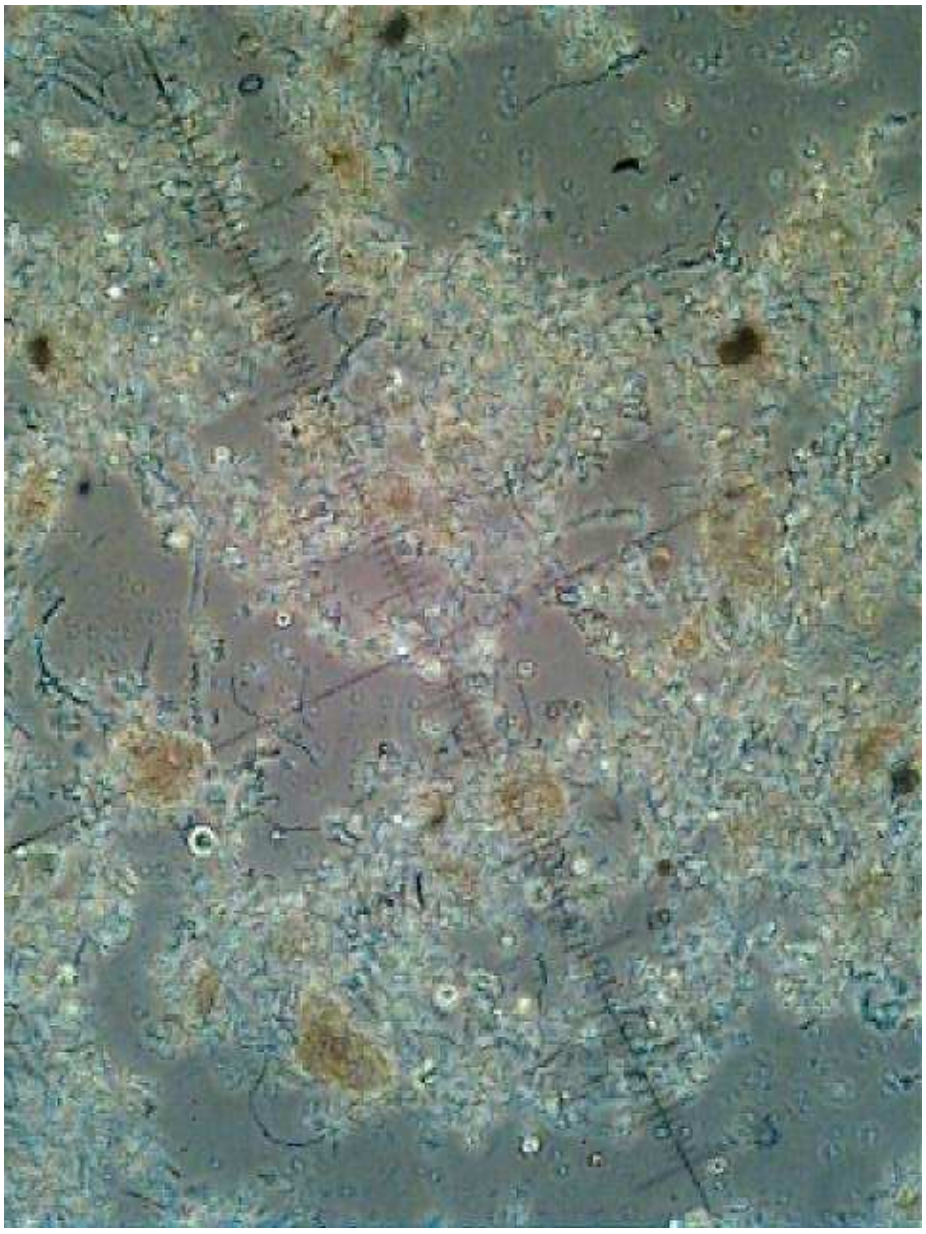

Figure 10: Untreated waste activated sludge at 50 x magnification. Mixture of 20 mL sludge and 60 mL water.

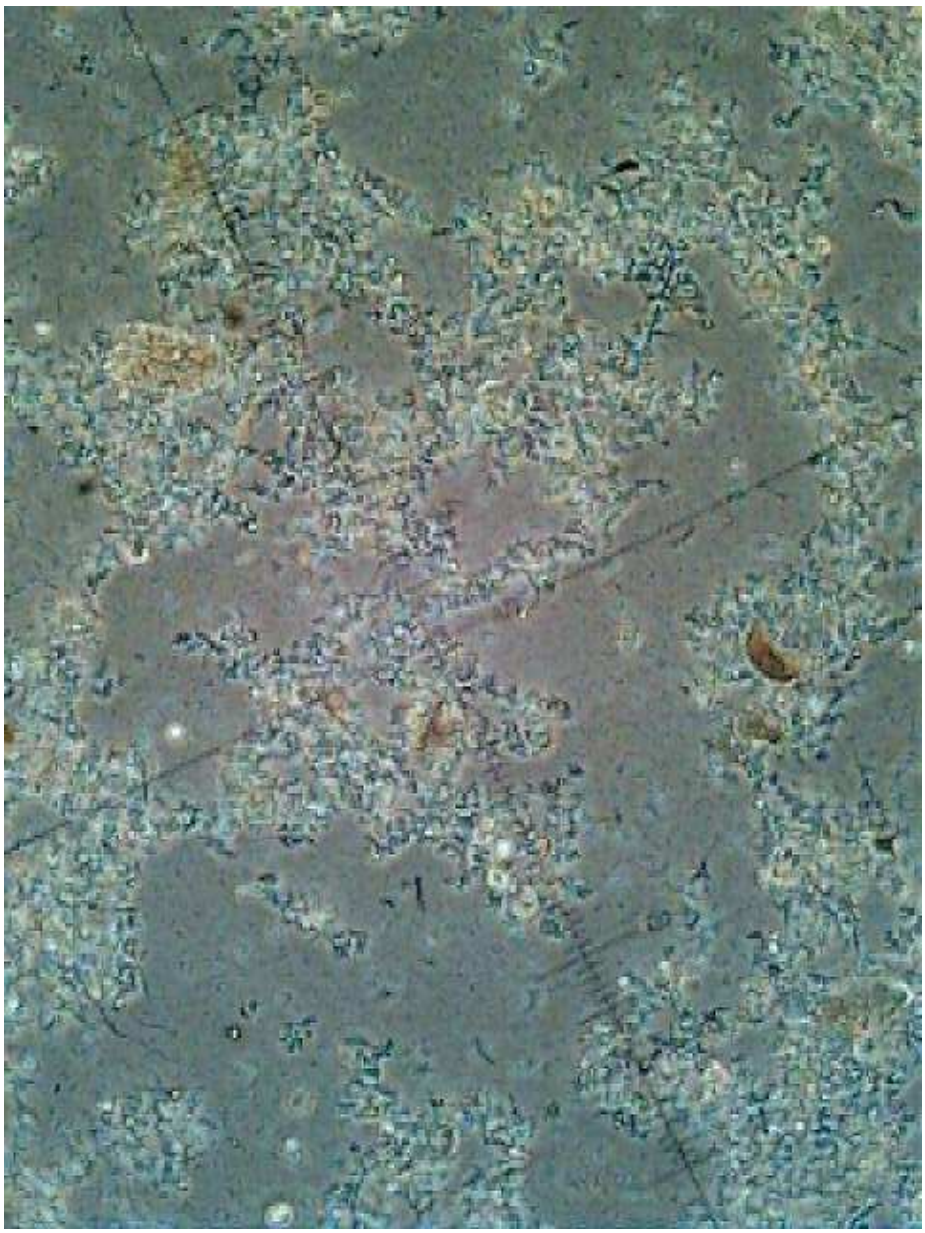

Figure 11: Waste activated sludge treated with ultrasound for 10 min (14 kWh/m^3), at 50 x magnification. Mixture of 20 mL sludge and 60 mL water. Compared with the untreated sludge flocs are disintegrated, filaments have been shortened and areas with a high degree of compactness are fewer.

compactness. A longer sonication time of 6 min (8.4 kWh/m^3) started to have a shortening effect on the protruding filaments (extended filament length) and the areas with a high degree of compactness were smaller and fewer. After 10 min (14 kWh/m^3) the flocs were clearly disintegrated and all filaments were shorter. Areas with a high degree of compactness were almost not found. Figure 10 and 11 show the untreated sludge and the sludge after 10 min of ultrasonic treatment respectively.

When handling and pouring the treated waste activated sludge it was clearly noticed that the viscosity decreases with increased treatment time. Thus, the ultrasonic treatment start to break up the floc structure of waste activated sludge relatively quick. To shorten the filaments takes longer, six minutes or more with the equipment used in this study. However, it should be noted that the occurrence of filamentous bacteria in the sludge was not extremely high to start with. Slottshagen sewage treatment plant did not have any foam-related problems through out the experiment. A more prominent effect might have been seen in a sludge with more and longer filaments.

The study of the reactor effluents showed no difference in the prevalence of filamentous bacteria between the reactors receiving ultrasonically treated sludge and the reactors receiving untreated sludge. Both the control and the test reactors had low occurrences of filamentous bacteria. The full result from the microscopic sludge analysis can be found in table 11 and table 12 in Appendix E.

5.1.7 Energy balance

Table 6 illustrates the results from an energy balance calculation with different TS and upper, lower limits of the confidence interval and estimated median of the increase in gas production. The complete calculation is given in Appendix D.

Table 6: Result of energy balance calculation. With lower (9.8 %), upper limits (16.5 %) and estimated median (12.8 %) of the increase in gas production, at different total solids (TS) and a volatile solids (VS) of 75.8 %.

	Energy gain (kWh/m^3)		
	Increase in gas production (%)		
TS (%)	9.8	12.8	16.5
3.5	−4.0	−2.7	−1.0
5.1	−1.9	0.0	2.5
6.0	−0.8	1.5	4.4
7.0	0.5	3.2	6.5

With a TS of 3.5 % there is a loss of energy because more energy is needed for the ultrasonic treatment than the potential energy from the increase in gas production. But if the TS is increased, the same amount of energy is used to treat more material. Break even (from the view point of the estimated median)

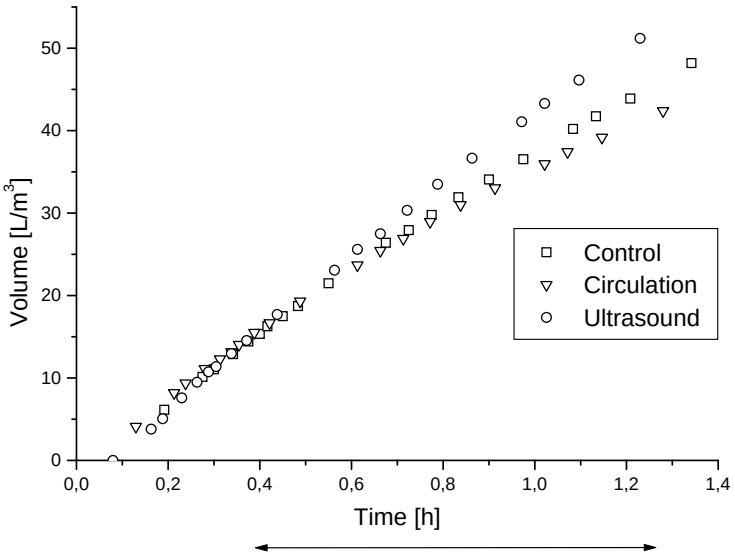

Figure 12: Degassing of digested sludge during 1.4 h. The arrow represents when the ultrasound was on.

is reached with a TS of 5.1 %. If the TS of the experiment is doubled to 7.0 %, the energy gain is 3.2 kWh/m^3 waste activated sludge.

5.2 Degassing experiment

The first trial failed because the modified gas meters were too sensitive and could not keep up with the gas flow. The second trial was successful. When the results were to be verified in a third trial there was a problem with the filling of the barrels and the trial could not be completed.

Figure 12 shows the degassing during the first 1.4 h. The arrow in figure 12 illustrates when the ultrasound was on. As seen in figure 12, more gas does leave the sludge treated with ultrasound during the first one and a half hours, but the effect is not prominent. The difference start to show after about 0.64 h, which equals that approximately 35 % of the sludge has been treated at least once. The trial needs to be repeated to verify, if the difference is a direct consequence of ultrasonic degassing or not. But clearly there is no burst of gas from the sludge as a result of the ultrasound.

Figure 13 shows the gas emissions during the whole trial. After 22 h, during the second measurement period it is clear that more gas has been emitted from the sludge treated with ultrasound compared to the two others. This is probably due to a post digestion induced by the ultrasonic treatment. Thus, some part of

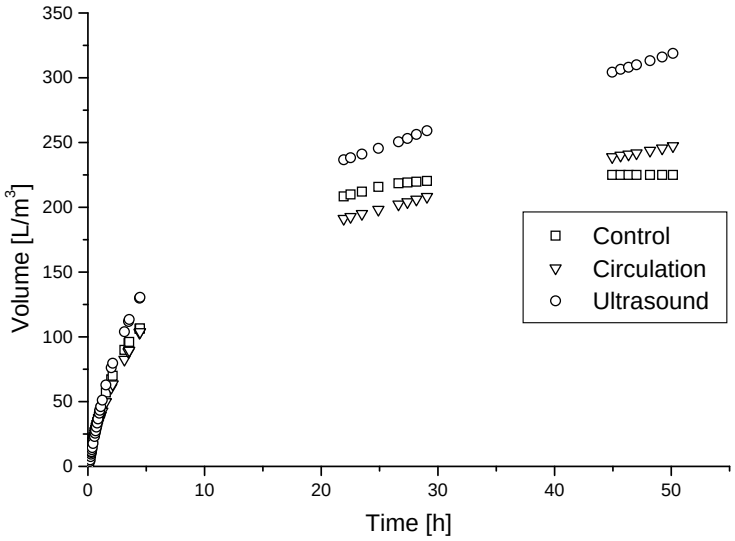

Figure 13: Degassing of digested sludge during 2 d.

the difference in emitted gas is presumably because of newly produced gas. It is somewhat surprising that up to and including measurement period two, more gas has left the barrel without circulation than the one with circulation. However, the difference is not that large and could result from differences between the gas-meter readings.

By the time of measurement during period three, after 45 h, gas has stopped flowing from the barrel without circulation and the total amount sums up to 225 L/m^3 sludge. The barrel with circulation continued to emit gas over measurement period three, but in a rather slow pace and after 50 h a total of 247 L/m^3 sludge had been emitted. Gas also continued to flow from the barrel with ultrasound and at a higher pace. After 50 h, 319 L/m^3 sludge has left sludge treated with ultrasound.

One obvious source of error in these measurements is that the degassing that occurs during the filling of the barrel, before the barrel is plugged, was not measured. It takes less then three minutes to fill and plug a barrel.

Three likely conclusions drawn from the experiment are:

- Gas emission from digested sludge during 48 h (usually the maximum retention time in the storage tank) is about 235 L (m^3 sludge)$^{-1}$.
- About 80 L (m^3 sludge)$^{-1}$ more gas is emitted from the sonicated sludge during 48 h.

- Ultrasonic treatment induces a post digestion of the sludge.

5.2.1 Energy balance

The energy input after 36 min effective treatment time is 1.4 kWh/m^3. If the methane content is assumed to be 60 % and the same potential energy of methane, as in Appendix D is used, the difference in gas emission equals 0.48 kWh/m^3. Thus, more energy is used in the treatment than what is potentially gained in increased gas emissions.

5.2.2 Slottshagen sewage treatment plant

The storage tank for digested sludge at Slottshagen has a volume of 450 m^3 and a retention time of 1–2 d. If the two-day retention time is used, a gas emission rate of 235 L gas/m^3 sludge for two days, equals a total amount of

$$450\,\mathrm{m^3\,sludge} \cdot 235\,\mathrm{L\,gas/m^3\,sludge} = 105\,750\,\mathrm{L\,gas} \approx 100\,\mathrm{m^3\,gas}$$

That is about 50 m^3/day, corresponding to 1.18 % of a daily gas production of 4500 m^3. 1.18 % is in the same order as the 1.75 % estimate of methane loss from sludge tanks at the Bromma sewage treatment plant (Starberg and Welin, 2004).

5.3 Concluding discussion

The increase of treatment time from 45 s to 2 min 14 s and finally to 6 min was probably necessary to reach a conclusive estimate of the increase in gas production, with the material and equipment at hand. Even after successful modification of the gas meters, it is doubtful that a gas-production increase originating from one of the shorter treatment times could have been measured with desired accuracy.

For the few references from which the energy inputs can be calculated, they are generally higher (>100 kWh/m^3) than in this study. Chu et al. (2001) saw an increase of fCOD from 0.5 % to 20 % of total COD, with an energy input of 660 kWh/m^3. In the present study, an energy input of 8.4 kWh/m^3 raised the fCOD 375 %, which is similar to the study by Lafitte-Trouqué and Forster (2002), where the energy input and fCOD increase was 12 kWh/m^3 and 354 % respectively. Other comparisons, e.g. of increased gas production, are difficult to do due to unclear presentation of parameters in other studies.

A suitable way to continue the work on ultrasonic pretreatment of waste activated sludge, would be to perform digestion experiments on a thicker sludge, i.e. TS of 5–7 %. This would verify the estimated energy gain from the calculation of the energy balance. Preferably, the experiment would be performed on a larger scale than in this experiment, which would make it easier to get reliable gas measurements.

Since only one experiment of degassing digested sludge was successful, the experiment needs to be repeated with interchanged gas meters to verify the results and to exclude calibration errors. Also, gas samples need to be analysed to determine the methane content, because it is the loss of methane that is most important — both economically and environmentally. If the results acquired are correct, ultrasound is probably not the best way to chose to degas digested sludge. But since a post-digestion most likely is induced in the digested sludge, it is evident that ultrasound can increase the amount of digestible material in the sludge. Perhaps it would be better to treat a sub flow of digested sludge with ultrasound and then lead it back into the digestion chamber. In this way the post-digestion effects would be utilised inside the digestion chamber.

6 Conclusion

The questions from section 3.2 are answered as follows.

Anaerobic digestion of waste activated sludge treated with ultrasound

- Treated waste activated sludge produce more gas. The gas yield is increased by 13 % at an energy input of 8.4 kWh/m^3.
- The methane content of the gas is not affected by treatment, but a longer study is needed for conclusive results.
- The filtrated chemical oxygen demand (fCOD) is increased by the treatment. An energy input of 8.4 kWh/m^3 increased the fCOD by 375 % compared to untreated sludge, or from 2.8 % to 16 % of total COD.
- The filamentous bacteria are affected by treatment, but a relative long treatment time (6–10 min, i.e. 8.4–14 kWh/m^3) is needed to shorten the filaments.
- There was no difference in the occurrence of filaments between ultrasonically treated or control reactors. However, the occurrence of filaments was generally low in all the reactor effluents.
- There seemed to be no increase in VS reduction, but the parameter needs to be studied over a longer period to verify the observation.

Degassing of digested sludge

- The degassing of digested sludge is at least 115 L/m^3 sludge and day
- There seems to be a small effect on degassing from circulation, but the trial needs to be repeated for verification.
- There seems to be no prominent direct degassing due to ultrasonic treatment, but the trial needs to be repeated for verification.
- Most likely the ultrasonic treatment induces a post digestion in the digested sludge.

Thus, hypothesis one has been confirmed; ultrasonic pretreatment does increase the biogas yield of waste activated sludge. To confirm or reject hypothesis two — that ultrasound speeds up the degassing of digested sludge — more work needs to be done.

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Appendices

Appendix A
Raw data

Raw data from the digestion experiment is shown in table 7, 8 and 9. Raw data from the degassing experiment is shown in table 10.

Table 7: Total solids (TS) and volatile solids (VS) of digested sludge used as substrate.

	Digestion chamber 1		Digestion chamber 2	
Day	TS (%)	VS (%)[a]	TS (%)	VS (%)[a]
2	3.1	63	3.2	64
8	3.1	63	3.1	62
16	3.3	63	3.4	63

[a] Day 16 is a mean of VS measurements from day 2 and 8

Table 8: Total solids (TS), volatile solids (VS), total chemical oxygen demand (total COD) and concentrations of filterable chemical oxygen demand (fCOD) of waste activated sludge from sub-experiment 1.

			fCOD (mg O_2/L)							
TS	VS	Total COD	Ultrasonic treatment time (mm:ss)							
(%)	(%)	(mg O_2/L)	00:00	00:45	01:00	01:30	02:14	05:00	08:00	10:00
3.1	77		730	1110		1410				
3.4	76		658		1120		1660	2940		
3.8	76	42 400	1200				1850	3790	5770	6760

Table 9: Gas production, methane content and pH, total solids (TS), volatile solids (VS) and organic acids of reactor effluents from the experimental period.

		Day													
	Reactor	1	2	3	4	5	7	8	9	10	11	12	14	15	16
Gas production	C1	256	256	281	281	290	333	314	285	304	295	322	322	311	311
(mL/g VS)	C2	270	279	289	289	298	300	282		257	257	285	314	309	285
	U1	245	280	297	315	323	350	342	302	322	302	332	332	329	305
	U2	261	268	292	292	308	361	344	322	344	310	344	355	336	309
Methane content	C1			57						57					
(%)	C2			60						56					
	U1			60						59					
	U2			58						58					
pH	C1	7.4			7.4			7.6			7.4				
	C2	7.4			7.4			7.6			7.4				
	U1	7.4			7.3			7.7			7.4				
	U2	7.4			7.4			7.6			7.4				
TS	C1	2.5			2.6			2.7			2.5			2.5	
(%)	C2	2.4			2.6			2.6			2.4			2.5	
	U1	2.5			2.6			2.6			2.5			2.6	
	U2	2.4			2.6			2.6			2.4			2.5	
VS	C1	64			65			64			63			64	
(%)	C2	64			64			64			64			63	
	U1	64			65			64			64			64	
	U2	65			65			64			63			64	
Organic acids	C1		51						62						
(mg/L acetic acid)	C2		<50						<50						
	U1		<50						78						
	U2		<50						92						

Table 10: Gas volume emitted from untreated (control), circulated and ultrasonically treated digested sludge.

Control				Circulation				Ultrasound			
Time (h)	Gas volume (L/m^3 sludge)	Time (h)	Gas volume (L/m^3 sludge)	Time (h)	Gas volume (L/m^3 sludge)	Time (h)	Gas volume (L/m^3 sludge)	Time (h)	Gas volume (L/m^3 sludge)	Time (h)	Gas volume (L/m^3 sludge)
0.19	6.14	2.23	70.0	0.13	4.09	2.17	63.4	0.08	0	2.12	79.6
0.28	10.1	3.24	90.0	0.21	8.18	3.18	82.4	0.16	3.79	3.13	104
0.30	11.1	3.59	95.2	0.24	9.35	3.53	88.3	0.19	5.06	3.48	112
0.34	12.9	3.65	96.1	0.28	11.1	3.59	89.4	0.23	7.58	3.54	113
0.38	14.4	4.53	106	0.31	12.3	4.47	103	0.26	9.48	4.42	130
0.40	15.3	4.58	107	0.34	13.2	4.51	104	0.29	10.7	4.46	131
0.42	16.3	22.03	208	0.35	14.0	21.97	191	0.30	11.4	21.92	237
0.45	17.5	22.63	209	0.39	15.5	22.56	193	0.34	13.0	22.51	238
0.48	18.7	23.60	212	0.42	16.7	23.54	195	0.37	14.54	23.49	241
0.55	21.5	25.01	216	0.49	19.3	24.95	198	0.44	17.7	24.90	246
0.68	26.4	26.73	216	0.61	23.7	26.67	202	0.56	23.1	26.62	251
0.73	27.9	27.50	219	0.66	25.4	27.44	204	0.61	25.6	27.39	253
0.78	29.8	28.27	220	0.71	26.9	28.20	206	0.66	27.5	28.15	256
0.83	31.9	29.18	220	0.77	28.9	29.11	208	0.72	30.3	29.06	259
0.90	34.1	45.05	225	0.84	31.0	44.99	239	0.79	33.5	44.94	304
0.98	36.5	45.77	225	0.91	33.0	45.70	240	0.86	36.7	45.65	307
1.08	40.2	46.45	225	1.02	36.0	46.39	241	0.97	41.1	46.34	308
1.13	41.7	47.13	225	1.07	37.4	47.07	242	1.02	43.3	47.02	310
1.21	43.9	48.29	225	1.15	39.2	48.23	244	1.10	46.1	48.18	313
1.34	48.2	49.33	225	1.28	42.4	49.27	246	1.23	51.2	49.22	316
1.65	57.1	50.26	225	1.59	49.7	50.20	247	1.54	62.9	50.15	319
2.11	67.2			2.05	60.8			2.00	76.2		

Appendix B
Calculation on methane content

The best way to explain the methane content calculations is through a description of the methane content analysis. The methane content of the reactor is x. A sample (volume 1.1 mL) is injected into a test bottle (volume 30.8 mL). Thus the methane content of the test bottle, y, is

$$y = \frac{1.1}{30.8} \cdot x = a \cdot x$$

When the gas from the test bottle is run in the GC the integrator gives the result as an area: *testarea*. The *testarea* is compared with the area of a control sample: *controlarea*.

The methane content of the control sample is ~ 2 %. The control sample is made by injection of 3 mL of methane and 20 mL of nitrogen into a control bottle (volume 123.5 mL). Thus the methane content of the control bottle, c is

$$c = \frac{3}{123.5 + 20} = 0.0209\ldots \approx 2\,\%$$

testarea, *controlarea*, y and c are related through the following expression

$$\frac{controlarea}{testarea} = \frac{c}{y}$$

The above relations combined give a formula which gives the methane content of the reactor (in percent):

$$x = \frac{c}{a} \cdot \frac{testarea}{controlarea} \cdot 100$$

Appendix C
Calculation on VS degradation

VS reduction is an indicator of how much organic material is being degraded in the reactor. It is defined as

$$\frac{(\text{added organic matter} - \text{organic matter in effluent})}{\text{added organic matter}}$$

The mean VS reduction of the control reactors during day 7–12 is 32 %. During the same period, the mean gas production is 290 mL (g added VS)$^{-1}$. Thus, the gas production in mL per g *degraded* VS is

$$\frac{290}{0.32} = 906$$

The mean daily increase in gas production during the same period is 95 mL. This means that

$$\frac{95}{906} = 0.1\,\text{g degraded VS}$$

corresponds to the increase in gas production, i.e. 0.1 g more VS is being degraded per day in the test reactors.

Appendix D
Energy balance calculation

This is an example of an energy balance calculation.

To begin the following is needed:

Sonicator power	P_S	420 W
Volume of treated sludge	V	5 L
Treatment time	T	6 min

From that we can calculate:

Power input	$P_I = P_S/V$	84 W/L
Energy input	$E_I = P_I \cdot T/60$	8.4 Wh/L

From the average methane content and the average gas production we can determine the average methane production:

Methane content	m	58.1 %
Gas production[a]	g	293.1 mL/g VS
Methane production	$p = m \cdot g/1000$	0.17 L CH_4/g VS

To continue we need to know how much potential energy one litre of methane has. We also need how much the gas production increased due to ultrasonic treatment and some sludge properties.

Energy content (CH_4)	E_{CH_4}	10 Wh/(L CH_4)
Gas production increase[b]	Δg	12.8 %
Sludge Total Solids[c]	TS	3.5 %
Sludge Volatile Solids[c]	VS	75.8 %
VS per sludge volume	$VS_m = 1000 \cdot TS \cdot VS$	26.2 g VS/(L sludge)

Now we can calculate the methane increase from sonication, the potential energy it yields and thus the energy gain:

Methane increase	$m_{incr} = p \cdot \Delta g \cdot VS_m$	0.57 L CH_4/(L sludge)
Energy increase	$E_{incr} = E_{CH_4} \cdot m_{incr}$	5.7 Wh/(L sludge)
Energy gain	$E_{gain} = E_{incr} - E_I$	−2.7 Wh/(L sludge)

[a] Without sonication
[b] Estimated median
[c] Experimental average

In this case the result was an energy loss of 2.7 Wh/L (kWh/m^3). The calculation can also be done with the upper and lower limits of the gas production increase confidence interval and with different TS values. The result is illustrated in table 6 on page 30.

Appendix E
Microscopic sludge analysis

Table 11: Microscopic sludge analysis of waste activated sludge treated with ultrasound.

Date		Treatment time	Extended filament length	Total filament abundance	Floc firmness
Sample	Analysis	(mm:ss)	(0–6)	(0–6)	(0–4)
04–10–18	04–10–18	00:00	3	4	1
Comments: Mixture of 40 mL sludge and 50 mL water. Some areas with a high degree of compactness					
04–10–18	04–10–18	00:45	3	4	1
Comments: Mixture of 40 mL sludge and 50 mL water. Less compactness in flocs. Looks more disintegrated. Smaller and less connective flocs compared to the 00:00-sample. Possibly thinner consistence.					
04–10–18	04–10–18	01:30	3	4	1
Comments: Mixture of 40 mL sludge and 50 mL water. No (significant) difference in filament abundance can be seen compared to sample 00:00 and 00:45. Flocs look less disintegrated compared to the 00:45-sample, could be due to mixing? Less compactness in flocs compared to sample 00:00.					
04–11–01	04–11–01	00:00	3	4	
Comments: Mixture of 40 mL sludge and 100 mL water					
04–11–01	04–11–01	02:00	3	4	
Comments: Mixture of 40 mL sludge and 100 mL water. Disintegrated flocs. Same (disintegrated) feeling with the filaments eventhough they do not look fewer.					
04–11–22	04–11–24	00:00	3.5	4	1
Comments: Mixture of 20 mL sludge and 60 mL water. Higher total filament abundance compared to previous samples.					
04–11–22	04–11–24	10:00	3	4	1
Comments: Mixture of 20 mL sludge and 60 mL water. Flocs look disintegrated. Less compactness in flocs. Filaments are shorter.					
04–12–07	04–12–09	00:00	3	4	1
Comments: Mixture of 40 mL sludge and 80 mL water.					
04–12–07	04–12–09	06:00	3	4	1
Comments: Mixture of 40 mL sludge and 80 mL water. Smaller and fewer areas of high compactness. Shorter extended filaments.					

Table 12: Microscopic sludge analysis of reactor effluent.

Date			Extended filament length	Total filament abundance	Floc firmness
Sample	Analysis	Reactor	(0–6)	(0–6)	(0–4)
04–10–18	04–10–18	C1	3	4	1
		Comments: Mixture of 40 mL sludge and 70 mL water. No limpid water between flocs. Small flocs. Floc firmness near zero. Very few areas of high compactness.			
04–10–18	04–10–18	C2	3	4	1
		Comments: Mixture of 40 mL sludge and 70 mL water. Like C1 sample.			
04–10–18	04–10–18	U1	3	4	1
		Comments: Mixture of 40 mL sludge and 70 mL water. Floc firmness near zero.			
04–10–18	04–10–18	U2	3	5	
04–12–06	04–12–09	No noticeable difference between control reactors (C1, C2) and test reactors (U1, U2) with regard to filamentous bacteria.			

Appendix F
Wilcoxon signed rank test for difference in gas yield

A daily mean for the gas yield from each reactor type was calculated. The control means were subtracted from the test means. This gave us six values, $\delta_1 \ldots \delta_6$, representing the difference in gas yield between the treated and the untreated reactor. The data is given in table 13.

Table 13: Gas yield data from the digestion experiment used for statistical analysis.

	Gas yield (mL/g VS)				
Day	C1	C2	U1	U2	δ_n
7	333	300	350	361	39.1
8	314	282	342	344	44.9
9	285		302	322	27.0
10	304	257	322	344	52.1
11	295	257	302	310	30.3
12	322	285	332	344	34.8

Minitab® 14.13 was used to calculate a confidence interval and an estimated median for δ (Delta). The result of the Wilcoxon signed rank test was:

```
                                        Confidence
                 Estimated    Achieved    Interval
              N     Median  Confidence  Lower Upper
    Delta     6       37.6        94.1   28.7  48.5
```

Note: The test includes day 9 despite the missing value for C2. Only the value from reactor C1 is used instead of a mean between C1 and C2. Since C2 produced less gas than C1 the missing data ought not to be a problem. If C2 had been included it had most likely lowered the mean. So the result from the test is in all likelihood an underestimate of δ.