

**Temperature Phased
Anaerobic Digestion
of
RDC WWTP Biosolids**

Final Test Report

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Abbreviations

°C	degree centigrade
COD	chemical oxygen demand
d	days
DCC	Dunedin City Council
G	gauge
g	grams
GC	gas chromatography
h	hour
HRT	hydraulic residence time
l	litre
M	molar
m	mill
min	minute
mm	millimetres
n	number of data points
ND	not determined
ppm	parts per million
PS	Primary Sludge
RDC	Rotorua District Council
rpm	revolutions per minute
STP	standard temperature and pressure (0°C, 101.3 kPa)
TCD	thermal conduction detector
TPAD	temperature-phased anaerobic digestion
TS	total solids
VFA	volatile fatty acids
VS	volatile solids
w/w	weight/weight
WAS	Waste Activated Sludge
WWTP	Wastewater treatment plant

Executive Summary

In late 2003 Waste Solutions Ltd (WSL) proposed to stabilise the combined and pre-thickened biosolids from the Rotorua District Council (RDC) Wastewater Treatment Plant (WWTP) with modern Anaerobic Digestion (AD) technology, the Temperature Phased Anaerobic Digestion (TPAD). Treatment by TPAD initially sanitises and hydrolyses the biosolids in a thermophilic stage at 55-65 °C and subsequently produces most of the biogas in mesophilic (35-40°C) anaerobic digestion of the thermophilic digestion residues. The expected advantage of the thermophilic TPAD process over standard mesophilic sludge digestion is thus a higher VS reduction, especially with WAS, and the reduction of faecal coliform concentrations substantially below 10³ viable counts/g dry matter. Dedicated digestion tests to determine the suitability of the RDC biosolids were commissioned by RDC in September 2004.

The results from this test program demonstrate that RDC biosolids are suitable as feedstock for anaerobic stabilisation by a TPAD treatment process. The expected biogas quality is adequate for power generation (2700 kwh/day or about 120 KW_{el} subject to H₂S removal to residual levels of < 800 ppm). The electricity production is less than the daily power consumption at the RDC WWTP and the possibility for full internal use of the produced electricity is expected. Details need to be determined in a site specific scoping study based on the daily load profile of the RDC WWTP.

The biogas yield for the combined PS and WAS in RDC biosolids was slightly lower than initially expected but an average annual production of 470,000 m³ biogas is expected with a lower calorific value of 21.5 MJ/m³ (STP). Surplus heat from the biogas utilisation would be adequate to heat the thermophilic process stage and the hot digestate of the thermophilic pretreatment would be adequate for heating of a thermally well insulated mesophilic sludge digester in the final polishing stage.

The observed lower digestability of the biosolids when compared to other sewage biosolids materials may be caused by the nutrient removal treatment at the WWTP and a consistently higher refractory content of WAS biosolids.

The salient features of the TPAD based anaerobic stabilisation of RDC biosolids established here were:

- Good volatile solids (VS) removal and stabilisation with 53 % VS removal efficiency is possible at a thermophilic hydraulic residence time (HRT) of 6 days and mesophilic residence time of 15 days.
- Digestion inhibition by biosolids constituents in batch or continuous tests in either thermophilic or combined thermo/mesophilic TPAD treatment was insignificant.
- RDC biosolids were not unusually refractory to anaerobic digestion and displayed characteristics that were in line with expectations.
- Contrary to expectations, the H₂S content in the biogas was only moderate (1300 +/- 100 ppm) and was comparable to the biogas composition of food waste digestion systems.

- The biogas showed a high methane content (about 60 %) which remained stable during more than 2 months of continuous digestion testing.
- RDC biosolids are suitable for anaerobic stabilisation and energy (biogas) extraction.
- The tested temperature/time regime in the TPAD is suitable to reduce pathogenic bacteria to a US-EPA 503 class A status when the thermophilic effluent is used as feed material to the mesophilic stage. If the material is then not re-infected in the mesophilic stage, the effluent and sludge from the total system should also have comparable low pathogen counts.
- The mesophilic stage effluent is low in VFA and is stable. The overall COD removal from RDC biosolids treated in a continuous TPAD process was about 45 % and the volatile solids reduction 53 %.
- The TPAD system was quite resilient to organic shock loads (step up procedure).
- Design calculations showed that the TPAD process would be capable of accepting raw feed sludges with 93-95 % water content (4-6 % VS). The ability to digest raw sludges prior to dewatering could contribute a substantial sludge dewatering cost saving for the WWTP operation.
- Design calculations based on the sludge analysis established that compared to the current situation a substantial sludge mass reduction is expected in the TPAD process due to the volatile solids reduction and improved sludge dewatering properties after digestion. We expect a TSS content in the dewatered anaerobically digested sludge of about 20 % and a total reduction of the dewatered sludge mass by 55 – 60 % when compared to the dewatered incoming biosolids. This is also expected to reduce polyelectrolyte consumption in the dewatering. This direct benefit from the anaerobic digestion pre-treatment prior to dewatering could become another substantial cost saving for the WWTP operation.

Based on the conservative electricity production estimate and the expected cost savings in the sludge handling operations at the RDC WWTP it is recommended that RDC proceed with implementation of a TPAD solution by initiating a scoping study and options review to determine the most cost effective way to utilise existing assets for integration of a TPAD biosolids treatment step into the operations of the WWTP.

1. Introduction

Biosolids from wastewater treatment plants (WWTP) are a normal byproduct of standard sanitary engineering practices i.e., primary and secondary wastewater treatment. Biosolids may be tainted with concerning levels of toxic heavy metals, typically carry substantial amounts of pathogens such as coliform bacteria, parasites, viruses and also significant amounts of nitrogen (N) and phosphorous (P) nutrients. For example, viable counts of faecal coliform bacteria can be up to 10^9 / g dry matter and N and P nutrients 2-3 % of dry matter. Biosolids from primary treatment of wastewater are also referred to as primary sludge (PS) and biosolids from secondary treatment as secondary sludge or waste activated sludge (WAS). WAS contains mainly bacterial cells which are typically more recalcitrant to biological degradation than primary sludge.

Suitable treatment options for WWTP biosolids are composting, thermal drying & pelletisation, lime stabilisation, incineration or thermophilic/mesophilic anaerobic digestion or a combination of these options. Thickened biosolids from the Rotorua WWTP may contain primary sludge and WAS in variable proportions but typically about 1/3 WAS and 2/3 primary sludge. An example of a typical analysis of thickened biosolids from the Rotorua WWTP provided by RDC is given below (Table 1).

**Table 1: Key chemical composition parameters for Rotorua WWTP biosolids
Average analysis of 8 RDC sludge samples from Dec 2003 to Jan 2005**

As	C (total)	Cd	Cr	Cu	Hg	N	Ni
mg/kg	%	mg/kg	mg/kg	mg/kg	mg/kg	% w/w	mg/kg
9.7	41.9	1.1	28.5	280.0	0.9	6.3	11.7

P	Pb	TS	VS	Fixed Solids	Zn	C/N
% w/w	mg/kg	%	%	%	mg/kg	ratio
12.8	32.8	15.9	13.7	3.3	410.0	6.0

WWTP biosolids are also an underutilised resource for energy production in New Zealand. Their comparatively high water content (70-98 %) - even after mechanical thickening and dewatering - makes incineration unattractive and treatment by composting requires net energy. Under ideal circumstances, stabilisation of primary solids by anaerobic digestion (AD) can achieve a volatile solids (VS) reduction of 50-65 %. This depends on the nature and composition of the biosolids (WAS versus primary sludge) and the chosen anaerobic digester system.

The achievable VS reduction from WAS is typically about 2/3 of the VS reduction achievable from primary sludge. Anaerobic digestion treatment of suitable WWTP biosolids, if properly designed, could thus recover up to 60-70 % of the total chemical energy (COD) in the biosolids in the form of biogas. Typically, destruction of 1 kg VS in primary biosolids would yield up to 0.9-1.1 m³ (STP) of biogas with a lower heating value of 23.3 MJ/m³ [1-4].

Provided that the methane content in the biogas is sufficiently high and the H₂S content is below 800-900 ppm, biogas from biosolids digestion is suitable for on site combined heat & power (CHP) generation to produce electricity for the WWTP operation and heat for the digester operation. It was thus a main objective of these tests to determine the biogas quality (methane content and H₂S content) and methane yield (quantity of usable fuel per unit of waste loaded) that was attainable from RDC biosolids under practical operating conditions using temperature phased anaerobic digestion.

AD treatment of biosolids is a common practice in Europe and in various sewage and industrial WWTP's in Australasia but is not yet widely implemented in New Zealand. Various larger cities and communities in NZ use anaerobic digestion of sewage biosolids with variable success mainly due to operational issues.

Why Anaerobic Treatment?

Anaerobic Digestion (AD) is now an established technology for the treatment of solid waste and wastewater. Great progress has been made in the past 25 years in the areas of digestion process (bio) technology and microbiology and AD is now a mature, robust and cost effective wastewater treatment option. The final product is biogas, a mixture of mainly methane (55 - 75 vol %) and carbon dioxide (25 - 45 vol %) with traces of other gases. It can be used as boiler fuel for steam generation, upgrading to natural gas quality or for cogeneration of electricity and heat. Digester installations are simple with low energy and space requirements. Anaerobic digestion systems currently operating in Europe have a total capacity of 1500 MWel. Worldwide, a capacity of up to 20,000 MWel can be realised by 2010 [5].

Thermophilic systems such as the temperature phased digestion (TPAD) tested here have shorter hydraulic and sludge residence times when compared to mesophilic digester systems [1-4]. This reduces the digester size and investment costs – the higher operation temperature also offering improved pathogen destruction efficiency.

The increasing popularity of anaerobic processes worldwide can be attributed to a number of important advantages in comparison with conventional aerobic and physico-chemical treatment processes. These are

- No aeration needed and thus low in energy demand
- Production of energy-rich biogas
- Reduced production of residual surplus sludge for final disposal
- General low demand of chemicals and nutrients
- High loading rates in terms of BOD removal per unit installed reactor volume
- Stable process performance under irregular load conditions
- Suitable for seasonal operations as the anaerobic bacteria remain viable during extended periods of plant shut-down
- Containment of malodour as the processes take place in closed tanks
- Compatible with aerobic post-treatment processes designed for nitrogen and phosphorus removal or composting.

AEROBIC TREATMENT

ANAEROBIC TREATMENT

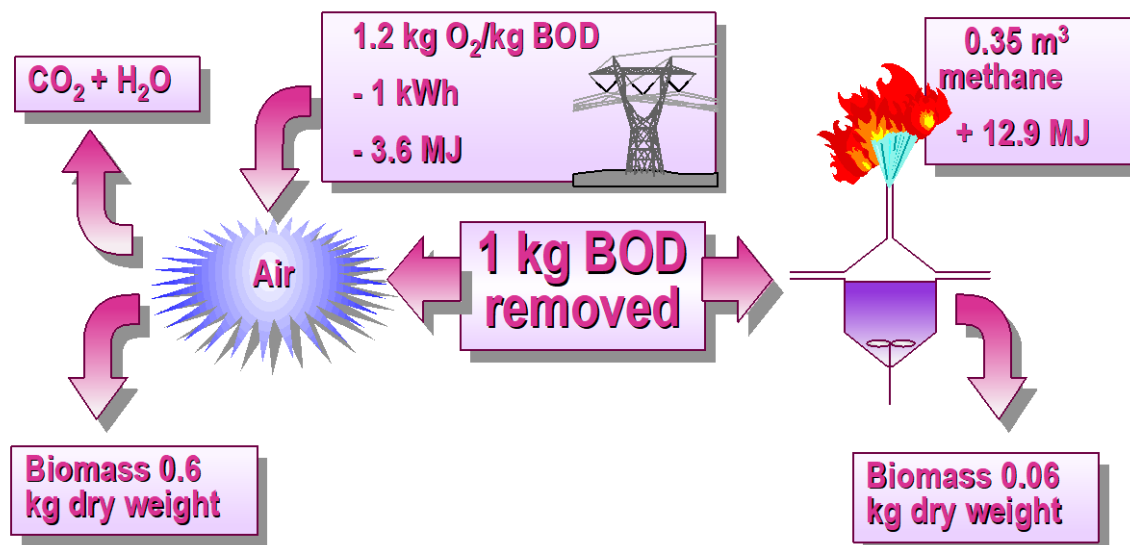


Figure 1.1. An energy comparison between aerobic and anaerobic treatment.

In Figure 1.1 a general energy comparison is made between traditional aerobic treatment methods such as activated sludge treatment (and composting) and anaerobic treatment. Complete oxidation of 1 kg of BOD requires 1.2 kg of oxygen and at least 1 kWh or 3.6 MJ of aeration energy (depending on aeration method). Also, 0.6 kg of additional sludge bacteria is produced as new waste under aerobic conditions. Under anaerobic conditions 0.35 m³ of methane is produced with an energy value of 12.9 MJ instead. Waste sludge production is much lower, typically 1/10 of the amount produced under aerobic conditions.

Anaerobic Decomposition of Organic Material

Organic material is decomposed in anaerobic reactors by a microbial process. In this process, solid and dissolved organic matter is being degraded during a number of steps, in which many different species of bacteria are involved. There are three main steps, in which four main groups of bacteria are involved. The products from one conversion are the feed material for the next group of bacteria, so that the entire process can be seen as a food chain. Two major classes of bacteria can achieve a full degradation of organic material. *Mesophilic bacteria* function best at temperatures of 25 – 40 °C and are killed at higher temperatures. *Thermophilic bacteria* function best at temperatures of 50 – 70 °C and are killed at higher temperatures. However, *thermophilic bacteria* tolerate low temperatures and easily survive storage at ambient temperature. *Initial thermophilic* anaerobic digestion is thus attractive for treatment & sanitation of biosolids because it sanitises the incoming waste, produces some biogas and conditions the material for improved final stabilisation in mesophilic sludge digesters.

A conceptual diagram of anaerobic digestion is shown in Figure 1.2. The first step is often designated as *Fermentation*, *Liquefaction* or *Hydrolysis*. The fermenting microorganisms in the reactor (bacteria and fungi) produce enzymes which are released into the fermentation liquor. The enzymes gradually "dissolve" the solid material in the wastewater by "cutting up" large complex organic molecules into smaller ones. The products of this initial liquefaction step are sugars, fatty acids, peptides and other products. The **fermentative microorganisms** ingest the dissolved products and carry out a fermentation process. Many different species of bacteria and fungi are involved in this fermentation process and many different end products may be the result. The most common products of this fermentation process are organic acids such as formic, acetic, propionic and butyric acid (often designated as *Volatile Fatty Acids*, or, in short, *VFA*), lactic acid or alcohol. Gases may also be produced including hydrogen and carbon dioxide. Fermentative microorganisms grow relatively fast and are also frequently used as starter cultures in dairy processing (yoghurt, cheese), brewing (yeast) and industrial alcohol production (food grade alcohol and gasohol fuel).

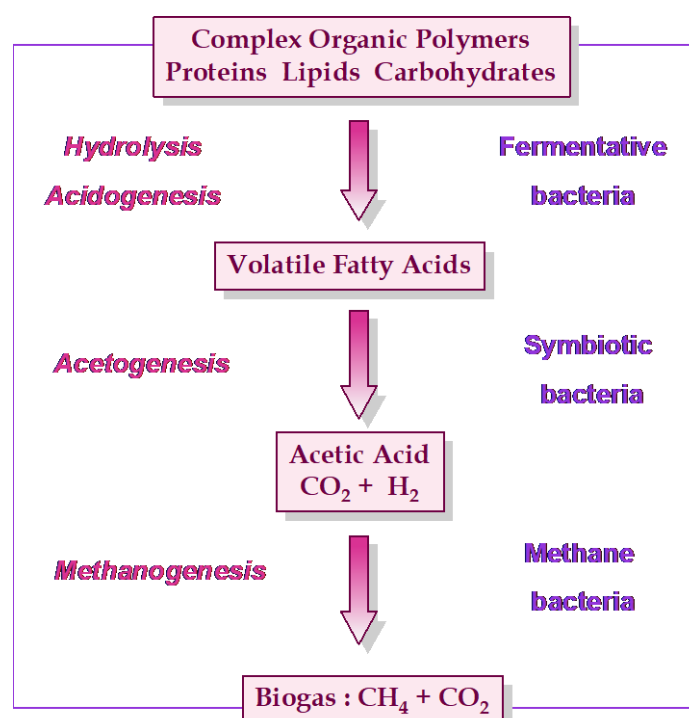


Figure 1.2. Main conversions during anaerobic decomposition of organic matter.

During the second step of anaerobic digestion the organic acids and alcohols from the fermentation are further metabolised by a special group of **symbiotic fermentative bacteria** that exist in symbiosis with some of the methane bacteria (the bacteria which produce the actual biogas at the end of the food chain). This group of bacteria, designated as *Syntrophic Acetogenic Bacteria* (SAB) convert organic acids and alcohols into acetic acid, formic acid and hydrogen gas.

The third and final step of the food chain is the actual production of biogas, a mixture of methane and carbon dioxide (usually 55-75 % methane and 45-25 % carbon dioxide). Biogas may also contain traces of other gases such as hydrogen sulphide, hydrogen phosphide, carbon monoxide and nitrogen gas. The biogas is produced by

methane bacteria, which is again a diverse group of many different species. Methane bacteria are slow growing bacteria which require a complete absence of oxygen and work best at a temperature of either 25 - 40 °C (= *mesophilic* methane bacteria) or grow at higher temperatures, typically in the 50-70 °C range (= *thermophilic* methane bacteria). Under thermophilic conditions one usually obtains faster rates of methane formation than under mesophilic conditions [6-9].

The only disadvantage is that one needs thermophilic bacteria from another thermophilic digester for rapid start of the thermophilic treatment process. This thermophilic seed sludge can also be produced from mesophilic anaerobic digester sludge through a process of gradual adaptation [7].

Brief and Objectives for the testing work

In late 2003 Waste Solutions Ltd (WSL) proposed to stabilise the combined and pre-thickened RDC WWTP biosolids with modern AD technology, Temperature Phased Anaerobic Digestion (TPAD) [1,4]. The situation at the RDC WWTP is unique because about 2/3 of the biosolids is primary solids and 1/3 are WAS from the biological nutrient removal (BNR) operation. Treatment by TPAD initially sanitises and hydrolyses the biosolids in a thermophilic stage at 55-65 °C and subsequently produces most of the biogas in mesophilic (35-40°C) anaerobic digestion of the thermophilic digestion residues. The expected advantage of the thermophilic TPAD process over standard mesophilic sludge digestion is thus a higher VS reduction, especially with WAS, and the reduction of faecal coliform numbers substantially below 10³ counts/g dry matter [1-4,13].

In initial discussions prior to the initiation of the digestion tests reported here, RDC staff mentioned that their own earlier AD trials with biosolids from the RDC WWTP had been disappointing. It was suggested, that the geothermal activity in the area may produce inhibitory substances that preclude effective anaerobic digestion. Therefore it is a major objective of the present work to evaluate the suitability of combined RDC biosolids for stabilisation and energy recovery by anaerobic digestion.

Subsequent discussions with FRI (Dr Per Nielsen) and RDC (Dr Alison Lowe, Dr Sean Barnes) resulted - in September 2004 - in the acceptance of the WSL proposal to conduct a two stage bench scale digestion test

- to establish whether RDC biosolids are suitable as potential feedstock for treatment by the TPAD method and
- to determine the key process design parameters that would allow a continuous anaerobic treatment system of RDC biosolids with a TPAD process.

The major objective of the initial batch digestion testing was to evaluate whether RDC biosolids contain substances that inhibit anaerobic digestion and thus could preclude effective treatment. The main function of the continuously operated temperature phased digestion test was to determine key process parameters that would be useful for the preliminary design of a full scale biosolids digestion facility for a two step TPAD process for the RDC biosolids. This report is the final report for both stages of the test program.

2. Materials and Methods

Thermophilic seed sludge: Thermophilic seed sludge (55°C) was obtained from the Dunedin City Council (DCC) thermophilic biosolids digester at the Green Island wastewater treatment plant. We would like to thank the site manager for his assistance to provide active seed culture for these tests. The active seed sludge was concentrated by centrifugation from 1.7 % to a final TS content of about 12 % (20°C, anaerobic conditions). The seed sludge was well digested indicated by a VS content of 65 % of TS. The RDC biosolids were received as a thickened paste with about 15 % TS content and 12 % VS content. The COD content of RDC biosolids was about 1.7 g COD/ g TS (Table 2).

Biological Methane Potential (BMP) Measurement: The BMP was evaluated in 500 ml Schott bottles with dedicated gas tight seals. The tests contained about 3-6 % TS as final concentration and were made of 25 ml seed sludge + 5, 10 or 15 g (wet wt) of RDC biosolids (as received by weekly courier) and water as the balance. The total test volume was 100 ml. The bottles were flush/evacuated with O₂ free N₂ (99.999 % N₂) and were incubated at 55°C in a shaking water bath. Daily samples of the headspace and liquid were analysed for total gas production. The methane, CO₂, N₂ content in the biogas was determined using standard laboratory procedures at Waste Solutions Ltd. Volatile fatty acids and pH were determined daily from samples of the mixed liquor. The TS and VS content in the mixed liquor of each test bottle was determined at the beginning and at the end of the tests. Typically, each test condition was tested in triplicate in independent BMP tests.

Data Evaluation: The cumulative methane production in each test system was determined for each day using the measurements and a calculation spreadsheet. The results were graphically presented and, where possible were screened for consistency using a mass and COD balance over the BMP test system.

Feed sludge: Feed sludge samples were provided by the client, Rotorua District Council (RDC). The samples consisted of about 1/3 waste activated sludge and 2/3 primary sludge. Samples were stored at 4°C until used. Table 1 above gives the parameters of the typical feed sludge (average of 8 sludge samples from December 2003 to January 2005). Table 2 (below) shows key composition parameters of RDC sludge according to our own laboratory analysis of the incoming samples (average of 4 sludge samples from December 2004 to January 2005)

Table 2: Analysed parameters of RDC biosolids received for the tests

COD	Total Solids (TS)	Volatile Solids (VS)
g/kg TS	%	%
1.75	15.1	12.3

The analysis demonstrates that the sludge samples used for the tests were quite consistent with the expected average composition (Table 1).

Temperature Phased Anaerobic Digestion (TPAD) reactor system: The TPAD test reactor consisted of two 5 litre glass bottles (Schott, Mainz, Germany), which were incubated in separate shaking water baths (Figure 2.1). The produced gas from

both test compartments was collected at one point. In order to test the effect of the hydraulic residence time in each compartment on the volatile solids removal and total methane yield, the working volumes in each compartment were varied from 1.7 to 3 litres (thermophilic stage) and 3 to 4.5 litres (mesophilic stage). Feed addition and liquor transfer between both compartments was with Masterflex neoprene tubing No 15 and No 18 respectively (Masterflex, Chicago, USA). Heidolph pump motors and speed controllers (Heidolph, Schwabach, Germany).

At the outset, the TPAD headspace was flushed with a 6-fold headspace volume of oxygen-free nitrogen. In addition, it was occasionally necessary to flush the system while operating. For this purpose, a nitrogen gas cylinder (pure nitrogen, oxygen-free) was permanently installed with the system to enable quick reaction to possible oxygen leaks.

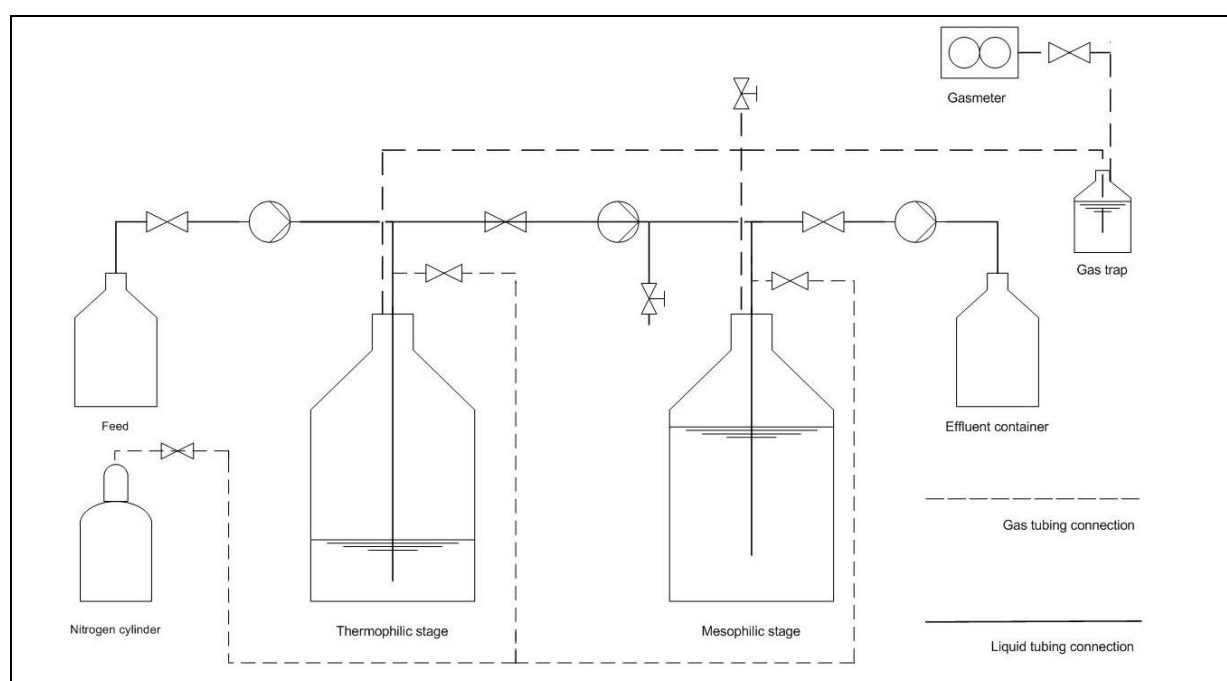


Figure 2.1: Experimental TPAD setup. See appendix 2 for photographs of the setup.

The TPAD had sample ports for both liquids (between the thermo- and mesophilic stage) and gases. Samples of the mesophilic stage were taken out of the effluent tubing.

The TPAD was fed once per day by first withdrawing 300 ml effluent out of the mesophilic stage. Then, the same amounts of mixed liquor were transferred from the thermophilic to the mesophilic stage, where a sample was taken, if required. Finally, 300 ml of fresh feed mix were fed through the feed pump into the thermophilic stage. While feeding, the gas tubing leading to the gas trap was closed to prevent water inflow to the system from the gas trap.

For start-up, the TPAD system was incubated and fed with 5 g COD/litre/day for 10 days with $HRT_{thermo} = 10$ days and $HRT_{meso} = 5$ days. The loading rate was calculated based on the volume of the thermophilic stage. Due to the Christmas holiday period, the system was shut down and kept at room temperature for 17 days. Three days

before shutdown, the system was not feed any more, but pH and VFA were monitored.

When restarting the system by heating up to process temperature, the HRT's were changed to $HRT_{thermo} = 6.7$ days (after running the system for 7 days) and $HRT_{meso} = 10$ days (immediately). After restart, the organic loading rate to the thermophilic stage was 6.7 g COD/litre/day, then it was raised to 14 g COD/litre/day (based on the volume of the thermophilic stage). After the TPAD was performing for 14 days under these loading rates, they were changed once more to $HRT_{thermo} = 5.7$ days and $HRT_{meso} = 15$ days, while the daily organic load was not changed. After 11 days of stable operation under these test conditions, the TPAD system was shut down.

During TPAD operation, the pH was kept above 7 by adding 0.135M $Ca(OH)_2$ (lime) to the feed, as $Ca(OH)_2$ was most likely to be the process base in the full-scale plant. The amount was equivalent to 56 kg $Ca(OH)_2$ per tonne loaded COD. During critical times, the pH was measured daily by taking samples of the effluent and using the sample port between the thermophilic and the mesophilic stage.

Reactor gas: The gas production in the batch setup was monitored by using a mariotti flask [10], filled with 1 mM HCl (pH=2.9) to prevent dissolving CO_2 in the water. The flask could measure gas volume increases from 1 ml up to 60 ml by displacing water and had an initial dead volume of 3 ml. Calibration tests showed that the error of this device was less than 5% error. The gas production of the TPAD was measured by using a wet gas meter (Ritter, Bochum-Langendreer, Germany). The reactor gas first had to pass a break tube (= "gas trap") and then entered the gas meter. The produced gas was analysed using gas chromatography. Two systems were used to detect nitrogen, oxygen, carbon dioxide and methane:

- Varian 3700 GC with a CTR I column, TCD detector and HP 3395 Integrator. The carrier gas flow was 30 ml/min Hydrogen. The temperatures of the column and injector were 40°C and 200°C and the detector voltage was at 150 mA.
- SRI GC with CTR I column and TCD detector. Carrier gas flow was 88.9 ml/min Hydrogen. The temperature of the column and injector was room temperature, the TCD temperature was 100 °C. Chromatograms were analyzed with PeakSimple II software.

The H_2S content was measured once at the end of the batch-tests and several times during the TPAD operation (via gas sample port) with Dräger hydrogen sulphide 100/a Short-term Tubes (Dräger Safety, Lübeck, Germany) with a range from 100 to 2000 ppm H_2S . The manufacturer specified an error margin of $\pm 5-10\%$ for individual measurements.

Volatile Fatty Acids (VFA): The observation of VFA (acetic acid, propionic acid, butyric acid, valeric acid) is useful for direct specific loading rate adjustments and preventing the reactor from overloads. A fast increase in VFA, primarily propionic acid, indicates stress of the reactor. VFA's also cause malodour in effluents, and an acceptable limit in effluents is 500 to 1000 mg/l [3,11].

One millilitre of the daily sample was frozen for VFA determination both in the batch and the continuous system. The TPAD was monitored daily during critical operation

periods, at other times monitored at least weekly. The VFA were analysed with a Varian 3700 gas chromatograph on a 185cm x 2mm ID glass column packed with chromosorb 101 80/100 mesh. The temperatures of the oven, injector port and detector were 180°C (isothermal), 210°C and 210°C. The carrier gas was hydrogen with a flow rate of 20ml/min. Chromatograms were quantitatively analyzed with an HP 3395 integrator. See appendix 1 for the detailed method.

Chemical Oxygen Demand (COD): The COD of the feed sludge and the TPAD effluent were determined. Due to the relatively high COD content, the samples were diluted 1:100 before analysed. COD balance calculations were made to test the methane build-up and the VS removal results for consistency in batch run and continuous system. See appendix 1 for the detailed method.

Total Solids and Volatile Solids (TS/VS): See appendix 1 for the detailed method.

3 Results

Part 1: Digestability of RDC biosolids at thermophilic temperatures (batch conditions)

3.1 Assessment of Digestion Inhibition by RDC Biosolids

Biological methane potential (BMP) tests at 55°C were seeded with thermophilic anaerobic sludge from the DCC WWTP at Green Island. The seed sludge was mixed with RDC biosolids in an approximately 1:1 ratio (VS basis) at a final strength of about 4 % VS in the test system. Increasing amounts of RDC sludge were added to the seed sludge in three BMP assays to test for negative effects on the methane production. A negative control without added RDC sludge was also included. To ensure that the methane formation was not limited by a lack of suitable substrates, low levels of ethanol (2 mM final concentration) were also daily added into the treatments and the control. The endogenous methane production determined from inoculated controls (with seed sludge + ethanol and without RDC biosolids addition) was subtracted from the presented data. Figure 3.1 demonstrates that RDC biosolids display properties that are not inhibitory to anaerobic digestion

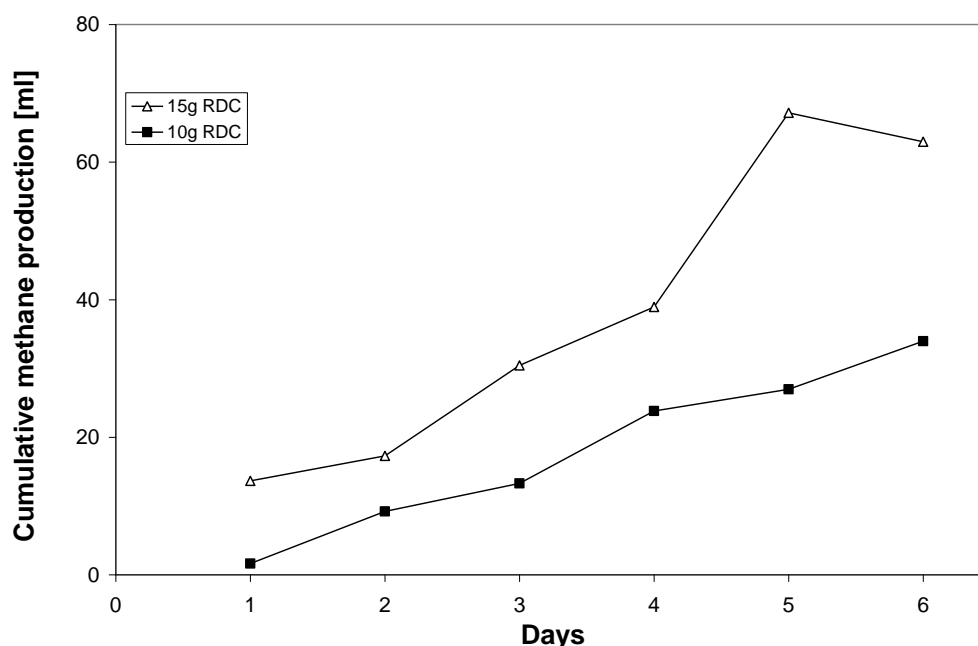


Figure 3.1: Inhibition test of RDC biosolids on thermophilic anaerobic sludge.

Thermophilic seed sludge from the DCC Green Island WWTP was combined with thickened RDC biosolids at 0 days in an approximately 1:1 (w/w) volatile solids ratio using a digestion test system with 100 ml working volume. Every day additional ethanol was added to each test to give each day a final level of 2 mMol/L. The cumulative methane formation in a negative control with seed sludge + 2 mMol/L ethanol is already subtracted from the curves.

Methane formation initiated immediately without any acclimatisation period. The cumulative methane production from ethanol + RDC biosolids increased with increasing biosolids additions in a dose dependent manner (Figure 1).

The data supported the conclusion that

- RDC biosolids do not contain significant amounts of substances that preclude thermophilic anaerobic digestion of soluble substrates because otherwise an increasing content of RDC sludge should have suppressed the methane production.
- RDC biosolids were amenable to anaerobic digestion by thermophilic bacteria.

Figure 3.2 shows the results of repeat thermophilic BMP tests (triplicates, mean +/- SD in error bars) conducted in the absence of added ethanol. Again, thermophilic methane formation from RDC biosolids started without delay in a dose dependent manner. The delayed onset of methane formation in the controls with thermophilic seed sludge and without added RDC biosolids shows that the centrifuged seed sludge contained a low amount of readily degradable organic matter. Thermophilic hydrolysis and digestion of seed sludge solids caused an added delayed methane formation in controls and in all treatments after 4-6 days.

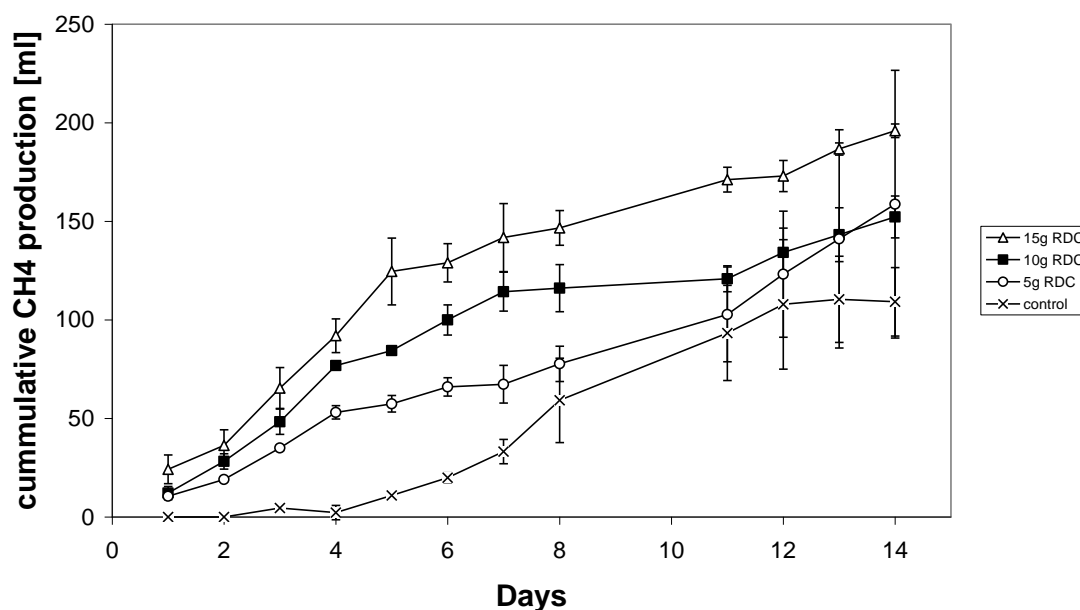


Figure 3.2: Thermophilic digestability test of RDC biosolids

Thermophilic seed sludge from the DCC Green Island WWTP was combined with thickened RDC biosolids in an approximately 33, 48 and 60 % (w/w) volatile solids ratio (standard digestion test systems with 100 ml working volume). The mean and standard deviation of the cumulative methane production in triplicate parallel tests are shown. The cumulative methane production of the control in the absence of added RDC biosolids is also shown for comparison.

Figure 3.3 confirms the dose dependency of thermophilic methane production from RDC biosolids in BMP tests without added ethanol. Tests were conducted essentially as shown in Figure 3.2. The methane formation in the controls without added RDC biosolids is already subtracted from the data. Only the early phase of the tests (0-6 days) is shown to highlight the RDC biosolids dose dependent methane production.

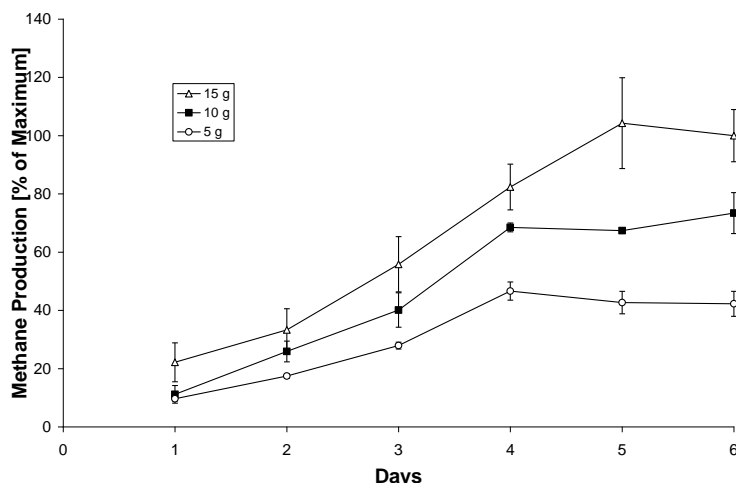


Figure 3.3: Thermophilic biological methane potential of RDC biosolids

Thermophilic seed sludge from the DCC Green Island WWTP was combined with thickened RDC biosolids at 50, 100 and 150 g/L RDC biosolids (wet weight) in an approximately 67, 52 and 40 % (w/w) volatile solids ratio. The mean and standard deviation of the cumulative methane production in triplicate parallel tests (100 ml test system) are shown. The endogenous methane production and solubilisation of the DCC seed sludge was determined in separate controls and was subtracted from the data. After 6 days incubation each treatment produced about 15-20 % VS removal (measured as methane) and about 30-35 % total VS solubilisation of the loaded biosolids (measured as methane + dissolved volatile fatty acids).

The data shown in Figures 3.1-3.3 thus established that the RDC WWTP biosolids samples provided for these tests were suitable for stabilisation by thermophilic anaerobic digestion. An increasing dose of RDC WWTP biosolids stimulated thermophilic methane production.

As the biosolids samples provided for the tests were representative for the expected average composition over more than a year, these results support the conclusions that

- (a) RDC biosolids are unlikely to contain constituents that significantly inhibit anaerobic digestion
- (b) RDC biosolids are suitable for treatment by thermophilic anaerobic digestion

3.2 Potential Thermophilic Digestibility of RDC Biosolids

The anaerobic digestibility of WWTP biosolids depends to some extent on the ratio between primary sludge (PS) and waste activated sludge (WAS) in the biosolids. Biosolids with about 50 % each of PS and WAS typically show about 50 % VS reduction whereas biosolids with 80 % PS and 20 % WAS show significantly higher VS reduction [1-3]. While the assessment of key process parameters for the thermophilic digestibility of RDC biosolids in a continuously operated TPAD process is the topic of part 2 of this report, thermophilic batch digestion test data similar to the data presented in Figure 3.3 were analysed to provide a preliminary indication of the achieved VS reduction. Triplicate BMP tests were started with thermophilic seed sludge and RDC sludge using the volatile solids (VS) ratios specified in the legend for

Figure 3.4. Tests were incubated at 55°C for 14 days until no further significant methane formation was observed. pH control was effected by addition of NaOH when necessary and the pH was stabilised around pH 7.4-7.6. The achieved VS removal and VS solubilisation contributed by the seed sludge in the tests was determined in separate controls and was subtracted from the data presented in Figure 3. 4.

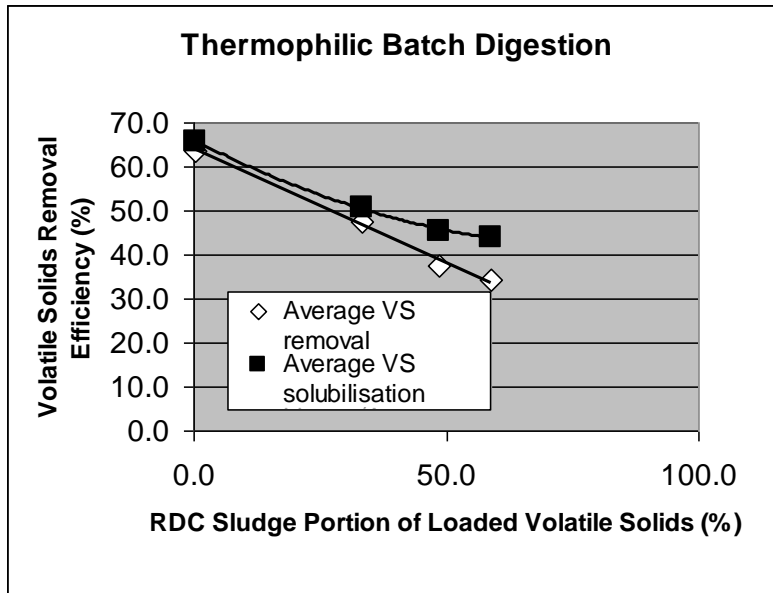


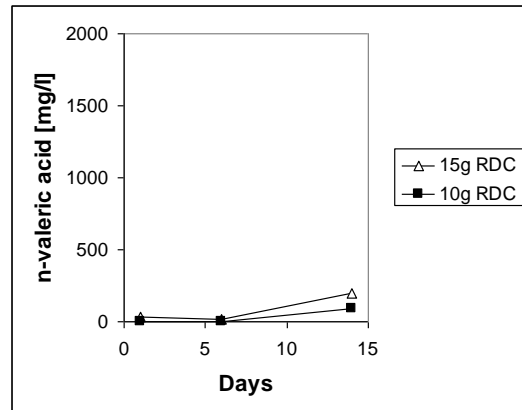
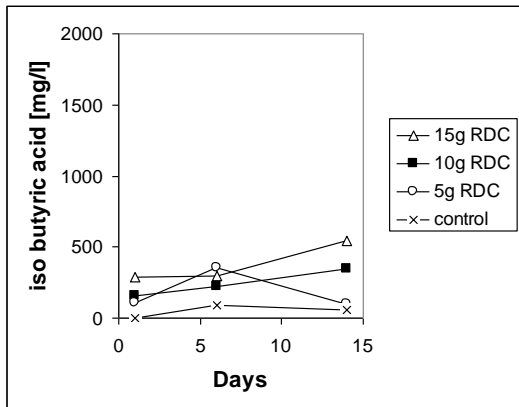
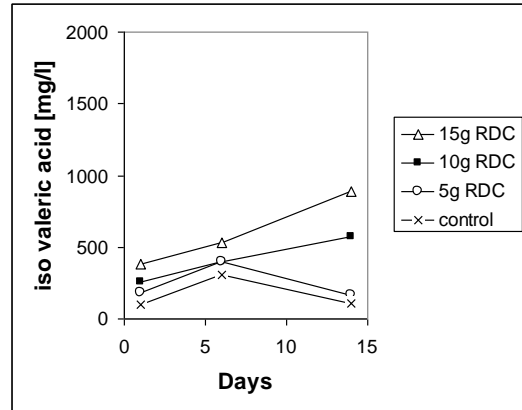
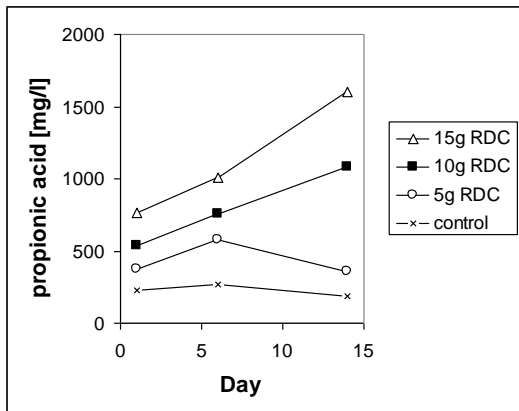
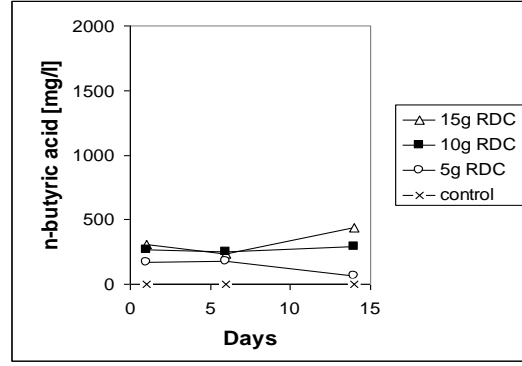
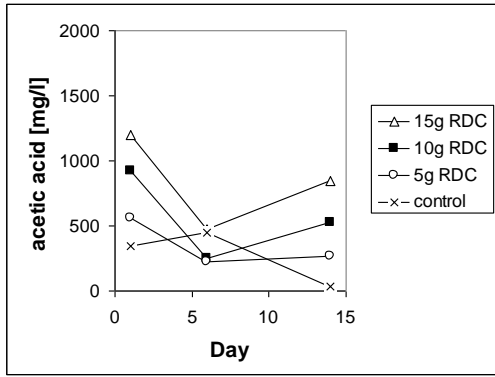
Figure 3.4: Thermophilic volatile solids removal of RDC biosolids

Thickened RDC biosolids were combined with thermophilic seed sludge from the DCC Green Island WWTP in an approximately 33, 48 and 60 % (w/w) volatile solids ratio (standard digestion test systems with 100 ml working volume). Each bottle contained the same total amount of volatile solids in the beginning. The mean and standard deviation of the cumulative methane production in triplicate parallel tests after 14 days incubation at 55°C are shown. Please note that the seed sludge (data points at 0 % RDC sludge portion) has a significantly higher degradability than the RDC biosolids.

The data demonstrate that the thermophilic biodegradability of RDC sludge in batch tests is substantially less than the biodegradability in the acclimatised seed sludge from an operating thermophilic digester such as the DCC Green Island plant (0 % RDC sludge portion of loaded VS in Figure 3.4). The data in Figure 3.3 suggest that the reduced thermophilic digestibility of RDC biosolids was not due to presence of inhibitory substances

The average overall VS solubilisation achieved under thermophilic conditions (Figure 3.4, filled squares) was determined by combining the measured biogas production (17 % conversion of loaded VS) and the residual volatile fatty acids found in the digestate after thermophilic treatment of RDC biosolids (see Figure 3.5 below). The high VFA accumulation under thermophilic conditions suggests thus that a subsequent mesophilic treatment of the thermophilic digestate is necessary.

Figure 3.5: Volatile Fatty Acid production, Thermophilic batch digestion



These data from the batch tests suggest that only 30-35 % of the RDC biosolids VS were solubilised under thermophilic batch digestion conditions. The low solubilisation could have partly been caused by digestion inhibition from the elevated VFA levels in the mixed liquor of the thermophilic sludge hydrolysis. The high residual volatile fatty acid (VFA) levels found after thermophilic treatment of RDC biosolids in batch (see Figure 3.5, above) suggest that a two step treatment would be advantageous with a mesophilic final stage to remove VFA formed under thermophilic conditions.

3.3 Quality of Biogas from Batch Digestion of RDC Biosolids

The major biogas constituents formed after 14 days of thermophilic treatment of RDC biosolids were analysed by gas chromatography. H₂S was determined by Dräger test tubes (200 – 2000 ppm range). The results are presented in table 3. The results showed that the biogas from the thermophilic treatment was of good quality. It should be noted that these results of the obtained biogas quality were preliminary. They were essentially confirmed by the continuous TPAD treatment of RDC biosolids shown in part 2 of this report.

Table 3: Indicative quality of biogas from thermophilic digestion of RDC biosolids

Biogas composition, batch conditions			
RDC Biosolids	% CH₄	%CO₂	ppm H₂S
control	60.5	39.5	ND
15 % (w/w)	53.6	46.4	900
10 % (w/w)	56.6	43.4	ND
5 % (w/w)	61.3	38.7	ND

ND: not determined

The slight decrease in the methane content in the formed biogas with the increasing RDC sludge concentration is consistent with the respective higher VFA levels found in the digestate (Figure 3.5).

Part 2: RDC biosolids digestability in continuous systems (TPAD, Temperature phased conditions)

3.4 Key Process Parameters for Temperature Phased Anaerobic Digestion of RDC Biosolids

A key objective for the digestion test in this report was to establish certainty that the proposed continuous TPAD system was suitable and effective for stable anaerobic digestion of RDC biosolids. A flexible test system simulating the two stage digestion process was assembled (see section 2 and appendix 2 for details). The system was used to evaluate a range of hydraulic residence times (HRT, i.e. relative system size for a given organic load) and their effect on the methane yield, total volatile solids reduction and digestion process stability.

Hydraulic residence time and organic loading rate: The test system and operation routine allowed to independently vary the HRT in the initial thermophilic and subsequent mesophilic stage. To make most effective use of the limited funding made available, the TPAD digestion process was evaluated concentrating mainly on the mesophilic stage. The starter culture was acclimatised to the test conditions (temperature and feed material) with exclusive feeding of RDC biosolids at a strength of 4 % VS (5 % TS) with about 85 kg COD/m³. The HRT in the thermophilic stage was 5.7-6.7 days and the mesophilic HRT 10 days. This translated into a COD loading rate of the thermophilic stage of 12.7 – 15 kg COD . m⁻³.day⁻¹. The organic loading rate to the mesophilic stage alone was initially 7.2 kg COD/m³.day⁻¹ (assuming 15 % VS removal in the thermophilic stage, section 3.3) and 4.1 – 5.1 kg COD.m⁻³.day⁻¹ as loading rate to the total system at 15 days HRT in the mesophilic stage.

The shortest HRT evaluated under these conditions (15.7 days) was 66-80 % of the typical HRT recommended for mesophilic sludge digestion (20-25 days). Despite high total volatile fatty acid levels in the thermophilic stage (Table 4) and high organic loading rates, the mesophilic stage at 10 days HRT achieved a reasonable volatile fatty acid (VFA) removal (Table 5). It is significant that the TPAD digestion process was able to adjust to the mesophilic load challenge of 7.2 kg COD.m⁻³.day⁻¹ within one hydraulic residence time. This is indicated by decreasing levels of acetic and propionic acid in the mixed liquor at 10 days mesophilic HRT which was observed within one HRT after the load challenge (Figure 3.6, next page).

Resilience to high organic loading rates: The mesophilic culture showed resilience in the TPAD system against shock conditions at shorter HRT's. This confirmed that RDC biosolids did not carry significant proportions of inhibitory constituents. The mesophilic culture responded to the load robustly with increased growth and biogas production indicated by the decreasing levels of acetic and propionic acid (Figure 3.6, next page) and increased gas production (data not shown). Relaxing the mesophilic HRT from 10 days to 15 days coincided with rapid and nearly quantitative consumption of practically all system effluent VFA to very low levels (Table 5 and

Figure 3.6, days 30 – 45; next page) despite continued loading with RDC biosolids to the thermophilic first stage.

Table 4: VFA levels in the thermophilic stage at different HRT's. VFA are given in mg/l. n = number of data points.

HRT [days]	n	acetic acid	propionic acid	iso butyric acid	n-butyric acid	iso valeric acid	n-valeric acid	total VFA
6.7	7	480 ± 110	1680 ± 70	570 ± 30	470 ± 120	970 ± 30	170 ± 20	4340 ± 250
5.7	6	650 ± 160	1770 ± 90	590 ± 40	438 ± 120	990 ± 50	170 ± 30	4600 ± 150

Table 5: VFA levels in the mesophilic stage at different HRT's. VFA are given in mg/l. n = number of data points.

HRT [days]	n	acetic acid	propionic acid	iso butyric acid	n-butyric acid	iso valeric acid	n-valeric acid	total VFA
10	14	460 ± 430	910 ± 150	380 ± 100	30 ± 40	510 ± 70	0	2290 ± 330
11	1	140	580	460	0	550	0	1730
12	1	270	490	460	0	470	0	1690
13	1	270	470	530	0	360	0	1630
14	2	400 ± 55	530 ± 30	255 ± 160	0	165 ± 130	0	1350 ± 330
15	3	140 ± 120	130 ± 130	0	0	0	0	260 ± 100

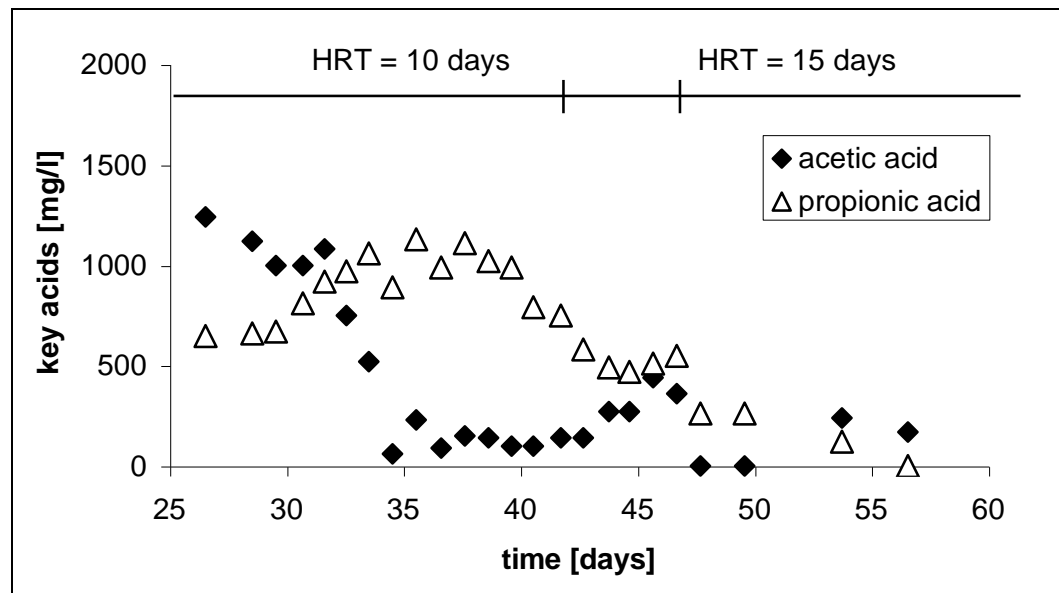


Figure 3.6: Time course of acetic acid and propionic acid levels in the mesophilic stage mixed liquor at 10 and 15 days HRT

These results demonstrated that a continuous TPAD system can be operated with RDC biosolids at HRT's in the thermophilic stage of about 5-6 days and mesophilic HRT's between 10 and 15 days. This was very encouraging and showed that RDC biosolids are suitable for treatment in a TPAD process with an overall HRT between 16 and 20 days.

The limited funding that was made available for these tests did not allow to further explore the minimum mesophilic HRT that permit stable TPAD operation, thus the results above are to be seen as preliminary. However, the fact that the mesophilic TPAD stage did stabilise when challenged at 10 days HRT demonstrates that a conservative design for about 15 days HRT in the mesophilic TPAD stage would be a reasonable design choice that would also provides reserve digestion capacity. It is expected that such a system would be able to accept load fluctuations with maximum daily loads up to 150 % of the average daily biosolids load to the TPAD system.

Operating temperature: The operating temperature in these tests with RDC biosolids was 55°C and 35°C for thermo and mesophilic stage respectively and thus in a suitable range to heat the mesophilic stage directly with the pre-digested effluent from the thermophilic stage. The limited resources made available for this report did not allow to test a different temperature regime.

3.5 Performance of the Temperature Phased Anaerobic Digestion of RDC Biosolids

Volatile solids (VS) destruction: The VS destruction and the methane yield are indirectly correlated and depend on the nature and biochemical composition of the biosolids. While the literature on the expected methane yield from biosolids digestion varies depending on the nature of the biosolids (depends on primary versus secondary sludge & fat content: 0.5 – 0.7 l CH₄/g VS destroyed, [1,11,12]), it is recognised in the industry that a higher content of WAS in the biosolids will reduce the methane yield [1]. The VS destruction efficiency was determined from the comparison of the VS content in the TPAD feed and the effluent from the mesophilic stage. The system was able to remove 53.4 ± 1.4 % of the loaded VS (n=14, measured over 20 days). The VS content in the system remained stable during that period indicating that VS accumulation/storage in the reactor contents was insignificant. This result compared favourably with the VS destruction achieved in other TPAD systems for mixed primary and secondary treatment sludge (45-50 % destruction, ref 1) or standard mesophilic anaerobic digesters (50-55 % destruction, ref 11,12). There was no significant difference in the VS removal efficiency in the TPAD system at various overall system HRT's with RDC sludge (16.7 to 20.7 days) indicating that some volatile fatty acids (Figure 3.6) may have been lost from the effluent sample during the initial drying step of the VS determination technique.

Methane yield: The methane yield (l methane/ g VS destroyed) was determined by direct measurement of the daily biogas production & composition and expressed for standard temperature and pressure (STP). The results are shown in Figure 3.7. The methane content in the combined biogas from thermophilic and mesophilic process stages was typically 59 – 62 % methane and 38- 41 % CO₂ (average range over 2 weeks of operation).

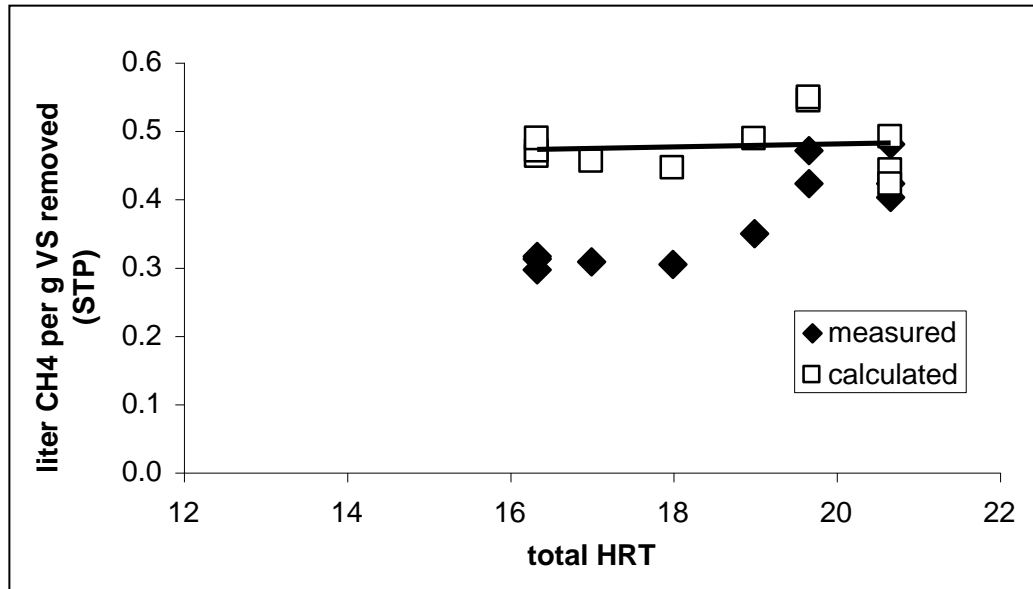


Figure 3.7: Measured and calculated methane production in liters methane (STP) per g VS removed at various HRT's. The calculated figures were based on the captured methane gas + the additional methane that would have been produced from the total VFA that were measured in the discharged effluent from the mesophilic stage.

To determine the practical methane yield from RDC biosolids, the HRT of the total system was increased from 16.7 to 20.7 days. In these conditions the maximum methane yields recorded were 0.49 l methane/ g VS destroyed. The average methane yield at 20.7 days HRT was 0.44 +/- 0.04 l methane/g VS removed. This result was slightly lower than the performance of other TPAD systems (0.5 l methane/ g VS destroyed, ref 1) and at the lower end of methane yields reported in the technical literature for anaerobic digestion of sewage biosolids (0.49 – 0.73 l methane/ g VS destroyed; ref 11,12). This lower methane yield of RDC biosolids despite a reasonable VS reduction during TPAD digestion is likely a consequence of the higher content of more oxidised aerobic WAS in the RDC biosolids that were supplied for the tests. It is known in the technical literature that aerobic WAS has a lower specific gas yield than primary sludge [1].

COD removal efficiency: The COD removal efficiency for the TPAD system was determined at two consecutive days after the TPAD system had stabilised at a mesophilic HRT of 15 days. The results are given in Table 6.

Table 6: COD removal efficiency achieved with the TPAD system (mean +/- SD, n = 3).

Sample	Feed mix COD (g COD/l)	Effluent COD (g COD/l)	COD remov. efficiency (%)
20.7 days HRT, sample 1	395 +/- 26	228 +/- 23	42
20.7 days HRT, sample 2	395 +/- 26	207 +/- 23	47

The data demonstrate that the COD removal efficiency was somewhat less than the VS removal efficiency that was achieved (53.4 ± 1.4 %). Therefore we conclude that the TPAD treatment preferentially removed more oxidised (= less COD/VS) components. This conclusion is in agreement with the lower methane yield that was observed. A lower COD reduction %-age in comparison with the VS reduction %-age is consistent with a lower specific methane yield (1 methane/g VS removed).

Suitability of the biogas for power generation: H₂S levels in the biogas above 800 ppm will somewhat compromise the lifetime and operation costs (oil change frequency) of genset equipment with internal combustion engines or gas microturbines (threshold depends on manufacturer). Details need to be requested from individual manufacturers as these vary from brand to brand and between different technologies. Six different biogas samples on three different days (duplicate determination on each day) were collected over a 7 day period when the TPAD system had been operated for more than 30 days with RDC biosolids. The system HRT during the gas sampling was between 19 and 20 days.

At this point more than 90 liters of biogas from RDC biosolids digestion had been produced into a system headspace of less than 6 liters and the TPAD system was thus thoroughly flushed with the authentic biogas (15 fold volume). The H₂S levels recorded were 1300 +/-100 ppm. While these H₂S levels were higher than desirable, it is common experience in the industry that air injection (< 5 % v/v) into the biogas buffer or into the digester headspace of the mesophilic stage will reduce slightly elevated H₂S levels. Therefore the quality of the raw biogas from TPAD of RDC biosolids (about 60 % methane, < 1400 ppm H₂S) is suitable for enduse in genset equipment provided that measures are taken to reduce the H₂S levels below 800 ppm.

Maximum liquefaction of RDC biosolids: When the total system HRT was reduced to 15.7 days, the methane yield was reduced to 0.31 liter methane/g VS removed (Figure 3.7, filled squares). In order to determine, whether the reduced methane yield at shorter HRT's than 20 days (Figure 3.6) was due to decreased biosolids liquefaction or was caused by apparent "methane losses" through discharge of undegraded VFA's, the effluent VFA content at each HRT was determined by gas chromatography and the respective total COD content of the discharged VFA species calculated. Using

standard calculation procedures (0.35 l methane produced/g COD destroyed), the potential VFA-methane loss incurred with discharged VFA was determined for each HRT and then was added back to the measured values. The results (Figure 3.7., open squares) suggested that the reduced methane yield at short system HRT's was mainly due to discharge of undegraded residual VFA in the effluent from the mesophilic stage and not due to reduced solids liquefaction at shorter HRT's. The calculated figures gave a straight line at 0.45 l methane/g VS removed and the value was virtually independent of the HRT (Figure 3.7., open squares). As the sum of the produced methane and the produced VFA is a direct measure of the achieved total liquefaction of sludge matter in the TPAD system these results showed that 6 days HRT under thermophilic conditions + 10 days HRT under mesophilic conditions are sufficient to stabilise the RDC biosolids if complete VFA degradation can be achieved at 10 days HRT in the mesophilic stage. A minimum HRT of 16 days was in agreement with the technical literature [1,12] and confirmed the conclusion that RDC biosolids do not contain significant amounts of constituents that inhibit the liquefaction or the methane production step in anaerobic digestion.

A thermophilic HRT of 6 days combined with a mesophilic HRT of 10 days are thus likely adequate for substantial biological stabilisation of the RDC biosolids.

Pathogen and vector attraction reduction: It is well established that thermophilic operation of anaerobic digesters leads to substantial reduction in the viability of pathogenic bacteria [13]. The US-EPA standards for the use or disposal of sewage sludge (PART 503, subpart D, US-EPA para 503.30) provide a calculation procedure for the minimum holding period at 55 °C to achieve class A pathogen reduction. The minimum holding period at 55°C would be 2.6 days to achieve class A pathogen reduction for sewage sludge with more than 7 % solids and less than 1 day for sewage sludge with less than 7 % solids. Therefore, it is highly likely that a 6 days HRT in the thermophilic TPAD stage at 55°C for biosolids at a strength of less than 7 % solids is sufficient to substantially reduce residual pathogenic bacteria from the incoming biosolids. Only experimental verification of the microbiological status of the effluent from the thermophilic stage (residual pathogen counts of *Salmonella* and fecal coliforms) will allow the final assessment of this aspect. This task was outside the scope for this testing report and will have to be left for future work during the detailed design stage for a TPAD system for RDC biosolids.

Bioenergy potential and suitability of existing tanks:

An important objective for this report was to establish the approximate bioenergy potential that could be generated from anaerobic processing of RDC biosolids. Table 7 below summarises the four year average biosolids production figures provided by RDC for this purpose. The expected biogas methane energy potential from processing of the average daily throughput is calculated as:

Bioenergy production. At 1,281,000 kg VS/annum, 50 % VS removal and 0.44 m³ methane/kg VS removed we expect with a TPAD at 20 days HRT annually the production of 281,820 m³ of methane or 469,700 m³/annum of biogas at 60 % methane (Lower heating value H_u :21.5 MJ/m³). This is sufficient to generate about 120 KW electricity on a continuous basis.

Table 7: Preliminary system requirements and existing tankage at RDC WWTP.

RDC WWTP Sludge				
Year	mass t wet	TS %	Monthly Average mass VS t (est.)	mass t dry
2001	11044	17.8	140	1982
2002	9163	16.2	105	1486
2003	8368	15.1	89	1258
2004	9022	15.1	96	1352
Daily average (4-yr)	26.4		3.6	4.2
Monthly average (4-yr)	783	16.0	107	126
Annual average (4-yr)	9399		1281	1519
Daily maximum (2004)	46.2		6.3	7.4
Monthly max (4-yr)	1378		188	249
Annual max (4-yr)	11044		1506	1982

Average daily flow at 5 % TS	83	t/day
Maximum daily flow at 5 % TS	148	t/day
Mesophilic digester tank size for average flow(m3)	830- 1250	m3 for mesophilic stage
available tankage, 2 x 700 m3	1400	m3

Suitability of existing decommissioned reactor tanks:

The available information at the moment suggests that the existing tanks need to be substantially altered/expanded to accommodate the proposed TPAD system.

A site visit in 2004 established that two open tanks are available in close proximity to the current sludge dewatering building. Based on drawings made available and subject to confirmation of the structural integrity and water tightness of the tanks by a certified engineer, we estimate that each tank would provide about 600 m³ working volume for the purpose of mesophilic or thermophilic anaerobic sludge digestion. Both tanks would be sufficient to provide the reactor volume for the mesophilic step – a new tank would be required for the thermophilic stage.

A preliminary assessment of the drawings suggested that it might be possible to seal the tanks with a HDPE cover and HDPE structural hoop at the top providing in both tanks together enough working volume for the mesophilic stage to digest the average RDC biosolids load. Each tank would have to be provided with adequate mixing (immersible mixer or circulation pump) and adequate penetrations + pumps/pipework for sludge conveyance. A separate design inspection is required to determine whether most of the pipework/penetrations could be ducted through the new tank cover. Insulation to the existing tanks would have to be provided.

Alternatively, only one of the existing tanks could be converted to the thermophilic digester with the same constraints/alterations as suggested above and the other tank could be dismantled/demolished. A site inspection needs to be carried out to establish whether sufficient area exists to site a 1200 m³ mesophilic digester on the foundation of the demolished tank. The options and the relative cost estimates need to be established separately and were outside the scope for this report.

4. Conclusions and Recommendation

The results from this test program demonstrate that RDC biosolids are suitable as feedstock for anaerobic stabilisation by a TPAD treatment process. The expected biogas quality is adequate for power generation (2700 kwh/day or about 120 KW_{el} subject to H₂S removal to residual levels of < 800 ppm).

The biogas yield for the combined PS and WAS in RDC biosolids was slightly lower than initially expected but an average yearly production of 470,000 m³ biogas is expected with a lower calorific value of approximately 21.5 MJ/m³ (STP). Surplus heat from the biogas utilisation would be adequate to heat the thermophilic process stage and the hot digestate would be adequate for heating of a thermally well insulated mesophilic sludge digester in the final polishing stage.

The observed slightly reduced digestability of the biosolids may be caused by the nutrient removal treatment at the WWTP and a consistently higher content of refractory WAS biosolids.

The salient features for the TPAD based anaerobic stabilisation of RDC biosolids established here were:

- Good volatile solids (VS) removal and stabilisation with 53 % VS removal efficiency is possible at a thermophilic hydraulic residence time (HRT) of 6 days and mesophilic residence time of 15 days.
- Digestion inhibition by biosolids constituents in batch or continuous tests in either thermophilic or combined thermo/mesophilic TPAD treatment was insignificant.
- RDC biosolids were not unusually refractory to anaerobic digestion and displayed characteristics that were in line with expectations and at the lower end of the typical range.
- Contrary to expectations, the H₂S content in the biogas was only moderate (1300 +/- 100 ppm) and was comparable to the biogas composition of food waste digestion systems.
- The biogas showed a medium methane content (about 53-59 %) which remained stable during more than 2 months of continuous digestion testing.
- RDC biosolids are thus suitable for anaerobic stabilisation and energy (biogas) extraction.
- The tested temperature/time regime in the TPAD is suitable to reduce pathogenic bacteria to a US-EPA 503 class A status when the thermophilic effluent is used as feed material to the mesophilic stage. If the material is then not re-infected in the mesophilic stage, the effluent and sludge from the total system should also have comparable low pathogen counts.
- The mesophilic stage effluent is low in VFA and is stable. The overall COD removal from RDC biosolids treated in a continuous TPAD process was about 45 % and the volatile solids reduction 53 %.
- The TPAD system was quite resilient to organic shock loads (step up procedure).

- Design calculations showed that the TPAD process would be capable of accepting raw feed sludges with 93-95 % water content (4-6 % VS) under such conditions. Anaerobic digestion of raw sludges prior to dewatering could result in a substantial sludge dewatering cost saving for the WWTP operation.
- Design calculations based on the sludge analysis and the achieved volatile solids reduction established that a substantial sludge mass reduction is expected in the TPAD process. This is due to improved sludge dewatering properties after digestion. We expect a TSS content in the dewatered anaerobically digested sludge of about 20 % and a total reduction of the dewatered sludge mass by 55 – 60 % when compared to the dewatered incoming biosolids. This direct benefit from the anaerobic digestion pre-treatment prior to dewatering could become another substantial cost saving for the WWTP operation.

Based on the reasonable electricity production potential and the expected cost savings in the sludge handling operations at the RDC WWTP it is therefore recommended to proceed with implementation of a TPAD solution by initiating a scoping study and options review to determine the most cost effective way to utilise existing assets for integration of a TPAD biosolids treatment step into the operations of the WWTP.

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Appendix

1 Methods:

1.1 Total Solids and Volatile Solids (TS/VS)

- 1 Description Heating samples at 103°C – 105°C drives off the water contained in sludge and sediment samples. Ignition of the dried sample at 550°C leaves the inorganic portion of the sample.
- 2 Interferences Subject to negative error due to the loss of ammonium carbonate and volatile organics.
- 3 Apparatus Drying oven at 103°C – 105°C
Evaporating dishes 90mm porcelain
Analytical balance 3 decimal place.
Muffle Furnace at 550°C
- 4 Procedure: with a Approximately half fill a pre-weighed (B) evaporation dish well mixed sample and weigh (C).
Record weight.
Analyse in duplicate.
Place in oven overnight.
Remove from oven and allow to cool to room temperature in a desiccator and weigh. (A)
Record weight.
Place in muffle furnace and ash.
Remove from furnace and allow to cool to ~100°C then place in a desiccator to cool to room temperature and weigh. (D)
Record weight.
- 5 Calculation
$$TS [\%] = (A - B) * 100 / (C - B)$$
$$VS [\% TS] = (A - B) - (D - B) / (A - B) * 100$$
$$VS [\%] = TS [\%] * VS [\% TS] / 100$$

A = weight of dry residue plus dish
B = weight of dish
C = weight of wet sample plus dish
D = weight of ash residue plus dish.
- 6 Reference: APHA 2540 G 20th edition 1998.

1.2 Chemical Oxygen Demand (COD)

1	Description the	When a sample is digested the dichromate ion oxidises carbonaceous material in the sample. This results in a change in the valency of the chromium ion from the hexavalent (VI) state to the trivalent (III) state both of these species are coloured so a spectrophotometer can be used to quantitate the amount of either species.
1	Interferences High	Samples should be preserved at pH <2 with H ₂ SO ₄ . levels of chloride may interfere. This method is to be used for COD determination in wastes and wastewaters with COD,s greater than 40mg/l.
3	Apparatus	Spectrophotometer (600nm), 150mm x 10mm screw cap test tubes. Digestion block at 150°C capable of holding the above tubes.
4	Reagents Digestion Solution Catalyst Solution Standard KHP	Add 10.216g K ₂ Cr ₂ O ₇ (dried for 2h at 105°C) to ~500ml distilled water. Slowly add 167ml conc. H ₂ SO ₄ (98%) and 13.3g HgSO ₄ . Allow to dissolve and cool then make to 1000ml. Dissolve 24.75g AgSO ₄ in 2.5l conc. H ₂ SO ₄ (98%) Dissolve 425mg Potassium Hydrogen Phthalate (dried for 2h at 105°C) in ~900ml distilled water. Make to 1000ml. Dispense into 50ml aliquots and freeze. Thaw and discard at least monthly. Store in fridge. The theoretical COD of this solution is 500mg/l.
5	Standard Curve	To duplicate tubes add 0, 0.5, 1.0, 1.5 and 2.0ml of standard to duplicate tubes and make to 2.5ml final volume with distilled water.
6	Procedure	To 2.5ml sample/ standard add: Add 1.5ml digestion solution. Add 3.5ml catalyst solution. Screw on cap firmly and invert to mix. <i>CAUTION- Wear face shield and gloves when adding catalyst solution and mixing tubes.</i> Digest at 150°C for two hours in heating block in fume hood.

Remove tubes from block, place in test tube rack and allow to cool to room temperature in fume hood.

CAUTION- *Wear face shield and gloves when removing tubes from block*

Measure absorbance at 600nm.

- | | | |
|---|-------------|--|
| 7 | Calculation | Plot a standard curve and read concentration off this curve. |
| 8 | Refs: | Standard Methods for the Examination of Water and Wastewater. APHA, 5220 D, 20 th edition 1998. |

1.3 VFA determination in digester liquors by gas chromatography

Description

Volatile fatty acids (C2 to C7) are critical to the control of anaerobic digesters especially during start up.

These acids are separated on the aromatic polymer Chromosorb 101.

Reagents

1 Protein Precipitant.

Dissolve 47g meta phosphoric acid in 150ml reversed osmosis (RO) water.
Add 62.5ml 100% Formic acid and make to 1000ml with RO water.

2 Internal Standard.

Weigh 2.43g 3-methyl valeric acid into a 500ml volumetric flask and make to volume with RO water.

3 VFA Standard mix.

Acetic acid weigh 6.0g into a 100ml volumetric flask and make to volume with RO water.

Propionic acid weigh 7.4g into a 100ml volumetric flask and make to volume with RO water.

Iso butyric acid weigh 0.8g into a 100ml volumetric flask and make to volume with RO water.

n-butyric acid weigh 8.8g into a 100ml volumetric flask and make to volume with RO water.

Iso valeric acid weigh 1.0g into a 100ml volumetric flask and make to volume with RO water

n-valeric acid weigh 1.0g into a 100ml volumetric flask and make to volume with RO water.

From these stocks prepare the following

To a 200ml volumetric flask add

- 20ml acetic acid
- 5ml propionic acid
- 5ml n-butyric acid

10ml iso butyric acid
10ml n-valeric acid
10ml iso valeric acid
and make to volume.

Working solution.

To 30ml of the above, mix 10ml protein precipitant, 10ml internal standard and 20ml RO water.

This will give final concentrations of:

3600 mg/l Acetic acid
1100 mg/l propionic acid
260 mg/l iso butyric acid
1320 mg/l n-butyric acid
306 mg/l iso valeric acid
306 mg/l n-valeric acid

Sample clean up.

Centrifuge sample at 13,000 rpm for 10 minutes. To 0.5 ml of the supernatant add 0.1ml protein precipitant and 0.1ml internal standard.
Centrifuge at 13,000 rpm for 10 min.
Inject 1 µl of the clear supernatant onto the column.

Gas Chromatography.

Varian 3700 gas chromatograph.

Column 185cm x 2mm ID glass packed with Chromosorb 101 80/100 mesh.

Oven temp 180°C isothermal.

Injector temp 210°C

Detector temp 210°C

Carrier gas Helium at 20ml/min

FID

FID gas flows:

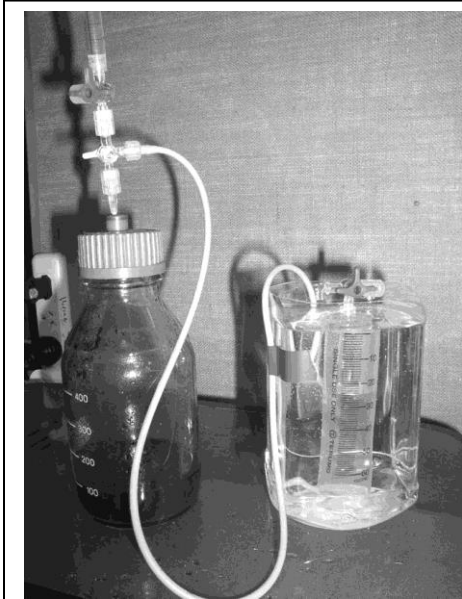
Hydrogen at 30ml/min

Air at 300ml/min

Quantitation.

Internal standard using a Hewlett Packard 3390 integrator.

2 Photographs of the experimental work



Batch test bottle connected to mariotti flask and sample syringe for gas production measurement.

