University of Southampton
Faculty of Engineering, Science and Mathematics
School of Civil Engineering and the Environment

The Development of a Mesh Bioreactor for the Anaerobic Digestion of Biodegradable Municipal Waste

Mark Walker

Thesis for the Degree of Doctor of Philosophy

September 2008

Supervisors:
Prof. Charles J Banks and Dr. Sonia Heaven

UNIVERSITY OF SOUTHAMPTON

ABSTRACT

FACULTY OF ENGINEERING, SCIENCE AND MATHEMATICS

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Doctor of Philosophy

THE DEVELOPMENT OF A MESH BIOREACTOR FOR THE ANAEROBIC DIGESTION OF BIODEGRADABLE MUNICIPAL WASTE

by Mark Walker

A laboratory scale prototype mesh bioreactor (MeBR) for the two-stage anaerobic digestion (AD) of biodegradable municipal waste (BMW) was successfully designed and tested.

The development involved a number of preliminary stages; creation and characterization of a synthetic BMW (SBMW), exploration of its single-stage AD characteristics under both methanogenic and hydrolytic conditions, and AD trials of a two-stage reactor system where SBMW was fed to a 1st stage hydraulic flush (HF) reactor and centrifuging was used as a method to produce liquid effluent which was fed to a 2nd stage anaerobic filter (AF) reactor.

The single stage digestion of SBMW suffered from process instability at very low organic loading rates (OLR) of 2-2.5 $gVSl^{-1}d^{-1}$ whilst the two-stage HF/AF system was robust up to a maximum OLR of 7.5gVS/ld. The HF reactors became methanogenic due to the effect of effluent recycling.

After this, two different prototypes designs of MeBR were built and tested in continuous two-stage AD trials (AF 2nd stage). The aim was to replace the centrifuging of the HF reactors with continuous mesh filtration whilst maintaining the stable and robust digestion process. The first design confirmed the ability to filter SBMW digestate through nylon meshes of pore size 30-140 µm at an OLR of 3.75 $gVSl^{-1}d^{-1}$. The mesh system operated similarly to the HF/AF system and efficient two-stage AD of the SBMW was shown. Problems with stirring thick digestate limited the OLR on both the mesh and HF systems.

To address this limitation on OLR, a 2nd MeBR was designed which employed a rotating drum for low effort mixing and 100 µm nylon mesh sections on the drum surface for filtration. This reactor system operated stably at an OLR of up to 15 $gVSl^{-1}d^{-1}$ albeit with reduced specific methane production.

Application of this type of system will be dependant on requirements for high plant throughput, system robustness and a compact process to make up for slightly lower methane production and waste stabilisation compared to single stage digestion.

Keywords: Anaerobic Digestion, Biodegradable Municipal Waste (BMW), Membrane, Mesh, Hydraulic Flush, Two-stage.

Contents

List of Tables

List of Figures

Authors Declaration

I, Mark Walker declare that the thesis entitled THE DEVELOPMENT OF A MESH BIOREACTOR FOR THE ANAEROBIC DIGESTION OF BIODEGRADABLE MUNICIPAL WASTE and the work presented in the thesis are both my own, and have been generated by me as the result of my own original research. I confirm that:

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- where any part of this thesis has previously been submitted for a degree or any other qualification at this University or any other institution, this has been clearly stated;
- where I have consulted the published work of others, this is always clearly attributed;
- where I have quoted from the work of others, the source is always given. With the exception of such quotations, this thesis is entirely my own work;
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- where the thesis is based on work done by myself jointly with others, I have made clear exactly what was done by others and what I have contributed myself;
- Parts of this work have been presented and published as conferences papers:

Walker, M., C. J. Banks and S. Heaven (2007). Effects of pH Control and Methanogenic Effluent Recycle on the Two-Stage Anaerobic Digestion of BMW. 11th IWA World Congress on Anaerobic Digestion, Brisbane, Australia, IWA.

Walker, M., C. J. Banks and S. Heaven (2008). Development of a Coarse Membrane Bioreactor for Two-Stage Anaerobic Digestion of Biodegradable Municipal Solid Waste. 5th international symposium of anaerobic digestion of solid waste & energy crops, 25-28 May 2008, Hammamet, Tunisia, IWA.

Signed: ……..

Date:…….

Acknowledgements

Professor Charles Banks and Dr Sonia Heaven are acknowledged for their supervision. Constructive and insightful advice was always delivered with enthusiasm and wit.

Secondly I would like to acknowledge all members of the environmental research group for countless discussions both in group meetings and in more social environments.

I also acknowledge the environmental laboratory technical staff for their practical support and Department of Farming and Rural Affairs (Defra) for financing this research under the Technologies Research and Innovation Fund (TRIF).

Abbreviations and Acronyms

AD	Anaerobic Digestion
AF	Anaerobic Filter
BMP	Biochemical Methane Potential
BMW	Biodegradable Municipal Solid Waste
C:N	Carbon to Nitrogen Ratio
CSTR	Continuously Stirred Tank Reactor
EPS	Extracellular Polymeric Substances
EU	European Union
FVW	Fruit and Vegetable Waste
HRT	Hydraulic Retention Time
HF	Hydraulic Flush
IA	Intermediate Alkalinity
LCFA	Long Chain Fatty Acid
MBR	Membrane Bioreactor
MeBR	Mesh Bioreactor
MFBR	Mesh Filter Bioreactor
MSBMW	Mechanically Sorted Biodegradable Municipal Solid Waste
MSW	Municipal Solid Waste
OD	Outer Diameter
OLR	Organic Loading Rate
PA	Partial Alkalinity
PAC	Powered Activated Carbon
PVC	Poly Vinyl Chloride
RDMBR	Rotating Drum Mesh Bioreactor
RPM	Revolutions Per Minute
RRF	Resource Recovery Forum
RUDAD	Rumen Derived Anaerobic Digestion
SAMBR	Submerged Anaerobic Membrane Bioreactor
SBR	Sequence Batch Reactors
SBMW	Synthetic Biodegradable municipal solid waste
(S)COD	(Soluble) Chemical Oxygen Demand
SMP	Specific Methane Production
SRT	Solids Retention Time

SSBMW	Source Segregated Biodegradable Municipal Waste
STP	Standard Temperature and Pressure
TA	Total Alkalinity
TMP	Trans-membrane Pressure
TRIF	Technology Research Innovation Fund
TS	Total Solids
TSS	Total Suspended Solids
UASB	Upflow Anaerobic Sludge Blanket
VS	Volatile Solids
VSS	Volatile Suspended Solids
VFA	Volatile Fatty Acid

1 Introduction

Anaerobic Digestion (AD) offers the potential of diverting large quantities of biodegradable waste from landfill and could form part of a sustainable waste management policy in the UK. Approximately 60% of the Municipal Solid Waste (MSW) produced in the UK is suitable for AD (Poll 2003). The solid and liquid materials produced can be used as soil conditioners and bio fertilizers. Additionally, AD yields methane which can provide renewable energy (Chynoweth and Pullammanappallil 1996).

Unfortunately, current digestion technology for wet solid materials can have shortcomings, such as low waste loadings and long retention times leading to large plant installations; and in certain cases potential process instability (Banks and Wang 2000). This research project explored the possibility of alleviating some of these problems by using a two-stage mesh/membrane bioreactor system.

1.1 Project Background

The project was funded by the Department for Environment and Rural Affairs (Defra) as part of the Technologies Research Innovation Fund (TRIF). This funding was created to stimulate the development of technologies with the potential to divert biodegradable municipal waste (BMW) away from landfill. This is part of the UK government's response to the EU (European Union) Landfill Directive (1999) which sets a number of targets to reduce the amount of landfilled waste relative to 1995 levels (EU 1999).

Another way in which the UK government is discouraging the use of landfill is the introduction of a landfill tax, designed to encourage a reduction in waste production and the recovery of any remaining value from waste materials. Landfill tax has two bands, the lower being for inert or stabilized waste, and the higher for other wastes including biodegradable waste (Defra 2007b).

According to the Defra municipal waste statistics for 2006/2007 the majority of municipal waste in the UK is still landfilled (58%) , although most collection

authorities have non-landfill routes for the disposal of source segregated green waste, paper/card and other components of MSW amounting to a total recycling rate of 31% (Defra 2007a).

1.2 Anaerobic Digestion

The key advantage of AD as a disposal technology, in comparison with other methods, is the production of biogas, which is a mixture containing methane and carbon dioxide. This gas is flammable and can be combusted to produce energy for a variety of applications such as heating, electricity generation or as an automotive fuel in gas engines. The production of methane means AD can be much more energetically favourable when compared with landfilling and composting of organic wastes (Gijzen et al. 1987a; Gallert et al. 2003) and avoids the large environmental impact typically associated landfill sites (Mata-Alvarez et al. 2000).

For many years, AD has been used for the treatment of sludge from wastewater treatment works and in 2000 around 36,000 anaerobic digesters were being used for this purpose in Europe (Mata-Alvarez et al. 2000). There are an increasing number of plant installations for the treatment of solid wastes and other organic materials and research is continuing into different aspects of AD of these materials in areas such as process modelling, digester performance, chemical inhibition/toxicity, parametric studies, collection and pre-treatment options, and reactor configurations such as two and multi-stage systems (Mata-Alvarez 2003a).

Most of the AD installed capacity worldwide is in the form of Continuous Stirred Tank Reactors (CSTR). This is the traditional design of digesters and is simply a large tank that is mixed by mechanical stirring, digestate pumping or biogas recirculation. Unfortunately, this design of reactor does not give the most efficient biological digestion process for solid waste materials, and a number of factors shown in Table 1 lead to low loading rates, long retention times and process instability (Gerardi 2003g). Without sufficient monitoring the biological system can irreversibly fail (Vandevivere et al. 2003a).

Table 1 Disadvantages of CSTRs When Digesting Solid Waste

Factor	Consequence
Slow growth rate of methanogenic organisms (A)	Long retention time and large tank (High capital and operating costs)
Sensitivity of microorganisms to hydrolysis/acidification intermediates and the pH change caused by these chemicals (B)	Low organic loading and unstable process
Hydrolysis is rate limiting (C)	Long retention time and large tank (High capital and operating costs)

CSTRs are being outperformed by many newer and more innovative reactor systems fed with various materials. Examples of these are plug flow reactors (Liu and Ghosh 1997), Anaerobic Filters (AF) (Ahn and Forster 2000), Sequenced Batch Reactors (SBR) (Chynoweth et al. 1992), high solids reactors (Kayhanian 1995) and a variety of novel two-stage digester designs (See section 2.3).

1.3 The Two-stage Mesh/Membrane Bioreactor

The main hypothesis of this work is that the problems cited in Table 1 could be alleviated by using a two-stage mesh/membrane bioreactor system as shown schematically in Figure 1. The key feature of each of the stages is that the solid retention time (SRT) and hydraulic retention time (HRT) are uncoupled, and unlike a CSTR (SRT=HRT) can be controlled independently by controlling the filtration flux.

In the 1st stage, or hydrolysis reactor, the purpose of the mesh is to allow long SRT as required by the rate limiting hydrolysis process, while allowing a short HRT to remove the volatile fatty acids (VFA) which are potentially inhibitory to the process. This means both the organic loading rate (OLR) as well as the total hydrolysis rate can be maximised. Treated process water, or methanogenic effluent, is recycled back to the 1st stage to replace the filtrate removed through the mesh, thus creating a hydraulic flush (HF). Reactors operated in this mode have been shown to allow much greater loading rates, and a more stable

process, than the CSTR equivalent whilst still maintaining similar material breakdown (Wang and Banks 2000).

The purpose of the membrane in the 2nd or methanogenic reactor is for the retention of biomass. As cited in Table 1, the slow growth rate of methanogenic organisms means that in a CSTR, long HRT/SRT are required to maintain a healthy population within the reactor. In the 2nd stage reactor the pore size of the membrane is such that the methanogenic archea cannot leave the reactor, and instead form an attached layer upon the surfaces, which exposes them to the feed solution. This biomass retention means that the reactor can be operated at low HRT, high SRT and high loading rate. This type of reactor has been shown to have high performance in terms of loading rates and COD removal in high strength wastewater (Hu 2004), and it is hypothesized that this could be extended to the liquid effluent produced from the breakdown of BMW.

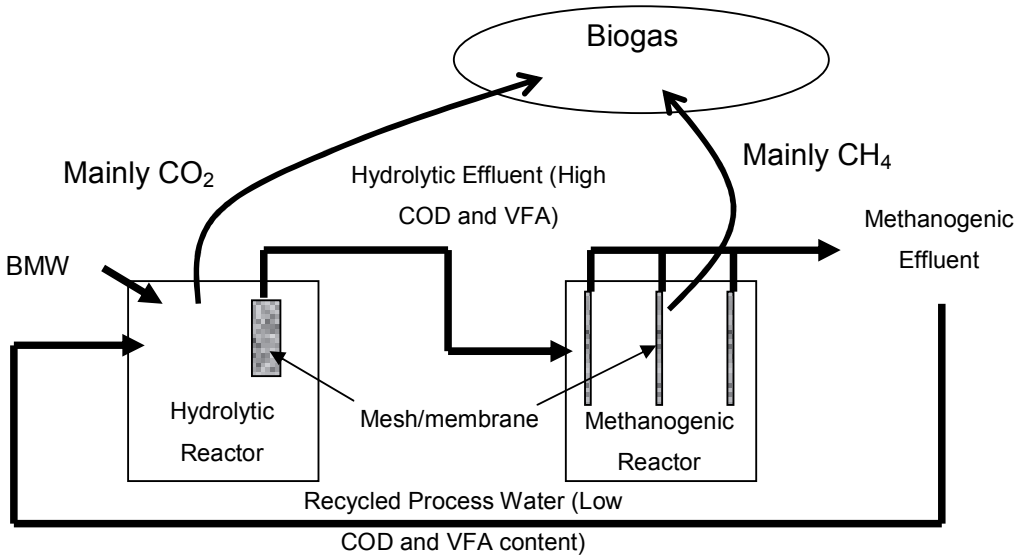


Figure 1 Schematic of the Two-stage MBR System

1.4 Project Aims and Objectives

The development of a two-stage mesh/membrane bioreactor system for the anaerobic treatment of BMW proposed for the original Defra-funded project was split into two discrete parts. Development of the 1st stage (hydrolysis) reactor and of the 2nd stage (methanogenic) reactor were performed at Southampton

University and Imperial College, London respectively. This report is the culmination of work performed on the development of the 1st stage reactor.

1.4.1 Aims

- Develop design criteria for a 1st stage HF mesh bioreactor capable of stabilizing ball-milled BMW at high loading rates and investigate its ability to be part of a two-stage AD process delivering high rates of organic waste stabilisation and energy recovery.

- Explore the possibility and potential problems and/or issues relating to the scale-up of the above reactor system and its use in the AD industry in the medium term.

1.4.2 Objectives

The above aims will be met by achievement of the following objectives, to:

- Create a feed material similar in composition and chemical make up to BMW that can be created and replicated in the laboratory, characterize this feedstock in terms of its biochemical methane potential (BMP), and chemical composition and compare this characterisation with literature cited examples.

- Understand the effects of various physical properties and reactor operational conditions on the hydrolysis rate of the feedstock and to investigate the AD performance of a conventional CSTR when being fed on SBMW.

- Gain insight into the HF process, and investigate the effect on the hydrolysis rate of various possible process modifications such as HRT, effluent recirculation, buffer addition and OLR.

- Design, build and test laboratory scale prototypes of novel mesh bioreactors able effectively to digest BMW at high OLR.

- Identify a suitable mesh material and pore size and investigate the filtration performance parameters, such as maximum flux and fouling, and operate a suitable flux maintenance and/or mesh cleaning strategy for sustainable filtration.

- Compare the two-stage process with a single-stage CSTR equivalent fed on the same material in terms of important process characteristics such as specific methane production (SMP), material stabilisation, process stability, and maximum OLR.

- Explore and discuss the potential problems, issues, strengths and weaknesses of the mesh bioreactor process especially with relevance to the scale-up of this technology.

1.4.3 Structure

This thesis follows a conventional structure; a review of the relevant literature, a description of the material and methods used, experimental results followed by discussions, conclusions and finally suggestions of further work.

In general the work has been presented in the order that it was performed. From the beginnings of creating and characterising the SBMW, though to the design and implementation of a laboratory scale rotating drum bioreactor two-stage AD system, the trend has been to start with the simple and to gradually increase the complexity of the experimental and equipment design as well as the interpretation of the results.

2 Literature Review

2.1 Anaerobic Digestion Background

AD is the breakdown of organic materials by microorganisms such as bacteria, archea and protozoa in the absence of oxygen. This process is responsible for the natural decomposition of organic matter under anaerobic conditions and takes place in manures, wetlands, aquatic sediments, and rice fields as well as the intestines of animals. The degradation involves a series of chemical reactions resulting in production of various gases such as methane, carbon dioxide and hydrogen sulphide as well as other soluble substances such as ammonia (Gerardi 2003a).

The four major processes in AD are hydrolysis, fermentation, acetogenesis and methanogenesis. Microorganisms are grouped in relation to the process they are responsible for, namely as hydrolytic, fermentative, acetogenic and methanogenic organisms. The nature of the anaerobic food chain is such that each of these groups relies on the previous one for its substrate and on the next one to avoid accumulation of its products (Gerardi 2003b). Because of the relative specialisation of anaerobic organisms, a plethora of different species need to be present in anaerobic digesters for complete breakdown of complex organic matter. This is in contrast with aerobic organisms which often can often oxidize complex organic molecules to carbon dioxide. Figure 2 shows the chemical pathways followed during the conversion of complex organic material to methane (Siegrist et al. 1993).

Absence of free molecular oxygen is important for the cultivation of anaerobic organisms and even the presence of certain ions which can accept electrons (nitrate, nitrite, sulphate) can discourage methane production since they allow more thermodynamically favourable oxidation reactions to take place. The oxidation-reduction potential of the environment should be around -300 to -400 mV for optimal AD. Above -100 mV sulphate reduction can be used to degrade organic compounds, and above -50 mV (anoxic conditions) nitrate and nitrite ions can be used (Gerardi 2003b).

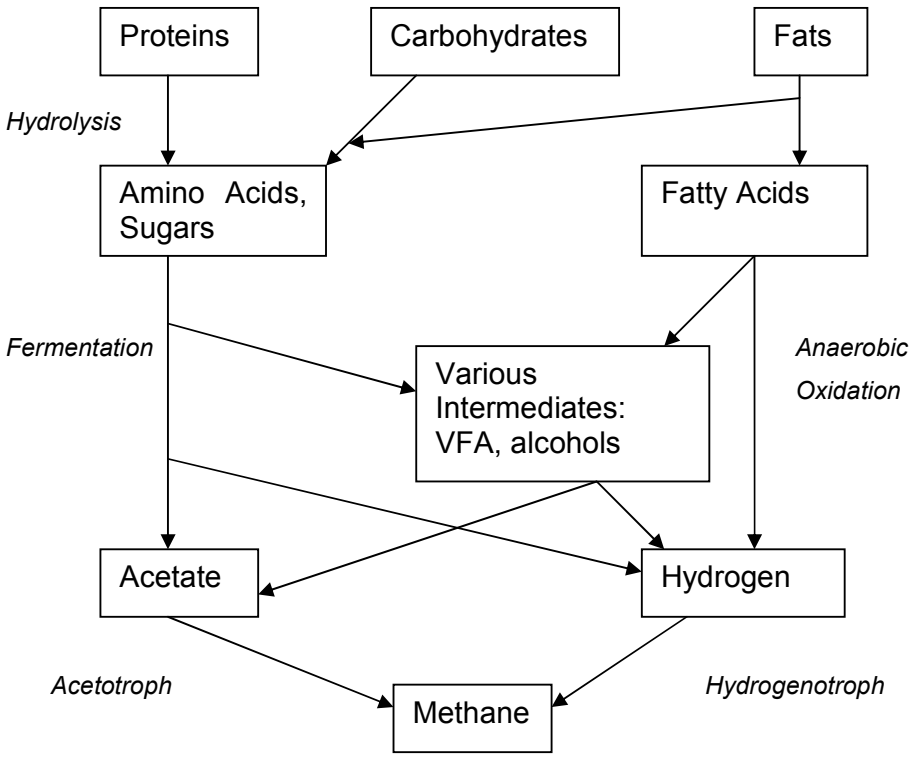


Figure 2 Biodegradation of Complex Organic Matter to Methane

(Siegrist et al. 1993)

2.1.1 Hydrolysis

The process of depolymerisation of particulate complex organic matter into simpler soluble molecules is commonly referred in the literature as hydrolysis. Hydrolytic organisms secrete enzymes called hydrolases and lyases which break bonds in polymeric molecules resulting in shorter chain organic molecules (Mata-Alvarez 2003b). Reactions for the hydrolysis of a simple lipid and of pectin by a lyase enzyme are shown in Figure 3 (Chynoweth and Pullammanappallil 1996). Hydrolysis products include carbohydrates, proteins, and lipids and where possible these are then further broken down into monosaccharides, amino acids, LCFA and glycerol (Vavilin et al. 2008). Although a small proportion may be readily fermentable the bulk of the material that composes BMW requires solubilisation by hydrolytic organisms. For example, a two tonne sample of MSW collected from an Australian waste

transfer station contained 8% readily soluble material (on a chemical oxygen demand (COD) basis) (Nopharatana et al. 2007).

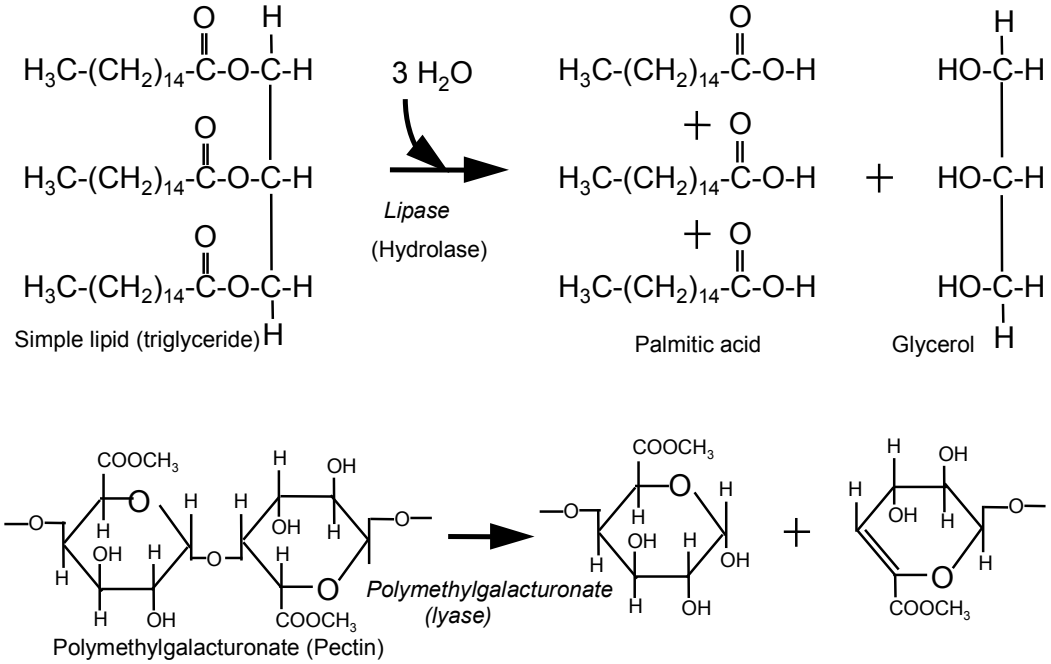


Figure 3 Two Examples of Enzymatic Depolymerisation

(Chynoweth et al. 1993a)

Lignocellulose Degradation

Cellulolytic bacteria, which are the hydrolytic organisms responsible for the breakdown of the various forms of cellulose, perform a vital role in the degradation of BMW since its fibre content can be as high as 71% in the form of cellulose, hemicellulose and lignin (Kayhanian 1995), while lignocellulose digestion can form up to 90% of the methane potential in this type of material (Chynoweth and Pullammanappallil 1996). Cellulose is made up of thousands of glucose monomers whereas hemi-cellulose is made from many different sugar monomers. The main linkages between the monomers in both cases can easily be broken by hydrolytic enzymes. Lignin however can only be degraded slowly under anaerobic conditions (Odier and Artaud 1992) and furthermore the structure of plant material often means that the lignified structure of the cell wall reduces the bioavailability of cellulose and other degradable materials, reducing the total biodegradability and methane potential.

It has been observed in microbiological studies that cellulolytic bacteria act by attaching themselves to the substrate particles using extracellular polymeric substances (EPS), forming a bio-film of cells (O'Sullivan et al. 2005). Only once the full surface area of the available substrate has been covered by the bacteria does the degradation proceed optimally. Microbial hydrolysis occurs through cell-attached (via EPS) or cell-free enzymes, the former being thought to be responsible for up to 90% of the total degradation. In the AD of cellulose-rich wastes it was found that methanogenic bacteria formed ball shaped colonies within the bio-film of hydrolyzing bacteria (O'Sullivan et al. 2005; Song et al. 2005).

Hydrolysis in the Rumen

It has been found that cellulolytic populations in the rumen of animals and in anaerobic digesters are different (Rivard et al. 1991), with the main rumen cellulose degraders coming from the *Fibrobacter succinogenes*, *R. albus* and *Ruminococcus flavifaciens* species, while in digesters *Firmicutes*, primarily *Clostridia* prevail (O'Sullivan et al. 2008). Organisms found in the rumen have a faster colonisation speed and a hydrolysis rate of up to twice that of sewage-based organisms (Song et al. 2005). It has been suggested that the large differences in cellulolytic activity could be explained by the higher biomass concentrations in the rumen compared to anaerobic reactors. In batch trials O'Sulivan et al (2008) found that the 1st order hydrolysis constant of cellulose was much higher for rumen-inoculated vials than those inoculated with material from a biowaste digester and went on to show that the cell density in the media correlated well with the hydrolysis constants. It was hypothesized that if hydrolytic cell densities could be enriched somehow hydrolysis rates typical for the rumen could be realized in industrial applications.

2.1.2 Fermentation, Acetogenesis and Methanogenesis

Acidogenesis or fermentation is the breakdown of soluble materials produced by the hydrolysis process: the main products of these organisms include VFA, alcohols and hydrogen. Fermentation is generally considered to be the fastest of the individual steps in the anaerobic process (Vavilin et al. 2008).

A syntrophic relationship exists between hydrogen producing fermentative organisms and hydrogen utilising organisms such as methanogens which is known as interspecies hydrogen transfer. The methanogens rely on the fermenters/acetogens to provide them with their required substrates of carbonate, hydrogen and acetic acid, while the acetogens rely on the methanogens to remove hydrogen as the chemical reactions they perform are only thermodynamically favourable at very low hydrogen concentrations (Chynoweth et al. 1993a).

The significantly unfavourable thermodynamics of the breakdown of fatty substances means that the anaerobic oxidation of LCFA will proceed at a lower rate without the removal of the shorter chain acids (Fox and Pohland 1994) and hydrogen (Beccari et al. 1996). Furthermore the organisms responsible for the breakdown of fatty acids have some of the slowest growth rates in anaerobic digesters, along with those of acetoclastic methanogens (Zinder 1993). These two groups of organisms are in most danger of being washed out of anaerobic digesters whilst being vital to the maintenance of a healthy digestion process.

Methanogens are from the evolutionary domain of archea, formally archeabacteria, and are distinct from other bacteria in many ways in their biochemistry and genetics. The defining characteristic of this group of organisms is their ability to produce methane (Boone et al. 1993). Many different species of methanogens have been identified, able to degrade a wide range of methylated compounds to methane including hydrogen, carbon dioxide, carbon monoxide, ethanol, methanol, acetate, and formate as well as methylated amines and sulphides (Zinder 1993). Methanogens usually found in anaerobic digesters come from only a very limited section of these (Sekiguchi et al. 2001) and the main reactive pathway is via acetic acid, with the pathway via hydrogen/carbon dioxide (or formate) also being important. Methanogens are grouped as acetoclastic and hydrogenotrophic according to the substrates that they can utilize.

Methanogenic Conditions

Methanogenic organisms are considered the most sensitive of the anaerobes and require particular physical conditions for efficient methane production

(Gerardi 2003c). The most effective methanogens are suited to neutral conditions (Jones et al. 1987) although methanogenic organisms have been found in habitats with pH between 4 in peat bogs (Williams and Crawford 1984) and 9.2 in a hypersaline lake (Mathrani et al. 1988). Methane production can occur at a wide range of temperatures, but optima around 35°C and 50-60°C (mesophilic and thermophilic) mean that most digesters are operated in these ranges (Gerardi 2003d). Methanogenic bacteria are strict anaerobes and will function best at low dissolved oxygen concentrations and are killed by solutions with redox potential above -300 mV.

Competition for Methanogenic Substrates

Methanogenic bacteria can often be outcompeted by other microorganisms for their substrates of hydrogen, carbon dioxide and acetate. Organisms such as sulphate reducing bacteria, hydrogen consuming acetogens and iron reducing bacteria can all utilize methanogenic substrates (Zinder 1993). In substrates containing a large amount of sulphate, sulphate reducing bacteria can compete with methanogens for hydrogen and acetic acid (Paulo et al. 2004) but the amount of acetate used by sulphate reducers decreases as the ratio of acetate to sulphate increases (Bhattacharya et al. 1996).

2.1.3 Inhibition

A substance is said to be inhibitory here and throughout if it causes an adverse shift in the microbial population or reduces the rate of bacterial growth. Many substances are known to be inhibitory to the common types of microorganisms found in anaerobic digesters, and common signs are a reduction in the methane production of the reactors or an accumulation of VFA in the digestate (Kroeker et al. 1979). Inhibitors reported in the literature include ammonia, sulphide, light and heavy metal ions and various organic substances. Although there is agreement that these substances can cause problems in digesters, reported values of inhibitory concentrations vary considerably (Chen et al. 2008) probably due to various antagonistic effects between inhibitors. It is also possible to increase the tolerance to potentially inhibitory substances by gradual acclimatisation of biomass (Cuetos et al. 2008).

Ammonia

Ammonia is the end product of the anaerobic food chain for nitrogenous organic substances (mainly proteins and urea), and therefore problems associated with ammonia inhibition usually occur where a substrate has a high nitrogen content, such as slaughterhouse waste (Cuetos et al. 2008) or poultry manure. Nitrogen is a requirement for microbial growth and therefore concentrations of up to 200 mg l^{-1} ammonia are considered beneficial (Gerardi 2003e). Additionally ammonia provides pH buffering which can provide additional stability against pH drop. Digesters accustomed to low ammonia loads can be successfully acclimatised to higher concentrations (Calli et al. 2005). Free ammonia, which dominates above pH 7.5 is more toxic than ionic ammonia (Kroeker et al. 1979).

2.2 The Hydrolysis Process

During the breakdown of highly lignocellulosic materials such as BMW, the hydrolysis process is not only the rate-limiting step (O'Sullivan et al. 2005), but also determines the maximum degradability of the substrate and the ultimate methane yield (Chynoweth and Pullammanappallil 1996). Optimisation of the hydrolysis process is therefore key to the optimisation of an AD system treating this type of material (Chynoweth et al. 1993a). The rate of hydrolysis can be affected by a number of parameters such as temperature, pH, material composition, available surface area, pre-treatment, acclimatisation of the biomass and the presence of VFA (Gavala et al. 1999).

2.2.1 Particle Size

Biological hydrolysis is a surface related mechanism, in that hydrolytic bacteria are believed to attach to the surface of the material and use extracellular enzymes for digestion (O'Sullivan et al. 2005). Theoretically the particle size of the feed can therefore have a large impact on the rate of reaction since this is directly related to the available surface area. This hypothesis has been proven in a number of experimental studies. Hills and Nakano (1984) found that methane production was inversely related to the product of the particle size and the sphericity of the particles of tomato waste in a continuous process, whereas Kayhanian and Hardy (1994) reported reaction rate to be inversely proportional to the average particle size. In a later study regarding various mechanical treatments on a number of organic materials, it was found that reduction in particle size could improve biogas production by up to 18% and reduce required digestion time by up to 59% (Palmowski and Muller 2000). Similar results were found by Mshandete et al (2006) where a reduction in the size of sisal fibre to 2 mm increased the methane production by 23% relative to the untreated waste; the effect of particle size on the ultimate methane yield in this case is shown in Figure 4

On this basis it has been suggested that in plant design, particle size reduction may present opportunities for increased degradation and biogas production as well as improved reaction kinetics (Delgenes et al. 2003).

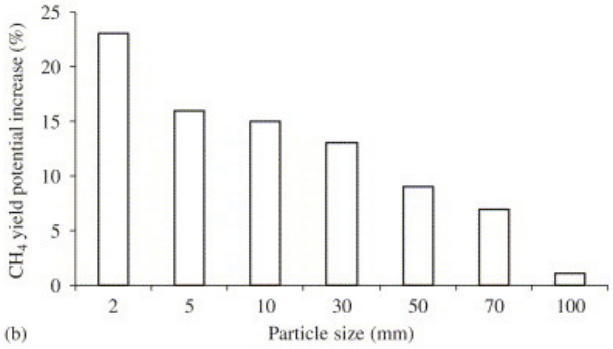


Figure 4 Effect of Particle Size on Methane Yield from Sisal Fibre (Mshandete et al. 2006)

Contrary to this, some work has shown that particle size has very little influence on the rate of breakdown or the ultimate biodegradability of MSW. Nopharatana et al. (2007) found no difference in the lag time, rate constant or methane potential for two samples of the same MSW with 2 mm and 50 mm average particle sizes. One explanation for this could be that a large proportion of MSW is made up of flat particles, such as paper and card, where reducing the size of the major dimension does not expose a significantly greater surface area.

2.2.2 Temperature

Temperature has an impact on the rate of reaction in anaerobic digestion. There are two main temperature ranges of high methanogenic activity, mesophilic (30-35°) and thermophilic (50-60°C), whereas hydrolysis is much less temperature sensitive. The main effect on the rate of reaction comes from temperature based enzyme activity (Gerardi 2003d), and as such can usually be modelled by an Arrhenius relationship. Veeken and Hamelers (1999) found that the 1st order hydrolysis constant increased by 3 to 5 fold in the range of 20 to 40ºC. Above this range the hydrolysis rate appears to continue to increase through to thermophilic temperatures, although it is questionable whether the improved rate would be sufficient to compensate for increased heating costs on a large scale (Llabres-Luengo and Mata-Alvarez 1988).

2.2.3 pH

With regard to pH, hydrolysis optima are reported mainly in the range of 6-7 (Babel et al. 2004; Hu et al. 2004). It has been found that controlling the pH in a hydrolytic reactor can double the biodegradation relative to an uncontrolled process (Zhang et al. 2005). Figure 5a shows how the degree of solubilisation of kitchen waste was changed by pH control over the range of 5 (no control) to 11. Figure 5b shows cellulose degradation by rumen organisms with varying pH (Hu et al. 2004). Many studies use hydrolytic reactors with pH control (e.g. (Gijzen et al. 1989)) to promote optimum hydrolysis.

In an enzymatic study, the effect of pH and acetate concentration on the rate of hydrolysis by the enzyme amylase which converts starch to glucose was studied. pHs of 5, 6, 7, 8 and 9 were considered and the rate of reaction was from higher to lower was pH 7 > 8 > 9 > 6 > 5 measured by concentration of soluble carbon in the reaction vessel (He et al. 2007).

Whilst the above research supports the idea that neutral pH is optimal for hydrolysis, other sources contradict this. In another enzymatic study of the degradation of solid potato waste, amylase was found to have pH optima of 6 and 9, and other hydrolytic enzymes were found to have optima between 5 and 6, except protease which was optimal at a pH of 7 (Parawira et al. 2005).

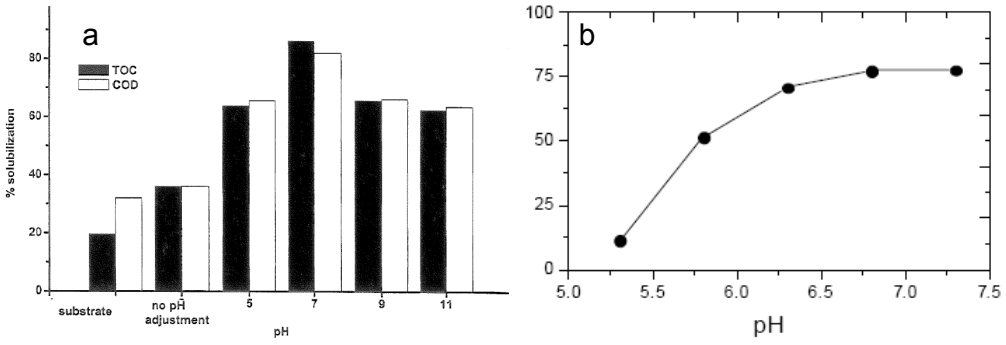


Figure 5 The Effect of pH on the Degree of Solubilisation (Hu et al. 2004; Zhang et al. 2005)

2.2.4 Volatile Fatty Acids

There is some disagreement concerning the inhibition of the hydrolysis process by VFAs as it is not yet clear whether the major effect is the VFAs themselves, or the consequent low pH or a combination of both (He et al. 2007). It has been noted that methanogens, especially acetate degraders, are particularly resistant to VFAs, with concentrations of at least 10 g l^{-1} required to give significant inhibition (Aguilar et al. 1995) as long as pH remains favourable.

A number of authors have commented that the undissociated forms of VFAs, found in greater quantities at lower pH, are much more inhibitory to acidogenesis (Garcia et al. 1991) and hydrolysis (Llabres-Luengo and Mata-Alvarez 1988) than their ionic counterparts. In one microbiological study, undissociated acetic acid was found to completely inhibit acetic acid production from acidogenic bacteria below pH 5, and it was postulated that the acetic acid was responsible for acidifying the cytoplasm and thus restraining the cell metabolism (Baronofsky et al. 1984).

In a study on the anaerobic degradation of pineapple waste, it was found hydrolysis was inhibited by VFA at concentrations of 6000, 12000 and 21000 mg l^{-1} at pH of 5, 6 and 7 respectively. This means as the pH decreases, the sensitivity of the hydrolytic organisms to VFAs increases. This is because the undissociated/dissociated equilibrium is pH dependent, and lower pH means a higher proportion of undissociated VFAs. The authors went on to calculate the concentrations of undissociated acids at the pH and VFA concentrations above and found them to be 2300, 650 and 120 mg l^{-1}, at pH 5, 6 and 7 (Babel et al. 2004). This suggests that hydrolytic organisms are actually more tolerant of undissociated VFAs at lower pH, but the ionic equilibrium means that much higher concentrations will be present for a given total VFA concentration so the observed effect is the opposite. In other work VFAs were found to be inhibitory to cellulose hydrolysis at neutral pH at much lower concentrations than cited above. In the digestion of filter paper cellulose at neutral pH inhibition of hydrolytic and cellulolytic activity began at VFAs of 2000 mg l^{-1} and at 12000 mg l-1 there was no measurable cellulose hydrolysis (Siegert and Banks 2005).

In contrast, other studies have found that VFA have no effect on the hydrolysis process up to concentrations of 30000 mg COD l^{-1} and that the apparent inhibitory effect of VFA is purely a pH related mechanism (Veeken et al. 2000). In this work, the effects on hydrolysis of pH and VFA concentration were isolated by the control of these two parameters during the batch hydrolysis of biowaste. VFA concentrations of 3000-30000 mg COD l^{-1} and pHs 5,6 and 7 were tested and the only statistically significant effect on the calculated hydrolysis constants was from pH. It was suggested that the model proposed by Llabres-Luengo and Mata-Alvarez (1988), in which the hydrolysis rate is inversely proportional to the VFA concentration, was able to successfully predict hydrolysis rates because the pH drop caused by the VFAs themselves was not considered.

Another finding of this work was that at pH 6 and 7, the production of soluble COD was equal to the production of VFA, indicating that hydrolysis rather than acidogenesis/fermentation was the rate limiting step. However at pH 5, COD greater than the VFA concentration was found, indicating that fermentation was the rate limiting step. This finding has also been seen in other systems such as leach beds digesting maize (Cysneiros et al. 2007).

2.2.5 Non-Biological Hydrolysis

Some researchers have considered other options for the replacement or enhancement of the biological anaerobic hydrolysis process. Performance gains have been reported from ultrasonic (Tiehm et al. 1997; Bougrier et al. 2005), alkaline (Chiu et al. 1997; Lin et al. 1997), aerobic (Brummeler and Koster 1990) and thermal pre-treatments. Steam pressure injection has also been used to increase the total methane potential of MSW by 40% (Liu et al. 2002); this treatment however was applied after an initial AD process, to break down lignocellulosic materials and allow a 2nd stage of methane production.

2.2.6 Modelling Hydrolysis

A 1^{st} order kinetic equation is often used to describe the hydrolysis process, based on the assumption that the substrate decay rate is proportional to the concentration of the substrate. Although it has been commented that for

complex organic material 1st order mechanics is not a good model, since substrate degradation appears to depend on other things such as biomass concentration (Vavilin et al. 2008), the 1st order hydrolysis model has become widespread in the simulation of AD processes. It has been suggested that hydrolysis can be described by this type of model only where the reaction rates are limited by the number of active sites on the solid material, and both enzymes and biomass are in excess (Sanders et al. 2003b). Where hydrolysis is rate limiting and no build-up of process intermediates occurs, methane production can be used to calculate the hydrolysis rate. This approach cannot be used however where another stage of process become rate limiting, as was the conclusion of a study on the co-digestion of coffee waste and sewage sludge (Neves et al. 2006). It is probable that in this case the rapid breakdown of readily hydrolysable material meant the methanogenic population was inhibited by a low pH caused by excess VFA production.

Other hydrolysis models have been suggested and used, for example using substrate concentration in conjunction with enzyme concentrations for the prediction of hydrolysis rates of materials containing lignocellulose (South et al. 1995); although when four different models (including 1st order) were compared, they all fitted experimental data for the degradation of swine waste, sewage sludge and cattle manure comparatively well (Vavilin et al. 1996).

2.3 Two-stage Anaerobic Digestion

The underlying concept of two-stage AD is that each reaction vessel can be controlled or operated at the optimum conditions for the particular consortium of bacteria within it. Many different configurations and designs have been proposed for two-stage systems in order to optimize hydrolysis/acidification and methanogenesis. Although conceptually the syntrophic reactions between different sets of bacteria could be disrupted by housing them in separate locations, this rarely occurs in practice since microorganisms are rarely completely segregated. This partial phase separation usually alleviates any problems due to separated syntrophic reactions e.g. hydrogen accumulation (Fox and Pohland 1994).

Single-stage CSRTs dominate the market, with only 10% of the installed capacity currently made up of two-stage systems (De Baere 2000). This is perhaps mainly for economic reasons, since two-stage requires greater capital investment. Some authors have also questioned the performance benefits gained from a two-stage system and remarked that the main advantage is greater process stability at high loading rates (Gerardi 2003g; Vandevivere et al. 2003b), especially for feed materials with high nitrogen content (Banks and Wang 1999). It has also been suggested that two-stage AD is especially useful for dealing with materials with carbon to nitrogen ratio below 10 or with little natural buffering capacity, but a single-stage design is best for ratios above 15 where protein degradation produces ammonium/ammonia which naturally buffers the pH (Weiland 1993).

2.3.1 Two-stage Reactor Types

Two-stage AD systems can be split into three main types, as shown in Figure 6. The simplest form of two-stage reactor system, based on kinetic phase separation, is the combination of two CSTR, plug flow or similar reactors. Logically there is no advantage to this type of system over a single-stage mixed digester, since any loading and/or retention time applied to either reactor is still subject to the same constraints as the single-stage - namely the maintenance of biomass and the rate of hydrolysis of solid waste.

However process benefits have been shown in the application of kinetic phase separation to the AD of Fruit and Vegetable Wastes (FVW) (Bouallagui et al. 2005); process gains in terms of higher OLRs were achieved over the single-stage equivalent (Pavan et al. 1999). This is because of the particular characteristics of FVW where readily degradable carbohydrates make up a large proportion of the material and methanogenesis rather than hydrolysis is the rate limiting step. Therefore the main advantage of the kinetic phase separation is the buffering of the organic loading in the 1st stage, allowing more continuous and homogeneous feeding to the second where the sensitive methanogenic bacteria reside (Lissens et al. 2001). It has been noted that with careful control and proper homogenisation of the feed material, a single-stage reactor should perform as well as its kinetic two-stage equivalent (Vandevivere et al. 2003b).

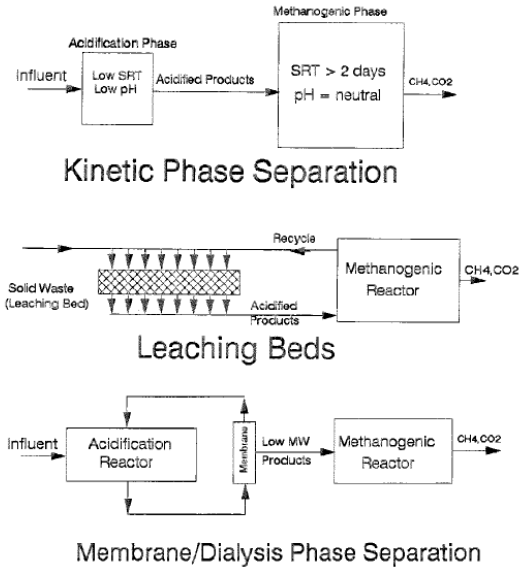


Figure 6 Two-stage Reactor Configurations (Fox and Pohland 1994)

2.3.2 Biomass Retention

With a biomass retention scheme in the methanogenic stage, as shown in the leaching bed and dialysis reactor systems in Figure 6, the HRT in the methanogenic reactor can be reduced below that which the slow growth rate of methanogens allows in a mixed reactor. Various reactor designs exist to perform this function, such as attached growth reactors (for example AFs); and

UASB reactors, where the settlement characteristics of granular methanogenic sludge are used to retain biomass. Also in development are MBRs (Hu 2004), where microfiltration is used to prevent biomass washout.

The use of a biomass retention scheme in the methanogenic stage allows a reduction in overall retention time. Also high biomass concentration reduces the sensitivity of the reactor to toxic effects and allows the application of high OLR (Gerardi 2003g). In an experiment on the digestion of acidogenic effluent from food waste in an UASB reactor, loadings of 15.8 g COD g VS l^{-1} d^{-1} and COD removal efficiency 96% were achieved (Shin et al. 2001).

Most biomass retention configurations, however, can only operate with feed materials containing low suspended solids, meaning that solid materials removed from the 1st stage cannot be input into the 2nd (Fox and Pohland 1994). Therefore alternative processes such as aerobic treatment need to be carried out on the solids to remove residual biodegradability with the result that, while waste throughput is maximized, this is at the price of biogas productivity and lower bio-stabilisation (Vandevivere et al. 2003b).

2.3.3 Leach Bed Reactors

In a leach beds, solid material is fed into a reactor in batch mode and liquid is drained at the base through a mesh of appropriate pore size, producing leachate whilst retaining the bulk of the solids in the reactor. Liquid is fed into the top of the reactor to provide moisture required for digestion. The use of leach beds is mainly confined to three principal modes of operation: single-stage, sequencing batch reactors and in a two stage system linked to a liquid feed reactor. In single-stage mode the quantity of inoculum added is important, since too little inoculum can lead to low degradation being realized due to low pH/high VFA concentration inhibiting the methanogenic population (Lehtomäki et al. 2008). If too much inoculum is used the volumetric gas production of the reactor will be affected since a significant proportion of the working volume will be taken by the inoculum itself. For example in a study on straw digestion in leach bed reactors, the increase of inoculum from 5 to 11% only increased SMP

by 7% leading to a decreased volumetric production (Torres-Castillo et al. 1995).

In sequenced mode the problems of process inhibition and inoculum addition are somewhat alleviated. In this case a bed is packed with fresh organic material and leachate from a previously stabilized leach bed is pumped into the top of the new reactor. This provides inoculum to the new reactor, which goes through an acidic stage followed by a methanogenic stage. At this point the old reactor is disconnected and the leachate is simply recycled around the new reactor until the material within it is stabilised. After this the leachate can be used to inoculate the next reactor (Lai et al. 2001). In this mode of operation, rather than having a hydrolytic and methanogenic reactor, each leach bed goes through stages of mainly hydrolysis and later methanogenesis.

This system is successful since the recycled leachate provides nutrients, microorganisms as inoculum and moisture required for efficient degradation to take place (Chynoweth et al. 1992). Problems were found to occur in a study when a stabilized bed was ineffective as an inoculum source to a new reactor due to the low biomass concentration in its leachate (Nopharatana et al. 1998). In this work it was suggested that methanogenic activity tests be used to ascertain when the start-up of a new bed is possible using the leachate of an old one. Lai et al (2001) suggested that the optimum time for inoculation of new sequenced reactor from an old one is when the methane production rate has peaked. This was based on observations that the cellulolytic activity of the leachate followed closely the methane production rate, since the process at this point was balanced and no build up of soluble COD (SCOD) was observed.

Another mode of operation of leach bed reactors is to feed the leachate to an attached growth methanogenic reactor. When crops and crop residues were digested in a number of configurations including single-stage leach bed, two-stage leach bed with UASB and AF with and without pH control, the two-stage with UASB reactor was found to give the best overall performance (Lehtomaki 2006).

2.3.4 Hydraulic Flush Reactors

The low solids content requirement of a methanogenic reactor with biomass retention means that, when digesting solid waste materials, the hydrolysis/acidification reactor must retain the solid (to allow complete degradation) while allowing solubilised material to leave the reactor to be degraded further to methane. This decoupling of the solid and hydraulic retention time is referred to as a HF. In Figure 6, this is most closely approximated by the membrane/dialysis reactor, although the particular method of solids/liquids separation is not necessarily the same.

A number of pieces of work have attempted to simulate the reactor system denoted membrane/dialysis reactor in Figure 6. A HF reactor, with separation provided by a coarse mesh, was used as a hydrolysis stage in investigations into the AD of abattoir waste (Banks and Wang 1999), wood and paper (Banks and Humphreys 1998) and MSW (Wang and Banks 2000). In each case, the process gains attained were above that of a single-stage CSTR in terms of maximum loading rate and solid breakdown.

2.3.5 Rumen Derived Anaerobic Digestion

Multiple studies have been performed on the application of rumen micro-organisms in a rumen derived anaerobic digestion (RUDAD) system, with very promising results. This reactor configuration is a HF reactor, with solid and hydraulic retention times uncoupled by means of a mesh. These studies have mainly focused on crops and crop residues as well as some work on MSW, and are characterised by good solids breakdown at extremely high loading rates.

The meshes used in these rumen derived processes had a 30 μm pore size. At the reported OLRs of up to 40 g VS l^{-1} d^{-1} and 90 hours SRT, the feed concentration equates to 15% volatile solids (VS), with the in-reactor concentration lower than this due to the 60% VS destruction. No further information on any practical/mechanical issues encountered is given in the published material, although personal communication with the authors revealed that problems were encountered at scale-up of this process because the required area of mesh meant it would occupy a greater volume than the reactor

itself, due to the low flux limitation of the meshes (Gijzen et al. 1987a; Gijzen et al. 1987b; Gijzen et al. 1988; Gijzen et al. 1989; Gijzen et al. 1990; Kivaisi et al. 1992; Kivaisi and Eliapenda 1995; Hu and Yu 2005).

Each of the rumen-derived processes uses a specific nutrient/buffering solution, both to maintain pH at optimum levels and to provide the rumen organisms with the nutrients needed. This maintains digester pH between 6 and 7, thus optimising hydrolysis (Hu et al. 2004). The exact composition of the nutrient medium varies for each study, but of importance are phosphate, carbonate and chloride. Some artificial saliva compositions are shown in Table 2.

2.3.6 Effluent Recirculation

The effects of effluent recirculation in SBR systems are well documented (Lai et al. 2001), but this is not the case for a non-sequenced two-stage system. Recirculation in SBR systems has been shown to improve performance of AD systems, and explanations for this include higher pH caused by maintenance of buffering in the system, no washout of nutrients or biomass except in wasted digestate, small biomass transfers between reactors leading to micro-inoculation, and no loss of partially degraded and/or soluble materials in the wasted methanogenic effluent (Chynoweth et al. 1992; Nopharatana et al. 1998).

When using a rotational drum reactor to decouple the solid and liquid retention times, it was found in a number of pieces of work that the recirculation of methanogenic liquor back into the hydrolysis/acidification reactor increased the VFA production and solids destruction. It was thought that the main enhancement effect of the effluent recycle was the elevation of pH in the hydrolytic/acidogenic reactor, leading to a decrease in the levels of undissociated VFAs (Jiang et al. 2005; Chen et al. 2007).

In a study of papermill sludge digestion in two-stage rumen derived AD (RUDAD) (Gijzen et al. 1990), coupling the hydrolysis reactor with a UASB methanogenic reactor and recycling the process water increased the overall methane production of the system by around 29%. In this work the increased

performance was attributed to the recycling of VFA in the methanogenic effluent which would otherwise have been lost.

Table 2 Artificial Ruminant Saliva Compositions

Source	Composition
(Hu and Yu 2005)	8 g/l $NaHCO_3$, 1 g/l KH_2PO_4, 3 g/l K_2HPO_4, 0.03 g/l $CaCl_2.2H_2O$, 0.08 g/l $MgCl_2.6H_2O$, 0.18 g/l NH_4 $_c$l. Additionally excess $CaCO_3$ in order maintain the pH above 6.0
(Broudiscou et al. 1999)	6.082 g/l $Na_2HPO_4.12$ H_2O, 5.293 g/l $NaHCO_3$, 0.566 g/l $KHCO_3$, 0.363 g/l NaCl, 1.333 g/l NaOH
(McDougall 1948) used in (Rufener Jr et al. 1962)	9.3 g/l $Na_2HPO_4.12$ H_2O, 9.8 g/l $NaHCO_3$, 0.47 g/l NaCl, 0.57 g/l KCl, 0.04 g/l $CaCl_2$, 0.06 g/l $MgCl_2$
(Rufener Jr et al. 1962), used in (Gijzen et al. 1989)	3 parts as above to 2 parts water
(Gijzen et al. 1987b)	As above but with additional 1.5 g/l NH_4 $_c$1 as a nitrogen source. additional trace elements at 0.2 mg/l as per (Vishniac and Santer 1957).

In the field of landfill research, the effect of leachate recycle is an increase in the waste degradation rate and generally the leachate is simply recycled from the bottom of the site, to the top. However, in one study, the effect of no recycled leachate was compared with both leachate recycling and recycling of the effluent from a methanogenic reactor fed on the leachate. The amount of methane produced was in the ratio 1.0:2.2:173 for no recycle, recycle and sludge recycle respectively: the main effect in this case was the inoculation of the simulated landfill and respective reduction in VFA concentrations (Bae et al. 1998).

2.4 Biodegradable Municipal Waste

2.4.1 Composition and Characteristics

The performance of an AD system is obviously dependent on the feed material with which it is supplied. In order to create a material representative of BMW in the UK with which to feed the digesters in this project, a literature search was performed on this topic. Unfortunately, few studies could be found giving detailed compositional data of this part of municipal waste.

In 2005, Open University students throughout the UK participated in a study looking at the amounts of waste generated and recycled (Jones et al. 2006). Students were required to log the weights and types of waste over four one-week intervals. The results from this study gave BMW as 68.1 % of total MSW generated and are shown in Table 3.

A report produced for the Welsh National Assembly in 2003 (Poll 2003) also gives detailed data on the composition of MSW. Nine out of 22 local authorities, selected to be representative of the whole of Wales, participated in the project and a total of 174 tonnes of waste was logged. The data presented in Table 3 represent the averages of 4 collections evenly distributed between October 2002 and July 2003 and show BMW to be 60.8 % of total MSW.

Another detailed study was performed by the Resource Recovery Forum (RRF) focusing on kerbside collection of dry recyclables. The collection area of Eastleigh, Hampshire was selected for monitoring for two collections (April and September). This area was chosen on the basis of the well-established dry recyclables collection scheme and the mixed socio-economic profile. Results for the organic fraction of waste collected (neglecting dry recyclables) are shown in Table 4 and give BMW as 48.8% of total MSW. This proportion is significantly lower than in other studies because dry recyclables were collected separately.

Table 3 Composition of MSW in the UK

(Jones et al. 2006) and (Poll 2003)

Waste Component	% By Weight of MSW (Jones et al. 2006)		Waste Component	% By Weight of MSW (Poll 2003)
cardboard & paper packaging	7.3		Newspaper and Magazines	9.4
non-packaging paper	13.1		Recyclable Paper	2.0
dense plastic packaging	3.7		Cardboard Boxes	6.1
miscellaneous plastic	2.1		Other Paper and Card	6.2
ferrous packaging	2.1		Refuse Sacks and Bags	1.9
aluminium packaging	1.0		Packaging Film	1.9
miscellaneous metal	2.6		Other film	0.2
glass packaging	7.9		Dense Plastic Bottles	2.5
textiles	2.1		Other Packaging	2.1
putrescible kitchen waste	15.7		Other Dense Plastic	1.5
garden waste	25.1		Textiles	2.4
sanitary wastes	2.6		Other Combustibles	2.1
misc. combustible waste	6.8		Packaging Glass	6.7
misc. non-combustible waste	4.7		Non Packaging Glass	0.5
fines	2.1		Garden Waste	8.3
			Kitchen Waste	25.0
Total BMW	**68.1**		Other Organics	1.8
			Ferrous Cans	2.5
			Other Ferrous Metal	1.1
			Non-Ferrous Cans	0.5
			Other Metals	0.5
			Fines	4.6
			Other	3.3
			Total BMW	**60.8**

Table 4 Composition of BMW from Eastleigh Not Including Dry Recyclables (RRF 2001)

Waste Component	April (%) By Weight (Of BMW)	September (%) By Weight (of BMW)	Average (%) By Weight (Of BMW)
Newspaper	6.9	13.3	**10.1**
Magazines	6.5	5.1	**5.8**
Recyclable Paper	4.2	3.5	**3.8**
Card and Paper Packaging	7.1	3.1	**5.1**
Cardboard	1.2	0.0	**0.6**
Card Non-Packaging	0.6	0.0	**0.3**
Liquid Cartons	0.9	0.5	**0.7**
Non-Recyclable Paper	13.8	29.1	**21.4**
Garden Waste	0.7	21.0	**10.8**
Kitchen Compostable	6.4	24.4	**15.4**
Kitchen Non-Compostable	51.8	0.0	**25.9**
Total BMW	47.0	50.6	**48.8**

Reported values of BMP, considered an important parameter in AD, are summarised in Table 5.

Table 5 Biochemical Methane Potential for MSW

Source	Type of Waste	BMP ($l\ g^{-1}$ VS added)
(Chynoweth et al. 1993b)	MSW (Various)	0.206-0.292
(Nopharatana et al. 2007)	MSW	0.24
(Zhang et al. 2008)	MSBMW	0.333-0.342

2.4.2 Anaerobic Digestion of Municipal Solid Waste

A summary of data from other continuous AD studies on BMW and similar feedstocks is shown in Table 6. There is a large variation in performance in terms of VS destruction, biogas/methane production and maximum OLR. These differences can probably be attributed to variation in composition of the

feedstock and the effects that this can have on the digestion process. For example a waste containing a high proportion of wood/paper/card material such as used by Banks and Humphreys (1998) not only shows low degradation due to its high lignin content but the consequent low nitrogen content means the material provides little buffering in the form of ammonia, and therefore can only be digested at a low OLR. A variety of operating conditions also can explain some of the variation between different AD studies. Unfortunately this means that only limited conclusions can be drawn from comparison between AD experiments with different feed materials.

Table 6 Digestion Characteristics of BMW and Similar Wastes

Source	Feedstock	% VS Break-down	Sp. Gas Prod. ($l\ g^{-1}$ VS added)	Max OLR (g VS $l^{-1}\ d^{-1}$)
(Hartmann and Ahring 2005)	MSW + Manure	69-74	0.67 (biogas)	4
(Gallert et al. 2003)	BMW	-	0.43 (methane)	9.55
(Davidsson et al. 2007)	SSBMW	80	0.35 (methane)	2.8
(Wang and Banks 2000)	MSW	71.5	0.18 (methane)	4.95
(Wang and Banks 2000)	MSW	75	-	15
(Bolzonella et al. 2003)	MSBMW	-	0.23 (biogas)	9
(Bouallagui et al. 2004)	FVW	-	0.44 (methane)	5.5
(Banks and Humphreys 1998)	Paper +Wood	53	-	1.5
(Chanakya et al. 1992)	FVW	95	0.42 (methane)	5.65
(Vaz et al. 2008)	SSBMW	76	0.41 (methane)	2.1

2.5 Membrane Bioreactors

The technology of Membrane Bioreactors (MBR) is growing rapidly on a worldwide scale (Yang et al. 2006), both in research and in full-sized plant installations. The majority of the market for these products is in the wastewater treatment industry, where an MBR plant can replace a conventional sewage works with a fraction of the land use, provide a much cleaner effluent (Yang et al. 2006) and reduce both energy use and sludge production (Bohdziewicz et al. 2008).

Much of the current research into anaerobic MBRs is directed toward the treatment of liquids or suspensions of low solids such as domestic sewage (Saddoud et al. 2007), industrial wastewaters (Choo and Lee 1996), and landfill leachate (Bohdziewicz et al. 2008) as well as a range of synthesised solutions (e.g. VFAs, glucose, sucrose). The applied membranes usually have pore sizes around 0.05-0.2 μm and most are made from organic materials such as polyamide, polyethylene, polyethersulphone and polyvinylidine fluoride (PVDF), although some are made from ceramic and stainless steel. The main topics of current MBR research are into operating conditions (such as HRT, SRT, OLR); performance (COD removal, methane production); flux maintenance and cake formation/fouling. Flux maintenance can be achieved by powdered activated carbon (PAC) addition (Akram and Stuckey 2008), gas sparging (Psoch and Schiewer 2006), back washing (Vargas et al. 2008), membrane rotation (Wu et al. 2008), and chemical additions (Koseoglu et al. 2008) as well as physical and chemical cleaning (Jeison and van Lier 2007), whilst fouling is mainly caused by attached biomass/EPS and struvite precipitation (Choo and Lee 1996).

The concept of critical flux was developed to aid the application of microfiltration membranes (Field et al. 1995), the hypothesis being that there exists a critical flux below which no irreversible fouling occurs. This work was done using membranes of pore sizes 0.14-0.2 μm, the filtrate being a yeast suspension. The results suggested that using constant flux filtration is superior in terms of flux maintenance compared with constant pressure filtration. Whilst much research into MBR technology is taking place, very little literature exists on

membranes/meshes for the filtration of high solids systems, to which the critical flux concept does not necessarily apply.

2.5.1 Aerobic Mesh Bioreactors

A number of publications address the use of mesh filters (30-100 μm pore size) for sludge removal in a so-called Mesh Filter Bioreactor (MFBR). These units have the potential to provide wastewater treatment, but do not produce the effluent quality of an MBR. Instead they produce similar quality effluent to traditional waste activated-sludge plants, at reduced land-use and cost (Kiso et al. 2005). These reactors avoid the major downfall of MBR; the high cost of the membranes and fouling problems since the mesh materials are significantly cheaper and the cake build-up and fouling layer performs a vital role in the filtration process. The cake layer reduces the effective pore size and increases filtration of smaller suspended particles (Kiso et al. 2000; Wang et al. 2006), thus no backwashing is performed.

This reduction in effective pore size has been extended to the concept of a self-forming dynamic membrane in which the colloidal material in the wastewater itself forms the active filter medium, supported by a more permanent and coarser meshes. In the work by Fan and Huang (2002), a 100 μm mesh was used to filter wastewater using only the pressure head of the water (TMP (Trans-membrane Pressure) was usually less than 10 Pa). Upon installation, or after cleaning the mesh, the dynamic filter formed over a period of around 30 minutes during which time the effluent was similar in characteristics to the influent and it was suggested that recirculation back to the inlet would be prudent during this stage. After this, for up to two days, filtration took place by the dynamic layer, which removed almost all of the suspended solids before eventually blocking the filter. It was found that back-aeration (in the opposite direction to wastewater flow) was sufficient to recover the meshes to their original state.

Satawali and Balakrishnan (2008) used a 30 μm mesh to filter a mixture of synthetic wastewater and brewery wastewater and found that at low reactor suspended solids (4-5 g l^{-1}) the mesh lasted in excess of two months before

replacement was necessary, whereas this time was reduced to two weeks when the suspended solids increased (10-12 g l^{-1}). Fuchs et al (2004) found that mesh blockage occurred suddenly and unpredictably after any time between a few days to 2-3 weeks when feeding on wastewater with a high suspended solids concentration. The time to blocking seemed to have no relation to the filtration rate, the digestate solids concentration or the aeration rate. High fluxes, up to 150 l m^{-2} h^{-1} were reached at low transmembrane pressures (TMP) of 30-100 Pa.

2.5.2 Anaerobic Mesh Bioreactors

The rumen studies on BMW (Gijzen et al. 1987b; Gijzen et al. 1989) both used the same 30 µm submerged filtration unit, as described in earlier work (Gijzen et al. 1986). A cylinder 6 cm high and 4 cm diameter was constructed of 0.3 mm steel mesh. This was then coated in a single layer of 30 µm nylon gauze. The total effective mesh area was around 90 cm^2. Personal communication with one of the authors revealed that the mesh was brushed daily to reduce fouling. This mesh was sufficient in all cases to filter the reactor contents and reduce the HRT to around 12 hours. In a 1.5 l working volume reactor, this equates to a total flux of 13.8 l m^{-2} h^{-1}, which is a relatively low figure compared with others in the literature.

This process was found to be unsuitable for scale-up since the ruman ciliates, crucial to the process, were too sensitive to pH changes and the presence of VFA, meaning a low HRT was obligatory and the mesh was unable to sustain the required flux for reactors above laboratory scale.

Dalhoff et al (2003) used a 1 µm polyethylene submerged membrane unit to run an anaerobic digester similar to the RUDAD reactors. Grass was used as the substrate. Physical cleaning of the membrane surface was more effective for flux maintenance than gas backwashing, but both were only able to maintain a flux of approximately 20 l m^{-2} h^{-1} for a period between cleaning of 4 hours. Greater flux of 120 l m^{-2} h^{-1} was obtained by decreasing the period between backwashing to 15 minutes. The difference between the concentration of VFA in

the reactor and filtrate was approximately 30%. The main loss of flux was caused by cake layer build-up rather than internal pore clogging.

2.6 Conclusion

Since only a handful of studies exist on the use of mesh filter bioreactors for the anaerobic digestion of solid waste materials, these being generally only preliminary and specialised in their purposes, it has been sought in this section to present the broad knowledge base that exists in the literature in the subject areas that overlap or are fundamental to the proposed thesis topic.

The first two sections of this chapter represent the building of a qualitative physical model of the most relevant aspects of the AD process. In section 2.1 the background microbiology was discussed, while in the focus in section 2.2 was anaerobic hydrolysis since this is considered the rate limiting step in the degradation of BMW. The knowledge presented is fundamental to the understanding of an AD system and its purpose in this work is to allow in-depth discussion of the results obtained.

A review of the existing work into two-stage AD is included in section 2.3. Analogies can be drawn between systems in this work and those in other studies despite them being not functionally equivalent, thus allowing further insight the experiments performed. In particular work which involves some sort of biomass or solids retention (e.g. leach bed, attached growth and hydraulic flush reactors) as well as where process effluent recirculation has been studied lends itself to comparison in this way. Similarly the work presented in section 2.5 on the use of membranes and meshes in various biological systems enhances the discussion of the experiments performed due to the comparison of analogous parts of the systems and additionally allowed the acquisition of preparatory knowledge of how continuous filtration in meshes can be sustained.

The few studies looking into the use of MeBR or similar reactors for the anaerobic digestion of high solids materials (see section 2.5.2) show promising results and yet this area has not been explored further. The series of papers into the RUDAD process by Gijzen and other researchers (Gijzen et al. 1987a;

Gijzen et al. 1987b; Gijzen et al. 1988; Gijzen et al. 1989; Gijzen et al. 1990; Kivaisi et al. 1992; Kivaisi and Eliapenda 1995; Hu and Yu 2005) represent almost all of the available knowledge on this topic but because of the use of a specialist and relatively sparsely available inoculum source (Rumen contents) have not explored fully the use of these type of reactors.

MeBRs show potential to provide an enhanced anaerobic digestion process but the further research is required especially with the use of a widely available inoculum source but also in other areas such as; the effect on the process of such parameters as HRT, OLR, MeBR mesh pore size; possible scale up issues and reactor designs; greater understanding of the filtration process and its effect compared to other solid/liquid separation methods; the addition of a 2nd stage biomass retention reactor and the effect of methanogenic effluent recirculation. The subsequent chapters of this thesis attempts to address these points through a series of laboratory experiments and discussion of the results obtained.

3 Equipment and Methods

Anaerobic systems fed on SBMW were studied using a series of controlled bench-scale experiments. Reactors of working volumes ranging from 15 ml to 5 litres were used and analysed for a variety of physical and chemical parameters in order to assess and compare the overall performance.

3.1 Anaerobic Reactors

In this work, 5 different reactor types were used, the designs of which are shown in Figure 7-Figure 13. The HF, MFBRs and rotating drum mesh bioreactors (RDMBR) were all designed and built specifically for this project.

Temperature control was provided by one of two methods. In the CSTR and AF reactors copper coils were placed around the reactors which were enclosed in an insulated wooden box. Water was heated and pumped around these coils using a thermo-circulating pump with an electronic temperature sensor. The temperature of the HF, mesh, rotating drum and small flask reactors was controlled by placing them in a water bath.

Collection of biogas was in most cases done using 3-10 litre Tedlar Bags, which were connected to the reactors by PVC tubing. Adequate stopcocks and valves were placed in the tubing such that the bag and/or reactor could be isolated for sampling and measurement of gas production. Reactors and tubing were checked for leaks under a small positive pressure before use.

3.1.1 Continuously Stirred Tank Reactors

The 1.5-litre CSTR design is shown in Figure 7. These reactors were used for the single-stage trial. 5-litre and 0.5-litre versions of this reactor were used for the 1st and 2nd BMP test respectively, the designs of which differed slightly from that of the 1.5-litre CSTRs but were functionally the same. The 1.5-litre reactors could be opened easily for feeding and sampling as the lid was sealed to the reactor body by a neoprene o-ring, and clamped down using bars attached to the wooden casing (not shown in Figure 7).

A draught tube was inserted through the lid of the reactor to house the stirring axle and was secured and sealed to the lid by a gland connector. Stirring was at 30 revolutions per minute (RPM) performed by a 12V DC motor connected via the axle to a stainless steel frame inside the reactor.

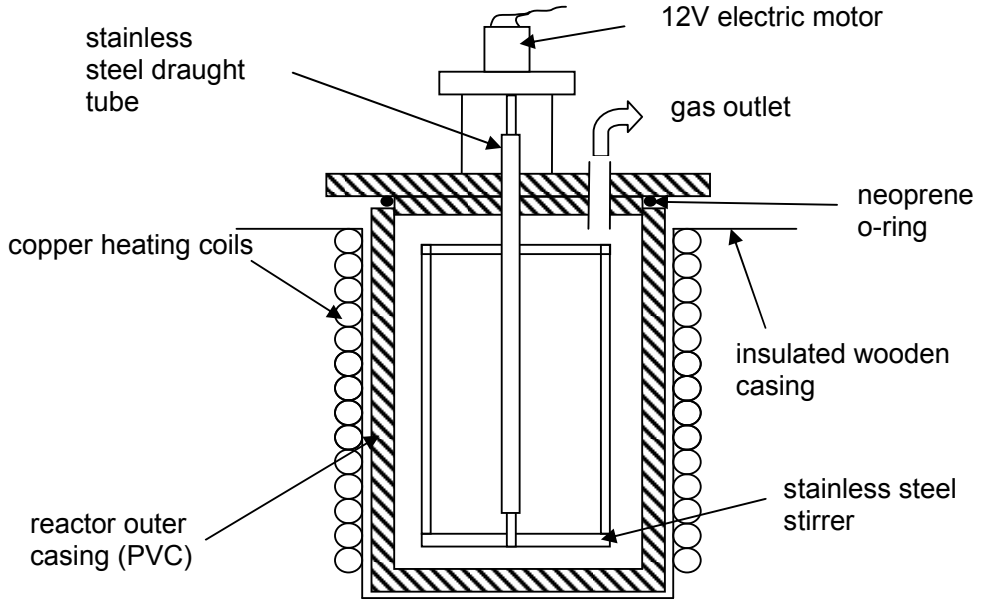


Figure 7 Continually Stirred Tank Reactor (CSTR) Design (1.5 and 5 litres)

3.1.2 Hydraulic Flush Reactors

Figure 8 shows the two types of HF reactor used (Type A and B). The two designs were functionally equivalent. The body of the reactor was simply a 1-litre centrifuge bottle (Nalgene, Hereford, UK). This meant the whole reactor could be centrifuged daily with minimal loss of contents. As with the CSTRs, stirring was performed by 12V DC motors with draught tubes, but this time using PVC stirring frame.

Type A was the original design used in the early single-stage HF trial, and had a tendency to leak as the cyclic transverse loading generated by the stirring of thick digestate tended to loosen the bung and allow some biogas to escape. The Type B modification created a more rigid seal between the bottle and the lid and thus was superior for the measurement of biogas composition and production. This design was used for the two-stage HF work.

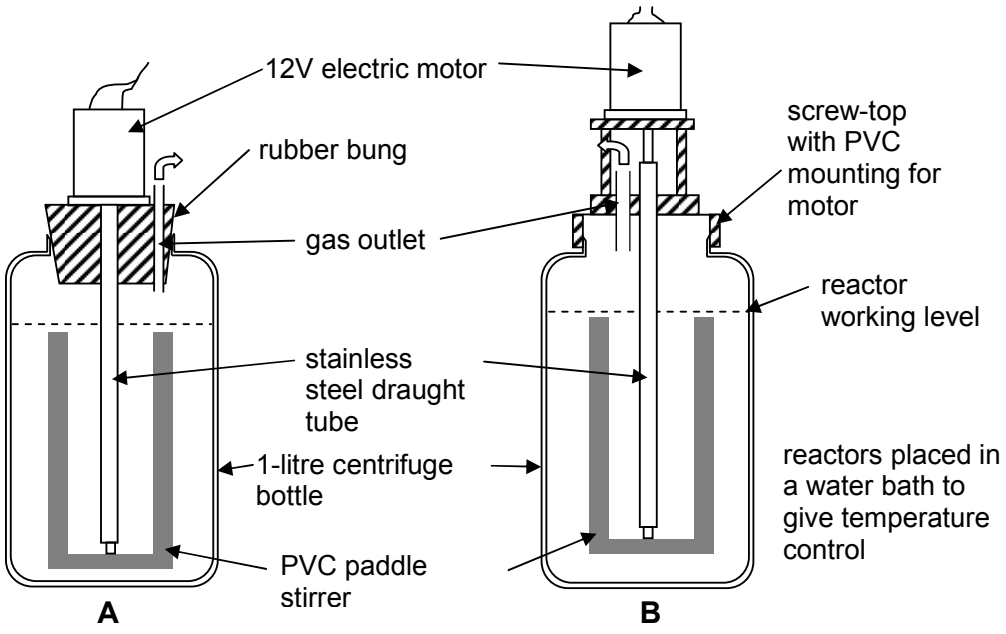


Figure 8 Hydraulic Flush (HF) Reactor Design (0.7-0.8 Litres)

3.1.3 Anaerobic Filter Reactors

The AF reactor design, used in the two-stage trials, is shown in Figure 9. The reactor was filled with a mixture of proprietary filtration media (Flocor, UK). Liquid or digestate with low solids content was pumped from the influent storage bottle by a peristaltic pump into the bottom of the reactor. The liquid then flowed through the filter medium until it reached the overflow tube at the top of the reactor where it drained into the effluent storage bottle. The pump speed was used to set the daily flow rate.

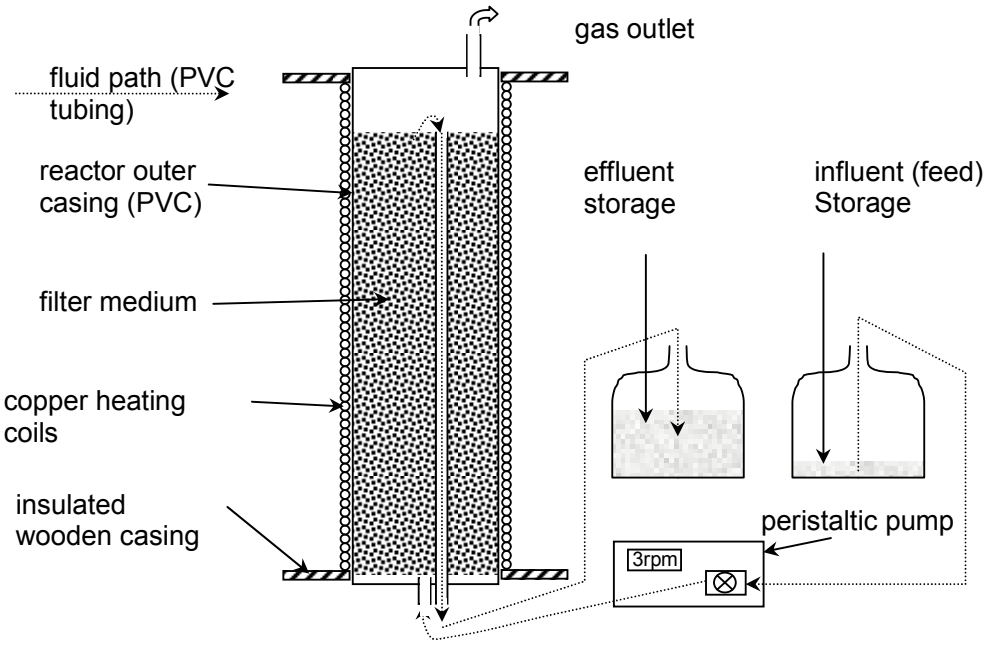


Figure 9 Anaerobic Filter (AF) Reactor Design (4 litres)

3.1.4 Mesh Filter Bioreactors

The design of the MFBR, which was a modified version of the 1.5-litre CSTR, is shown in Figure 10. The modification involved the addition of a mesh unit, the details of which are shown diagrammatically in Figure 11 and photographically in Plate 1. Other additions included liquid influent and effluent lines through the reactor lid and a nylon brush attached to the stirring frame. The mesh unit, which was made from a length of PVC tubing, was attached to the draught tube using gland connectors with rubber seals. A nylon mesh (Fisher Scientific, Loughborough, UK) was wrapped around the unit and attached using superglue. The brush was orientated such that the bristles gently scoured the surface of the mesh as the stirring frame rotated, removing any external fouling. Filtrate was pumped though the effluent line to a storage bottle using a peristaltic pump and the liquid removed in this way was replaced by liquid of equal volume though the influent line. The total filtration area was 14.1 cm^2.

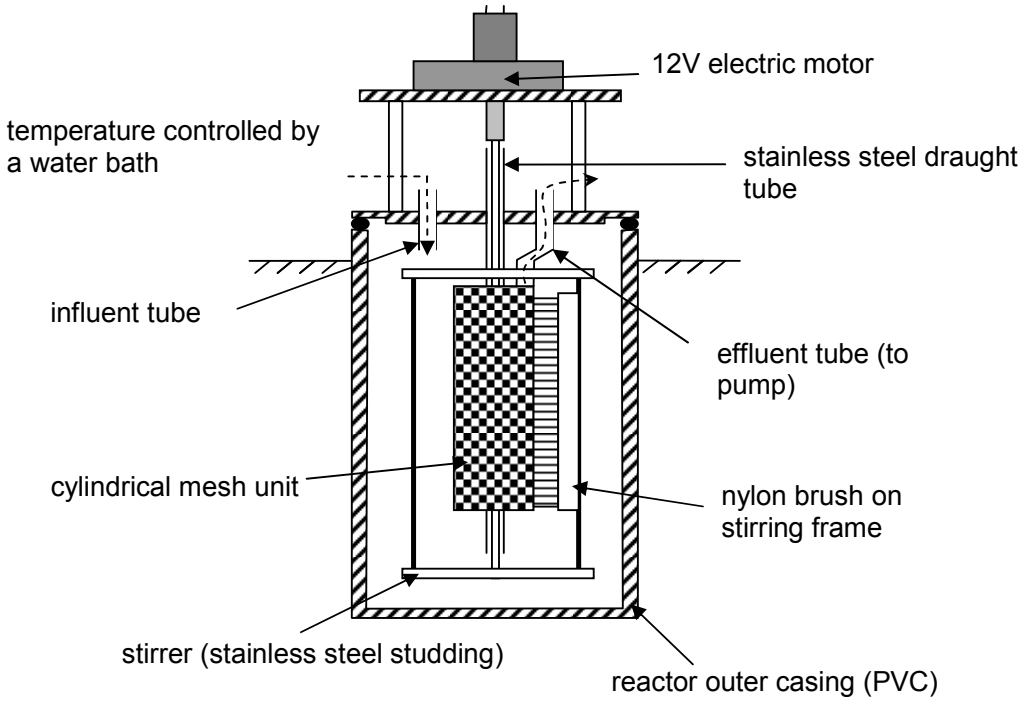


Figure 10 Mesh Filter Bioreactor Design (1.5 litres)

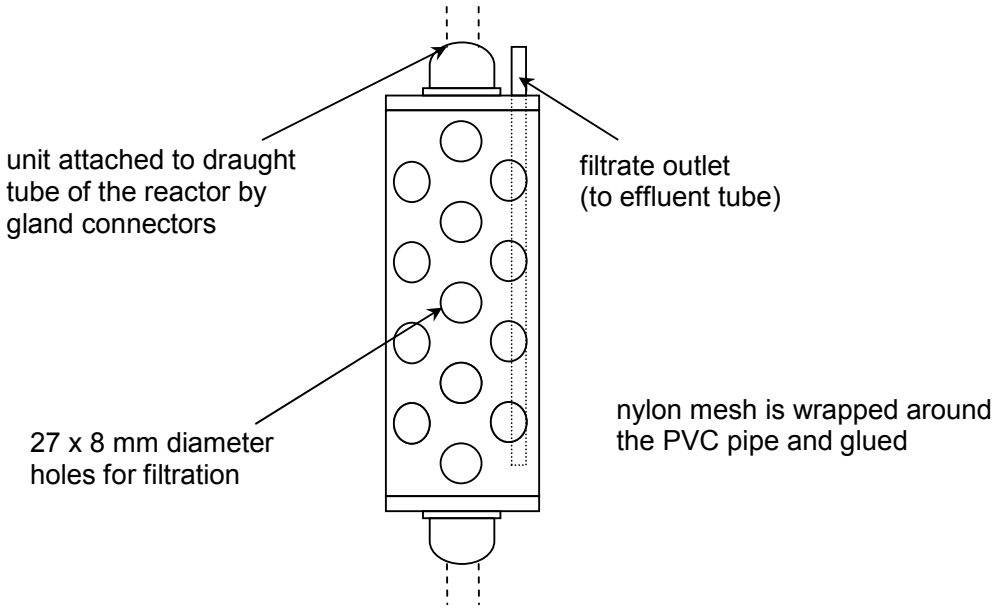


Figure 11 Mesh Unit Detail

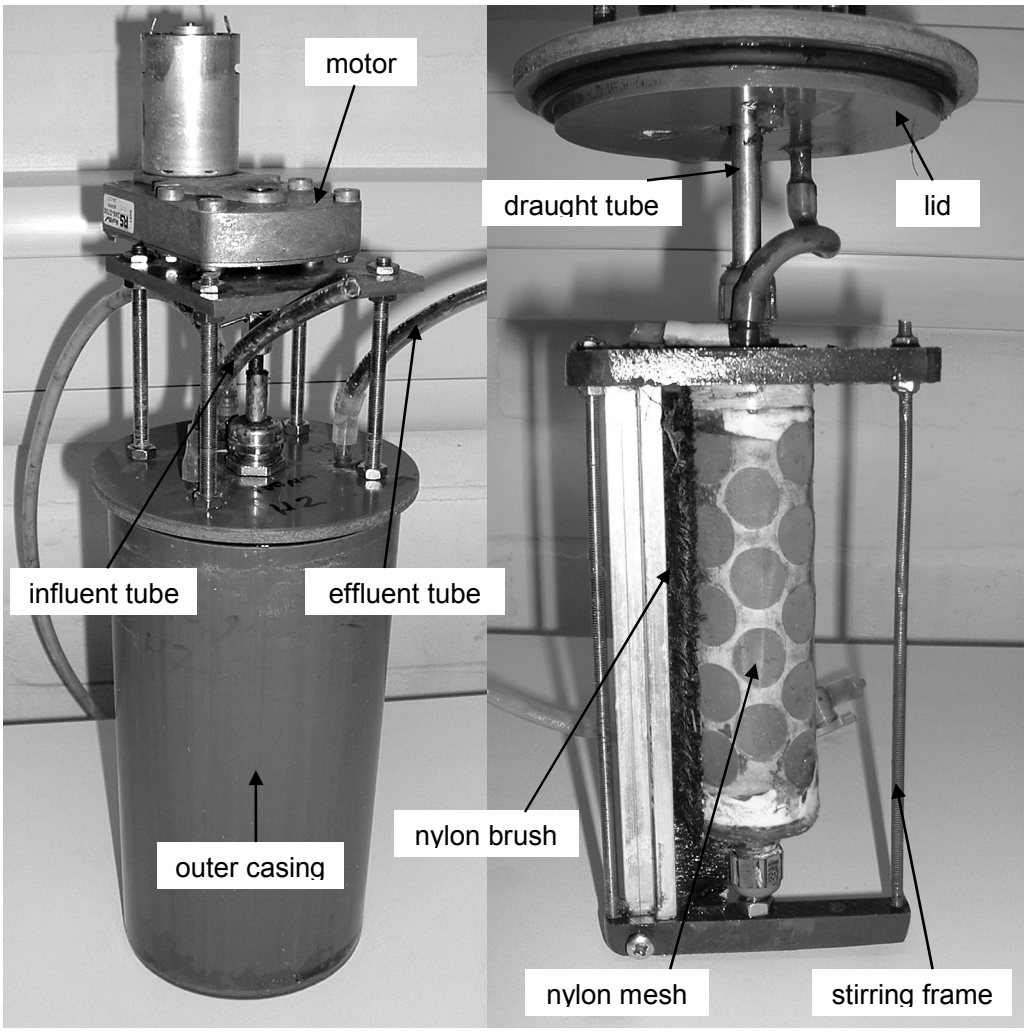


Plate 1 MFBR Reactor Photos

3.1.5 Rotating Drum Mesh Bioreactors

The RDMBR had the most complex design of the reactors used in this work. The full design is included in appendix 8.1 and only a brief description of the functionality is discussed here. A cross-section of the reactor, somewhat simplified, is shown in Figure 12. The reactor was made of two main parts: the rotating drum and the outer casing.

The 2-litre drum was attached to the outer casing such that it could rotate on a horizontal axis. Some of its outer surface was cut away and replaced with nylon mesh sections supported by stainless steel mesh sections of the same dimensions but with much larger pores (~1 mm), the total filtration area was 360 cm^2. Bars were fixed inside the drum, which were designed to agitate the digestate inside. An influent tube was fixed into the outer casing and entered

the drum via a sealed bearing allowing influent delivery to the drum interior. At the other end of the axis, a drive shaft was attached to the drum supported at the outer casing using another sealed bearing. At the end of this shaft a pulley wheel was driven by an electric motor attached to the top of the outer casing. One section of the surface of the drum was made into a hatch to allow access to the interior of the drum for feeding and digestate removal.

The outer casing was made from PVC and was essentially a box with a removable lid. Six wing nuts were used to compress a neoprene gasket between the lid and the rest of the box to ensure a gas-tight system. The casing was placed in a water bath to control the reactor temperature. Filtrate dripped down from the drum and collected in the outer casing until it was pumped out of the reactor via an effluent tube. Another tube coming out of the lid of the reactor allowed gas collection.

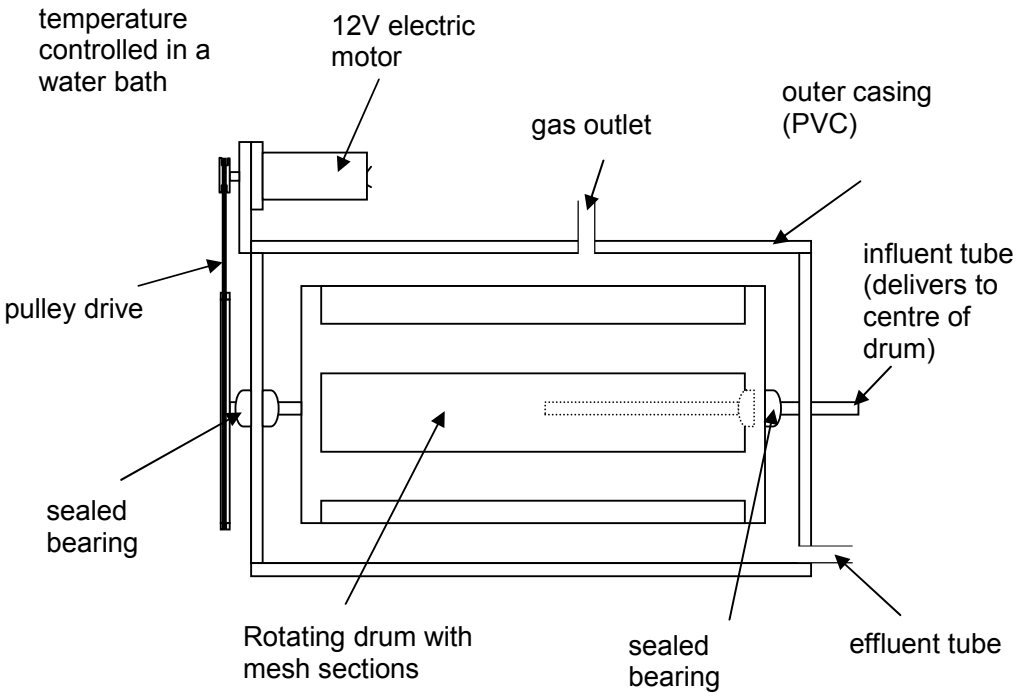


Figure 12 X-Section of RDMBR Design (1.5 litres)

(Simplified – see section 8.1 for full design)

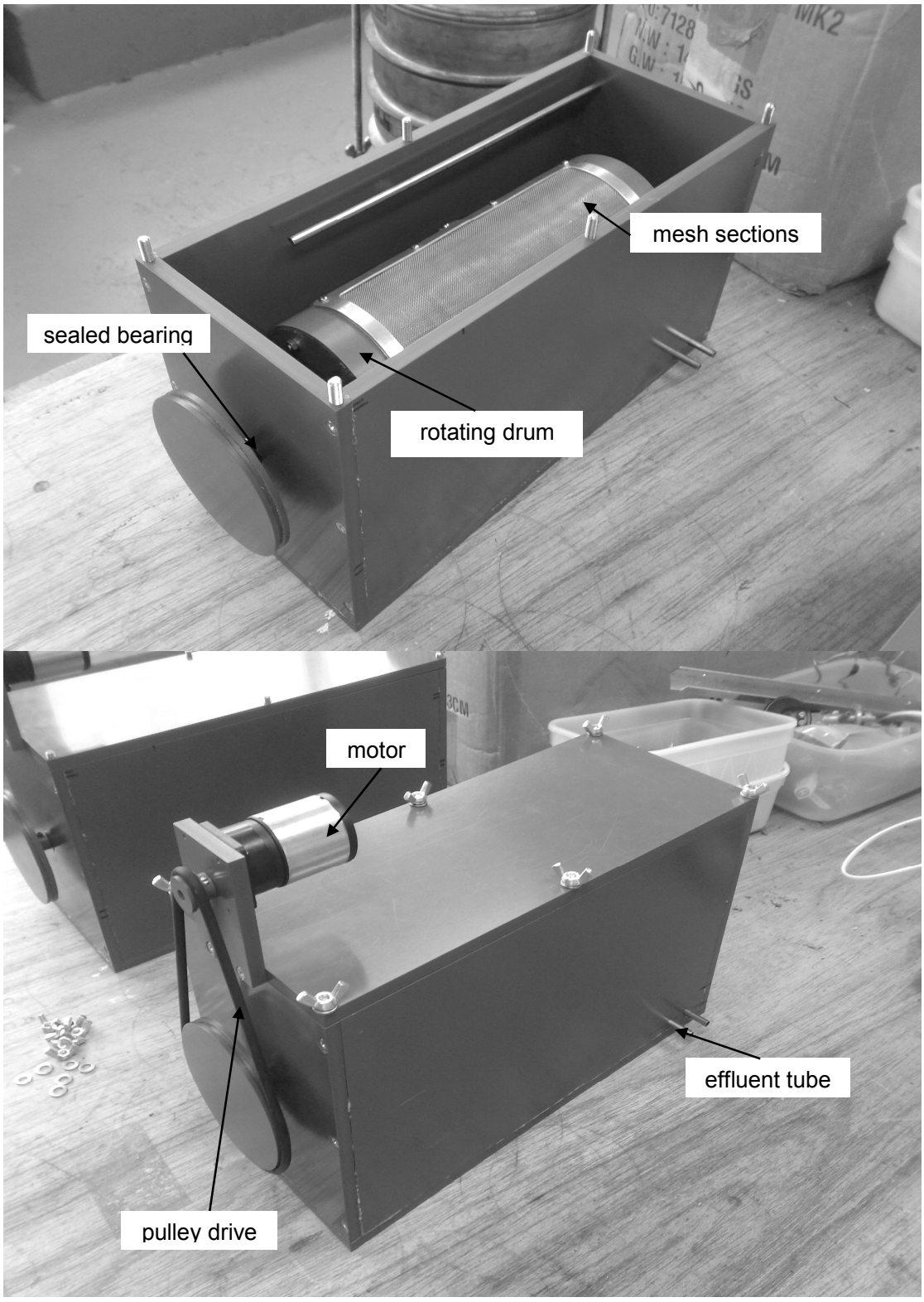


Plate 2 RDMBR Photos

3.1.6 Small Flask Reactors

Small flask reactors were used to measure residual BMP of digestate during the RDMBR trials: the design is shown in Figure 13. These were placed in a shaking water bath to provide temperature control and mixing. The construction involved modifying the lid of a 25 ml McCartney bottle to include a gland connector with a stainless steel tube for collection of biogas. Initially a needle and silicone septum was used as the gas line, but this proved to be unsuitable since movement of the bottles caused the septa to leak. The useful working volume of the bottles was approximately 15 ml, allowing enough headspace to ensure the digestate or contents did not block the gas line and cause a pressure build up inside the reactors. This was essential since the bottles were made from glass.

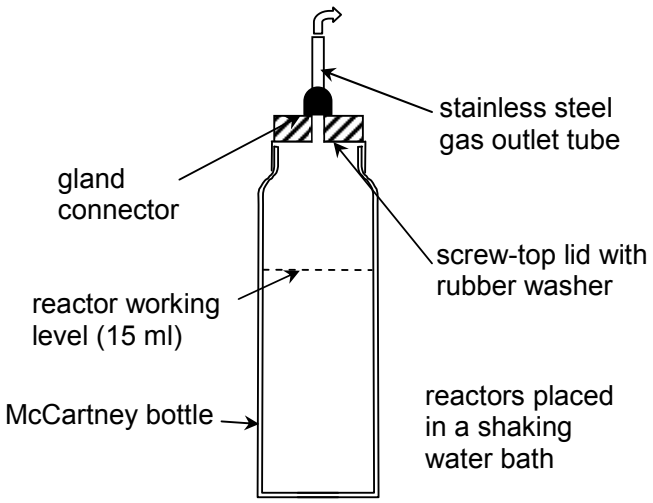


Figure 13 Small Flask Reactors (15 ml)

3.2 Feedstock Material and Inoculum

Feedstock

It was decided that a synthetic rather than real waste would be used for the digestion trials in this work. This decision was based on the fact that real biodegradable waste shows variation depending on a number of factors including time of year, collection method and affluence of the area of collection. Collection of a single batch of real waste for use over the whole project, while avoiding the variability problems, was not possible due to freezer space limitation. Use of a synthetic waste recipe allowed production of a material with repeatable and reproducible characteristics. This meant that different digestion experiments performed over the period of this work could be compared meaningfully. Additionally the risk of exposure to pathogens present in real BMW was avoided.

The digester feed material, SBMW, was made using components of kitchen waste, green waste, paper and cardboard in the proportions shown in Table 4. This recipe was chosen since it was thought that the waste audit from which these compositional data were taken best represents the composition of BMW in the UK because

- The collected waste came from a collection area with a mixed socio-economic population chosen for its representation of the UK population.
- Two collections took place, in spring and autumn, taking into account some aspect of seasonal variation.
- The audit took into account the separate collection of dry recyclables, important since all collection authorities in the UK provide some method of recycling these materials (Defra 2007a).

The kitchen/food waste used was sourced from the University of Southampton Staff Club, which includes a number of different catering establishments. The waste was collected in February and March of 2005 from the kitchen and dining areas of the Staff Club, where dedicated and labelled bins were placed for use by both staff and customers. Contaminants (packaging etc) were removed and the remaining mixture was homogenized using a commercial garbage grinder

(S52/010, Imperial Machine Company Ltd, UK) and thoroughly mixed in a large container before freezing in portions of approx 4 kg.

The green/garden waste was collected from Downend Quarry; a municipal composting site in Fareham, Hampshire. The waste collected had been delivered to the site the day before, and had only been subject to a coarse shredding operation. On return to the laboratory this waste was shredded in a household garden shredder (Alko-Kober Ltd, UK) and frozen.

The paper and card components of the synthetic waste were collected where possible from recycling bins at the University of Southampton. Non-recyclable paper components were bought. The different components of paper and card were weighed and mixed according to the recipe. The resulting mixture was then shredded in a commercial paper shredder (Scimitar). The whole mixture of paper/card, green and kitchen wastes was passed together through the commercial garbage grinder, with tap water addition to facilitate the pulping: to minimize the addition of water, the wetter components (such as the kitchen waste) were pre-mixed with the drier (newspaper/toilet roll) before grinding.

The resulting slurry of SBMW was around 10-20% solids and was homogenized in a large container before freezing in small batches. These smaller batches were defrosted as needed and during some parts of the project were air-dried before performing analysis or feeding to reactors. This intensive particle size reduction, pulping and freezing process, although not necessarily representative of a real process, was necessary to ensure homogeneity of feedstock which was considered important since the experiments performed as part of this work were at a relatively small scale compared to an industrial process.

Inoculum

Anaerobic digester sludge from Millbrook Wastewater Treatment Works (WWTW), Southampton, was used as inoculum. In order to reduce the amount of solid material in the sludge, it was sieved through a 1 mm mesh before use. At the beginning of each semi-continuous or batch trial, each reactor was filled to the working volume with a combination of the required feed and sieved sewage sludge.

3.3 Analytical Methods

All reagents used were laboratory grade except where stated.

3.3.1 Solid/Digestate Analysis

Total and Volatile Solids

TS and VS were measured gravimetrically using a fan-assisted oven (Vulcan-Hart, USA) at 105 ^{o}C and a muffle furnace (Carbolite, UK) at 550 ^{o}C according to Standard Method 2540 G (APHA 2005). Using a balance with sensitivity ± 0.1 mg the accuracy of TS is ± 3 mg TS kg^{-1} wet weight and the VS is ±7 mg VS kg^{-1} wet weight based on typical measurements made throughout this work (60g sample, 10% TS, 0.8 VS/TS). Precision according to the standard method gives a standard deviation for TS of 6 mg TS kg^{-1} wet weight (n=41) and for VS 11 mg VS kg^{-1} wet weight (n=40) (APHA 2005).

3.3.2 Liquid Analysis

pH

pH of samples was measured using a pH probe connected to a Jenway 3310 pH meter (Jenway, UK). The pH meter was calibrated before use with buffer solutions (pH 4, 7 and 9.2, Fisher Scientific general purpose grade) which were made up weekly and stored in sealed jars. Between measurements, deionised water was used to clean the probe. The measurement was taken within a short period of sampling to avoid the evaporation of volatiles or evolution of dissolved carbon dioxide, both of which could alter the pH reading. High solids samples were homogenized before measurement by either stirring or shaking. The accuracy of the pH meter was ± 0.01 pH unit although according to the standard method 4500-H^{+} (APHA 2005) under normal conditions expected accuracy of this method is ± 0.1 pH unit with a precision of ± 0.05 pH unit.

Alkalinity

The alkalinity of liquid samples was measured by titration with a 0.25N solution of sulphuric acid to selected pH endpoints measured continuously using the pH equipment described above. Magnetic stirring was used to ensure the pH probe did not suffer from fouling during the analysis. Cross contamination between sampling was reduced by thoroughly cleaning and visually inspection of the pH probe between different samples.

Three measures of alkalinity were used: partial, intermediate and total alkalinity (PA, IA and TA) as described by Ripley et al (1986). These are defined by the pH start and endpoints shown in Table 7. Due to the nature of the samples being analysed, PA and IA were useful concepts because the first is a measure of carbonate buffering, while the second is mainly a measure of volatile fatty acid (VFA) buffering. In AD the alkalinity ratio, defined as the ratio of partial to IA, gives a good measure of the stability of the process. Alkalinity is presented in equivalent concentration of calcium carbonate and is given by Equation 1.

According to standard method 2320 A (APHA 2005), on which the method described here is based, it is difficult to give a meaningful statement on the accuracy of this analysis. This is because the pH change by addition of a unit of acid changes greatly throughout the analysis and between samples of different sources. It is likely that the accuracy and precision are much greater than those involved with taking samples and sample handling before the analysis. pH endpoints were measured to within the accuracy and precision of the pH analysis described above, sample volumes and acid titre volumes were measured to accuracies of ± 1ml and ±0.01 ml respectively.

$$\text{Alkalinity (mg } CaCO_3\ l^{-1}) = \frac{Volume_{Acid}.Normality_{acid}.50000}{Volume_{sample}}$$

Equation 1 Alkalinity Calculation

Table 7 Definition of Alkalinities

Type of Alkalinity	pH Start Point	pH End Point
PA	pH of Sample	5.7
IA	5.7	4.3
TA	pH of Sample	4.0

Chemical Oxygen Demand

COD was measured by a titrimetric method (Westwood 2007) which is based upon standard method 5220 D (APHA 2005) but modified slightly to remove the use of mercury. The analysis was done in standard culture tubes with TFE lined lids. The sample preparation involved dilution to bring the COD to below 400 mg l^{-1} and to make the sample size up to 2 ml with distilled water, then the addition of 3.8 ml of FICODOX-plus reagent (Fisher Scientific Ltd, UK - see Table 8 for composition). Where SCOD was required the mixture was centrifuged at 14g (136.1 ms^{-2}) for 10 minutes to remove suspended material. To these samples was added 0.1 ml of 1000 g l^{-1} silver nitrate solution to compensate for the interference of chlorides.

The mixture was refluxed at 150°C for 2 hours in a culture tube with the lid secured. After cooling, a few drops of ferroin indicator were added (Fisher Scientific Ltd, UK - see Table 9) and the solution was titrated with acidified (2% Sulphuric acid) 0.025N ferrous ammonium sulphate solution which was made monthly and labelled with an expiration date. The end point was a colour change from blue to red. Blanks (distilled water only) and FAS standard (unheated) were used to calculate the COD by Equation 2 and Equation 3. A standard solution containing 3.8 g l^{-1} of potassium hydrogen phthalate was also diluted and titrated with each batch of samples tested. The COD of this solution was 4 g COD l^{-1} and this was used as a check against calculated values of COD. The standard concentration was chosen since this was the close to the COD measurements of the samples, meaning that to some extent dilution errors were accounted for since the standard and samples were diluted using the same equipment.

According to the standard method the precision of this analysis is within a coefficient of variation of 8.7% (n=240) in the absence of chlorides and 9.6% (n=240) with chlorides.

$$Normality_{FAS} = \frac{0.12884}{TitrantVolume_{FASstd}}$$

Equation 2 Normality Calculation

$$\text{COD (mg } O_2 \text{ l}^{-1}) = \frac{4000\left(TitrantVolume_{Blank} - TitrantVolume_{Sample}\right).Normality_{FAS}}{\left(Dilution\right)}$$

Equation 3 COD Calculation

Table 8 FICODOX-plus Composition

Chemical	Concentration
Potassium di-chromate	1.7 g l^{-1}
Silver sulphate	8.1 g l^{-1}
Sulphuric acid	81.1%

Table 9 Ferroin Indicator Composition

Chemical	Concentration
1,10-phenanthroline monohydrate	14.85 g l^{-1}
Iron (II) sulphate heptahydrate	6.95 g l^{-1}

Volatile Fatty Acids

Volatile fatty acids (VFA) were quantified in a Shimazdu 2010 gas chromatograph, using a flame ionization detector and a capillary column type SGE BP 21 with helium as the carrier gas at a flow of 190.8 ml min^{-1}, with a split ratio of 100 giving a flow rate of 1.86 ml min^{-1} in the column and a 3.0 ml min^{-1} purge. The GC oven temperature was programmed to increase from 60 to 210 ^{o}C in 15 min, with a final hold time of 3 min. The temperatures of injector and detector were 200 and 250 ^{o}C, respectively. Preparation of samples involved centrifuging at 14g (136.1 ms^{-2}) for 10 minutes, then dilution to required concentration and preservation in formic acid (10% concentration). Three standard solutions containing 50, 250 and 500 mg l^{-1} of acetic, propionic, iso-butyric, n-butyric, iso-valeric, valeric, hexanoic and heptanoic acids were used for VFA calibration. This solution was prepared by diluting the pure VFA with deionised water up to 1 l. This was date labelled with an expiry date a month later after which a fresh solution was made. The accuracy of the instrument using the 250 mg l^{-1} standard solution over 26 measurements was found to be ± 21, 11. 20, 25, 27, 32 and 36 mg l^{-1} with standard deviations (n=26) of 7, 5, 9,

10, 14, 15, 20 and 22 mg l^{-1} for acetic, propionic, iso-butyric, n-butyric, iso-valeric, hexanoic and heptanoic acids respectively.

Suspended Solids

Suspended solids content was measured by passing a sample of known volume through a glass fibre filter paper (Whatman, UK) of known dry weight (pore size 0.4 µm). After drying at 105^{o}C the paper was again weighed and the difference calculated as per method 2540 D (APHA 2005). Using a balance with sensitivity ±0.1 mg an accuracy of ±12 mg l^{-1} was obtained for typical samples measured in this work (3 g l^{-1}, 20 ml sample size), the precision according to the standard method gives a standard deviation of 5.2 mg l^{-1} (n=80).

3.3.3 Gas Analysis

Gas Composition

Biogas composition was measured by two different methods. The first, for large samples, used a Infra-Red gas analyser (Model GA 94A, Geotechnical Instruments, Leamington Spa, UK) to measure methane and carbon dioxide. This instrument was calibrated by the manufacturer as per the recommended schedule and was checked weekly against a standard gas of 35% CO_2 and 65% CH_4 (BOC, Guildford, UK). The accuracy of this device was ±1% for compositions between (5-15%) and ±3% for compositions above 15%.

The composition of smaller gas samples was measured using a Varian CP 3800 gas chromatograph with a gas sampling loop using argon as the carrier gas at a flow of 50 ml min^{-1}. The GC was fitted with a Haysep C column and a molecular sieve operating at a temperature of 50 ^{o}C. The GC was calibrated using a standard gas containing 35% CO_2 and 65% CH_4 (BOC, Guildford, UK).
20 replicate measurements of a mixture of the standard gas and air were made; the accuracy of methane and carbon dioxide were found to be ±1.8% and ±1.0% and the precision of these measurements resulted in standard deviations of 1.1% and 2.5% (n=20) respectively.

Gas Volume

Except during the first BMP test, biogas was collected using Tedlar bags, which were then emptied into water displacement gasometers for volume analysis.

The use of Tedlar bags meant the diffusion of biogas components through barrier solutions was not an issue as the gas was only in contact with the gasometer liquid (acidified water) for a short time. Continuous use of gasometers using water as the barrier solution was avoided as the solubilisation and diffusion of biogas components can lead to incorrect volume and composition readings.

Gas volumes quoted in this work are corrected to STP (0°C, 100 kPa). Two similar designs of gasometer were used for emptying Tedlar bags; one where gas entering the column displaced water into a trough below the column (trough type) and the other where water displaced was weighed on a balance (weight type). These are shown in Figure 14. In both designs, measurements of water column height and liquid weight along with atmospheric temperature and pressure were used to calculate the volume of gas introduced. Governing equations for the volume calculations are given in Equation 4 and Equation 5. Saturated Vapour Pressure (SVP) is calculated using the Goff-Gratch relation shown in Equation 6 (Goff and Gratch 1946). The notation for the volume calculations is given below. The derivation of the governing equations is given in appendix 8.2.

The using the gasometer governing equations and values of the constants for the equipment used in this work gives an accuracy of ±28 ml and ±8 ml for a trough and weight gasometer respectively for a gas measurement typical of those made in this work (1 litre biogas). The precision of these measurements was estimated to have a standard deviation of 10 ml (n=12) based on comparing a weight of water used to displace a (therefore) known volume of gas into a Tedlar bag which was then measured using a weight gasometer.

Gas Volume Notation

V	Volume (m^3)	H	Total height of column (m)
p	Pressure (Pa)	T	Temperature (K)
A	X-section of gasometer (m^2)	m_b	Mass of barrier solution (kg)
ρ	Density (kg m^{-3})	h	Distance from the top of the gasometer to the barrier solution level.

1, 2, stp, atm, H_2O, b, t, c

Subscripts refer to condition 1, condition 2, standard temperature and pressure, atmospheric, water, barrier solution, trough and column respectively.

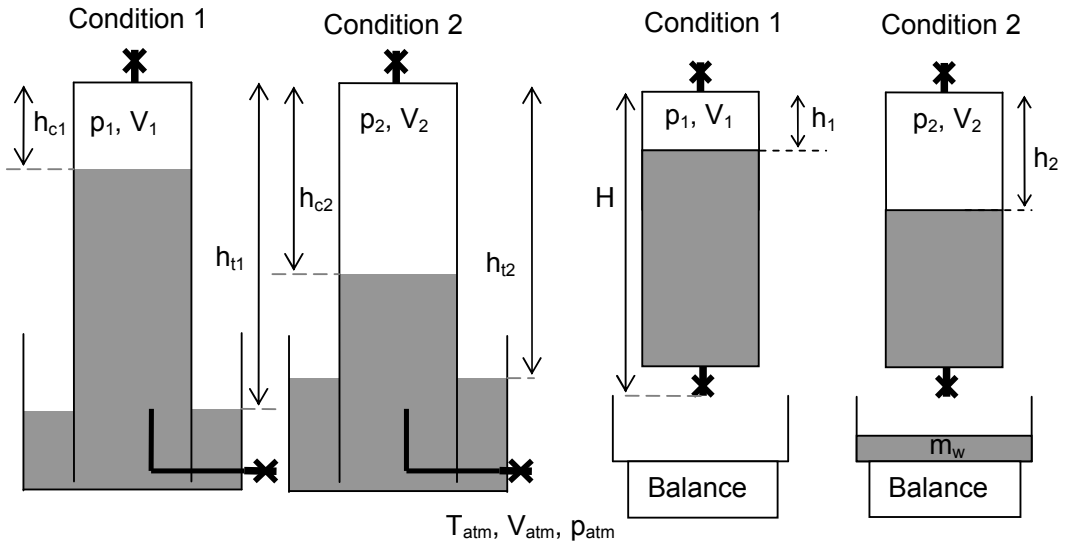


Figure 14 Gasometer Design and Notation

$$V_{stp} = \frac{T_{stp} A}{T_{atm} p_{stp}} \left(\left(p_{atm} - p_{H_2O}(T_{atm}) - \rho_b g \left(h_{t2} - h_{c2} \right) \right) h_{c2} - \left(p_{atm} - p_{H_2O}(T_{atm}) - \rho_b g \left(h_{t1} - h_{c1} \right) \right) h_{c1} \right)$$

Equation 4 Trough Gasometer Volume

$$V_{stp} = \frac{T_{stp} A}{T_{atm} p_{stp}} \left(p_{atm} - p_{H_2O}(T_{atm}) - \rho_b g \left(H - \left(h_1 + \frac{m_b}{A\rho_b} \right) \right) \right) \left(h_1 + \frac{m_b}{A\rho_b} \right) - \frac{T_{stp} A}{T_{atm} p_{stp}} \left(p_{atm} - p_{H_2O}(T_{atm}) - \rho_b g (H - h_1) \right) h_1$$

Equation 5 Weight Gasometer Volume

$$p_{H_2O}(T) = 11324.6 \times 10^{z}$$

$$z = -7.9190\left(\frac{373.16}{T} - 1\right) + 5.02808 \log_{10}\left(\frac{373.16}{T}\right) - 0.00000013816\left(10^{11.34\left(1-\frac{373.16}{T}\right)} - 1\right) + 0.0081328\left(10^{\left(-3.49149\left(\frac{373.16}{T}-1\right)\right)} - 1\right)$$

Equation 6 Goff-Gatch SVP Calculation (Over liquid)

3.4 Process Modelling

The first order hydrolysis model was used on some experimental data collected in this work and therefore a brief mathematical description is presented bellow (Sanders et al. 2003a). The terms have the following meanings;

X - Substrate concentration

k - 1st order hydrolysis constant

f - Degradable fraction of substrate

R - Retention time (In a continuous system)

t - Time

$_{drg,\inf,eff}$ - Subscripts are degradable, influent and effluent respectively.

Equation 7 shows the first order hydrolysis model basis and can be rearranged to form Equation 8 and Equation 9 for batch and continuous systems respectively. These can be further manipulated to give Equation 10 and Equation 11 which give a method of determining the constant again in batch and continuous systems respectively.

$$\frac{dX_{dgr}}{dt} = -kX_{dgr}$$

Equation 7 First Oder Hydrolysis Model

$$X_{eff} = X_{\inf}(1-f) + fX_{\inf}e^{-kt}$$

Equation 8 Degradation Equation for Batch First Order Hydrolysis

$$X_{eff} = \frac{X_{\inf} f}{1+Rk} + X_{\inf}(1-f)$$

Equation 9 Degradation Equation for Continuous First Order Hydrolysis

$$kt = -\ln\left(\frac{X_{eff} - X_{\inf}(1-f)}{X_{\inf} f}\right)$$

Equation 10 Rearrangement of Equation 8

$$R = \left(fX_{\inf}\right)\left(\frac{R}{X_{\inf} - X_{eff}}\right) - \frac{1}{k}$$

Equation 11 Rearrangement of Equation 9

4 Experimental Procedures

4.1 Objectives and design

A summary of the experimental objectives and plan for each of the sections in this chapter is given below.

- 4.2 - BMP tests performed to characterise the SBMW used as the feed material subsequent trials.
- 4.3 - Preliminary trials to gain understanding into the AD characteristics of SBMW.
- 4.4 - Single-stage hydraulic flushing to measure performance gains of decreasing the HRT relative to the SRT.
- 4.5 - Two-stage HF to assess the total methane production of the system and understand the effect of the second stage effluent recirculation.
- 4.6 - 1st MFBR design, understanding the effect of mesh pore size under similar conditions to section 4.5.
- 4.7 - The design and implementation of a rotating drum bioreactor to overcome the problems with the previous design and investigation into the effect of higher loading rates on the two stage RDMBR/AF system.

4.2 Biochemical Methane Potential

Overview

The biochemical methane potential (BMP) of a particular substance provides information on the likely performance of a continuously operated process fed on that substance (Shanmugam and Horan 2009). For any particular anaerobic treatment system, the comparison of the BMP with the actual methane production can be used to access the degradation effectiveness. This is more relevant than comparing literature cited or industrial values, since for a material such as BMW, variations around the world, as well as other local and seasonal factors mean different samples of waste will have different physical, chemical and AD characteristics (Gunaseelan 1997) as can be seen in Table 6. Therefore it was considered important that the BMP of the SBMW was determined as part of this work.

Description

The BMP test was performed under conditions chosen to ensure full realisation of the methane potential, including:

- Active methanogenic inoculum
- Large inoculum:substrate ratio (2:1 inoculum VS to substrate VS)
- Long batch incubation (1st BMP - 60 days, 2nd BMP – 100 days)
- Gentle stirring to ensure good contact of inoculum biomass and substrate
- Isothermal mesophilic conditions

Before the start of the BMP the VS content of fresh sieved sewage sludge was measured and the amount of feed needed to give the correct inoculum to substrate ratio (on a VS basis) was calculated. Control reactors were fed on only sewage sludge in order to assess the background methane potential of the inoculum and the BMP was calculated as the difference in methane production between the reactors containing substrate and the control reactors at the end of the trial.

1st BMP

The first set of BMP tests were performed in 5-litre CSTR reactors. Two substrate reactors and two control reactors were used. Gas production was measured using a trough type gasometer (Figure 14). When the collection column became full of biogas, it was refilled with water and the gas was sampled for its composition. At the end of the test VS content of the substrate reactors was measured in order to estimate total degradability of the substrate.

This BMP had two main flaws; the first being the continuous use of water displacement gasometers to measure the gas production, which meant that the biogas production readings were subject to some errors due to diffusion and solubilisation of carbon dioxide and methane, leading to a probable underestimate of BMP (see section 3.3.3). The second was neglecting to measure the end VS content of the control reactors, meaning that overall degradability of the substrate could only be estimated based on the assumption that equal quantities of methane were produced from equal VS destructions in the inoculum and substrate.

2nd BMP

A 2nd BMP test was undertaken after the problems with the 1st were realised. Apart from the following points the test was the same;

- Gas measurements were made by collecting the biogas in 3 l Tedlar bags and measuring the volume and composition.
- The test lasted longer (100 days). Since gas production was not measured on a real-time basis the test ran for a longer period before the similar gas production from the substrate and control reactors was the same.
- 0.5 litre CSTRs were used since a total of 12 reactors were available allowing greater replication of results. 3 controls and 3 different batches of SBMW each in triplicate were tested.
- VS analysis was done on all of the reactors at the end providing a means to calculate maximum VS destruction in the substrate.

4.3 Preliminary Trials

The two trials in this section were not central to the subject of this thesis, and were simply used to gain insight into the digestion characteristics of SBMW. In a similar manner to the BMP test, it was thought that the comparison of the performance of more complex systems described later in this work with these simpler single-stage processes would be interesting and useful.

4.3.1 Single-stage

Table 10 Single-stage Trial Overview

Number of reactors	4 (2 duplicates)
Size and type of reactors	1.5 litre CSTR
OLR	2-2.5 g VS l^{-1} d^{-1}
Solids retention time (SRT)	30 days
HRT	30 days
Feed	Wet SBMW

Overview

For comparative purposes, the single-stage digestion performance of reactors fed on SBMW was investigated.

Description

4 reactors were seeded with sieved sewage sludge and were fed daily SBMW at 2 g VS l^{-1} d^{-1}.The solids and hydraulic retention times were set by removing 50 g of mixed reactor contents per day and the feed volume was made up to 50 g by adding either tap water or buffer.

Performance was assessed using gas production (daily), gas composition (weekly) and solids destructions (weekly). Stability was assessed using pH (daily) and alkalinity (weekly).

On day 30 of the trial, the OLR of two of the reactors was increased to 2.5 g VS l^{-1} d^{-1} and this was maintained until the end of the trial. On day 110, buffer

solution in the place of tap water was given to the two reactors that remained on the lower OLR; again this continued until the end of the trial.

4.3.2 Kinetic Hydrolysis

Table 11 Kinetic Hydrolysis Trial Overview

Number of reactors	10 (5 duplicates)
Size and type of reactors	1.5 litre CSTR
OLR	7.5 g VS l^{-1} d^{-1}
Solids retention time	2-10 days
HRT	2-10 days
Feed	Wet SBMW

Overview

The purpose of this trial was to investigate the effect of changing the SRT on the rate of hydrolysis of the feed material. Reactors were subject to a relatively high OLR of 7.5 g l^{-1} d^{-1} ensuring methanogenic bacteria were quickly inhibited by the abundance of VFAs and the consequent low pH in the reactors. This meant these reactors were the 1st of a hypothetical two-stage process with kinetic phase separation as described in Figure 6.

Description

10 reactors run in 5 duplicates were operated at retention times of 2, 4, 6, 8 and 10 days, all at the same loading rate. The reactors were subject to gradually decreasing retention times set by adjusting the amount of digestate removed on a daily basis. The retention times were reduced by 25% or less for each retention time running, thus allowing the biomass to adjust to the new flow conditions and avoiding wash out.

Hydrolysis performance was assessed using VFA and COD production (weekly), along with total and VS destruction (weekly). pH was also monitored (daily).

4.4 Single-stage Hydraulic Flush

Table 12 Single-stage HF Trial Overview

Number of reactors	10 (5 duplicates)
Size and type of reactors	0.8 litre HF
OLR	3.75 g VS l^{-1} d^{-1}
Solids retention time	20 days
HRT	1.6-20 days
Feed	Wet SBMW

Overview

This trial was a preliminary investigation into the effect of the HF on the anaerobic hydrolysis process. Reactors had a constant SRT and a range of HRT between 1.6 and 20. The main hypothesis was that hydrolysis rates could be enhanced by removal of VFA and other products as well as the resulting higher pH caused by the HF. In addition to this the effect of controlling the pH using buffer addition was investigated under the same HF regime.

Description

The design of the HF reactors allowed the stirring assembly to be removed and the reactor bottles to be centrifuged in a WIFUG 4000E (WIFUG, UK). In order to decouple the hydraulic and solids retention times the reactors were centrifuged at 8g (78.5 ms^{-2}) for 20 minutes daily. The working volume of the reactors was 800 ml. The SRT was set by the amount of mixed digestate removed before centrifuging (40 g) and the HRT by the amount of supernatant removed after centrifuging (0-450 g). After removing the digestate and supernatant the reactors were fed on SBMW and made back up to the working volume using tap water.

Four different HRTs were chosen between 1.6 and 20 days, and duplicate reactors were operated at each one. The condition HRT = SRT = 20 days was used as a control, to assess whether the hydraulic flushing had any effect. At this condition no supernatant was wasted after centrifuging and the SRT and HRT were simply set by removing mixed digestate.

A final set of duplicate reactors was used to assess the effect of correcting the pH to near neutral conditions using a buffer solution. The composition of this solution is shown in Table 13 and was based on the solution used by Rufener Jr et al (1962). Distilled water was used as the solvent.

Table 13 Buffer Used in Continuous Trials

Chemical	Concentration
Sodium hydrogen carbonate	5.8 g l^{-1}
Di-sodium hydrogen orthophosphate hexahydrate	3.7 g l^{-1}

These reactors were operated at a HRT of 1.6 days, using the same protocols as for the unbuffered equivalent except that buffer solution in place of tap water was used to make up the reactor to its working volume after feeding.

Hydrolysis performance was measured using VFA production (every 5 days), COD (measured at the end of the trial), along with TS and VS (every 5 days). pH was monitored daily. Since a significant proportion of the reactor solids could be removed in the supernatant, the suspended solids were also measured. Equation 12 was used to calculate TS and VS destruction.

$$\%TS,VS_{destroyed} = 100\left(1 - \frac{TS,VS_{removed}Sludge + TS,VS_{removed}Suspended}{TS,VS_{added}Total}\right)$$

Equation 12 Solids Destruction Calculation in Single-stage HF

4.5 Two-stage Hydraulic Flush

Table 14 Two-stage HF Trial Overview

Number of reactors	12 (4 x HF duplicates, 4 x AF)
Size and type of reactors	0.7 litre HF and 4 litre AF
OLR	3.75 and 7.5 g VS l^{-1} d^{-1}
Solids retention time	20 days (HF)
HRT	1.75 days (HF) 5 days (AF) 180 days (Overall)
Feed	Air dried SBMW

Overview

The purpose of this trial was to ascertain the two-stage performance of an anaerobic system using HF reactors as a 1st stage, and AF reactors as the 2nd. The particular effects investigated were that of adding alkalinity in the form of buffer solution, the effect of methanogenic effluent recirculation and the effect of increased loading.

Description

The HF reactors in this trial were operated using similar procedures to those in section 4.4, specifically 700 ml working volume with 35 g removed sludge, and 400 ml removed supernatant per day. This gave an SRT of 20 days and an HRT of 1.75 days.

Supernatant from each set of duplicate reactors was fed to an AF reactor, and effluent from these reactors was used to fill the HF reactors back to working volume after feeding, in place of the tap water used in the previous experiment. This meant that each AF reactor was fed 800 ml of supernatant each day, giving a retention time of 5 days; thus the liquid in the system was passed back and forth between the HF and AF reactors. Because the water content of the removed digestate was much higher than that of the air dried feed, a daily deficit in liquid was made up using either tap water or buffer solution in the

manner described below. The amount of tap water/buffer used was around 30 ml per day making the overall HRT of the two-stage system around 180 days.

Four duplicate sets of HF reactors were run under the following conditions. Two sets were fed at an OLR of 3.75 g VS/ l^{-1} d^{-1} and two were fed at 7.5 g VS l^{-1} d^{-1}. Tap water was used as the top up fluid in one of each set at each loading and the other was topped up with the buffer solution shown in Table 13. The high OLR unbuffered trial was started with a slightly different inoculum to the other reactors. This was a mixture of digestate from the low OLR unbuffered trial and fresh sewage sludge from Millbrook WWTW.

On day 64 the operation of the HF reactors at loading rate 7.5 g VS l^{-1} d^{-1} with water addition only was changed such that none of the supernatant was fed to the AF reactor, but instead it was retained in the reactor after centrifuging. This was done to determine whether the reactor could operate stably without the second stage reactor.

Performance of the system was mainly assessed using biogas production (daily) and composition (weekly), along with the TS and VS destructions (weekly). Other parameters measured included COD and VFA concentrations in the HF supernatant (weekly) along with COD, VFA and alkalinity concentrations in the AF effluent (weekly). pH was also monitored in both sets of reactors (3 times a week).

4.6 Mesh Filter Bioreactors

Table 15 MFBR Trial Overview

Number of reactors	6 (3 x MFBR and 3 x AF)
Size and type of reactors	1.5 litre MFBR and 4 litre AF
OLR	3.75 g VS l^{-1} d^{-1}*
Solids retention time	20 days
HRT	1.5 days (MFBR) 4 days (AF) ~300 days (Overall)
Feed	Wet SBMW

*After 85 days the OLR was doubled in the 30 and 100 µm reactors, and operation continued at the same SRT and HRT.

Overview

The purpose of this trial was to assess the viability of using filtration rather than centrifugation as the means to decouple the hydraulic and solids retention time in the 1st stage reactors. Three meshes of different pore sizes were used in the 1.5 l MFBRs. Filtrate from each MFBR was fed to an AF reactor, and the AF effluent was recirculated to the 1st stage reactors.

Description

Meshes made from woven nylon (Fisher Scientific, Loughborough, UK), chosen for their low cost and physical strength, of pore sizes 30, 100 and 140 µm were installed inside three 1.5-litre MFBR 1st stage reactors which were fed at an OLR of 3.75 g VS l^{-1} d^{-1}. Attempts were made to use a 10 µm mesh but this proved unsuccessful: the mesh quickly became clogged with the sewage sludge inoculum, and the resulting suction inside the mesh unit caused it to collapse.

The SRT and HRT were set at 20 days and 1.5 days respectively by the removal of 30 g of mixed digestate and 1 l of liquid effluent daily. The filtrate was fed to a second stage AF reactor and an equivalent volume of effluent was re-circulated back to each 1st stage reactor on a daily basis. The flux in the MFBRs in this work was 44 l m^{-2} h^{-1}.

Performance was measured by monitoring gas production (daily) and composition (weekly), and TS and VS destruction (weekly). For further insight into the operation of the system, the following parameters were also measured: SCOD (2 times a week), TCOD (weekly), 2nd stage alkalinity (2 weekly), VFAs (end), pH (3 times a week) and effluent suspended solids (end).

4.7 Rotating Drum Mesh Bioreactors

Table 16 RDMBR Trial Overview

Number of reactors	4 (2 x RDMBR and 2 x AF)
Size and type of reactors	1.5 litre working volume RDMBR and 4 litre AF
OLR	7.5-15 g VS l^{-1} d^{-1}
Solids retention time	See description
HRT	1.5 days (MFBR) 4 days (AF) ~300 days (Overall)
Feed	Air Dried SBMW

Overview

In this trial the RDMBR design was tested. A loading of 7.5 g VS l^{-1} d^{-1} was applied to these reactors and as before the liquid effluent was fed to an AF reactor. After stable operation had been reached one RDMBR was isolated from its second stage reactor and the OLR was doubled to 15 g VS l^{-1} d^{-1} on the other one.

Description

Two RDMBRs were operated at a working volume of 1.5 litres: the capacity of the drum was 2 litres, but headspace is needed to allow the digestate to tumble inside the drum as it rotates. The loading rate was based on the working volume. Reactors were fed daily, but digestate was removed three times a week as the process was time consuming and involved exposing the contents briefly to air and ambient room temperature. Digestate was removed only as required to keep the reactors at constant working weight (weight of reactor + weight of

digestate + weight of filtrate). Under this regime the reactors were in effect setting the SRT themselves based on the drainage characteristics of the digestate; although since solid analysis was done on both the digestate and the liquid effluent the SRT could be estimated.

Initially a mesh of pore size 30 μm was used but this was unable to filter the digestate and was replaced with 100 μm meshes. The filtrate which drained naturally into the base of the reactors was pumped into a storage bottle, and was replaced continuously through the influent line with an equivalent volume of liquid. On a daily basis, 1 l of filtrate from each RDMBR was fed to an AF reactor and the equivalent AF effluent was recirculated back to the 1st stage. The flux through the mesh was 3.5 l m^{-2} h^{-1}.

In a similar way to the two stage HF reactors in section 4.5, after 72 days of stable operation one of the RDMBRs was isolated from its AF reactor, in an attempt to see if stable digestion could be performed in this reactor without use of the second stage. At the same time the OLR on the other reactor was doubled to assess the behaviour of the two-stage RDMBR/AF system under higher loading.

Performance was measured by monitoring gas production (daily) and composition (weekly) and solids and VS destruction (3 x weekly). For further insight into the operation of the system, COD (weekly), 2nd stage alkalinity (twice monthly), VFAs (end), pH (3 times a week) and the effluent suspended solids (weekly) were also measured.

4.7.1 Digestate Residual Methane Potential

At the end of the RDMBR trial (before the OLR increase) some of the digestate was taken from the reactors and tested for its residual BMP. This was done by placing the digestate samples in small flask reactors, as described in section 3.1.6, which were placed in a water bath and left for 60 days connected to Tedlar bags. At the end of this period the gas production and composition were measured. Since solids analysis was performed on the digestate samples specific residual methane production could be calculated. Additionally an

estimate could be made of the specific residual methane production based upon the influent VS rather than digestate VS, which is more relevant since it reflects how much of the initial BMP is being lost in the digestate.

5 Results

5.1 Feedstock Characterisation

The results of feedstock characterisation are shown in Table 17. The substrate was made in large batches, each being used to feed reactors for a few months, and though much effort was made to keep the composition similar between each batch some differences were unavoidable. The largest difference between batches was the TS and VS, because different amounts of water were added to the substrate during mixing; this was not considered a problem since all OLRs and analyses were based on TS or VS and neglected the moisture content.

Table 17 SBMW Characteristics

(Standard deviations, where appropriate, in brackets)

TS (%)	12.6 - 21.2
VS (%)	10.8 - 17.8
VS/TS ratio	0.875 (0.011)

5.2 Biochemical Methane Potential Test

During the BMP tests the substrate was degraded in the reactors quickly, with approximately 75% of the total biogas production in the first 10 days of the test.

1st BMP Test

The 1st BMP test lasted for 58 days before there was no measurable difference in the daily gas production of the reactors containing substrate (Substrate 1 and 2) and those containing only sludge (controls). VS analysis was performed on the seed sludge at the beginning of the test in order to set the inoculum to substrate ratio at 2. TS and VS analyses were also conducted on the MSW sludge at the end of the test. The results from these are shown in Table 18.

Table 18 TS and VS of 1st BMP Sludge (Standard deviation in brackets)

	Inoculum	MSW End
TS (%)	Not measured	3.04 (0.02)
VS (%)	2.22 (0.03)	1.79 (0.02)

Biogas production curves are shown in Figure 15 and the methane production curves are shown in Figure 16 along with the derived BMP curve. The BMP and biogas potential of the SBMW were 0.28 and 0.45 l g^{-1} VS added respectively. The methane content of the biogas in the reactors increased throughout the test from 59% at the beginning of the test to 82% at the end.

Substrate VS breakdown can be estimated by using the initial inoculum VS content and by assuming that the methane produced per g VS destroyed is the same for both the substrate and the inoculum. The calculation is set out in Table 19 and gives a value of 68.9% VS destruction for SBMW.

Using Equation 10 the 1st order hydrolysis constant was estimated by assuming that methane production followed VS destruction for the feed under batch methanogenic conditions. The logarithmic part of Equation 10 is shown in Figure 17 with a linear fit to the curve giving the 1st order hydrolysis constant at 0.113 d^{-1}.

Table 19 Calculation of Ultimate Degradation in 1st BMP Test

VS contribution from the inoculum at the beginning of the test.	4 litres @ 2.22% = 88.8 g (A)
VS contribution from the feed at the start of the test	44.0 g (B)
VS at the end of test	5 litres @ 1.79% = 89.5 g (C)
Inoculum:substrate biogas	0.12:0.28 (D)
Total VS destroyed	(A+B-C) = 43.3 g (E)
Amount of VS reduction in substrate	(E*D) = 30.3 g (F)
VS Reduction in substrate	(F/B) = 68.9%

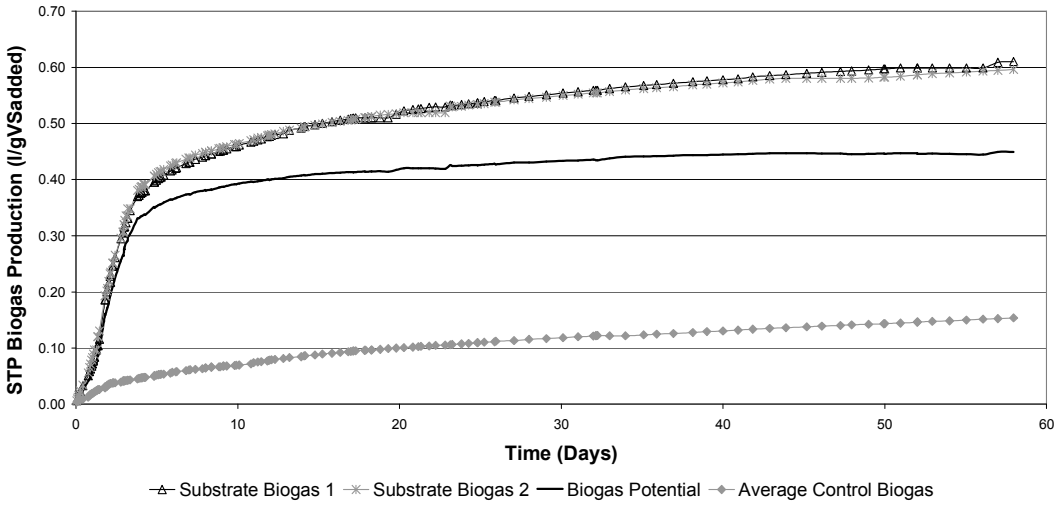


Figure 15 Biogas Production from 1st BMP Test on SBMW

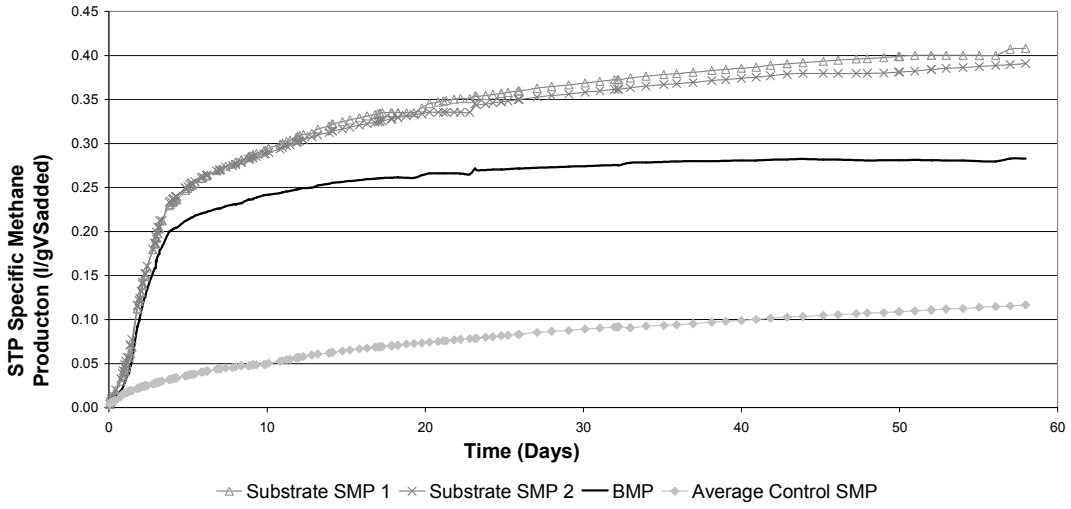


Figure 16 Specific Methane Production from 1st BMP Test on SBMW

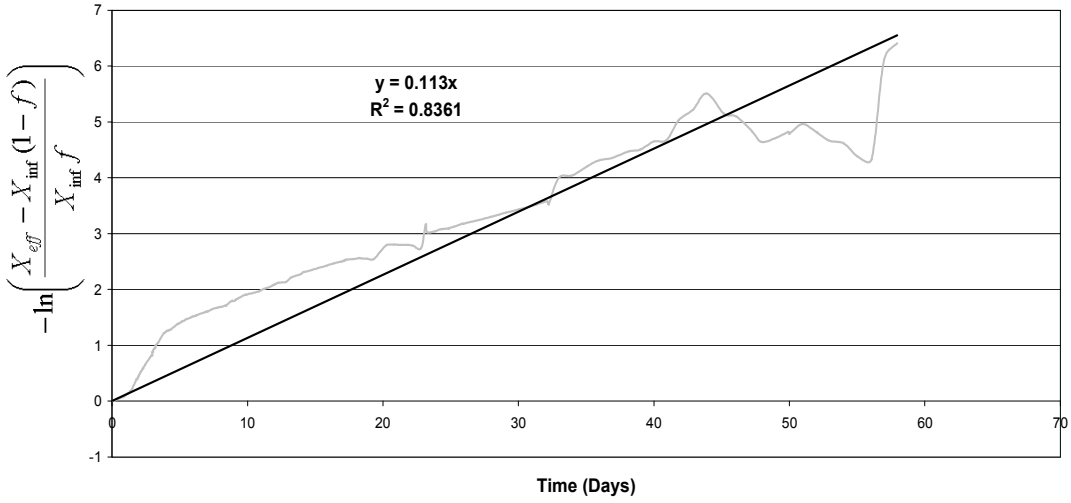


Figure 17 Line of Best Fit for Determination of the First Order Hydrolysis Constant from 1st BMP Test with SBMW

2nd BMP Test

The 2nd BMP test lasted 106 days. Gas volume and composition were measured batch wise using Tedlar bags in this test and therefore more time was needed to confirm that the control and substrate reactors were producing similar amounts of methane. Again the methane content of the biogas increased throughout the trial, from 62% to 75%. The results from the second BMP test are shown in Figure 18, with standard deviations shown in the error bars (n=3 for controls, n=9 for substrate reactors). The BMP and biogas potential calculated from this test were higher than for the 1st BMP test at 0.32 (S.D. 0.03) and 0.61 (S.D. 0.04) l g^{-1} VS added respectively. This time TS and VS analyses were performed on the sludge from all reactors at both the beginning and the end of the trial and therefore a better estimate of the ultimate degradability of the substrate could be made. The measured results and calculation steps are shown in Table 20. The only assumption required for this calculation is that the inoculum degradation characteristics were the same in both the control and substrate reactors. The results from this calculation gave the ultimate degradability of the SBMW as 53 and 62% on a TS and VS respectively.

Table 20 Calculation of Ultimate Degradation in 2nd BMP Test

(Standard deviation in brackets)

Measured TS, VS of the inoculum	3.71% TS, 2.32% VS (A)
Calculated TS, VS contribution of the inoculum	400 g Wet * =14.84 g TS, 9.28 g VS (B)
Measured TS, VS contribution from the feed at the start of the test	3.48 g TS, 3 g VS (C)
Measured TS, VS left in control reactors after the test	12.13 g TS(0.53) , 6.90 g VS (0.30) (D)
Measured TS, VS remaining in substrate reactors after the test	13.79 g TS (0.59), 8.04 g VS (0.19) (E)
Calculated substrate TS, VS remaining after the test	(E-D) = 1.66 g TS (0.35), 1.14 g VS (0.13) (F)
Substrate TS, VS degraded	(1-(C/F)) =53% (10), 62% (4)

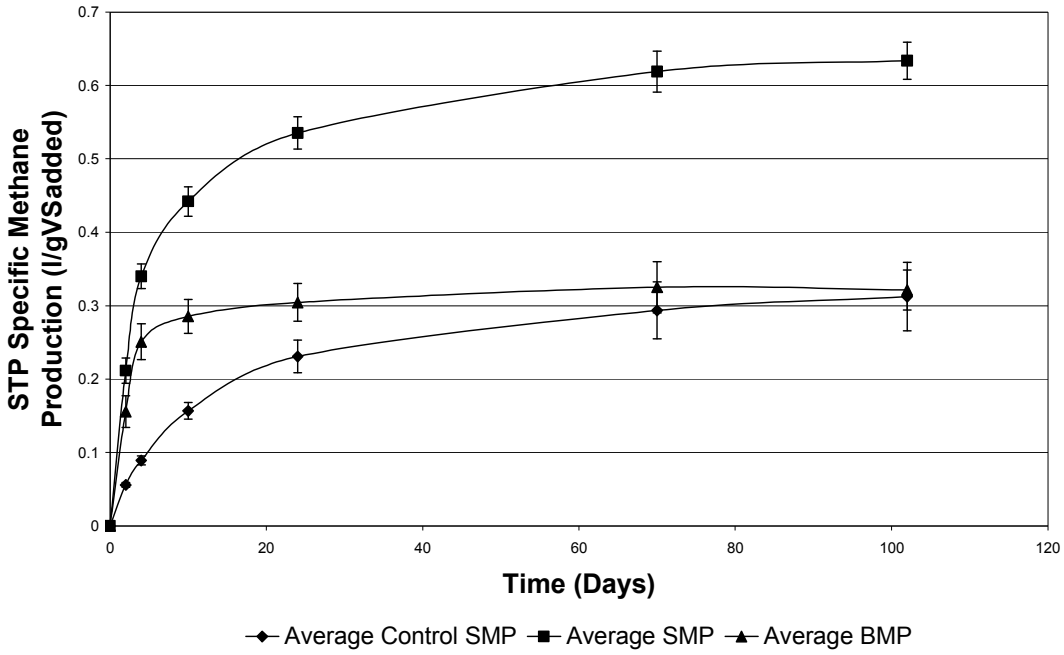


Figure 18 Specific Methane Production from 2nd BMP on SBMW

5.3 Preliminary Trials

5.3.1 Single-stage

The purpose of the single-stage trial was to investigate the AD performance of SBMW in CSTR mode. 4 Reactors were operated in duplicate, at OLR of 2-2.5 g VS l^{-1} d^{-1} and the HRT/SRT was 30 days. Details of the method can be found on page 71.

The trial ran for a total of 153 days, the first 100 of which are denoted the stable period of the trial and the final 50 days, the unstable period. The stable part of the trial was characterized by pseudo steady state conditions of biogas production, pH and alkalinity. However during the unstable part of the test, a there were some accidental disturbances to the reactors along with some changes in operating conditions meant that the process showed instability and in some reactors methane production dropped sharply as the process failed. The changes and disturbances are given in Table 21. A summary of some important results during the stable operational period is given in Table 22.

Table 21 Changes and Disturbances in the Single-stage Trial

Change or Disturbance	Time (Days)
MH3 and MH4 OLR increased to 2.5 g VS l^{-1} d^{-1}	30
Moving of some equipment in the laboratory meant the temperature of MH1 and MH3 was reduced by 4°C	103
Buffer solution added to MH1 and MH2	110
MH2 and MH4 moved to attempt to equalize temperature resulting in a second temperature change to these reactors.	148

The results from the reactors are not presented as averages, since the duplicate reactors showed large differences especially in the unstable period, partly due to these disturbances.

The pH of the low and highly loaded reactors for the duration of the trial is shown in Figure 19 and Figure 20 respectively. All four of the reactors reached a stable operational state at a pH of around 6.5-6.7. The specific methane production of the reactors is shown in Figure 21 and the TS destruction is shown in Figure 22. It should be noted that throughout the stable period, the methane production and solids destruction were steady and similar at the two loading rates.

Table 22 Summary of Single-stage Trial Results (Figures averaged over stable operational period, standard deviation in brackets)

Description	Low Loading (2 g VS l^{-1} d^{-1})	High Loading (2.5 g VS l^{-1} d^{-1})
Average SMP (STP l g^{-1} VS added)	0.28 (0.04)	0.27 (0.05)
Volumetric methane production (STP l l^{-1}Reactor)	0.56 (0.10)	0.64 (0.11)
Methane content of biogas (%)	53.5 (0.7)	53.6 (1.1)
Total Alkalinity (mg$CaCO_3$ l^{-1})	2216 (164)	2863 (335)
TS destruction (%)	50.9 (2.8)	55.3 (3.8)
VS destruction (%)	58.4 (3.2)	64.3 (4.4)

Figure 23 and Figure 24 show the alkalinity and IA:PA ratio for the low and high loaded reactors respectively. The reactor failures can been seen by the sudden increase in IA:PA representing an accumulation of VFAs in the reactor. Note

that in Figure 23, the effect of adding artificial buffering can be seen as an increase in TA in MH1and MH2.

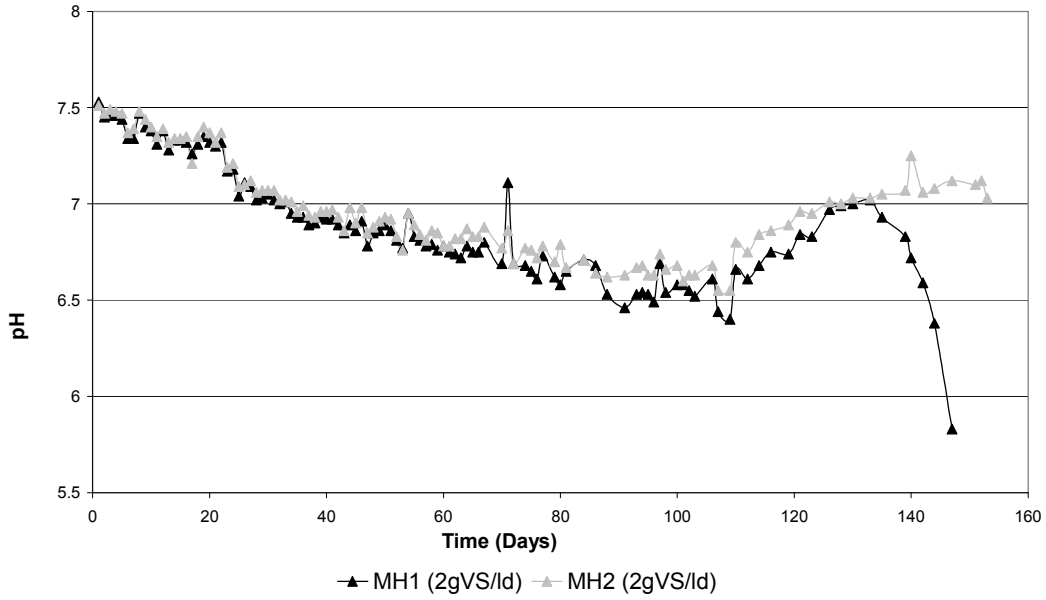


Figure 19 pH of Single-stage CSTR Fed SBMW at 2 g VS l^{-1} d^{-1}

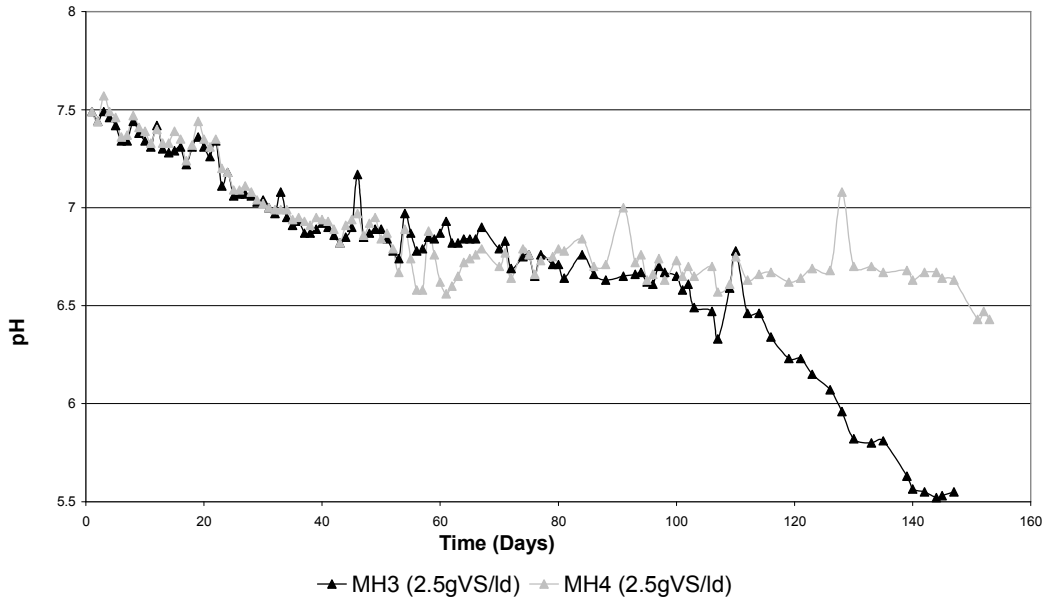


Figure 20 pH of Single-stage CSTR Fed SBMW at 2.5 g VS l^{-1} d^{-1}

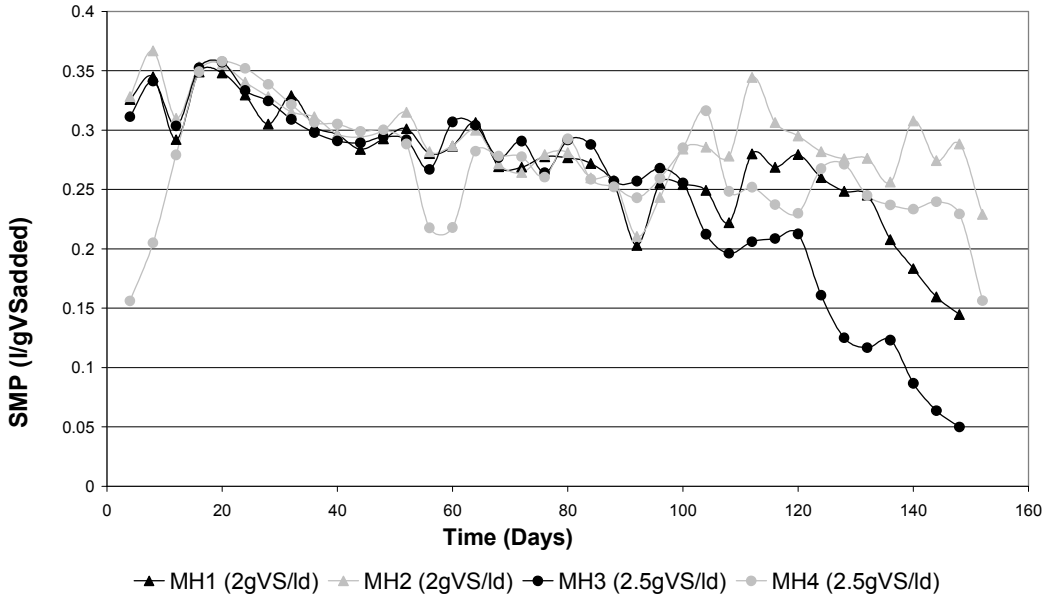


Figure 21 Specific Methane Production of Single-stage CSTR Fed SBMW

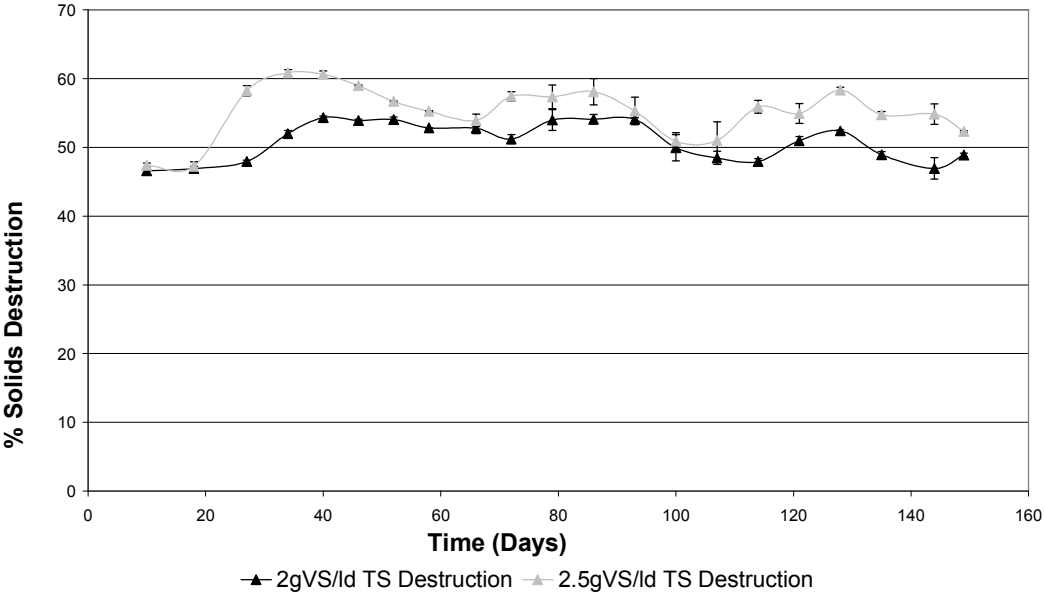


Figure 22 TS Destruction of Single-stage CSTR Fed SBMW

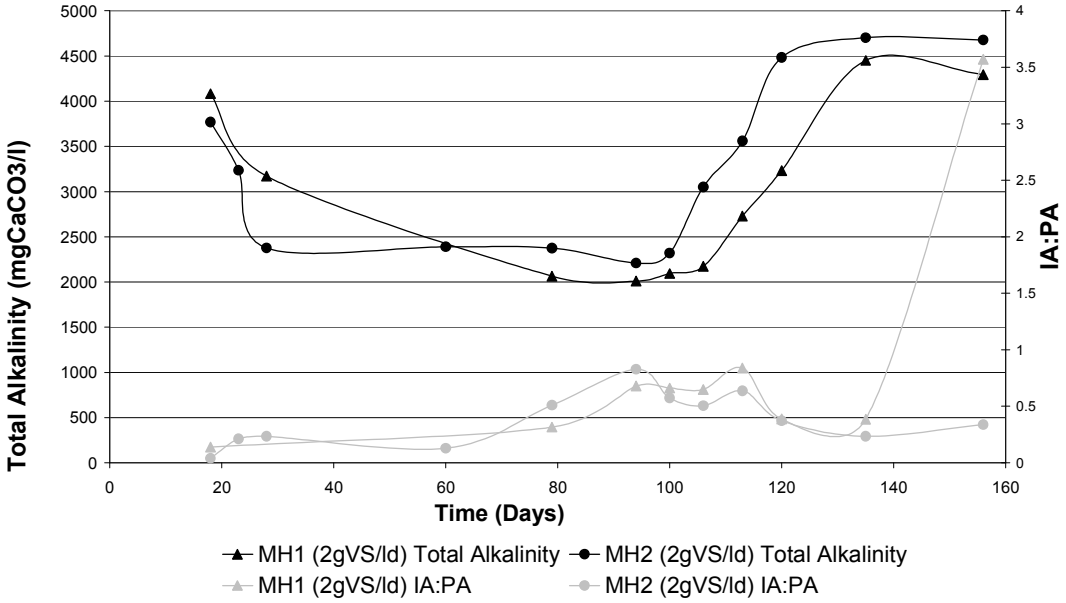


Figure 23 Total Alkalinity and IA:PA of Single-stage of CSTR Fed SBMW at 2 g VS l^{-1} d^{-1}

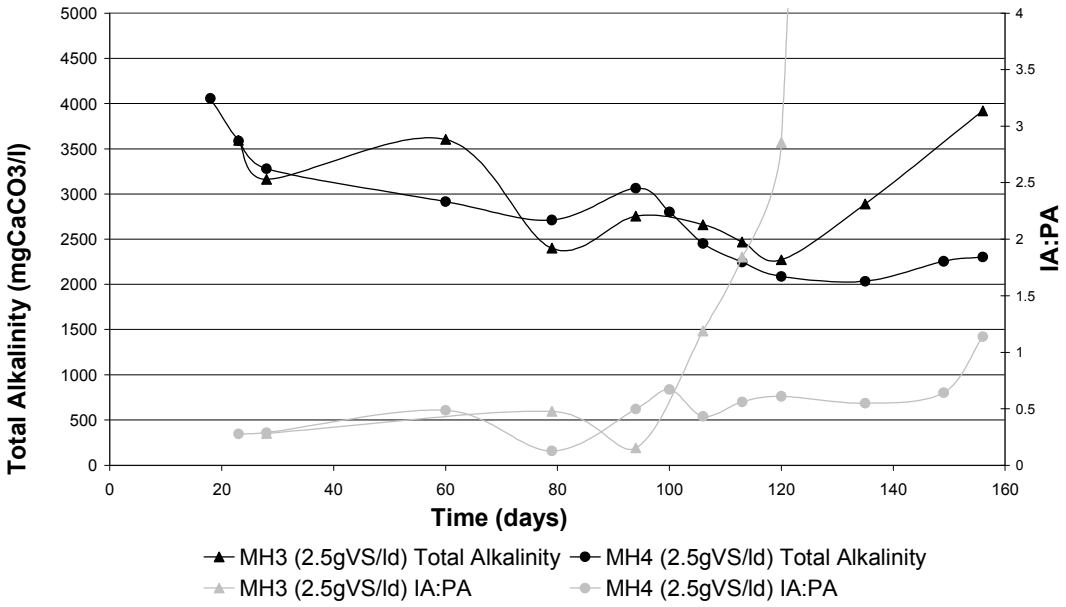


Figure 24 Total Alkalinity and IA:PA of Single-stage of CSTR Fed SBMW at 2.5 g VS l^{-1} d^{-1}

5.3.2 Kinetic Hydrolysis

This trial investigated the effect of changing the SRT on the rate of hydrolysis of SBMW. The OLR was 7.5 g l^{-1} d^{-1}. 10 CSTR were operated in duplicate at SRT of 2, 4, 6, 8 and 10 days. More details on the experimental method can be found on page 72.

The results presented for the kinetic hydrolysis trial are averaged over the final operational period for each set of duplicate reactors. The total time taken for the trial was 50 days, the first 25 of which was taken gradually reducing the retention times of the reactors to the desired operational conditions as displayed in Figure 25. During this time feeding continued for all reactors at the rate of 7.5 g VS l^{-1} d^{-1}. Each pair was operated at the final retention time for a period of at least three retention times such that stable operational conditions were achieved.

Very little biogas was produced in this trial: measured volumes were between 100 and 200 ml per day. The composition of the biogas was always above 90% carbon dioxide and less than 8% methane. This methane represents a COD of 23-46 mg, which was considered negligible in comparison with the reactor SCOD of between 2000 and 12000 mg/l and daily SCOD production rates of 1-1.35 g COD d^{-1}.

The variation of pH and solids destructions with SRT is shown in Figure 26, showing downward and upward trends with increasing SRT respectively. The error bars on the solids destruction curves are large, due to difficult sampling conditions. The digestate in the reactors was thick and spongy which meant collecting representative samples for solids analysis was difficult. Effort was made to reduce any sampling errors by using composite solids samples, but even so the variation between measurements remained.

The VFA concentrations and specific production rates are shown as COD equivalents in Figure 27. The increase in VFA concentration with increasing SRT correlates with the decreasing pH. The profile of the eight VFAs measured is given Figure 28 and shows no appreciable change or trend with SRT. COD

measurements were made periodically and it was found that the component of COD due to VFA made up 95-100% of the soluble COD.

An estimate of the 1st order hydrolysis constant can be made using by plotting the retention time against $\left(R / X_{\text{inf}} - X_{eff}\right)\left(fX_{\text{inf}}\right)$ and noting that the intercept of this line is $1/k$. This is shown in Figure 29 and gives a hydrolysis constant of 0.109 d^{-1}.

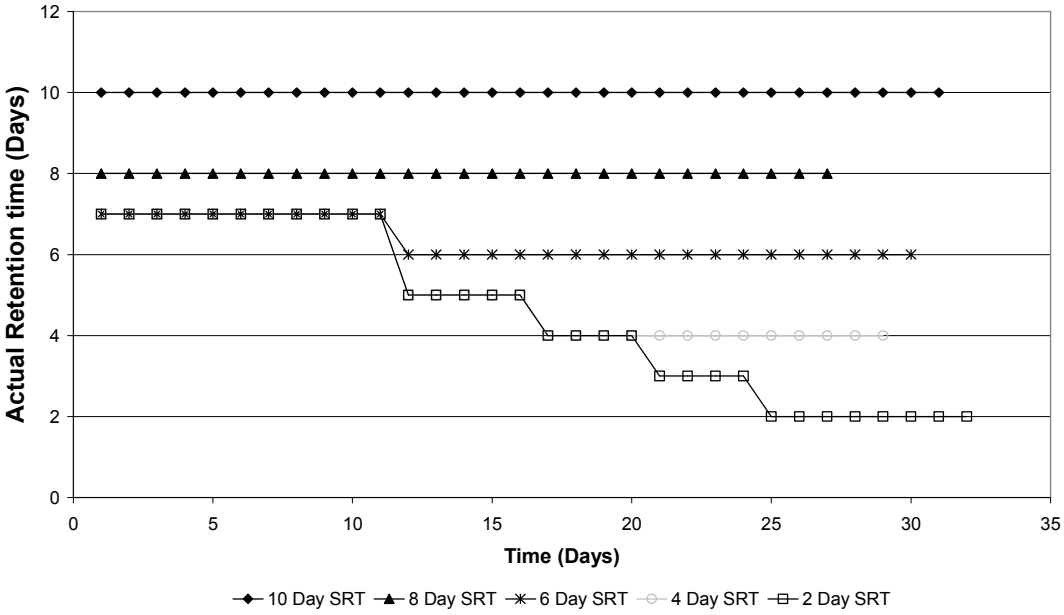


Figure 25 Retention Time Reduction Schedule

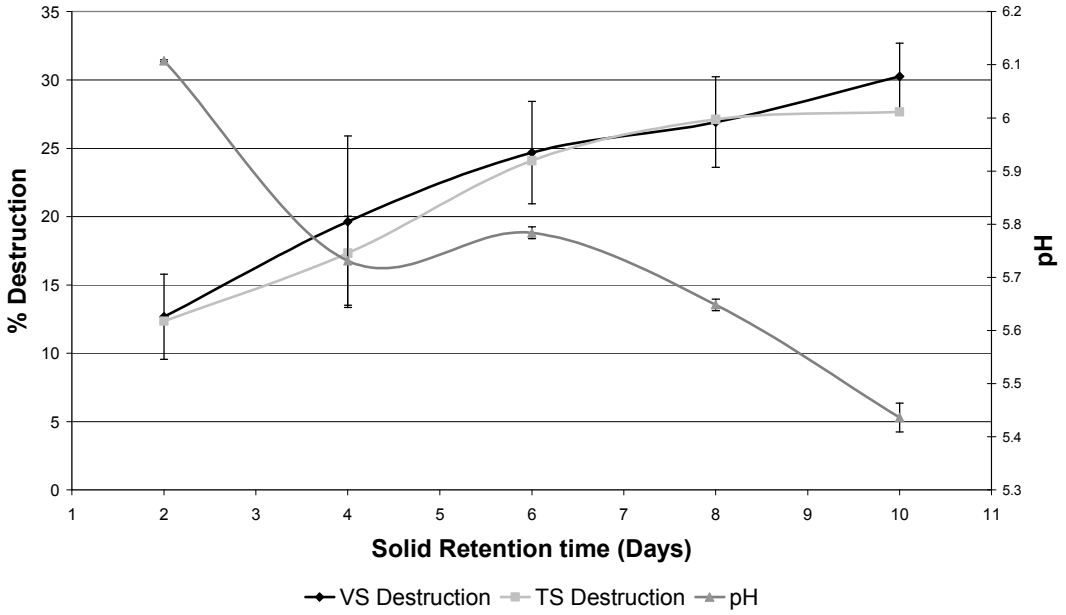


Figure 26 Kinetic Hydrolysis Solids Destructions and pH

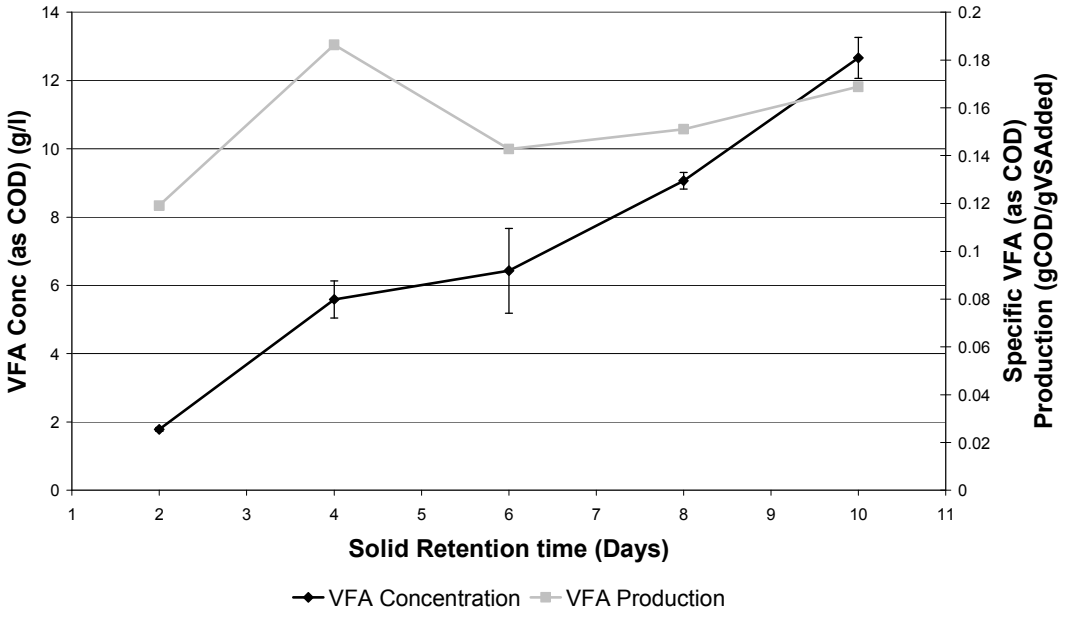


Figure 27 Kinetic Hydrolysis VFA Concentrations and Specific Productions

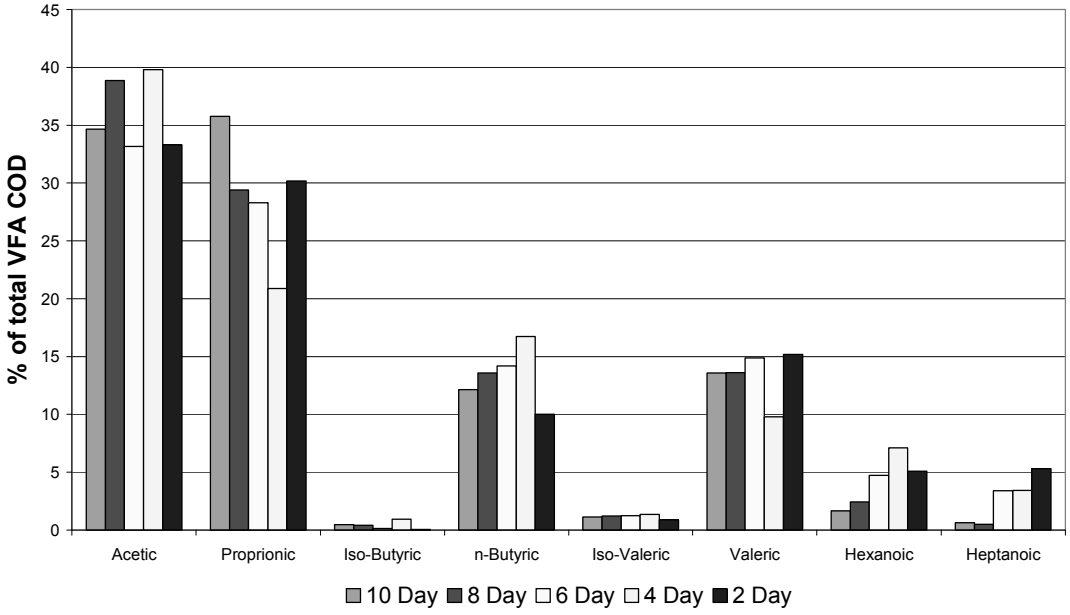


Figure 28 Kinetic Hydrolysis VFA Profiles

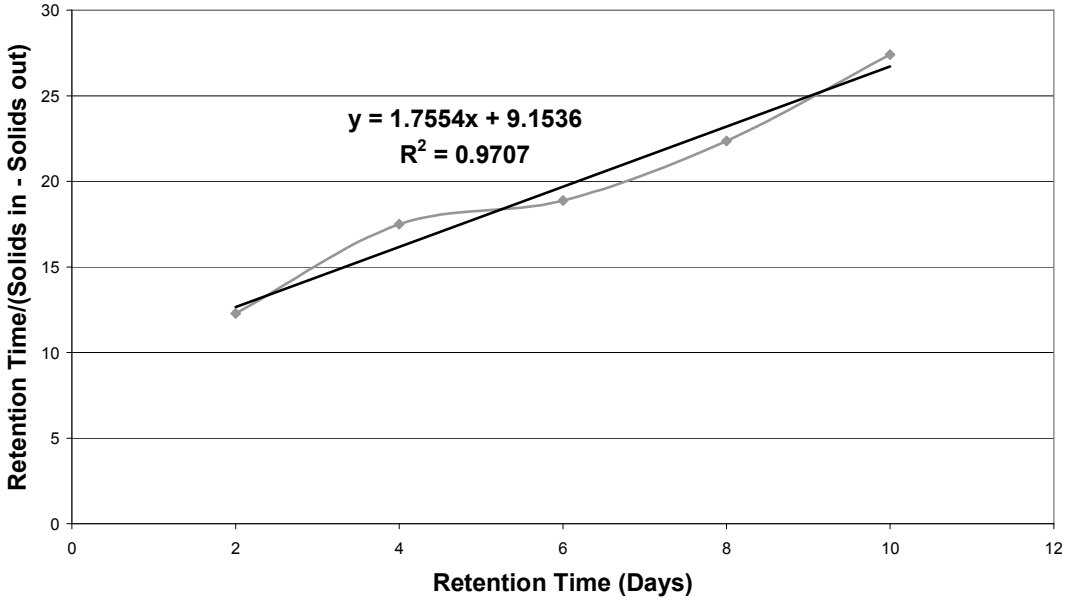


Figure 29 Calculation of the 1st Order Hydrolysis Constant in Continuous Conditions

5.4 Single-stage Hydraulic Flush

The results presented here come from two different trials. The eight reactors with no pH control (i.e. no buffer solution addition) were operated together, while the duplicate reactors with pH control were actually run in parallel with the two-stage HF trial. Full details of the procedures used in this trial can be found on page 73. The length of the first trial was 67 days and HF reactors of type A were used, whereas in the later run type B reactors were used (see Figure 8) and the trial lasted 85 days.

Table 23 shows a summary of important results from this trial. The main performance measures for this reactor system were solids destructions and VFA production, the latter giving a measure of how much methane could be derived from the effluents of these HF reactors. As can be seen in Figure 30, the pH of the reactors without buffer was low, whereas the buffered reactors showed stable almost neutral pH. On two occasions during the uncontrolled pH run the centrifuge needed servicing and so the reactors were not flushed on these days. These occasions were characterized by a sharp drop in pH followed by a recovery once flushing was resumed, and occurred on days 15-19 and 43-47.

The VFA concentration in the reactors is shown in Figure 31, and its relationship with HRT is shown in Figure 32. Specific VFA production is seen to be greater with hydraulic flushing, relative to the case where SRT = HRT = 20 days. The effect of the pH control, however, seems to have reduced slightly the VFA production (0.225 rather than 0.246-0.257).

With regard to solid destruction, shown in Figure 33, the case is similar, that flushing increases the solids breakdown; where as controlling the pH has a slightly negative effect relative to its unbuffered equivalent (40.0% compared to 41.6-43.2% VS destruction). As mentioned in the previous section, collecting representative solids samples was quite difficult, and therefore composite samples of reactor sludge were used for solids analysis. However, this did not remove all sampling errors and the variation between measurements was still relatively large as can be seen in the error bars are shown in Figure 33.

Table 23 Summary of Single-stage HF Results (Figures averaged over final operational period, standard deviation in brackets)

HRT (Days)	20	5.2	2.3	1.6	1.75
SRT (Days)	20	20	20	20	20
pH control (buffer)	No	No	No	No	Yes
VFA (as COD) concentration (mg COD l^{-1})	11840 (760)	3420 (410)	2150 (160)	1430 (240)	1540 (310)
VFA (as COD) specific production (g COD g^{-1} VS added)	0.166 (0.011)	0.228 (0.027)	0.257 (0.019)	0.246 (0.042)	0.225 (0.039)
pH	5.22 (0.17)	5.46 (0.22)	5.45 (0.25)	5.62 (0.30)	6.93 (0.15)
Average SMP (STP l g^{-1} VS added)	Not Measured	Not Measured	Not Measured	Not Measured	0.049 (0.025)
TS destruction (%)	27.6 (3.8)	37.2 (2.5)	38.7 (0.9)	37.4 (3.6)	38.3 (4.8)
VS destruction (%)	29.5 (4.4)	42.4 (2.4)	43.2 (3.0)	41.6 (6.0)	40.0 (4.4)

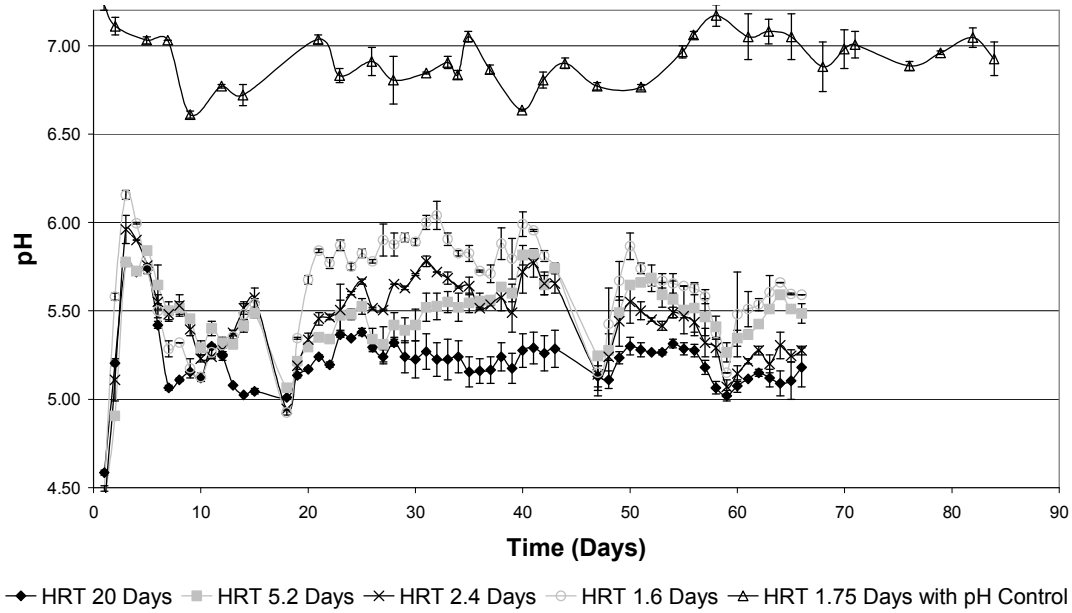


Figure 30 Single-stage HF pH

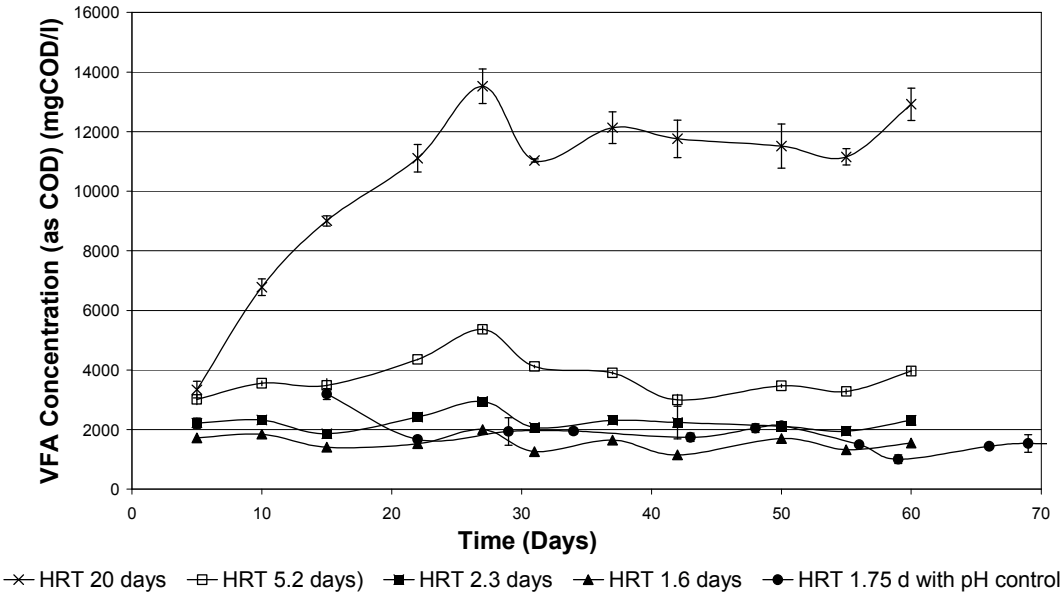


Figure 31 Single-stage HF VFA (as COD) Concentration

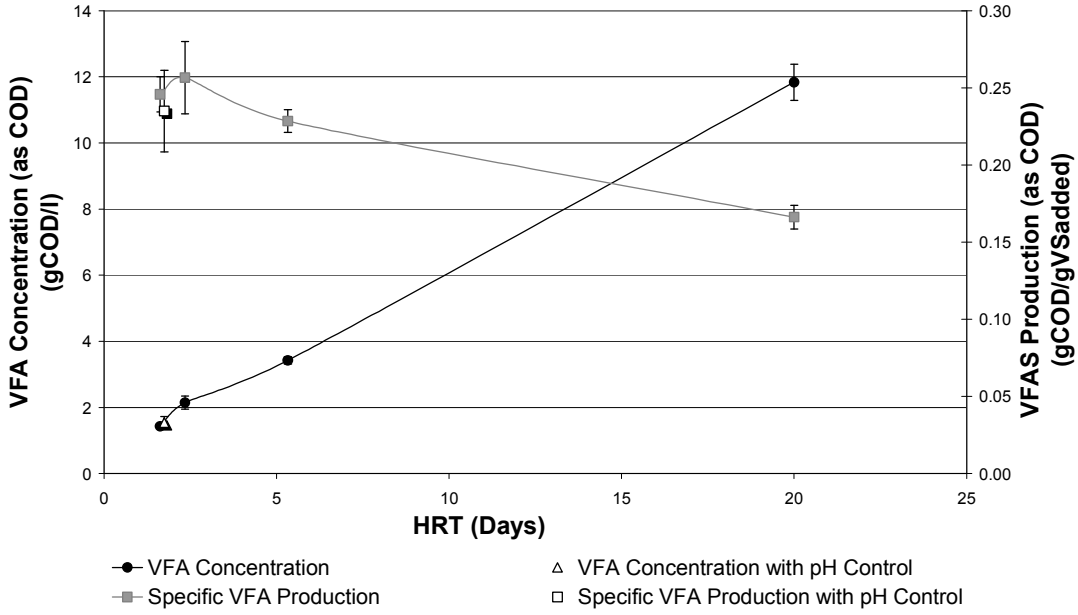


Figure 32 Single-stage HF VFA Concentration and Specific Production

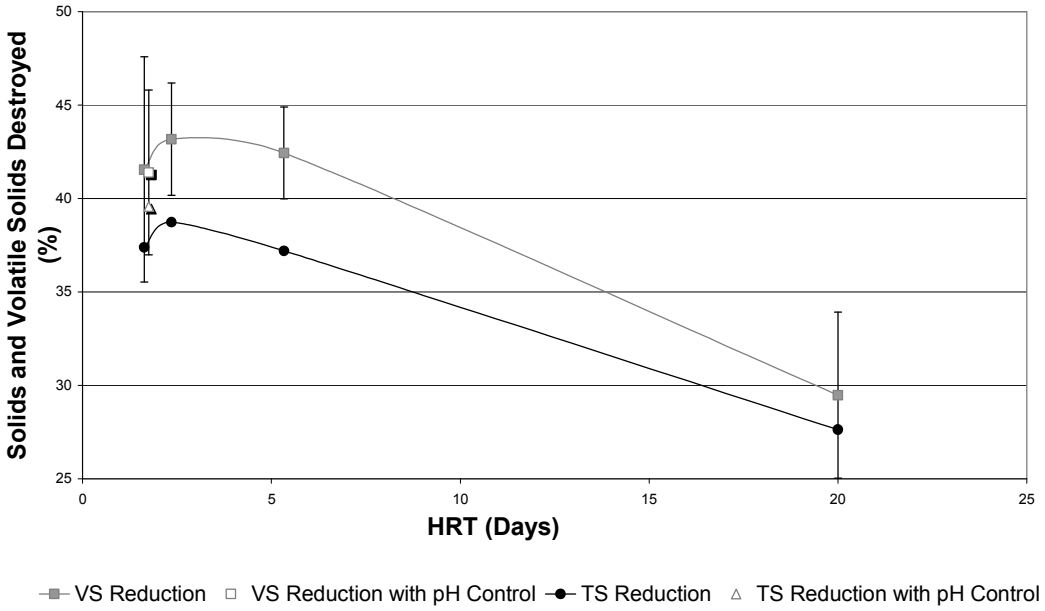


Figure 33 Single-stage HF Solids Reduction

5.5 Two-stage Hydraulic Flush

The two-stage HF trial consisted of 8 1st stage HF reactors and 4 2nd stage AF reactors. Duplicate HF reactors were coupled with a single AF reactor to form a two-stage process. The reactor systems are identified by whether the pH was controlled by buffer additions, and the OLR (either 3.75 or 7.5 g VS l^{-1} d^{-1}). A detailed description of the procedure used can be found on page 75. A summary of the results of the two-stage HF trial is shown in Table 24.

The trial lasted a total of 131 days although the non-buffered systems reached steady state at an earlier period were stopped after 90 and 85 days. The trial was originally planned to only last a total of 60-70 days but there were some operational issues during the test leading to large disturbances to the reactors early on in the trial. These were solved and the reactors were allowed to become stable again before the trial was terminated.

The large biogas production in the HF (1st stage) reactors was problematic because the thick digestate was able to trap the gas bubbles causing the level in the reactor to increase. This increase often caused blockages in the gas collection line which made digestate leak out of the top of the draught tube. This meant that on a number of occasions in the first 30 days of trial, a large quantity of digestate in the 1st stage was lost and had to be made up with water or buffer. This problem was eventually solved by optimizing the mixing speed and the stirrer design to allow the gas bubbles to escape by ensuring adequate agitation.

The pH which was close to neutral in the inoculum quickly decreased in all 1st stage reactors and for the first 40 days the reactors were relatively acidic as can be seen in Figure 34. The extent of this sharp decrease was related to the loading rate and buffer addition since the largest drop in pH was shown by the highly loaded reactors without pH control (pH 5.1) and the smallest was seen in the low loaded reactors with pH control (pH 6.0). This acidic phase was followed in all cases by a gradual recovery to the stable operational pH in the range 6.5-7.0 as shown in Figure 34.

Although the total biogas and methane production were quite erratic throughout the trial, the average values for all four two-stage systems was similar at 0.22-0.24 l g^{-1} VS added. The methane production measured on a daily basis is shown in Figure 35, and values averaged over 4 days are shown in Figure 36. Figure 37 shows the proportion of the methane produced in the1st stage throughout the trial and it can be seen to increase with pH in these reactors. At stable operating conditions 77-86% of the total system methane came from the 1st stage where methanogenic conditions had become prevalent.

The 1st and 2nd stage effluent COD concentrations are shown in Figure 38 and Figure 39 respectively. It can be seen that for much of the trial in the two buffered systems 1 - 2 g SCOD l^{-1} was circulated back and forth between the first and second stage reactors, without being degraded; the equivalent for the unbuffered systems was lower. Figure 40 shows the specific SCOD production from the 1st stage reactors, which in all cases started high and decreased to steady state values of around 0.05-0.15 g COD g^{-1} VS added.

The 2nd stage alkalinities remained stable throughout the trial and are shown in Figure 41. The intermediate alkalinities of all 2nd stage reactors were similar at around 400-800 mg$CaCO_3$ l^{-1} confirming the low VFA concentration of the 2nd stage effluents as shown in Table 24. The effect of increased loading (without pH control) was higher values for both partial and TA, and pH control increased these parameters further.

Temperature Shock

For two days starting on day 106 for the pH controlled systems and on day 26 for the highly loaded reactors without pH control, the temperature of the water bath containing the 1st stage reactors was accidentally changed from 35°C to 50°C. At first this was thought to mean the end of any useful results from this trial; but the reactors gradually recovered from this disturbance and returned to approximately the earlier operating conditions. The recovery of the reactors was similar to their response at the beginning of the trial. This effect can be seen in a change and then recovery in pH, total methane production, methane production in the 1st stage, 1st stage effluent and specific COD and 2nd stage

effluent COD, in Figure 34, Figure 36, Figure 37, Figure 39 and Figure 38 respectively, at the appropriate times in the trial.

Isolation of the HF reactor

At day 64 the 1st stage reactors at OLR of 7.5 g VS l^{-1} d^{-1} without pH control were isolated from the AF reactor. In the last 20 days of operation, the methane production decreased, the COD concentration increased and the pH decreased, all indicating the failure of the process. The trial was terminated on day 85.

Table 24 Summary of Two-stage HF Results (Figures averaged over stable operational period shown, standard deviation in brackets)

Description	No pH Control 3.75 g VS l^{-1} d^{-1}	pH Control 3.75 g VS l^{-1} d^{-1}	No pH Control 7.5 g VS l^{-1} d^{-1}	pH Control 7.5 g VS l^{-1} d^{-1}
Length of trial (Days)	90	130	85	130
Stable operational period (Days)	50-90	50-90	40-60	50-90
1st stage SCOD concentration (mg COD l^{-1})	1228 (342)	1354 (293)	2094 (585)	3744 (470)
HF SCOD specific production (g COD g^{-1} VS added)	0.159 (0.047)	0.081 (0.022)	0.109 (0.029)	0.173 (0.004)
% of SCOD as VFA in 1st stage	73	22	60	49
pH (HF, AF)	6.53 (0.09) 7.17 (0.14)	7.05 (0.07) 7.56 (0.14)	6.72 (0.04) 7.20 (0.07)	7.00 (0.07) 7.56 (0.10)
SMP (STP l g^{-1} VS added)	0.23 (0.04)	0.24 (0.06)	0.24 (0.05)	0.22 (0.04)
Methane content of biogas (%) (HF, AF)	54 (2) 87 (5)	58 (2) 89 (5)	52 (2) 63 (5)	51 (2) 86 (4)
% of methane produced in HF	77 (10)	86 (9)	87 (8)	87 (8)
TS destruction (%)	50 (2)	49(2)	51 (2)	55 (2)
VS destruction (%)	58 (2)	56 (2)		57 (2)
2nd stage SCOD concentration (mg COD l^{-1})	276 (96)	830 (267)	658 (288)	1496 (426)
% of SCOD as VFA in AFR	9	4	8	3

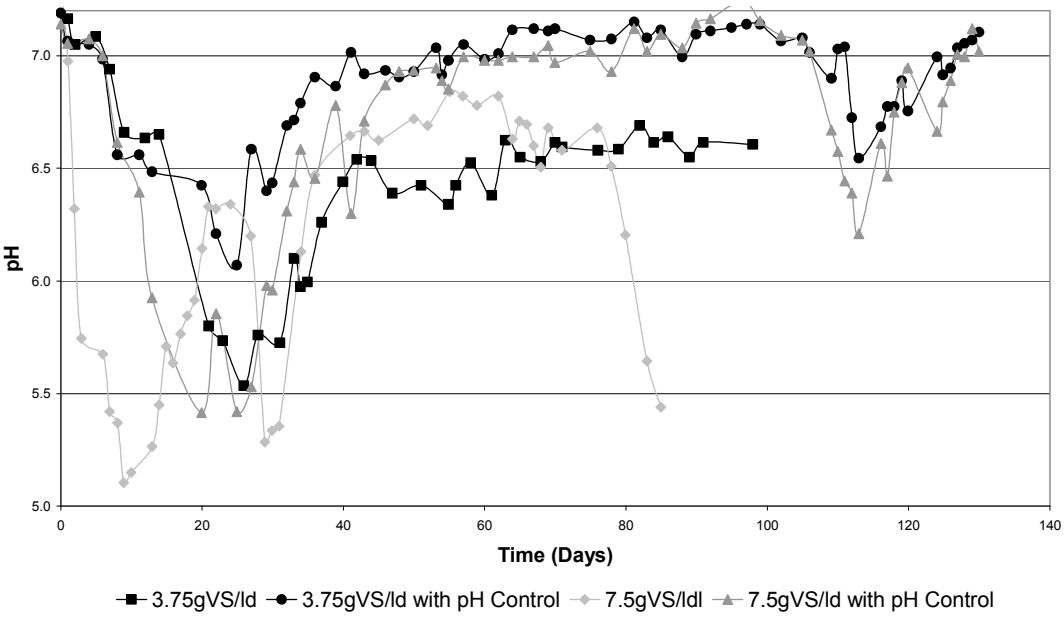


Figure 34 pH of the 1st Stage HF Reactors in Two-Stage HF/AF Systems

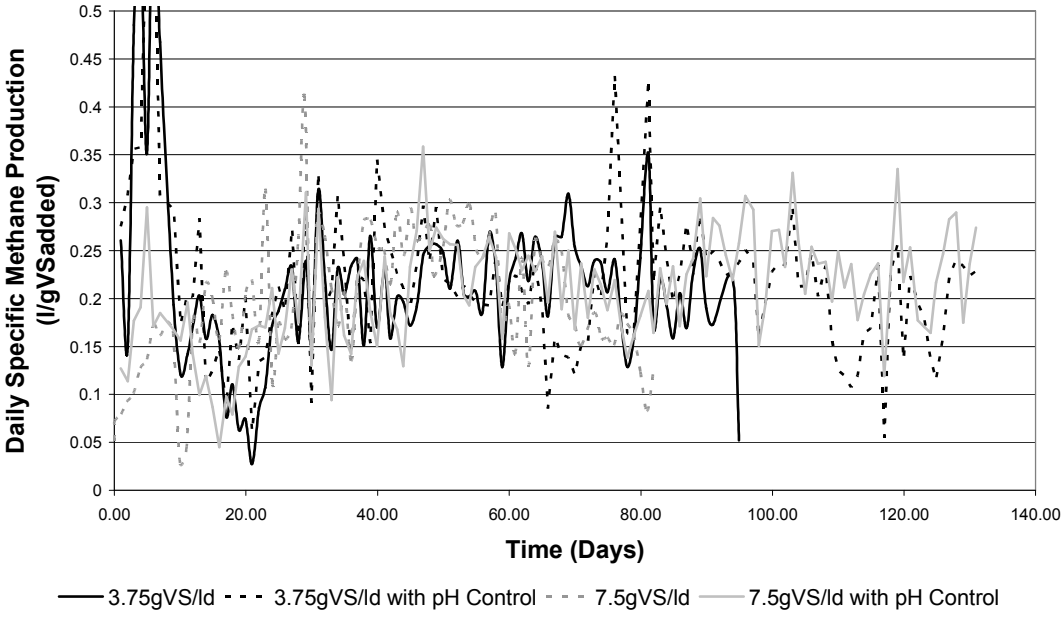


Figure 35 Total Specific Methane Production of the Two Stage HF/AF Systems

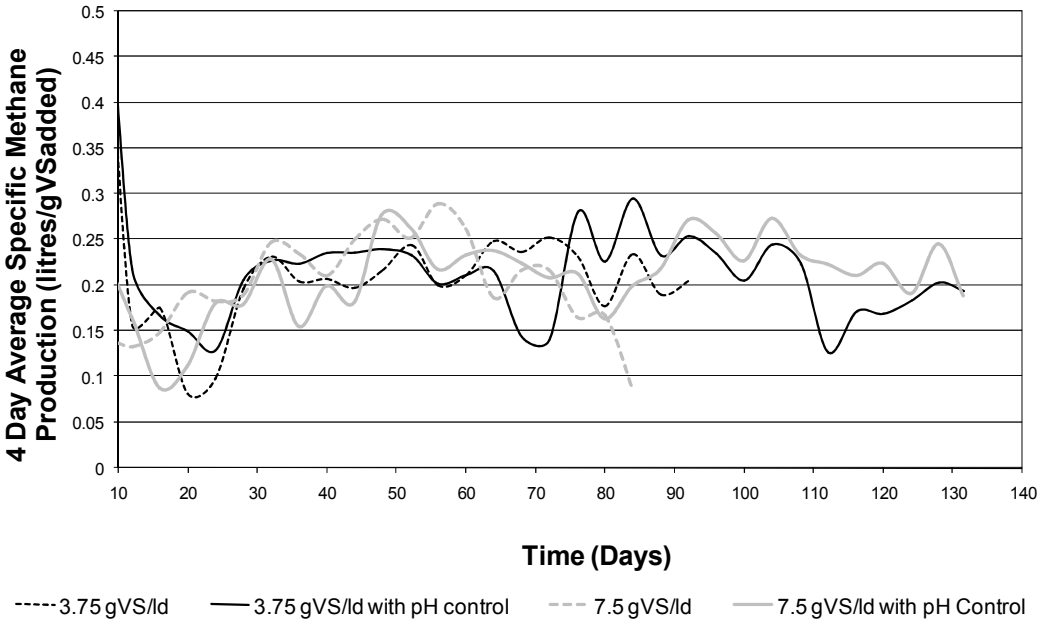


Figure 36 Total Specific Methane Production of the Two Stage HF/AF Systems (4 Day Average)

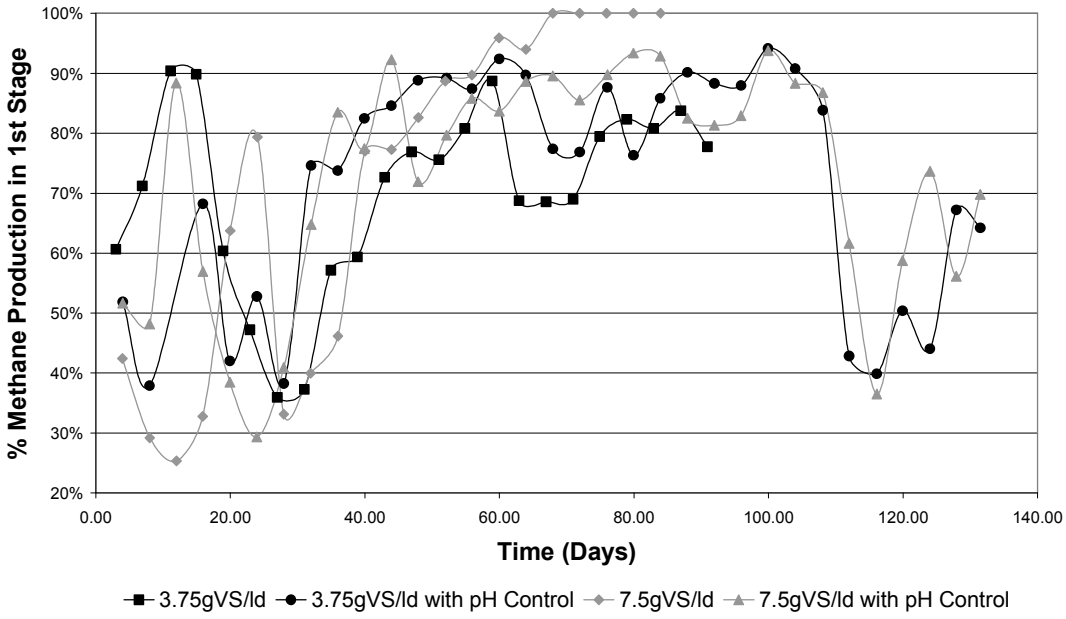


Figure 37 Methane Contribution of 1st Stage HF Reactors in Two-stage HF/AF Systems

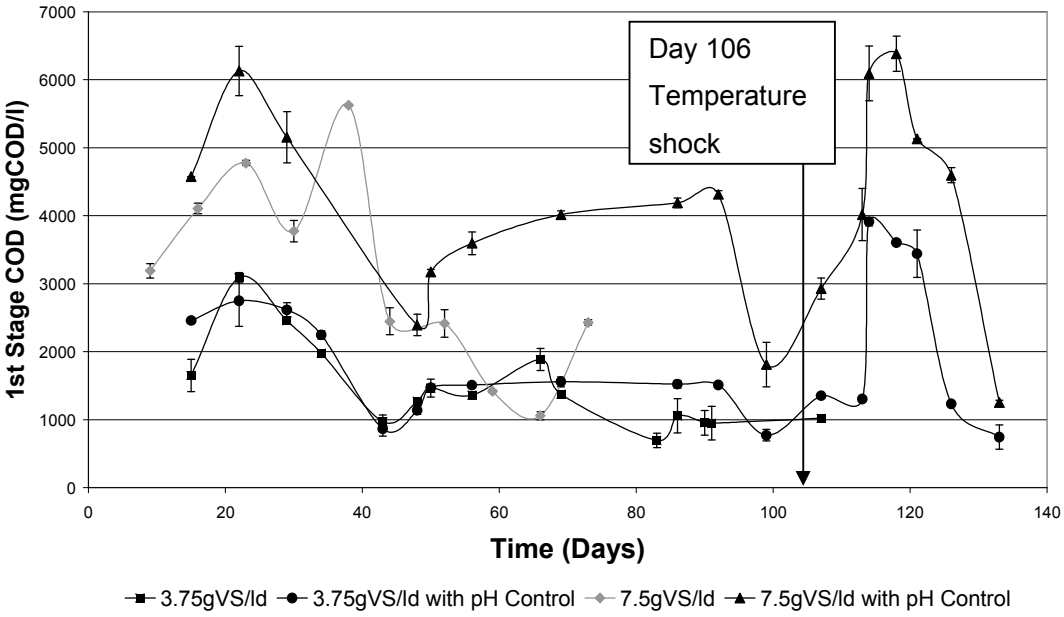


Figure 38 1st Stage HF Reactor Effluent SCOD in Two-stage HF/AF Systems

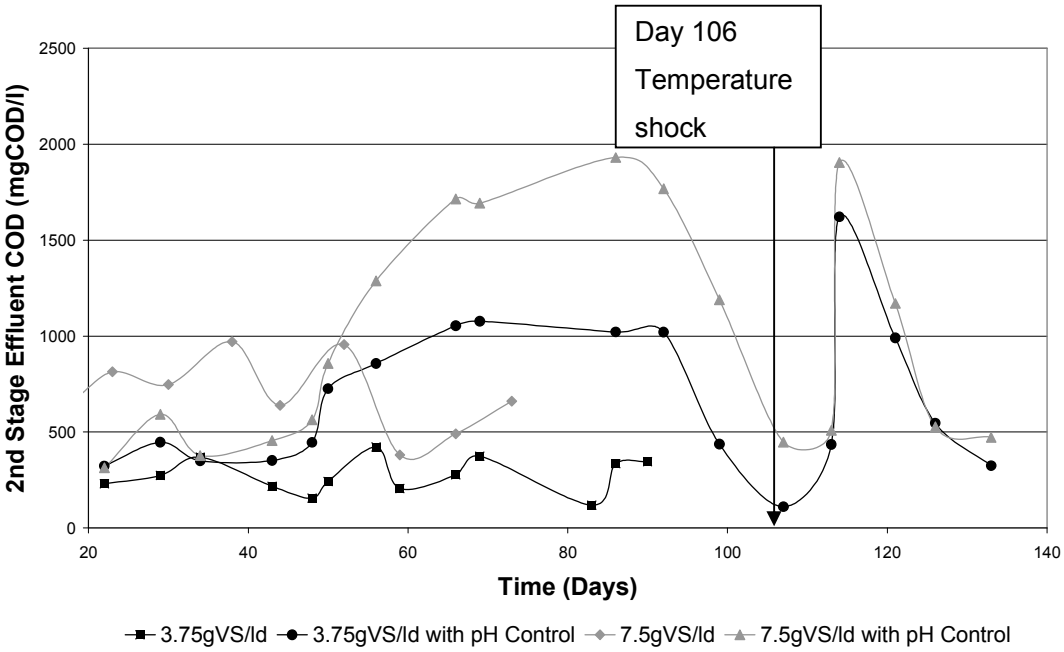


Figure 39 2nd Stage AF Reactor Effluent SCOD in Two-stage HF/AF Systems

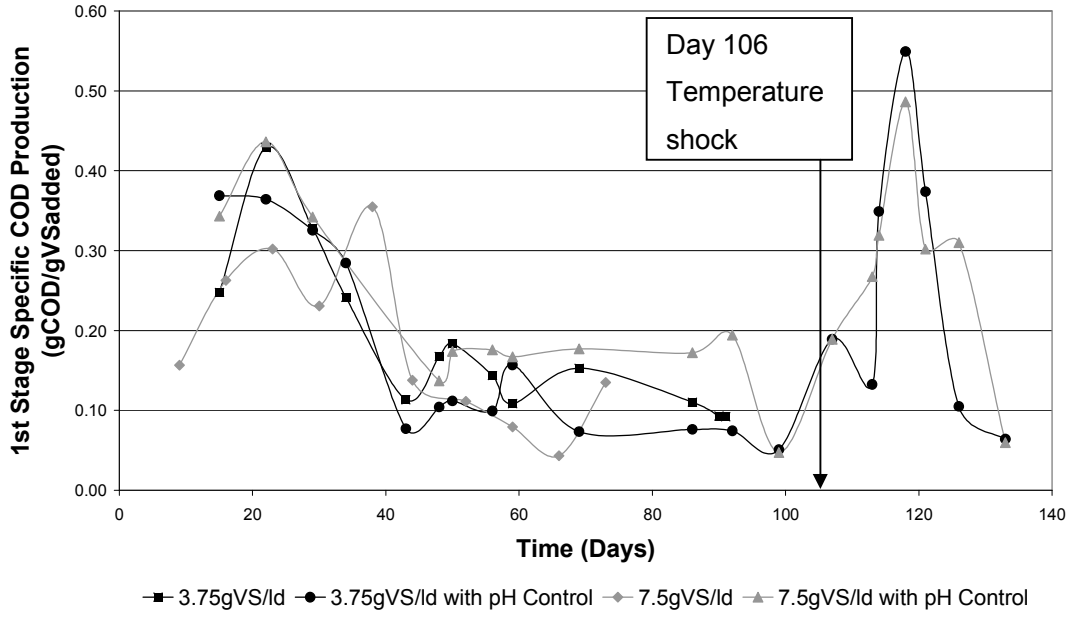


Figure 40 1st Stage HF Reactor Specific SCOD Production in Two-stage HF/AF Systems

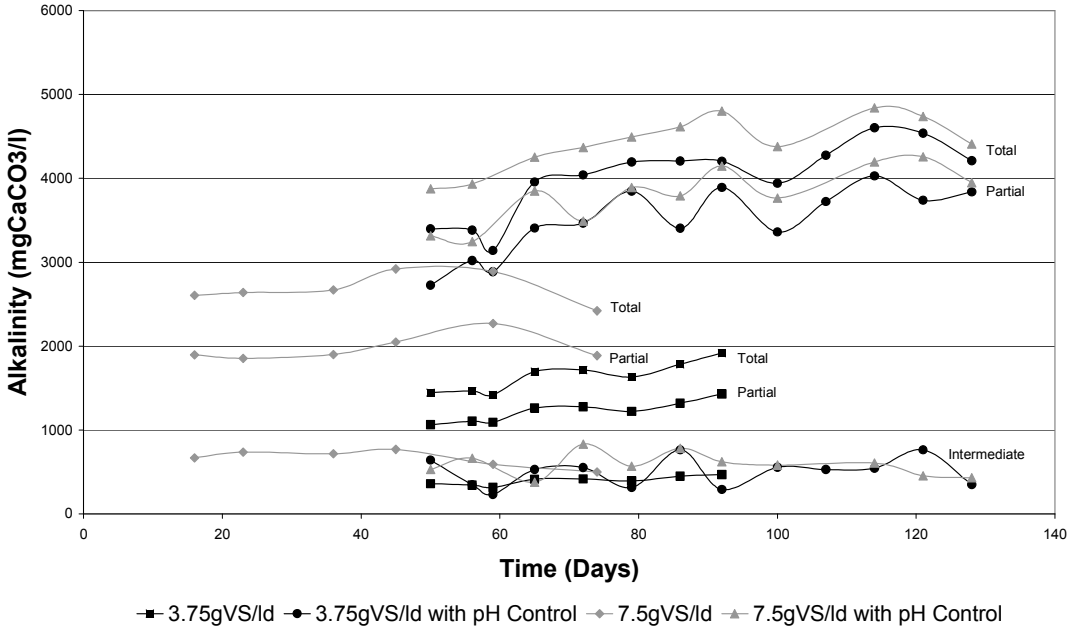


Figure 41 2nd Stage AF Reactor Total, Partial and Intermediate Alkalinities in Two-stage HF/AF Systems

5.6 Mesh Filter Bioreactors

In this trial the viability of using filtration rather than centrifugation as the means to decouple the hydraulic and solids retention time in the 1st stage reactors was tested. The full details of the procedures used can be found on page 77.

The MFBR trial lasted 85 days during which continuous filtration was sustained in the 30, 100 and 140 μm pore size MFBRs. No replacement of meshes was required during the operational period and no physical damage was seen on inspection when the mesh apparatus was removed from the reactors at the end of the run.

The main results are summarised in Table 25. Values are for samples taken from the reactors between days 65-85, designated as the steady state sampling period. Standard deviations are given in brackets where appropriate; the absence of these indicates that the value was from a spot sample rather than over the stable period since for some measurements, such as effluent VS content, a large sample was required. Removing this volume during the operational period could have significantly altered conditions in the reactors and thus these measurements were only taken at the end of the run.

During the early stages of the trial, all three 1st stage reactors went through a period of relatively low pH as shown in Figure 42. The mesh reactors reached minima of pH 6.05, 5.98 and 5.95 for 30, 100 and 140 μm respectively. In the 140 μm MFBR, an initial increase in pH was followed by a decrease towards the end of the trial whilst the other MFBRs and the HF reactor showed a gradual increase to the steady state values. pH remained steady in the AF reactors.

The daily SMP of the two-stage systems is shown in Figure 43 and showed considerable variation. The 7-day average of the same data in Figure 44 shows, however, that the three systems performed similarly in terms of overall methane production. This is confirmed by the average values in Table 25. The similarity in overall performance of the three systems is further confirmed by the TS and VS destruction rates.

The proportion of methane produced in the 1st stage reactor is shown in Figure 45 and as in the two stage HF trial (see page 101) shows a gradual increase with increasing 1st stage pH and decreasing 1st stage effluent COD, as shown in Figure 46. Analysis of the results in Table 25 reveals that with increasing pore size, pH became more acidic in both the 1st and 2nd stage, the soluble COD, TS and VS content of the 1st stage effluent was greater, while the methane production from the 1st stage became a smaller proportion of the whole.

When the OLR was increased to 7.5 g VS l^{-1} d^{-1} on the 30 and 100 µm MFBRs, filtration continued for approximately 8 days before the digestate became thick and the stirring motors failed. Since the stirrer motor also performed the scouring of the mesh, the meshes quickly became blocked and the test was terminated.

Table 25 Summary of MFBR Trial Results (Figures averaged over period 65-85 days, standard deviation in brackets)

Mesh size	30 µm	100 µm	140 µm
Two-Stage SMP (STP l g^{-1} VS added)	0.21 (0.04)	0.22 (0.03)	0.21 (0.05)
% of total methane produced in 1st Stage	72% (11)	50% (6)	49% (13)
SCOD concentration in 1st stage (mg l^{-1})	965 (257)	1365 (354)	1812 (44)
VFA concentration (mg COD l^{-1}) in 1st stage	297 (80)	922 (95)	470 (58)
SCOD concentration in 2nd stage (mg l^{-1})	434 (66)	466 (116)	341 (101)
VFA concentration (mg COD l^{-1}) in 2nd stage	<10	<10	<10
1st stage pH	6.56 (0.06)	6.44 (0.06)	6.21 (0.12)
2nd stage pH	6.99 (0.05)	6.98 (0.05)	6.79 (0.10)
TS destruction (%)	68% (2)	73% (1)	72% (3)
VS destruction (%)	71% (2)	74% (1)	72% (3)
Total alkalinity (mgCaCO3 l^{-1})	2527 (260)	2773 (72)	2549 (1460)
Alkalinity ratio	0.54 (0.03)	0.57 (0.02)	0.50 (0.11)
VS concentration in 1st stage effluent (g l^{-1})	3.54	5.42	7.81
VS concentration in 2nd stage effluent (g l^{-1})	1.92	2.20	2.48
Derived Quantities			
Specific methane production of AF reactors (STP l g^{-1}SCODdegraded)	0.61	0.68	0.40
VS reduction in AF reactor (g day^{-1})	1.63	3.22	5.33

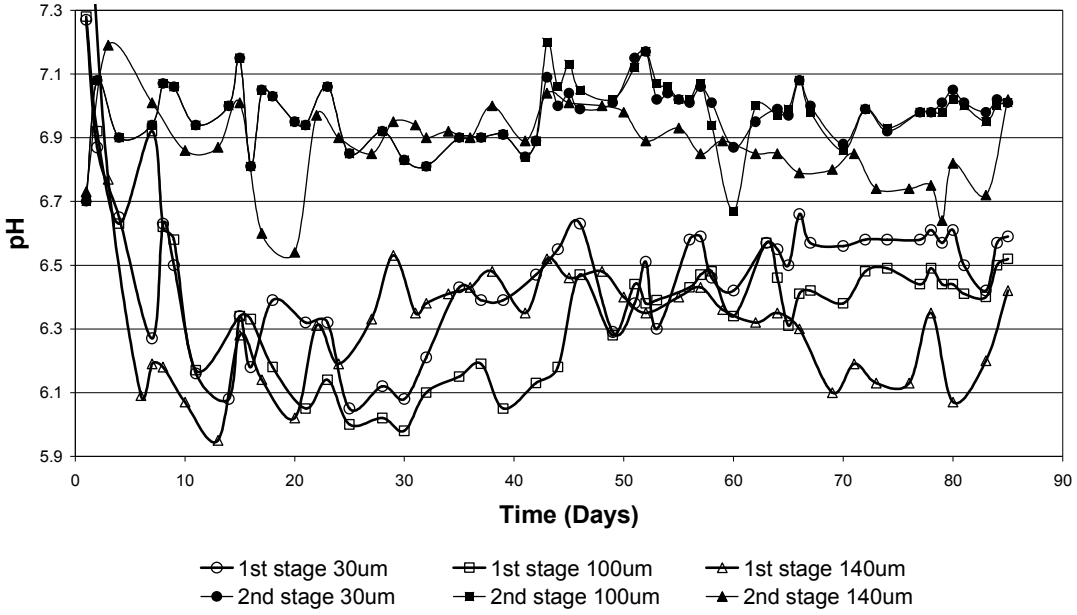


Figure 42 1st and 2nd Stage Effluent pH in Two-stage MFBR/AF Systems

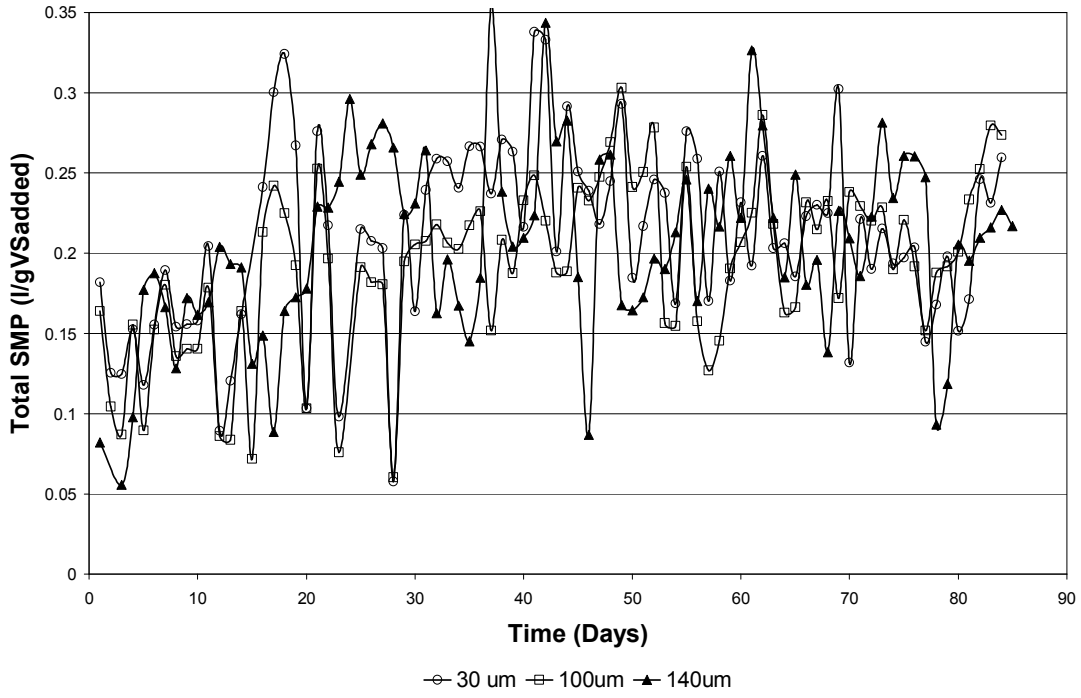


Figure 43 Total Specific Methane Production of Two-stage MFBR/AF Systems

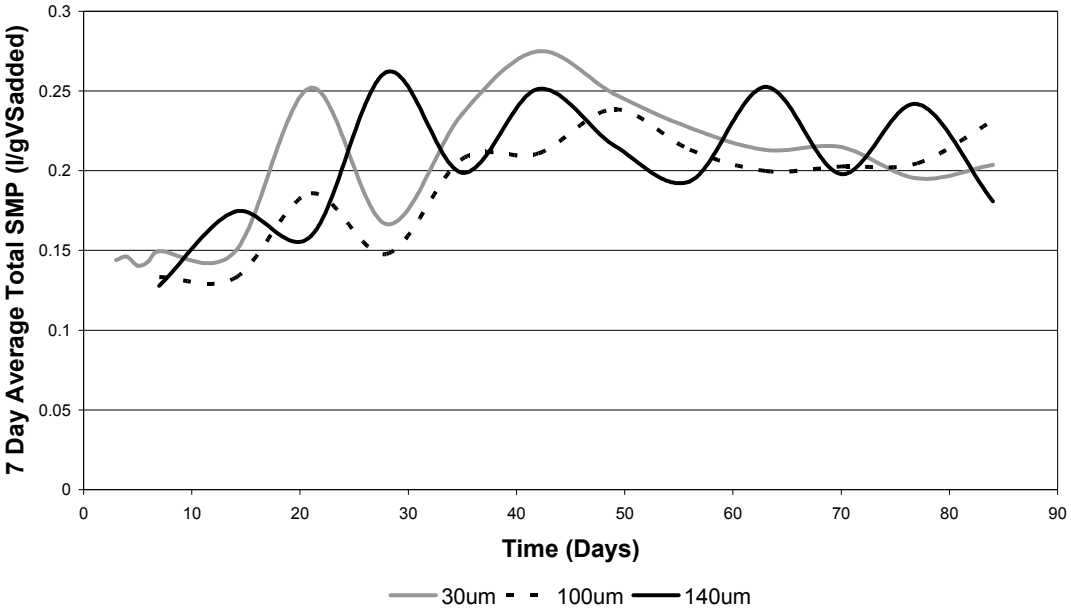


Figure 44 7 Day AverageTotal Specific Methane Production of Two-stage MFBR/AF Systems

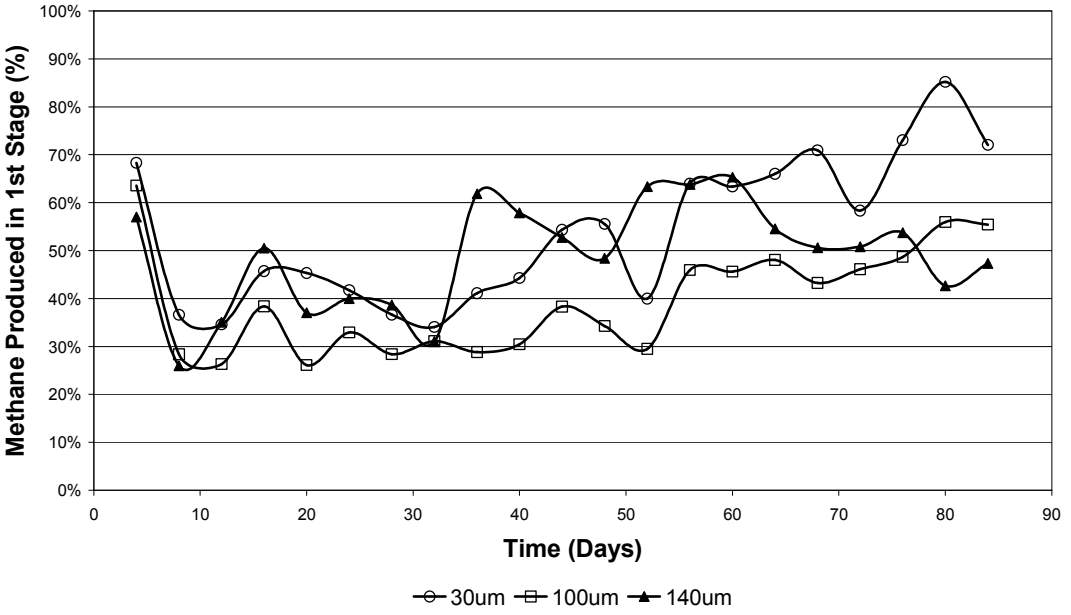


Figure 45 Methane Contribution of 1st Stage MFBR in Two-stage MFBR/AF Systems

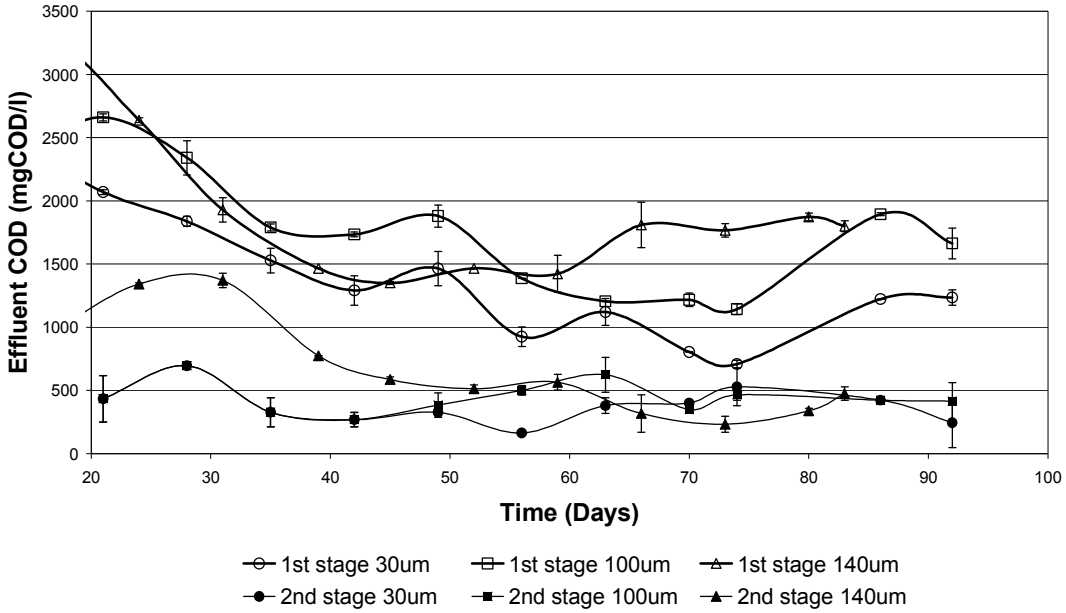


Figure 46 1st and 2nd Stage Effluent SCOD in Two-stage MFBR/AF Systems

5.7 Rotating Drum Mesh Bioreactors

In this trial the RDMBR design was tested. Two RDMBR and two AF reactors were operated in duplicate two-stage systems. An OLR of 7.5 g VS l^{-1} d^{-1} was applied to these reactors and the liquid effluent was fed to an AF reactor. After stable operation had been reached one RDMBR was isolated from its second stage reactor and the OLR was doubled to 15 g VS l^{-1} d^{-1} on the other.

A summary of the main operational parameters from the RDMBR trial is given in Table 26. The trial lasted for 87 days during which time the RDMBRs continuously filtered the SBMW digestate with no fouling problems. The rotating drum was able to mix the high solids digestate with little mechanical effort, avoiding the stirring problems encountered in previous trials.

As mentioned in section 4.7, during the first 72 days of the trial the reactor systems were fed at an OLR of 7.5 g VS l^{-1} d^{-1} in duplicate, and the values summarised in Table 26 are averages over the period 56-72 days which was deemed the steady state period. The overall performance of the systems, as indicated by the SMP over this period, was similar at 0.20 l g^{-1} VS added. The total system methane production is shown in Figure 48. The lower methane production of S2 can be attributed to intermittent leakage from RDMBR2, which was finally remedied on day 56 of the trial (Figure 49). The daily methane production was quite erratic although the rolling average showed much more consistent results.

The evolution of pH throughout the trial is shown in Figure 47 and as seen in previous trials the pH of the 1st stage reactors dropped at the beginning of the trial followed by a gradual recovery to steady state values. Also as in earlier trials the gradual increase in pH in the 1st stage reactors occurred simultaneously with a decrease in the SCOD of the effluent from these reactors, as seen in Figure 50.

Table 27 gives calculations regarding the COD inputs and outputs of the AF reactors for the whole duration of this trial. From the methane production and SCOD measurements made it is possible to perform a balance around these

reactors. It can be seen in this table that the total methane production of the AF reactors was greater than the theoretical production from SCOD alone, with the additional methane presumably coming from the solids present in the 1st stage effluent. If the methane production above theoretical is related to the degradation of VS in the AF reactor it can be seen that similar values of SMP occur in both systems (0.24 and 0.23 l g^{-1} VS destroyed).

A mass balance was performed around the reactor systems, the results of which are shown in Table 28. The period for the mass balance was chosen as the final week of the stable operational period, this being the closest to steady state conditions. Since the mass balance boundary was around the complete two-stage system, inter-reactor mass transfer was not taken into account. Over the period of one week, the deficit in solids for the systems was 4.3 and 1.1 g TS for S1 and S2 respectively and the direction of the deficit was such that more mass was seemingly added to the reactors than removed. Deficit is expected in an experimental mass balance since as well as the usual experimental errors in measuring the solids there are some solid removal routes which are difficult to quantify; The removal of volatile compounds (VFA, NH_3) in biogas and removal of small amounts of reactor contents during feeding and sampling the reactors (e.g. on stirring implements, beakers, spillages etc.).

After 72 days, RDMBR1 was isolated from its 2nd stage reactor for two weeks. The average SMP over this period decreased from the previous stage of the trial to 0.15 (0.02) l g^{-1} VS added. There was no evidence of process instability, as pH and SCOD remained stable over this period as can be seen in Figure 47 and Figure 50 respectively. For the same period, the OLR applied to S2 was doubled to 15 g VS l^{-1} d^{-1} and similarly, a decrease in SMP was observed to 0.16 (0.02) l g^{-1} VS added with no signs of process instability.

5.7.1 Digestate Residual Methane Potential

After 60 days of incubation at 37°C the residual methane potential of the digestate taken from the reactors on day 72 of the trial was measured. The results are summarised in Table 29.

Table 26 Summary of RDMBR Trial Results

(Figures averaged over period 56-72 days, standard deviation in brackets)

System number	S1 (RDMBR1, AF1)	S2 (RDMBR2, AF2)
Two-Stage SMP (STP l g^{-1} VS added)	0.20 (0.04)	0.20 (0.04)
Total methane produced in 1st Stage (%)	87 (2)	88 (4)
1st stage methane composition of biogas (%)	43 (2)	44 (0)
2nd stage methane composition of biogas (%)	68 (5)	72 (1)
SCOD concentration in 1st stage (mg l^{-1})	2101 (202)	2189 (282)
VFA concentration (mg COD l^{-1}) in 1st stage	15	23
SCOD concentration in 2nd stage (mg l^{-1})	1710 (218)	1696 (108)
VFA concentration (mg COD l^{-1}) in 2nd stage	<10	<10
1st stage pH	6.69 (0.05)	6.68 (0.07)
2nd stage pH	6.86 (0.05)	6.86 (0.05)
TS destruction (%)	58	55
VS destruction (%)	62	60
2nd stage total alkalinity (mg $CaCO_3$ l^{-1})	3103 (164)	2779 (68)
2nd stage alkalinity ratio	0.35 (0.11)	0.32 (0.10)
VS concentration in 1st stage effluent (g l^{-1})	4.51 (0.08)	4.62 (0.08)
VS concentration in 2nd stage effluent (g l^{-1})	3.59 (0.01)	4.35 (0.02)

Table 27 AF Calculations for RDMBR Trial (Figures averaged over period 0-72 days)

System Number	S1	S2
Total methane production (STP l)	58.1	53.2
Total COD degraded (g SCOD) (time based average)	107.3	109.7
SMP of 2nd stage (STP l g^{-1} SCOD degraded)	0.54	0.48
Theoretical SMP (STP l g^{-1} SCOD degraded)	0.35	0.35
Above theoretical methane production (STP l day^{-1})	0.28	0.20
VS reduction in AF reactor ($g\ day^{-1}$)	1.18	0.90
SMP of 2nd stage (STP l g^{-1} VS destroyed)	0.24	0.23

Table 28 TS Balance for the period 66-72 days

System Number	S1	S2
Solids Input		
SBMW 7.5 g VS l^{-1} d^{-1} VS/TS = 0.866 (g TS)	90.9	90.9
Solids Output		
Wet digestate removed (g)	284	294
Average TS of wet digestate (%)	13.4	13.7
Digestate removed (g TS)	38.1	40.3
Gaseous Output		
Volume of methane (STP l)	15.7	14.9
Volume of carbon dioxide (STP l)	19.0	19.8
Mass of methane (g)	11.2	10.6
Mass of carbon dioxide (g)	37.3	38.9
Total Output (g TS)	**86.6**	**89.8**
Mass Balance		
Mass deficit (Mass in - Mass out) (g TS)	4.3	1.1
Specific mass deficit (g TS g^{-1}TSAdded)	0.04	0.01

Table 29 Summary of Residual Methane Potential Results

System Number	S1	S2
Residual SMP based on digestate VS (STP l g^{-1} VS added)	0.122 (0.011)	0.090 (0.023)
Residual SMP based on input VS (STP l g^{-1} VS added) (assuming 60% VS destruction)	0.074	0.054
Reactor SMP + Residual SMP based on input VS (STP l g^{-1} VS added)	0.27	0.25

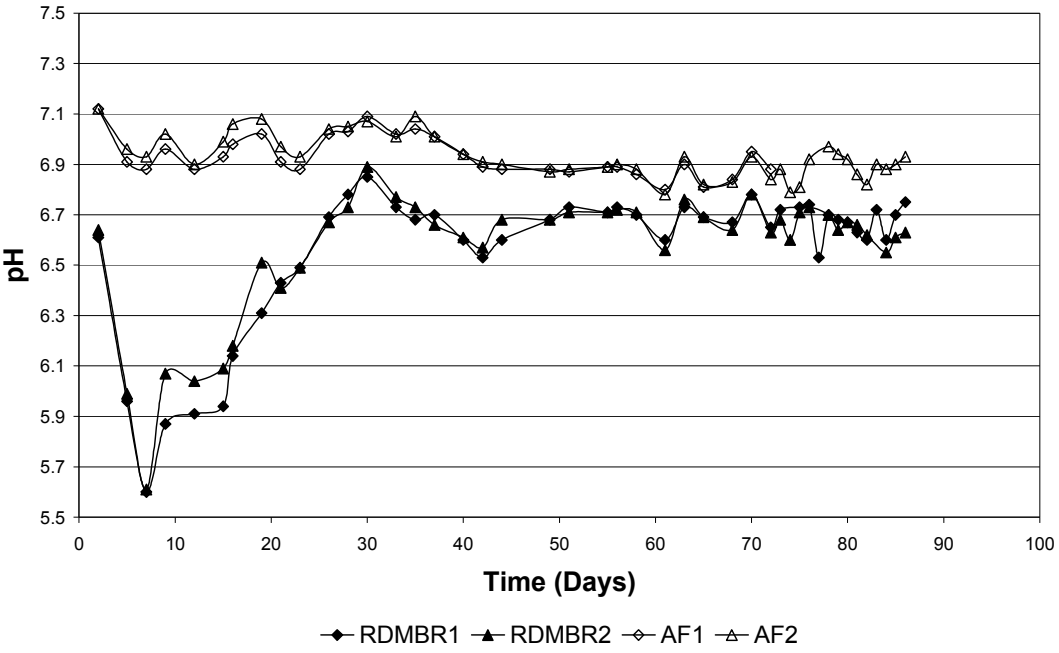


Figure 47 1st and 2nd Stage pH in Two-stage RDMBR/AF Systems

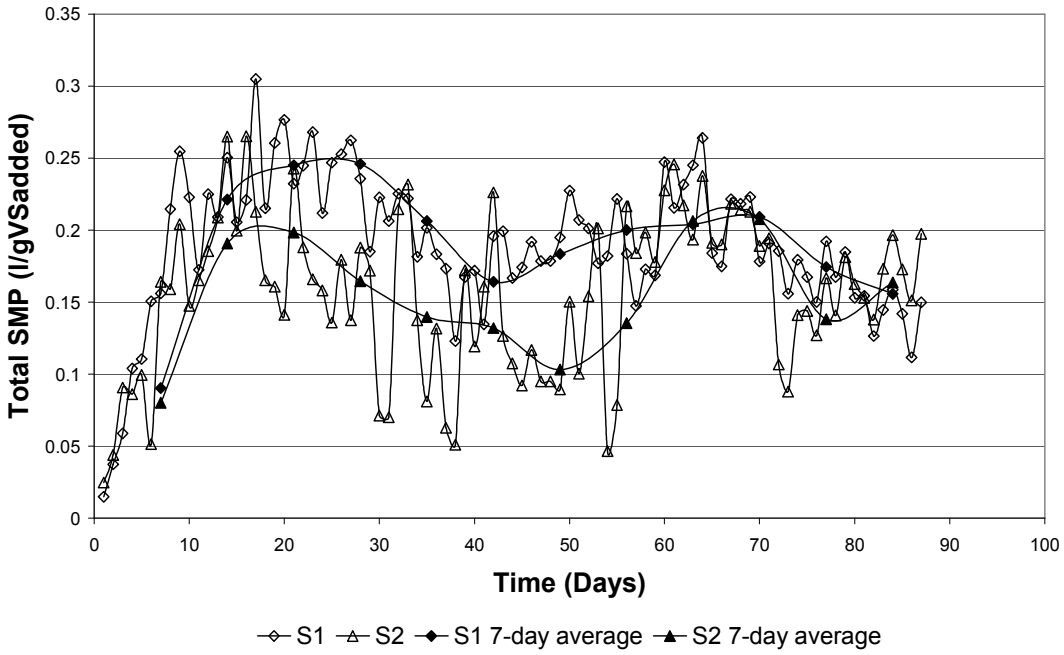


Figure 48 RDMBR Total System Methane Production

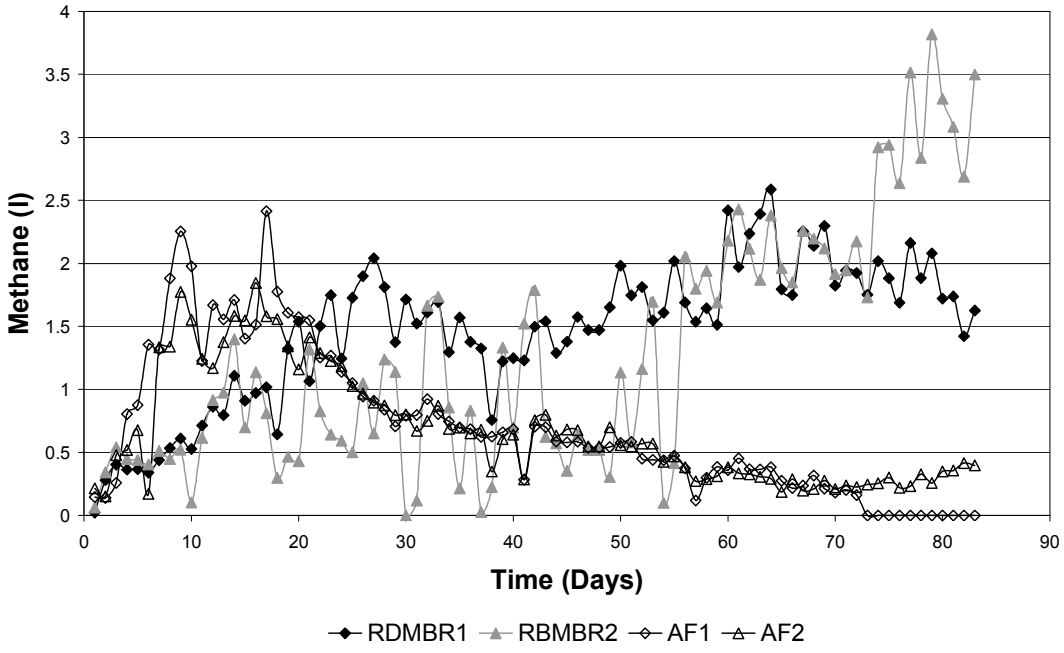


Figure 49 RDMBR and AF Individual Methane Production

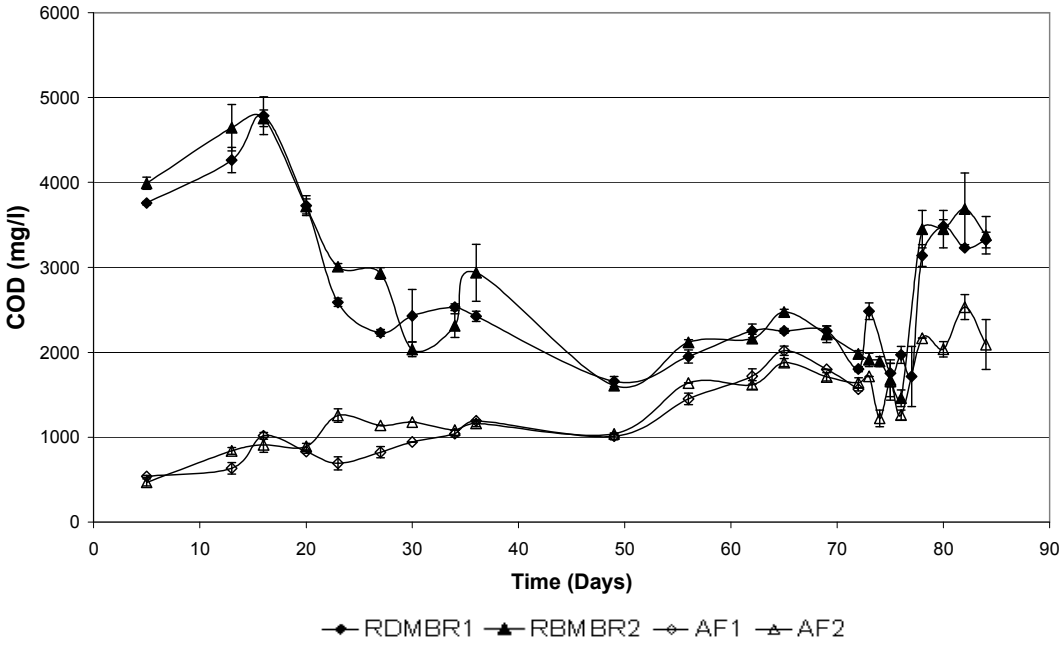


Figure 50 RDMBR and AF Reactor Effluent COD

6 Discussion

The results are discussed in order of presentation in the previous section. Initial results such as the characterisation of the feed and the preliminary trials, although not directly linked to the aims as cited in section 1.4.1, will enable insight to be gained into the AD process for this particular feed material. These initial findings will then referred back to during discussion of the two-stage HF and mesh trial results, allowing comparisons of the same degradation process under various operational and physical conditions.

6.1 Feedstock Characterisation

The BMP values of 0.28-0.32 l g^{-1} VS added for SBMW were both within the ranges found in the literature for BMW as shown in Table 5.

The results from the 2nd BMP test were considered more reliable than the 1st since the method of biogas collection and sampling was more rigorous. Additionally more replication was employed, with 3 control reactors and 9 substrate reactors. However the 1st BMP provides some additional insight since gas volume sampling was performed at much more regular intervals throughout the test. This allowed some dynamics of the degradation to be analysed, as will be discussed later.

The 1st BMP test produced methane at 0.28 l g^{-1} VS added, less than found in the 2nd test. The reason for this is thought to lie in the gas collection method which in the 1st BMP test was based on the use of water displacement gasometers of the trough type to continuously collect the biogas produced in the reactor. The barrier solution used in these gasometers was acidified tap water (pH 2), and since the biogas was in constant contact with the solution, which was in turn in contact with the atmosphere, some biogas was undoubtedly lost. On this basis it is suggested that this method of gas collection not be used where long term contact of the biogas with the gasometer solution occurs. Throughout the test the gas production rate decreased leading to an increase in the time between gasometer refills and increased contact time between the biogas and the barrier solution. Also the greater solubility of carbon dioxide (BS

2005) means that it is lost much more quickly than methane when a diffusion route is available through a barrier solution, and this explains why the apparent methane content of the biogas increased to a higher concentration in the 1st BMP than the 2nd.

The problem of biogas quantification in the 1st BMP would have been further exacerbated if samples for biogas composition were taken from the headspace of the reactor rather than the gasometer. This would have lead to additional errors because the produced gas and collected/stored gas have different compositions due to diffusion through the barrier solution. Fortunately methane, the gas of most interest, has rather low solubility and since the compositional samples were taken from the gasometer, where the volume was also measured, only the error caused by the solubility/diffusion of methane affects the BMP result. The problem of biogas diffusion can be reduced significantly by the use of acidified (pH 2) and saline (270 g l^{-1}) water in the gasometers. Carbon dioxide and methane are much less soluble in this solution and so the test gives a more accurate measure of both BMP and biogas potential (Yang and Speece 1986; Schonberg et al. 1997; Sponza 2003). Unfortunately saline solution was not used during the 1st BMP test leading to the biogas losses as described above.

The 2nd BMP test avoids the issues related to solubility and diffusion of biogas components through barrier solutions because the biogas was collected in Tedlar bags and measured periodically, when the gas bags became full. Water-filled gasometers were used to measure the volume of gas in the bags, but the contact time of the biogas with the gasometer liquid was short (<1 min).

The maximum degradability of the substrate in terms of VS destruction was 69% and 62% for the 1st and 2nd BMP test respectively. The method of calculation of the 1st relied upon the assumption that the specific methane production based on VS destruction of the inoculum and the substrate was the same. This was found not to be the case in the 2nd BMP where the VS destruction in both the substrate and control reactors was determined. The inoculum and substrate in this case produced methane at 0.39 and 0.52 l g^{-1}VS destroyed, accounting for the higher estimate of VS destruction in the 1st BMP.

If the calculation in Table 19 is updated to use the specific methane productions calculated in the 2nd BMP the ratio of VS destruction in the inoculum and substrate reactors comes out at 0.30:0.53 which equates to a VS destruction of 63%. This agrees well with the 62% calculated in the 2nd test.

The value of 0.113 d^{-1} for the 1st order hydrolysis constant found in the 1st BMP should not be greatly affected by the problems with measurement of methane production and VS destruction highlighted above. This is because calculation of the constant relies mainly upon the dynamics of the gas production rather than any absolute endpoint. The curve shown in Figure 17 indicates that the decay of the substrate does was not precisely conforming to 1st order mechanics as it is not straight, although a perfect fit could not be expected given the presence of the experimental errors. It can also be noted that the hydrolysis rate curve is above the line of best fit early in the test, when readily biodegradable components with greater hydrolysis constants are being hydrolyzed, and below the line when the degradation of more refractive components is taking place. Despite 1st order dynamics being only an approximation (R^2=0.84) of the hydrolysis process, the model is useful on a comparative basis and will be referred back to later.

The variation BMP values found in the literature, and the range of digestion characteristics shown in Table 6, mean that comparison of anaerobic systems treating different waste samples is difficult for benchmarking purposes. Instead the values of BMP, maximum TS and VS destruction and 1st order hydrolysis constant will be used as benchmarks with which to compare the AD performance of the subsequent trials.

6.2 Preliminary Trials

6.2.1 Single-stage

The single-stage trial was performed to acquire benchmarks of AD performance for SBMW with which more complex and novel reactor system could be compared. A description of the procedure used for this trial can be found on page 71 and the results are presented on page 87. The trial was characterized by a period of stable operation followed by a period of instability where disturbances in the reactor conditions caused three out of the four reactors to crash. The two periods will be discussed separately.

Stable Operation

All four reactors quickly reached a pseudo-steady state of operation where biogas production, pH, solids destruction and alkalinity were not changing significantly over time. The performance during this period was good, with specific methane production around 90% of the BMP of the feed material. Although the pH in the reactors was at the lower end of the optimal range for methanogenic bacteria (pH 6.5), it appears steady in Figure 19 and Figure 20. The specific methane yield at both loading rates was similar at 0.28 and 0.27 for OLRs of 2 and 2.5 g VS l^{-1} d^{-1} respectively. This suggests the process was running smoothly without any build-up of intermediates (Gerardi 2003f). Low VFA concentrations in all four reactors can be deduced from the low IA:PA ratios seen in Figure 23 and Figure 24 (Ripley et al. 1986). The solids destruction in the reactors was high at 50-55%, which was close to the maximum degradability as found in the BMP tests.

The daily specific methane production early in the trial as can be seen in Figure 21, was at times above that measured for the BMP test. Although initially this may seem unfeasible it can be explained given the methane produced by the inoculum. It can be seen in from the results of the BMP tests that the sewage sludge produces methane for a long time without extra feed, showing that it has available substrate within it. This theory is supported by the fact that the supposed surplus methane production decreases over time and the steady state methane production of the reactors (0.27-0.28 l g^{-1} VS added) is bellow the BMP value (0.32 l g^{-1} VS added).

The stable period of operation lasted around 100 days. At a 30-day SRT it would be expected that 96.7% of the inoculum of fresh sewage sludge would have been removed and replaced by MSW digestate, with only 3.3% of the original reactor contents remaining. Although in theory this should mean that the reactors were close to a steady state condition, during the later stages of the trial it was found that reactor conditions were very sensitive and easily disturbed to process failure.

Unstable Operation

During the unstable period of the trial, acidification and methanogenic failure occurred in three out of the four reactors (MH1, 3 and 4 at 134, 103 and 148 days respectively). This can be seen in the pH, methane production and alkalinity data shown in Figures 21-26. Disturbances in reactor working temperature documented in Table 21 can explain two of the failures. The temperature shocks occurred because another reactor trial, which was not part of this work, was terminated, and the reactors were emptied. Unfortunately these reactors were on the same heating loop as the four single-stage reactors and the difference in thermal inertia and heat losses in the system meant the internal reactor temperature dropped by 4°C.

After the first temperature change it can be clearly seen that the gas production and pH of reactor MH3 decreased, indicating a reduction in the activity of the methanogenic population, and the reactor crashed over the next few days. Although MH1 was subject to a similar shock and a drop in pH can be seen, it eventually recovered from this.

The sensitivity of anaerobic digesters to temperature is well known (Gerardi 2003d). If the methanogenic population are disturbed (e.g. by a temperature change) continued loading rates means a faster increase in concentration of VFA and therefore a greater likelihood of a reduction in pH leading to reactor failure.

After the second temperature shock, it can be seen that reactor MH4 suffered the same fate as MH3 and began to fail; the trial was terminated at this point.

Buffer solution was added to reactors MH1 and 2 in order to check that it would not kill or inhibit the digestion process. The successful application of a similar medium to support the growth of rumen protozoa in work on AD of BMW indicated that it was suitable (Gijzen et al. 1989). On day 130 of the trial reactor MH1 suffered process failure. The reason for this is not obvious given that the reactor conditions were undisturbed during this time. MH2, its duplicate, remained healthy for the rest of the trial and showed no signs of unstable operation and so it is unlikely that the crash was caused by the build-up of buffer solution components in the reactor. Additionally the buffer solution had only been used for the previous 20 days, meaning that concentrations in the reactor would be only around 50% of those in the solution itself. Further confirmation of the ability of the mixed culture to deal with the buffer solution can be seen in the results presented in section 5.5 where continuous two-stage systems were operated with buffer addition for over 130 days.

Stable single-stage digestion of BMW and other MSW-based materials has been achieved in many laboratory scale trials as well as in full-scale plants throughout the world (Mata-Alvarez et al. 2000). The fact that digestion of the SBMW used in this work was unstable in some cases suggests that it may have characteristics dissimilar to some other real wastes. The same food waste that was used as a component in the SBMW was also used by Climenhaga and Banks (2007), who found that single-stage digestion of this substrate was difficult without nutrient supplementation. These food waste digestion trials consisted of a long stable period followed by sudden failures. The authors suggested a number of possible causes for these failures, such as LCFA accumulation and trace element depletion or precipitation. A possibility is that the factors causing indigestibility of the food waste component also cause a similar failure in the SBMW fed reactors although this was not explored fully to ascertain this. In any case, for this feed material the shortcomings of single-stage digestion mentioned in the introduction to this thesis were highlighted in this trial. The maximum loading rate achieved in the single-stage system of 2.5 g VS l^{-1} d^{-1} is modest when compared with values cited in the literature making the SBMW used in this study an ideal material to test the stability of a two-stage system.

6.2.2 Kinetic Hydrolysis

The purpose of the kinetic hydrolysis trial was to gain insight into the hydrolysis process, in particular the effect of changing the HRT of completely stirred reactors between 2 and 10 days. The operational procedures are detailed on page 72 and the results are presented on 93. Methanogenic bacteria were washed out by the application of short retention times, and the methane production quickly decreased to almost negligible amounts. Lack of methanogenic conditions in all reactors led to an accumulation of VFAs to concentrations between 2 and 12 g l^{-1}, making the digestate very odorous.

Increased residence time in the reactor, which meant greater contact time between microorganisms and the substrate, led to greater solids destructions. It can be seen clearly in Figure 26 that the solids destruction increases with retention time. It is interesting to note, however, that the solids destruction is approximately linear, suggesting that the rate of destruction per day is roughly constant. Such a line would have an intercept of 9.6% which probably represents the readily degradable fraction of SBMW.

In section 6.1 it was commented that during the BMP test, the rate of hydrolysis was greater than predicted by first order mechanics in the early stages of the test and lower toward the end. This was attributed to different components of the substrate being more or less readily biodegradable. The solids destruction results shown in Figure 26 are another representation of this effect. The 2-day retention time reactor had a greater solubilisation rate because its main substrate was the readily biodegradable components of the feed material, the reactors with longer retention times also had access to these components, but they represented a smaller fraction of the total breakdown, thus leading to a lower solids destruction (per day retention time) rate.

Although this is a plausible physical explanation, the 1st order model applied to this solids destruction data gives some further insight; It can be seen in Figure 29 that the solids destruction data plotted in the form of gives a remarkably good straight line fit, suggesting that the hydrolysis process in the reactors was

indeed conforming to the 1st order model. This means the relatively high solids destruction rates seen in the 2-day retention time reactors can simply be explained by the nature of hydrolysis; that the highest rate always occurs during the early stages of degradation.

Another outcome of the kinetic analysis of the data from this trial was the calculation of the 1st order hydrolysis constant. Given the fit of the derived data shown in Figure 29, the hydrolysis constant can be calculated as the inverse of the intercept of the line, which gives a value of 0.109 d^{-1}, similar to the 0.113 d^{-1} obtained from the BMP test.

The main soluble products of hydrolysis were VFAs, accounting for 95-100% of the soluble COD in the reactors. The concentration of these was increased with the retention time of the reactor as shown in Figure 27. This is a direct consequence of the longer retention time and was caused by slower washout of soluble substances in the removed digestate. The increased acid concentration also caused the pH to decrease in the longer retention time reactors.

The specific VFA production was calculated for each of the sets of reactors and is also shown in Figure 27 and increases with retention time. There is a seemingly anomalous result at the 4-day retention time reactor, given that this increase in VFA production was not backed up by an increase in solids destruction. Discounting the anomalous result this data resembles the equivalent TS and VS destruction curves, which by a solids balance, are two different ways of assessing the same hydrolysis process. Again this data suggests a relatively constant rate of hydrolysis in the reactors, where increasing the residence time only increases the amount of degradation as expected by longer contact time between substrate and microorganisms. The similarity between the reactors is further illustrated by the VFA profiles shown in Figure 28, which show no trends with retention time, and show the main VFA products to be acetic and propionic acids.

At the beginning of this section it was remarked that the reactors conformed as expected to the physical model, and to a great extent this was true. For example it was expected that greater residence time in the reactor would lead to

a greater degree of solubilisation, greater VFA concentrations and lower pH. However, it was not expected, as summarized in the past few paragraphs that the reactors with different operational and physical conditions would have similar hydrolysis rates. Differences in pH, washout of microorganisms and VFA concentrations according to the literature do affect the rate of hydrolysis AD (Garcia et al. 1991; Babel et al. 2004; Hu et al. 2004), yet in this trial it seems that these have little difference.

Contrasting results were found in a similar investigation into the hydrolysis of maize (Heaven et al. 2008a), where over a range of HRT of 2-12 days a decrease in VS destruction was observed. The pH in these reactors was lower than in the kinetic hydrolysis trial (3.9-3.7) owing to the low natural buffering capacity of maize, which could have caused inhibition of the process at longer HRT where VFA and especially UVFA concentrations were greater.

An explanation of the similar hydrolysis rates seen in the kinetic hydrolysis trial in contrast to the maize study could be explained by the increased pH (5.4-6.1) leading to reduced inhibition of the hydrolysis process (Zhang et al. 2005). It is possible in this scenario that the effects of parameters such as washout, inhibition by pH/VFA, which vary with retention time, could be in balance. Biomass washout increases with decreasing retention time, as more organisms are being removed on a continuous basis. Conversely, the effects of pH and VFA inhibition increase with increasing retention time; VFAs are accumulated due to slower washout leading to low pH. Given that some inhibitory effects correlate positively with retention time and some negatively it is possible to imagine the opposing factors in balance and seemingly having no effect. This hypothesis could be further investigated by performing the kinetic hydrolysis trial at other OLR since this would alter the balance between the antagonistic effects of washout and VFA/pH.

The similarity of the hydrolysis constant under batch and continuous, methanogenic and non-methanogenic conditions suggests that hydrolysis is in fact the rate limiting component of the degradation, as suggested in the literature review (O'Sullivan et al. 2005). However, the constant rate under

various conditions perhaps suggests that enhanced biological hydrolysis rate by the alteration of physical conditions (such as HF) may be futile.

6.3 Single-stage Hydraulic Flush

This trial was a preliminary investigation into the effect of the HF on the anaerobic hydrolysis process. Reactors had a constant SRT and a range of HRT between 1.6 and 20. It was found that hydrolysis rates could be enhanced by removal of VFA and other soluble products as well as the resulting higher pH. There was no measured enhancement of the hydrolysis process by controlling the pH using buffer addition. Full details of the procedure used in this trial can be found on page 73 and the results are presented from page 97.

Early in the trial Methanogenic conditions were, as before, quickly eliminated in the reactors by the washout imposed by the HF itself. At the loading rate of 3.75 g VS l^{-1} d^{-1} the CSTR 'control' reactor quickly accumulated high concentrations of VFAs (12 g l^{-1}) and accordingly the pH decreased.

Comparison of the control reactor with the reactors from the kinetic hydrolysis trials gives additional insight into the hydrolysis process. At SRT = HRT = 20 days, i.e. a completely mixed process, 27.6% TS destruction was achieved, and the specific COD (as VFA) production was 0.166 g COD g^{-1} VS added. The equivalent performance measures for the 10-day SRT reactor from section 6.2.2 are 27.4% and 0.168 g COD g^{-1} VS added, which are similar - with double the retention time, the performance was actually no better. This is important as it appears that the particular conditions in this trial (possible low pH and high VFA concentration) are limiting the biodegradability of the substrate to around half of that under other more favourable conditions (such as methanogenic, neutral pH). A similar result was found in a study into the single- stage hydrolysis of maize; reactors with 12 and 20 days HRT showed very little difference in hydrolysis performance (Heaven et al. 2008a).

The effect of decreasing the HRT in the three sets of unbuffered reactors was to increase the pH and TS destruction up to a maximum of 38.7% at a HRT of 2.4 days as shown in Figure 33. Note that all three of the flushed sets of reactors performed similarly suggesting that although the HF was important, the actual HRT is not important within the range investigated (1.6-5.2 days). This is in agreement with previous studies where the flushing or a reduction in HRT has a

beneficial effect on the hydrolysis process bellow a critical value, bellow which any further reduction has little benefit. This was found to be around estimated to be around 4 days for MSW (Banks and Wang 2000) and between 4 and 8 days for maize (Heaven et al. 2008a).

This trend of similar hydrolysis performance is also repeated when looking at the specific VFA production, and when compared with the control reactor and kinetic hydrolysis reactors in the previous section; the HF reactors performed significantly better. Compare 0.228-0.257 g COD g^{-1} VS added (Figure 32) for the HF reactors with 0.119-0.168 for the simple hydrolysis reactors. Hydraulic flushing increases the pH and decreases VFA concentration (Figure 31), and both of these are beneficial to the hydrolysis process as has been suggested by various other authors (Llabres-Luengo and Mata-Alvarez 1988; Babel et al. 2004; Hu et al. 2004; He et al. 2007).

Although the HF was found to be beneficial to the hydrolysis process, in comparison with the CSTR control reactors, the performance of these reactors was still relatively poor. The VFA production of 0.228-0.257 g COD g^{-1} VS added equates to a methane production (COD equivalent) of 0.08-0.09 l g^{-1} VS added, which means the reactors were achieving around 25% of the maximum possible solubilisation. It is likely that these reactors were also producing some methane, unfortunately this was not measured. Given that the solids destruction and specific COD production were similar, it can be assumed that methane production in the buffered reactors was similar to the value of 0.049 litres g^{-1} VS added in the unbuffered reactors. This would bring the total methane production from the unbuffered reactors up to 0.13-0.14 l g^{-1} VS added, or only 40-44% of the maximum possible as measured by the BMP.

The effect of buffering on HF reactors was investigated to try and determine whether pH alone was causing the poor hydrolysis performance, and pH control has been used in other work to improve hydrolysis performance (Zhang et al. 2005). pH was controlled by the addition of buffer solution as described in Table 13. As mentioned in the results section, this part of the trial took place at a later date to the main part of the single stage HF trial in a response to the poor performance of the unbuffered HF reactors and also since previous

research had found neutral conditions to be beneficial to hydrolytic bacteria and the overall degradation process (Babel et al. 2004; Hu et al. 2004; Zhang et al. 2005). However this was not the case in this trial, as can be seen in Figure 32 and Figure 33: the buffered reactors showed a slight decrease in hydrolysis performance compared with the equivalent unbuffered HF reactors. This occurred even though pH was at near neutral conditions for most of the trial as shown in Figure 30. This supports the idea, suggested in section 6.2.2, that pH has no significant effect on the hydrolysis rate over the range in this trial. The pH difference between the buffered/unbuffered reactors means that although VFA concentrations were similar, the UVFA concentration in the unbuffered reactors was higher. No additional inhibitory effect of the increased UVFA concentration was detected in this experiment.

The reason for this poor performance of the HF reactors was most likely biomass washout by the HF, since the centrifuging operation did not completely remove the solids from the supernatant. Although hydrolytic organisms are generally considered to act by attaching to the surface of substrates (Song et al. 2005), and thus to be associated with the solid phase, there is some evidence to suggest that at lower HRT many hydrolytic organisms can be washed out of anaerobic digesters in the liquid phase (Cysneiros et al. 2007). It is possible that a proportion of the biomass was detached by the shear forces caused by centrifuging and was therefore removed with the supernatant. Colonisation of the whole surface area is important to gain maximal hydrolysis rates in cellulolytic materials (O'Sullivan et al. 2005).

6.4 Two-stage Hydraulic Flush Trial

The purpose of this trial was to ascertain the two-stage performance of an anaerobic system fed on SBMW using 1st stage HF reactors and 2nd stage AF reactors. Four two-stage systems were operated, each consisting of two HF reactors running in duplicate, with effluent fed to an AF reactor. Comparison with the single stage HF trial allows discussion regarding the effect of effluent recirculation. The four systems were at OLRs of 3.75 and 7.5 g VS l^{-1} d^{-1} with and without pH control by buffer addition. The experimental procedure is described on page 75 and the results begin on page 101.

The methane production from the two stage HF reactor systems was comparable to the single-stage reactors but was achieved at a higher OLR. As in the single-stage HF trial, pH control had no significant impact on the overall performance of the reactor systems. Systems with and without pH control were able to recover from a large system disturbance in the form of a temperature shock, displaying great process resilience. Firstly the stable operational period of the reactors will be discussed, then the disturbance period in section 6.4.5.

Although the reactors generally performed well, the mode of operation was unexpected. In all systems, methanogenic conditions were established in the 1st stage reactor as well as the 2nd, and 70-90% of the total methane production came from the 1st stage as can be seen in Figure 37. Gas production, shown in Figure 35 and Figure 36, was 0.23-0.24 l g^{-1} VS added, or around 75% of the BMP of the feedstock.

As can be seen in Figure 40, the specific COD production from the 1st stage reactors during the stable operation period was 0.088-0.159 g COD g^{-1} VS added and was lower than in the single-stage HF trial (c.f. 0.255-0.257). This is because a large quantity of the soluble COD production in the 1st stage reactors was being converted into methane rather than being removed from the reactors in the effluent. If the COD of the methane was taken into account (2.86 g COD l^{-1}), the 1st stage reactors were producing 0.665-0.740 g COD g^{-1} VS added and so performing much better than their single-stage counterparts.

Any accumulation of VFAs in the AF reactors would be apparent by an increase in total alkalinity, an increase in alkalinity ratio and a decrease in pH. The 2nd stage alkalinity and the alkalinity ratio demonstrate that the AF reactors were running stably for the period of the trial, as can be seen in Figure 41 and from the pH of these reactors shown in Figure 34.

Comparison of the data from the two different loading rates of 3.75 and 7.5 g VS l^{-1} d^{-1} reveals little difference with regard to overall performance, and both reactors were methanogenic in each system at both loading rates. As can be seen in Table 14 the SMP of the systems are similar. Double loading produced double biogas and methane, suggesting that the process was comfortably dealing with the higher loading and perhaps even had the capacity to deal with a greater OLR. The problem in this case would be stirring, since higher loading would mean high in-reactor solids. Stirring is discussed later in this section.

Gas production from the two-stage systems, as can be seen in Figure 35, was erratic throughout the test. It is thought that this was due to a number of factors. The first is that the reactors were not fed and sampled at exactly the same time each day, which could lead to variations in daily gas production. The second is that the reactors were subject to a substantial disturbance each day in the form of removal from the water bath, cooling, centrifuging and reheating, and although every attempt was made to make this process as repetitive as possible, this could not practically be guaranteed. In any case, Figure 36 shows that over a 4-day period the gas production is steadier, giving a clearer picture of the system performance. It is likely that with the use of a continuous filtration system, the erratic gas production would cease, since the reactors would be in a continuous steady state although any variability of the feedstock would still be present.

6.4.1 pH Control – The Effect of Buffering

The effect of adding buffer solution in an attempt to alter the pH was minor. The methane production and solid destruction rates were similar in unbuffered and buffered systems. Although these two systems had similar performance, the

effect of pH control was to change the mode of operation of the two-stage system.

At the OLR of 3.75 g VS l^{-1} d^{-1} the differences between the buffered and unbuffered system can be attributed to the fact that the buffer solution increased the pH in the 1st stage reactor and therefore improved environmental conditions for the growth and activity of methanogens (Jones et al. 1987). The buffered systems had greater methane production in the 1st stage, due to increased methanogenic activity, and lower soluble COD production, since more of this was being converted to methane in the reactor. It should also be noted that the working pH of the unbuffered 1st stage reactor (6.53) was close to the lower efficient working limit of common methanogenic bacteria (≈6.5) (Gerardi 2003c). Lower pH in the 1st stage may inhibit the methanogenic bacteria, and thus could result in a decreased overall system performance.

At the increased OLR of 7.5 g VS l^{-1} d^{-1}, without buffer addition, the pH of the HF reactor was higher than at the lower loading, and the proportion of methane higher at 86% of total. This can perhaps be attributed to increased acclimatisation, since the inoculum for the unbuffered, high OLR trial was a mixture of digestate from the lower loaded trial with fresh sewage sludge. This result could also have been caused by the higher alkalinity in the system due to greater feed addition and therefore increased production of ammonium into the digestate (Garcia-Heras 2003).

The effect of buffer addition can be seen in the high TA shown in Figure 41. Additionally both buffered systems had a large amount of COD, not in the form of VFAs, which was not being degraded in either reactor.

6.4.2 Recalcitrant COD Build-up

The COD results in Figure 38 and Figure 39 show that for a large part of the trial and throughout the stable operational period, a high proportion of the SCOD in the two buffered systems was not being broken down in the methanogenic reactor. This effect, although present in the unbuffered system, was less pronounced. Furthermore, in Table 24 it can be seen that the

breakdown in the methanogenic reactor, which is the difference between the SCOD of the 1st and 2nd stage effluents respectively, was slightly higher than the proportion of the COD present in the form of VFA. Along with the low VFA concentration in the methanogenic effluent this means that the 1st stage reactors, especially the buffered systems, produced some recalcitrant SCOD that was not readily degradable by the microorganisms in either reactor. It is speculated that the different physical conditions in the buffered and unbuffered systems led to a difference in microbiological ecology, which in turn led to the production of a hydrolysis or fermentation intermediate in the buffered system that was somewhat recalcitrant. It has been suggested that the breakdown of lignin can produce some intermediate products which are recalcitrant (Gerardi 2003e). This did not detract from the system performance, however, and toward day 105 it can be seen that this SCOD was decreasing in the buffered two-stage systems. Since the addition of buffer solution to increase the pH of the first stage reactor had no measurable benefit to the system the further exploration of this phenomenon was no performed.

6.4.3 Mixing Problems

As mentioned in the results section, early in the trial there were a number of problems with digestate mixing. The sludge matrix was so thick and gas production so high, especially at higher OLR, that bubbles of gas tended not to rise to the surface. Instead the digestate expanded, causing an increase in apparent working volume of the reactor, until overflow occurred. The problem of mixing at high solids levels needs to be addressed before any larger-scale implementation of this system could occur.

6.4.4 Comparison of One and Two-stage HF – The Effect of Recirculation

The main effect of the recirculation of process liquid on the HF reactors was a step increase in performance, as measured by methane production and solids breakdown. This change was also accompanied by the establishment of methanogenic conditions in the 1st stage reactors. The establishment of a methanogenic population is akin to the well-known effect found in SBRs, where this occurs after an initially acidic period as biomass is transferred from an old reactor (Lai et al. 2001). It is difficult to establish whether the recirculation itself

or the methanogenic condition enabled by the recirculation was responsible for the increase in performance. The latter could be the case, since accumulation of fermentation products such as hydrogen can inhibit hydrolytic/acidogenic organisms (Chynoweth et al. 1993), and the presence of an active methanogenic population can relieve this.

It is proposed that there are three mechanisms by which the addition of effluent recirculation could increase the hydrolysis performance of the HF reactors and create methanogenic conditions within them. These are: low alkalinity washout, low biomass washout, and micro-inoculation from the AF reactors. Which of these is responsible for the change in performance and/or establishment of methanogenic conditions in the 1st stage is uncertain, but the physical reasoning behind each explanation will be discussed.

Low alkalinity/biomass washout

Little liquid was removed from the two-stage systems with effluent recirculation. The only sources of removal were in digestate sampling, a small amount removed with the mixed digestate to give the 20-day SRT in the 1st stage reactor, spillages and evaporation. This meant that both biomass and alkalinity (provided by the feedstock, or otherwise added as buffer solution) were both washed out of the system slowly. High alkalinity is advantageous as it maintains high pH, thus creating optimum conditions for hydrolysis, and also allowing methanogenic organisms to populate the 1st stage reactors (Chynoweth et al. 1992).

Low biomass washout would also explain a change in system performance since biomass concentration, especially of hydrolytic biomass, will be strongly related to the degradation rate until excess biomass exists (Song et al. 2005). Although hydrolytic biomass is usually considered to act by attachment to solid surfaces (O'Sullivan et al. 2005) there is some evidence to indicate that the liquid phase is also important (Cysneiros et al. 2007) (see section 6.3).

From another point of view, low biomass washout means that even if the methanogenic population was not under ideal conditions, it could continue to work slowly, without being washed out of the system by the HF, since it was

returned with the recycled effluent from the 2nd stage. In general methanogenic organisms will gradually establish a neutral environment by utilisation of acetic acid and thus maintaining them in the system would certainly increase the chances of establishing predominantly methanogenic conditions.

Micro-inoculation

The effluent coming from the methanogenic AF reactors will undoubtedly contain a small amount of detached methanogenic biomass. The suspended solids content of this liquid was measured a number of times during the test and was found to be less than 0.4 g l^{-1} at all times. This continuous trickle of methanogenic inoculum could well be the reason that methanogenic conditions are established in the 1st stage and could be responsible for the performance step change. This effect has been seen in flushing bioreactor landfill research (Bae et al. 1998).

6.4.5 Sensitivity of Two-stage HF System

As mentioned in the results section, on day 106 of the trial for the buffered systems and on day 26 for the high OLR unbuffered system, the water bath controlling the temperature of the HF reactors was accidentally changed to 50°C for two days.

All of the effects of this disturbance can be explained by a decline in activity of the methanogenic populations in the 1st stage reactors which led to a drop in pH as a high VFA concentration accumulated (Gerardi 2003d). SCOD production, shown in Figure 40, increased during these periods due to reduced methane conversion in the 1st stage. The specific SCOD production at both loading rates increased to around 0.36-0.50 g COD l^{-1} d^{-1}, the highest value obtained in any of the trials in this work, demonstrating the effective hydrolysis process in these reactors. In all three cases the 1st stage reactors gradually recovered to their previous state after a period of 10-20 days. It is also interesting to note that during the period of reduced methanogenic activity in the 1st stage, the overall methane production of the system only decreased slightly, as the AF reactors took over as the major methane producers. The re-establishment of the methanogenic community in the 1st stage reactors could again be due to low

biomass washout, biomass regrowth, the effect of micro-inoculation from the AF reactors, or any combination of these.

Compared with the single-stage digesters, discussed in section 6.2.1, the HF/AF reactors were more robust. CSTR at only one quarter of the loading, a small temperature disturbance was enough to mean process failure. This provided confirmation of the disadvantages of CSTR and potential advantages of the two-stage MBR system as suggested in the introduction. The advantage of the two-stage HF/AF reactor system perhaps lies not in an increased or optimized hydrolysis rate, but instead in its stability, robustness and its ability to degrade effectively at high OLR.

6.4.6 Isolation of the 1st Stage Reactors

As the final part of the trial the high OLR unbuffered reactors were isolated from the AF reactor. This was attempted since these 1st stage reactors were producing a high proportion of the total system methane, and as such it was important to determine whether they could operate as a single-stage digester after the establishment of an enriched stable methanogenic population. The results for pH (Figure 34) and total methane production (Figure 35) show that the reactor quickly became acidified and methane production decreased significantly. This indicates that although the AF reactor in this system provides little in term of gas production, it was required to maintain system performance, probably due to a combination of the effects that allowed the methanogenic conditions to prevail in the 1st stage originally.

6.5 Mesh Filter Bioreactors

In this trial, filtration, rather than centrifuging, was used as the means to decouple the HRT and SRT 1st stage reactors. Four meshes pore sizes of 10, 30, 100 and 140 µm were used in the 1.5 l MFBRs. Filtrate from each MFBR was fed to an AF reactor, and the AF effluent was recirculated to the 1st stage reactors. A detailed description of the trial can be found on page 77 and the results are presented from page 109.

Of the four mesh pore sizes tested in the MFBR trial, only the 10 µm was unable to filter the digestate continuously. MFBRs with mesh pore sizes of 30, 100 and 140 µm worked continuously for the 85-day period of the trial at the OLR of 3.75 g VS l^{-1} d^{-1}. The reactor stirring mechanism was unable to mix the digestate at the OLR of 7.5 g VS l^{-1} d^{-1} as the torque required was greater than could be supplied by the DC motor. Because the stirring mechanism was also responsible for the clearance of fouling from the mesh surface, the pores became blocked and the trial was terminated.

The overall performance of the three two-stage systems was similar in terms of SMP (0.21-0.22 l g^{-1} VS added). This was a slight reduction compared with the same parameter for the two stage HF/AF systems (0.23-0.24 l g^{-1} VS added). As was seen in the two-stage HF trial, the MFBRs showed recovery of methanogenic activity after an initial drop in pH. Despite the low pH of 6.21 in the 140 µm MFBR during the stable operating period, this reactor still produced 51% of the system methane. This result was unexpected since the pH was below the limit where efficient methanogenesis usually takes place. This again demonstrates that the presence and activity of the methanogenic population in the 1st stage reactor is enhanced beyond its capabilities if effluent were not being recirculated from the AF reactor, probably by the effect of micro-inoculation as discussed in section 6.4.4.

The VFA concentration in the effluent from the AF reactors was low, showing that efficient degradation of these compounds was taking place. A proportion of the SCOD was not degraded and was returned to the 1st stage but showed no sign of build-up during the trial.

6.5.1 Filtration Discussion

The TMP was not measured directly, but was low on the 100 and 140 μm meshes. When the mesh units were taken out of the reactors, the small head of water above them (~10 cm) was enough to allow the filtrate to drain back through the mesh. A slightly higher TMP was apparent on the 30 μm mesh, as when the connecting tubes were opened the liquid was sucked back into the unit. This higher TMP is disadvantageous as it would increase the required complexity and energy use of any implementation of this type of system. Additionally the lower TMP of the higher mesh sizes suggests that they have a greater critical flux, meaning smaller mesh units could be used in relation to the reactor size.

No visible build-up of material was observed on the surface of any of the meshes, although inside the mesh unit there was a significant amount of aggregation and, upon emptying the reactors, some larger suspended particles (~1-2 mm diameter) were found. The effluent from these reactors often contained visible particles much bigger than the pore size. Additionally, on viewing through a microscope, long fibres were commonly seen in the effluent, which must have passed through the mesh lengthways, possibly being pushed through by the brushing action. These observations are important since in any larger-scale or longer time-scale implementation of this type of system, the problems of blockages in tubing / pipework / pumps etc caused by aggregation would need to be addressed.

6.5.2 The Effect of Pore Size

The trends of pH, SCOD and methane production in the 1st stage are all indicative of a varying degree of methanogenesis in the MFBRs. These trends are shown diagrammatically in Figure 51 and Figure 52, where data from the two-stage HF trial is also given to allow comparison of centrifugation with filtration as the solid/liquid separation method. The 30 μm MFBR showed the highest methane production, with 72% of the total produced in the 1st stage. With increasing pore size the MFBRs became less methanogenic, and instead produced a slightly higher strength effluent which was then degraded in the AF.

It was found that the degree of methane formation followed a trend inversely related to the TS and VS content of the effluent from the MFBR and HF reactor. A physical explanation of this is that the methanogenic behaviour of the 1st stage reactor was related to the biomass/fine solids retention characteristics of the solid/liquid separation process. Hydrolysing/acidogenic organisms were seemingly unaffected by the increased washout seen at higher pore sizes, which is expected as these organisms have generally higher growth rates than methanogens/acetogens (Zinder 1993; Vavilin et al. 2008) and their numbers in the reactors would be expected to remain high.

The HF/AF system acted similarly to the 30 µm mesh MFBR/AF system in most respects although the HF reactor showed slightly greater methane production, suggesting that the centrifuging operation was retaining a larger proportion of the biomass than the finest mesh used in this trial. This can be confirmed by the VS content of the HF effluent which was significantly lower than in any of the MFBR reactors.

The trends in soluble loading applied to the 2nd stage in these systems lead to the conclusion that the use of lower mesh pore sizes is advantageous. This is because the reduction in dependency on the AF reactor to deal with soluble loading means that in practice the required volume of the 2nd stage would be smaller, decreasing capital cost and increasing the volumetric methane production. Very modest loadings (around 0.25 g COD l^{-1} d^{-1}) were applied to the AF reactors in this trial since they were simply used as a means to degrade the 1st stage effluent. Unfortunately more appropriately sized AF reactors were not available in the laboratory at the time of these experiments. Further testing may be required to assess the response of an appropriately sized AF reactor before implementation of this type of system can commence.

6.5.3 Accumulation of Solids in the AF Reactor

The methane production from the AF reactors in the MFBR systems, shown in Table 25, was greater than the degradation of the SCOD alone based on a theoretical value of 0.35 l g^{-1} COD degraded, indicating that a proportion of the methane produced was actually coming from the solid component of the 1st

stage effluent. This was not the case in the HF/AF system, where the VS coming from the 1st stage was much lower. In the MFBR systems this additional methane production was not enough to account for the difference between the VS content of the AF influent and effluent, however, and although some VS degradation was apparent, there was also presumably some accumulation of solid material in the AF reactors.

The MFBR systems all had much higher apparent solids destruction rates than the HF systems, and higher even than the maximum degradation found in the BMP test. This is unlikely to be a physically correct result, and more likely to be another indication of solids accumulation in the AF reactors. This effect was most pronounced in the 100 and 140 µm MFBRs where the 1st stage effluent VS was the highest. Although the solids accumulation in the AF reactors caused no problems in the 85-day period of the trial, this could eventually cause the filter medium to become blocked and require service. As this is very undesirable in a large-scale process, for this reason again the 30 µm mesh pore size is recommended.

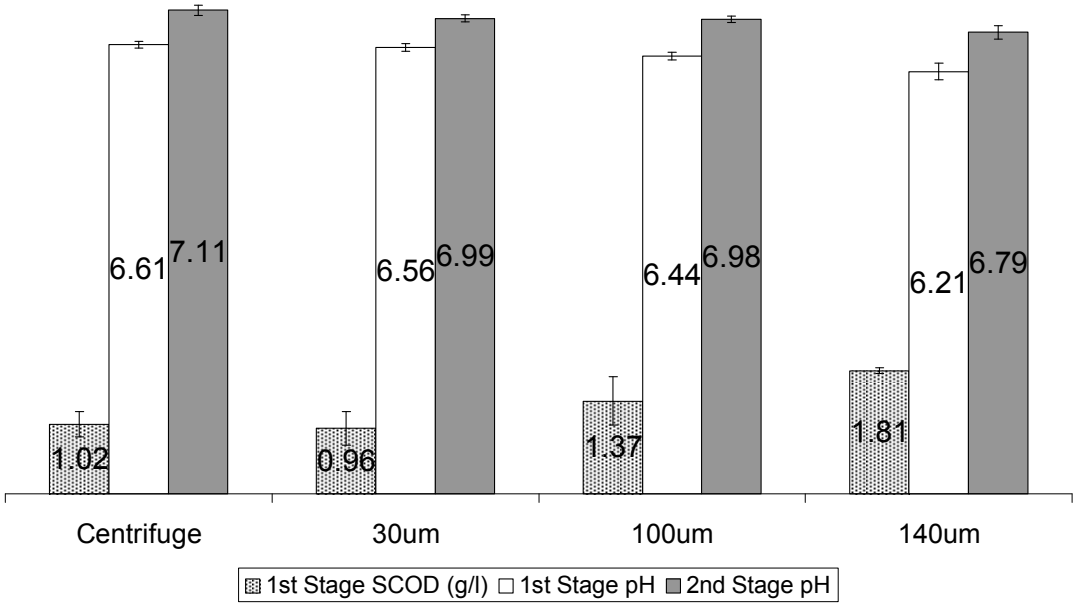


Figure 51 Trends of SCOD and pH in MFBR/AF systems

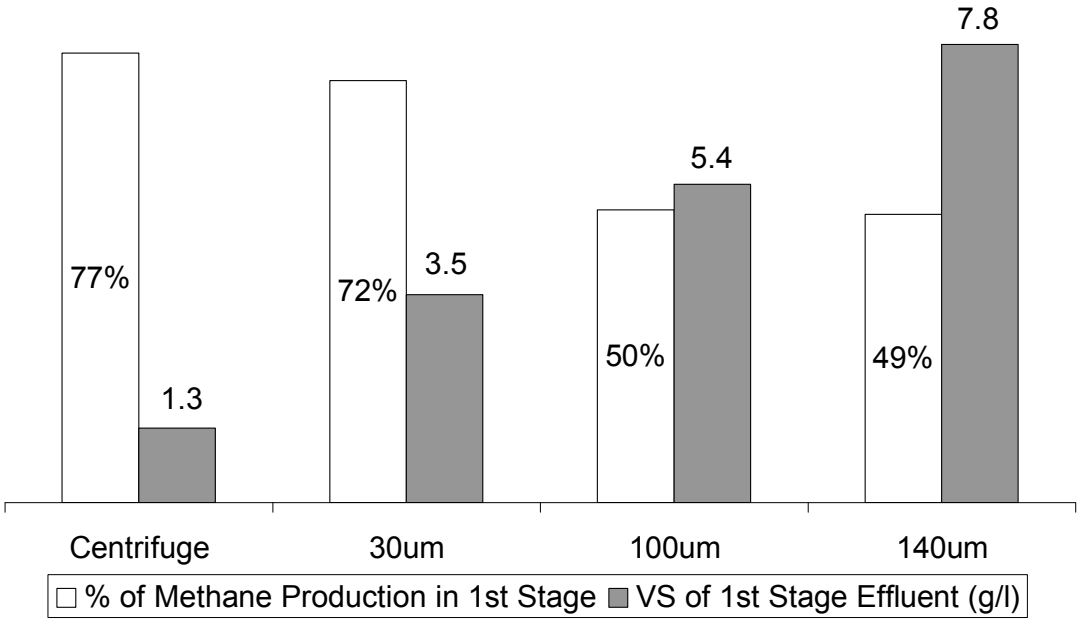


Figure 52 Trends of Methane Production and Effluent VS in MFBRs

6.6 Rotating Drum Bioreactors

In this trial ability of the two-stage RDMBR/AF system was tested. The RDMBR allowed continuous filtration of digestate while also being able to mix sludge of high solids concentration alleviating the problems of the MFBR design. A loading of 7.5 g VS l^{-1} d^{-1} was applied to these reactors and liquid effluent was fed to an AF reactor. After stable operation had been reached one RDMBR was isolated from its second stage reactor and the OLR was doubled to 15 g VS l^{-1} d^{-1} on the other one. The full description of this trial can be found on page 78 and the results are presented from page 147.

The two RDMBR/AF systems had SMP of 0.20 l g^{-1} VS added, lower than both the MFBR trial (0.21-0.22) and the two-stage HF trial (0.23-0.24). Despite this lower methane production, the system appeared stable at the OLR of 7.5 g VS l^{-1} d^{-1} and showed similar developments of pH and SCOD. The duplication of the two systems produced similar results except in the 1st stage gas production. The intermittent gas leak in RDMBR2 was finally fixed on day 56, after which the gas production of the systems became similar.

The pH in the RDMBR reactors (6.68-6.69) was higher than in the 100 μm MFBR (6.44), a trend which, although is not entirely expected, was also seen in the two-stage HF trial, where applying an increase in loading increased the pH and the methane production from the 1st stage reactor. This can be attributed to increased ammonia concentration since a greater quantity of substrate was being degraded.

VS destruction rates of 60-62% are e high relative to the maximum found by the BMP test (62%). Given that only 60% of the BMP of the feedstock was being realised in this trial, it seems likely that solids were being accumulated in the AF reactor, as was discussed in section 6.5.3 for the MFBR trial. If the VS removed in the AF reactor of 1.04 g d^{-1} is assumed not to be degraded but instead accumulated, the VS destruction rate of the system can be calculated as 52% which is more realistic.

6.6.1 Filtration Discussion

Although the results from the MFBR trial lead to a recommendation of the 30 µm mesh for further testing, initial trials with the RDMBRs showed that the increased TMP requirement in this pore size meant no filtration took place in this setup. Because of the design of these reactors, there was no way of forcing filtration across the mesh surface, since no pressure build-up was possible in either part (i.e. the drum or the outer casing). The only TMP applied to the meshes in these reactors was the digestate hydrostatic head (4-5 cm) as well as some dynamic pressure head caused by the tumbling of the digestate. The 100 µm mesh was found suitable for the RDMBRs because, as was observed in the MFBR trial, the TMP required for filtration was much smaller relative to the 30 µm mesh. Using this mesh continuous filtration of the digestate was possible for the duration of the trial at both loading rates of 7.5 and 15 g VS l^{-1} d^{-1}.

Because there was no direct way to control the flux rate in the RDMBRs, the filtration occurred at the natural rate under the rotating drum regime. The pumping speed was slightly higher than the flux rate, thus ensuring efficient recirculation of the process liquid. Additionally the RDMBRs had no mesh scouring mechanism, and simply relied on the weight and structure of the digestate to allow tumbling under the rotation of the drum. For these reasons the flux through the meshes on the RDMBRs settled at around 3.5 l $m^{-2}h^{-1}$ compared with 44 l m^{-2} h^{-1} for the MFBRs in the previous trial.

In an engineering implementation of the RDMBR system both pressure control and some kind of backwashing regime would allow filtration at higher rates than achieved in this trial. For example Dalhoff et al (2003) achieved a six fold increase in trans-membrane flux from 20 to 120 l m^{-2} h^{-1} by simply increasing the frequency of backwashing. The ability to achieve higher fluxes in scale up systems would be important since the surface area of the drum only increases with an exponent of 2/3 relative to the volume so the useful mesh surface becomes relatively smaller compared with the required flux to give the appropriate HRT.

As an example, a 500 m^3 rotating drum digester would have a surface area of 293 m^2 (assuming 2:1 aspect ratio of the cylinder). Assuming 50% of this area

is useful for filtration, the flux required to give a 1.5 day HRT would be 94 l m^{-2} h^{-1} which is greater than was achieved in the MFBR trial (c.f. 44 l m^{-2} h^{-1}). There is no evidence to suggest this is not possible, since a fully instrumented filtration system with backwashing and TMP control may be able to sustain greater fluxes than in the MFBR trial. It may be that the flux limitation defines the maximum scale at which this type of reactor could operate. As another example, the flux of 44 l m^{-2} h^{-1} would mean a maximum reactor volume (again assuming 2:1 aspect ratio and 50% filtration area) of 50 m^3 (49.94). Although this seemingly limits the scale-up of this type of system, the results found in the single-stage HF trial suggest that the HRT was not important within the range investigated (1.6-5.3 days HRT). Further testing would be required to determine whether a similar result would be the case for the two-stage system. In any case a maximum scale of 50 m^3 does not necessarily limit the application of this process since a required number of reactors could be operated in parallel while sharing ancillary systems such as process management, heating, pumping, maintenance etc.

6.6.2 COD, Solids and Methane Balance

The results from the COD balance around the AF reactors in Table 27 show a good agreement between surplus methane production in the AF reactors and apparent VS removal from these reactors. Throughout the full experimental period (to day 72), despite the overall methane production from AF1 being higher than AF2, it is shown that if ideal breakdown of SCOD to methane took place and the surplus was attributed to VS destruction, similar values of methane production specific to the VS removal occurred in both reactors. This does not mean that the VS was necessarily fully degraded in the AF reactors, but simply that some proportion of it was and that this proportion was similar in both systems. This similarity is to be expected since the reactors were fed and operated in the same way up until day 72 of the trial.

The solids balance applied to the whole system, as shown in Table 28 shows that a better balance was obtained for S1 than S2, where 1% and 4% respectively of input TS was unaccounted for in the solids balance. The direction of this solids deficit was such that apparently more solids were added

to the system than were removed. This is to be expected in small-scale laboratory trials since losses of small amounts of digestate occur when materials are manipulated outside the reactors, such as when transferring samples and mixing digestate.

The residual methane balance results show that 25-35% more methane could be produced under the conditions in the RDMBRs in this trial. According to this test, the results of which are shown in Table 29, the maximum methane production under these conditions is 0.25-0.27 l g^{-1} VS added, which is lower than the BMP of the feed (0.32).

6.6.3 Increased OLR and Isolation of the 1st Stage

The increase of the OLR to S2 and the isolation of RDMBR1 had similar results: a reduction in SMP from 0.20 to 0.15 and 0.16 respectively with no apparent process instability. Unfortunately due to time constraints this part of the trial had to be terminated earlier than would have been ideal. The two-stage SMP of S2 at the OLR of 15 g VS l^{-1} d^{-1} was showing signs of recovery the end of the trial, and it is unknown whether the SMP would have returned to its former value over time, in a similar way to the recovery of the two-stage HF systems from the thermal shock after a slight dip in overall SMP.

The fact that the 1st stage could be isolated in this type of system, where the equivalent centrifuge reactor could not, suggests that the centrifuge reactors relied more on the AF stage for inoculation and/or removal of SCOD. This indicates that the RDMBR reactors had a more stable methanogenic population despite the increased washout of VS (1.3 g l^{-1} in the HF, 4.51-4.62 g l^{-1} in the RDMBR). It is possible that the methanogenic population was affected negatively by the disturbance caused by the centrifuging operation and therefore growth rates were lower than in the RDMBR, where conditions were more stable under the continuous filtration regime. The ability of the RDMBR to operate in isolation from the 2nd stage suggests that the HRT of the 1st stage could be decreased as hypothesised in section 6.6.1 if required by flux limitations.

6.6.4 Potential Application of the RDMBR system

The results from this trial show that although the two-stage RDMBR/AF system has good stability at relatively high OLRs, the methane production (63% BMP) is lower than was produced in the single-stage trial (88% BMP). This of course could limit the potential for application of the two-stage system given that methane will be one of the major income generators for an AD facility. However, since waste disposal can also generate a large income this may make a highly loaded, compact process more desirable.

One possible application of the RDMBR system would be to introduce a 3rd stage reactor, where the solid residue (after dewatering) could be further digested under high solids conditions. The residual digestate would be a good candidate for a high loading high solids digestion since all of the readily degradable components have already been removed in the RDMBR and degraded where possible and therefore little build up of VFA would take place. Also since this 3rd reactor would only deal in high solids digestate, it would be reasonably compact and therefore not require much additional space; this type of system has been suggested for maize (Heaven et al. 2008b). This three-stage system is purely hypothetical and its application would depend on requirements for high waste throughput, low land use and the requirement for low residual biodegradability, to justify the increased level of technology required to run this complex reactor system relative to a single-stage digester. Further laboratory work would be required to confirm the feasibility of this type of system.

Another issue which may affect the use of this process on a larger scale is potential damage to the meshes. Careful protection of the mesh surfaces from coarse, heavy and sharp materials would be required. Removal of contaminants such as metal and glass would be of paramount importance since these could easily damage the nylon meshes used in this work; but also protection from the more tough biodegradable materials which form part of BMW, such as plant stems and branches, would need to be addressed. In the RDMBRs used in this work, the mesh was surrounded on both sides by a stainless steel mesh, but this was not entirely necessary since the synthetic waste used had been pulped and so no sharp materials remained. Sufficient particle size reduction would

prevent most damage to mesh surfaces, but at the expense of increased process energy and cost.

6.7 The Submerged Anaerobic Membrane Bioreactor

As part of the same Defra-funded TRIF project, the development of a 2nd stage submerged anaerobic membrane bioreactor (SAMBR) has taken place at Imperial College, London. This reactor uses a 0.4 μm membrane for biomass retention and has been able to operate under OLR up to 26 g l^{-1} at HRT as low as 2 days. The original plan was to have the 1st stage mesh bioreactor developed at Southampton University to be coupled with the 2nd stage membrane bioreactor. The development of methanogenic conditions in the RDMBRs, as presented in this thesis, meant that the organic loading to the 2nd stage reactor was always low, raising the question of whether it is necessary to have a complex 2nd stage to deal with the 1st stage effluent. Furthermore the membrane selection in the SAMBR was chosen for its ability to retain methanogenic biomass in the reactor, which may limit the establishment of methanogenic conditions in the 1st stage reactor and thus decrease the solids degradation characteristics of the process. This was the case in published work by Trzcinski and Stuckey (2008): at OLR of 4, 8 and 16 g VS l^{-1} d^{-1} the VS destruction was only 38, 25 and 9% respectively, and very little methane was produced in the 1st stage reactor.

7 Conclusions and Further Work

7.1 Conclusions

The following conclusions can be drawn from the work carried out:

- SBMW was created and characterized. The BMP of this material was found to be 0.32 l g^{-1} VS added, which compared well with literature values for real BMW samples.

- A single-stage digestion trial showed that a CSTR fed semi-continuously on the SBMW resulted in an unstable process even at low OLRs of 2-2.5 g VS l^{-1} d^{-1}. The reactors were operated at a retention time of 30 days, with 100 days of stable operation before the reactors eventually failed. Failure was cause by small disturbances in the temperature of the reactors, demonstrating the fragility of the process.

- CSTR reactors, operated at an OLR of 7.5 g VS l^{-1} d^{-1} and retention times between 2 and 10 days, showed almost zero methane production and hydrolytic/acidogenic conditions prevailed. These reactors, despite having varying pH and VFA concentrations, showed little variation in hydrolysis rate when a 1st order analysis was applied. It was clear that the only factor having a large influence on the solubilisation of solid material was the SRT.

- The single-stage HF experiment revealed that reduction of the HRT relative to the SRT induced a greater degree of hydrolysis. Even so, the single-stage HF reactors performed poorly in terms of solids breakdown and specific COD production, even with pH corrected to neutral conditions. This poor performance was thought to be due to biomass washout in the flushed liquid.

- The introduction of methanogenic effluent recirculation in the two-stage HF (HF/AF) system caused a step increase in performance relative to the single-stage equivalent. The system could be loaded up to 7.5 g VS l^{-1} d^{-1}

and still achieve around 50% TS destruction and SMP of 0.24 l g^{-1} VS added.

- Methanogenic conditions were established in the 1st stage reactor of the two-stage HF/AF process, which subsequently generated up to 86% of the total system methane production. Consequently the 2nd stage AF reactors were subject to very low loading.

- Controlling the pH to neutral in the single and two-stage HF systems had little impact on the reactor performance. It is thought that the extra alkalinity may be more important at higher loadings, where greater VFA concentrations and lower pH will induce greater stress on the methanogenic population.

- The unbuffered and buffered two-stage HF systems were both robust enough to deal with a large temperature shock in the 1st stage. Methanogenic conditions were temporarily eliminated from these reactors, and acidic conditions prevailed. During this period the 2nd stage reactor took over as the larger methane producer until eventually methanogenic conditions were re-established in the 1st stage and the mode of operation of the two-stage system returned to the conditions before the shock.

- A laboratory scale MFBR was designed and implemented in a two-stage system (MFBR/AF). The meshes used were made of woven nylon, and continuous filtration of digestate for 85 days (HRT 1.5 days, SRT 20 days) was possible at pore sizes of 30, 100 and 140 μm. No damage to the meshes occurred during this period. Filtration was not possible at a pore size of 10 μm.

- The MFBR/AF systems performed almost as well as the equivalent HF/AF systems, producing 0.21-0.22 l g^{-1} VS added at a loading rate of 3.75 g VS l^{-1} d^{-1}.

- The main impact of the different mesh sizes was to alter the level of methane production in the 1st stage reactor with 72, 50, and 49% of the total system methane generated in the MFBR (1st stage) at 30, 100 and 140 μm respectively. The level of methanogenesis in the MFBR was thought to be related to the level of biomass/fine solids retention provided by the mesh, with smaller pore sizes providing greater retention. This was indicated by an increasing VS content in the effluent with increasing pore size.

- At an OLR of 7.5 g VS l^{-1} d^{-1} the stirring mechanism in the MFBR was unable to mix the thick digestate and since the scouring of the mesh was performed by the same mechanism, the pores quickly became blocked and the trial was terminated.

- To solve the problems of stirring thick digestate a novel type of reactor was designed, in which a rotating drum provided the mixing and mesh sections placed around the drum provided filtration. The laboratory scale reactor had a working volume of 1.5 litres and used a nylon mesh of pore size 100 μm.

- The RDMBR/AF system was able to stably digest SBMW at the OLR of 7.5 g VS l^{-1} d^{-1} and continuous filtration was achieved for 86 days. Methane production during this time was 0.20 l g^{-1} VS added. The rotating drum provided adequate stirring without the fouling problems of the paddle stirrers used previously. It was shown that the RDMBR could be isolated from the AF reactor at this loading rate without any signs of process instability. Solids, COD and methane balances were successfully applied to the two-stage systems.

- Just before the RDMBR/AF trial was terminated, the OLR was increased to 15 g VS l^{-1} d^{-1}. The system showed no signs of instability in response to this shock increase in loading rate although the specific production of methane decreased from 0.20 to 0.15 l g^{-1} VS added.

- Despite the process stability at high OLR of the RDMBR/AF system, scale-up and further application may be limited by the fact that specific methane production was lower than in the single-stage reactors. Also the flux limitation of 44 l m^{-2} h^{-1} through the 100 µm mesh means a maximum reactor working volume of around 50 m^3, although it is possible that this could be increased by optimisation. It is thought that factors such as requirements for a compact, problematic feed materials or a highly loaded process, if present, may warrant the use of this technology.

7.2 Further Work

The laboratory trials performed as part of this work were terminated due to time limitations, and it was therefore not possible to further investigate the maximum loading rate, kinetic of operation at multiple loadings rates and under other filtration regimes. Extension of this work could provide further understanding and insight into the two-stage system developed. Specific suggestions of how the work could be extended are given below.

Confirmation of Maximum OLR and Kinetic Investigation

The operating time at the maximum loading rate of 15 g VS l^{-1} d^{-1} applied to the RDMBRs was insufficient to confirm stable operation of the two-stage system. After two weeks the system seemed to be recovering from the shock increase in loading. It would be of interest to determine the operational characteristics at a steady state condition at this loading rate and above if possible. At a higher OLR the feed may be need to be dried in order that its volume is not larger than the amount of digestate removed. Also given more time (and more reactors) a kinetic study into the response of the RDMBR/AF system to various OLR and more importantly HRT would be of interest. In particular the response of the system to lower flush rates (longer 1st stage HRT) would be important since the HRT determines the limit to the scale of the process (see section 6.6.1).

The Effect of Inoculum

As noted in the literature review, the activity of rumen hydrolytic microorganisms is up to twice that of those in sewage (Song et al. 2005). The successful

application of rumen inocula was also shown in the RUDAD studies (Gijzen et al. 1987a), although ciliate numbers always were lower in reactors than in the rumen and decreased with time. The effect of rumen inoculum in the RDMBR/AF system would be interesting since the comparatively long SRT (compared with RUDAD) may allow ciliate numbers to remain high. Key points would be the ability to sustain the high hydrolysis rates provided by the rumen inoculum and comparison with the sewage inoculated equivalent. Another interesting study in this and other anaerobic digestion processes would be co-digestion with rumen contents from a slaughterhouse, which may provide enhanced hydrolysis without the need for concern about the sustainability of ciliate populations since these would be added with the feed.

Use of Appropriately sized AF reactors and Continuous 2nd Stage Feeding

The AF reactors used in this work were oversized for their application, since the 0.8-1.5 litre 1st stage reactors provided neither a large volume nor a high strength effluent. This meant the OLR and HRT applied to the AF reactors was low compared with other applications in the literature. Furthermore the 4-litre volume of these reactors more than tripled the total volume of the two-stage systems.

It is possible that the use of these AF reactors allowed the system to operate more stably for two reasons: firstly there was spare capacity in the AF reactors to deal with the higher strength 1st stage effluent during the start up phase or after a temperature shock. Secondly the increased system volume meant that the build-up of VFA or other intermediates occurred more slowly, allowing the biomass extra time to deal with the change. In a larger-scale implementation it would be financially unviable to over-engineer the 2nd stage reactors and so some testing of a two-stage system with appropriately sized reactors is required. The method of feeding the 2nd stage reactors could become more important with appropriately sized reactors, since the reactors will have less spare capacity to deal with changes in conditions such as those experienced under batch feeding. With continuous feeding the inlet conditions remain steady over time, which may allow AF reactors to operate under conditions that would cause failure with batch wise transfer of the 1st stage effluent.

Mesh Filtration Monitoring and Flux Optimisation

So far only a superficial understanding of the flux characteristics of the MSW digestate has been gained. Given that the flux effectively limits the maximum possible size of the system as described in section 6.6.1, greater understanding and optimisation of this part of the process is required. TMP is an important parameter in filtration systems which can be used to monitor the build-up of a fouling layer on the surface of the filtration media, and therefore reactors with pressure sensors on both sides of the mesh surface would give some insight into this. Maximum flux in the RDMBRs was lower than in the MFBRs, probably because there was no applied TMP to encourage filtration or fouling removal in the former. Increasing the flux in RDMBRs requires that a method of direct flux control and maintenance be introduced. Flux could be maintained by a number of methods including liquid and gas backwash, although at laboratory scale all of these are rather difficult to implement.

A simple filtration test rig could bypass the difficulty of laboratory scale in situ testing of flux maintenance/optimisation options. This would allow trials of a number of different methods and regimes under controlled and monitored conditions, which would ideally lead to the development of routines to detect excessive fouling. Actual BMW digestate from a RDMBR could be used to ensure representative filtration characteristics.

Digestion of Other (Problematic) Feed Materials

The process stability demonstrated by the RDMBR/AF system may allow the digestion of potentially problematic feed materials such as those that undergo rapid breakdown (e.g. FVW, FW) or are naturally low in buffering, which may otherwise produce an unstable AD process at high organic loading rate. This hypothesis would need to be tested on a case to case basis but the FW used in the recipe for SBMW in this work was shown to cause process failure in single-stage digestion (Climenhaga and Banks 2007) and thus would be a good example of a problematic feed material.

Three-Stage System

As was described in section 6.6.4, the potential of a high solids post-RDMBR stage to obtain the residual methane should be explored. Of prime importance

is that this system provides high throughput of waste and also that the volumetric production and specific methane are both high. Therefore emphasis should be placed on finding the minimum size for the 3rd stage (and AF) reactors. One possible method for this would be to de-water the removed digestate, keeping the liquid in the RDMBR/AF system to moisten the feed, while allowing the partially stabilised solids to degrade in a high solids environment. As discussed earlier, since the rapidly degradable fractions of the BMW will have been removed in the 1st stage, the digestate is unlikely to undergo acidification when digested in a high solids reactor.

8 Appendices

8.1 Rotating Drum Mesh Bioreactor Design

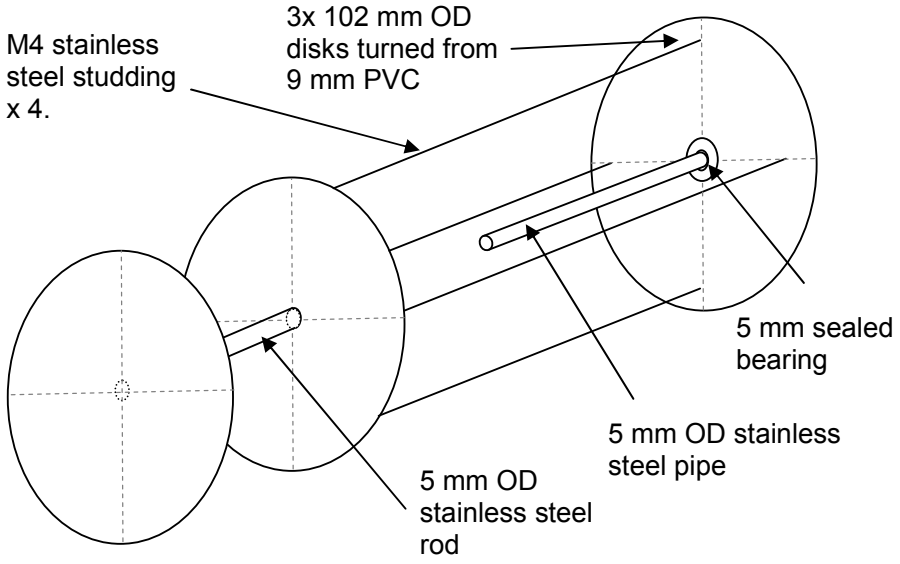
8.1.1 Assembly details

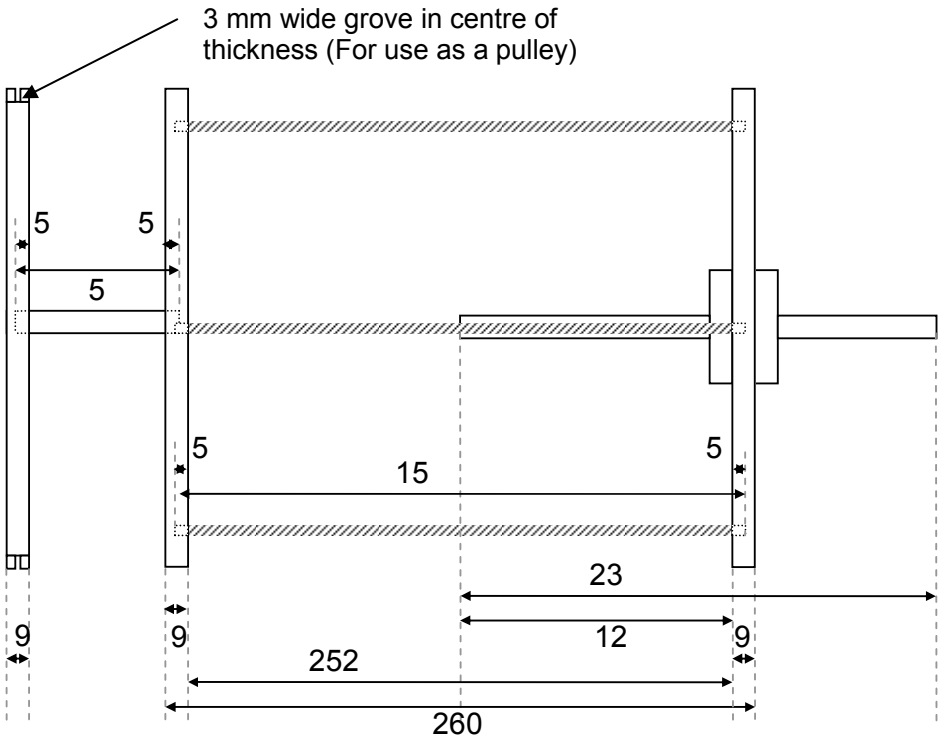
Complete Assembly	Sub Assembly	Part	Total Number needed
Rotating Drum Reactor			1
	Drum		1
		End Assembly	1
		Outer Shell	1
		Mesh Units	3
	Outer Casing		1
		Box	1
		Lid	1

8.1.2 Drum

Made up of **end assembly**, **outer shell** and 3 x **Mesh Unit**s. The mesh units attach to the outer shell and the shell fits over the end assembly and is secured using glue.

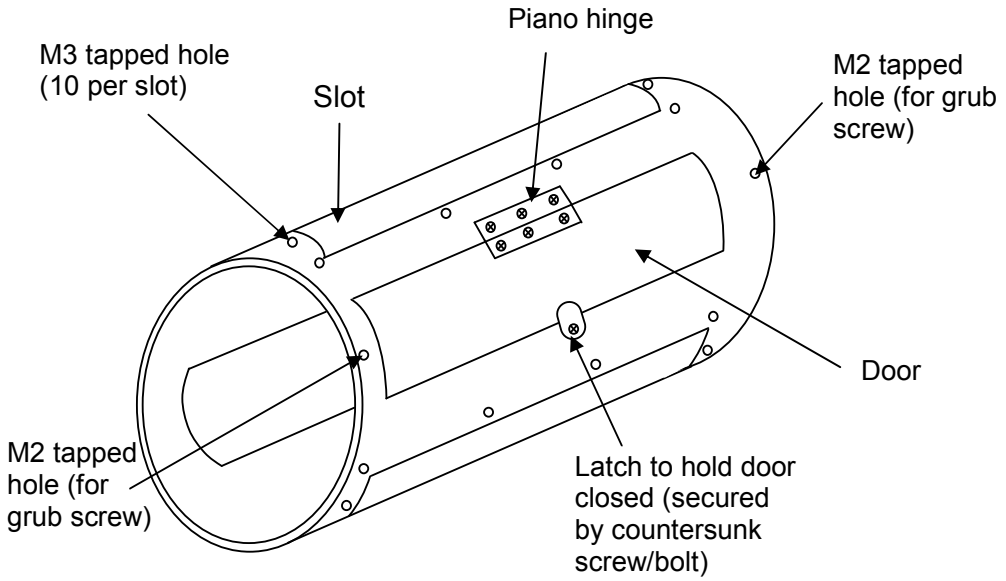
End Assembly

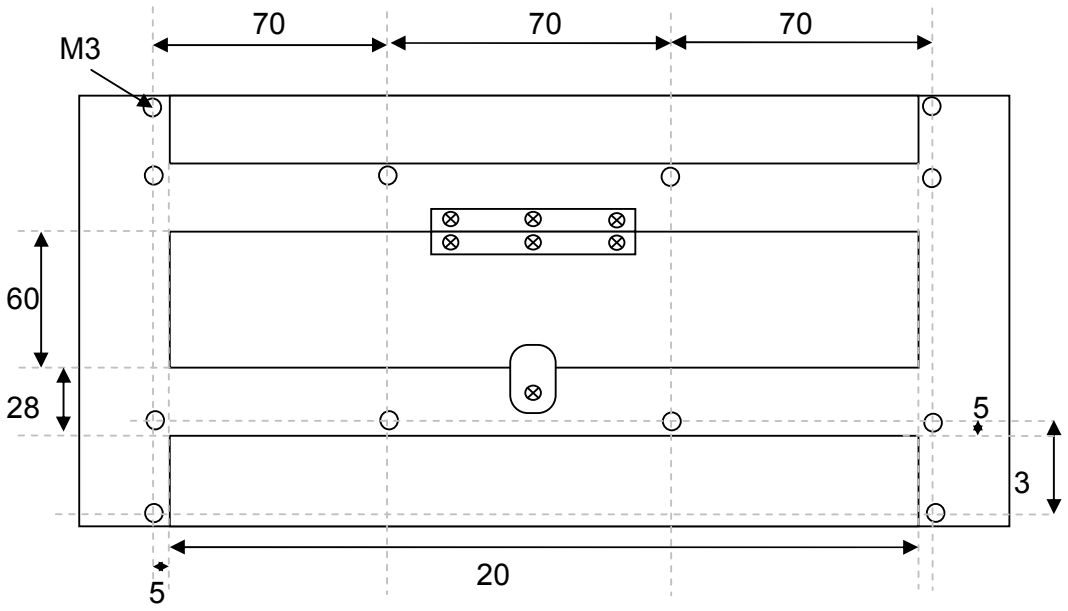




Outer Shell

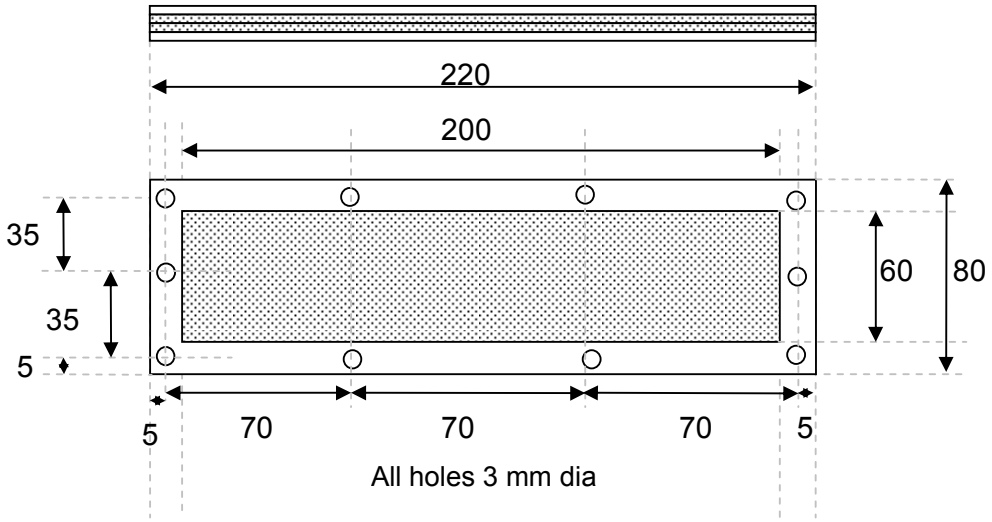
A piece of 110 mm OD drain pipe (4 mm wall thickness), with 4 slots cut out equally spaced around the circumference. One of the slots acts as a door, to allow access to the inside. The others are covered by mesh units, which in turn screw into the M3 tapped holes. Distances shown are measured around the arc surface.





Mesh unit

3 x needed per outer shell. These fit onto the slots cut into outer shell and are secured using M3 pan head bolts. The unit essentially is a sandwich of 2 sheets of 1 mm stainless steel mesh between 2 flexible stainless steel sheet ‘frames’.

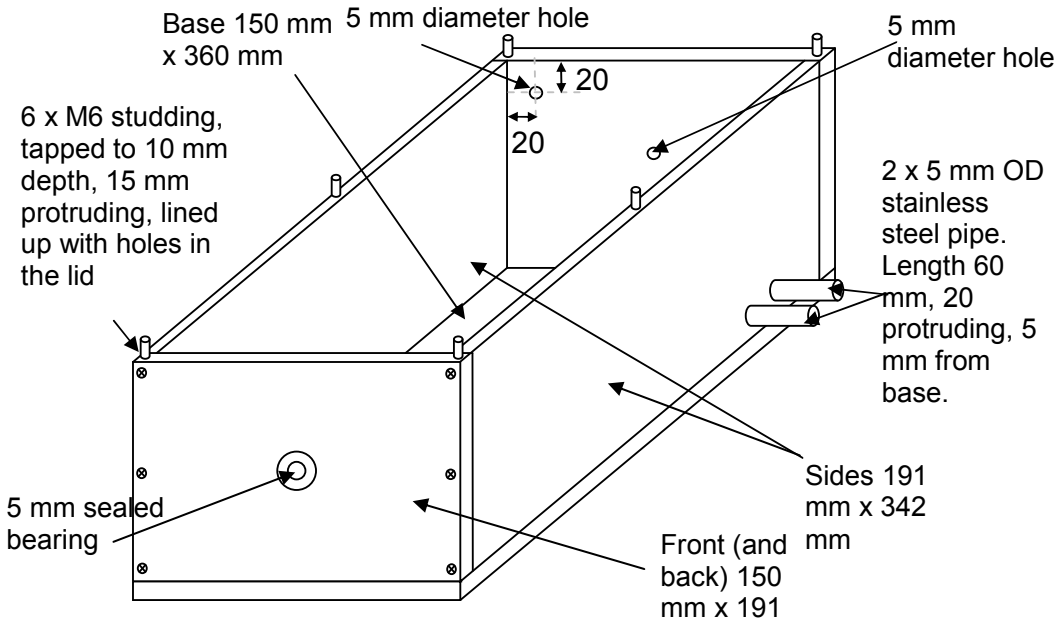


8.1.3 Outer Casing

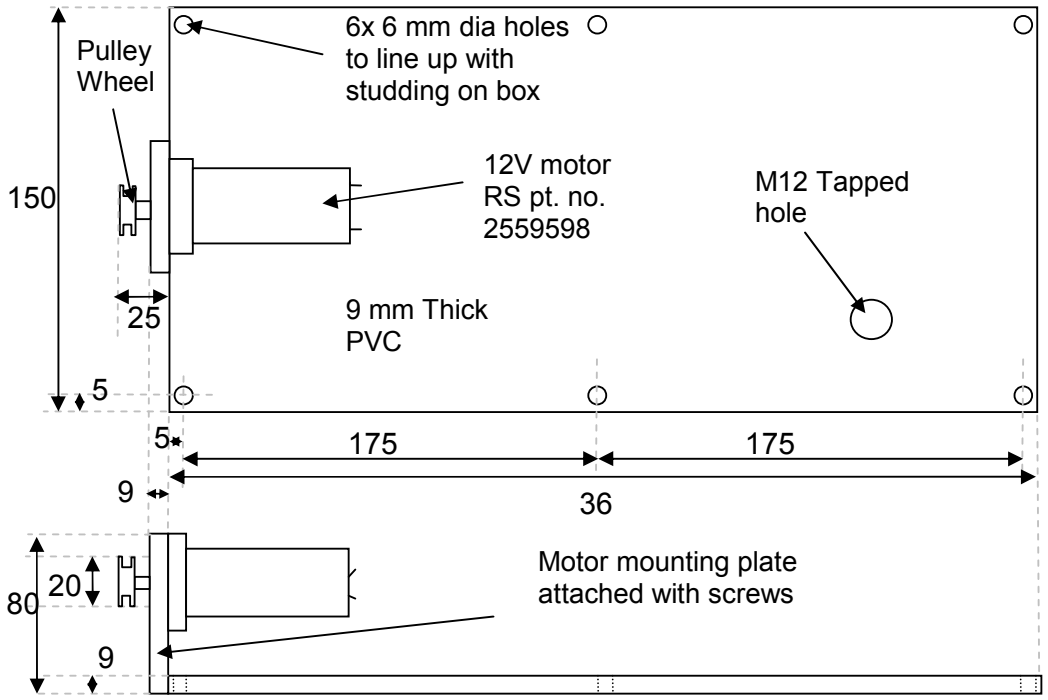
A **box** with a **lid** held on using wing nuts, sealed by a rubber gasket. The drum goes inside the casing and is driven using a pulley by a motor mounted to the lid. The box has two drain pipes (5 mm OD).

Box

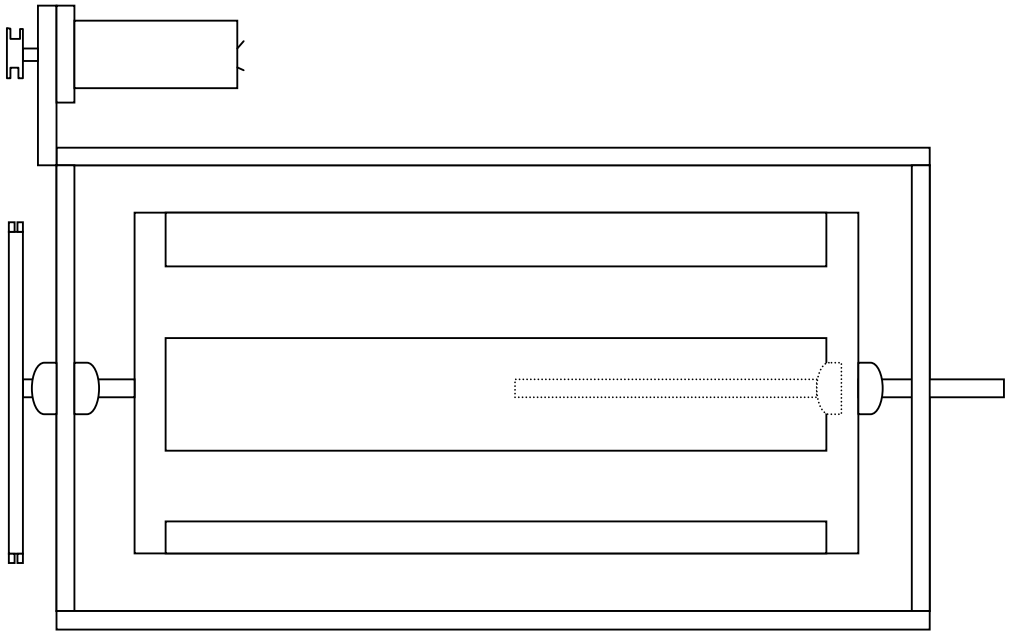
The box has outer dimensions of 150 mm x 200 mm x 360 mm and is constructed from 9 mm PVC and held together with countersunk screws and glue.



Lid



8.1.4 Full Assembly X-Section



8.2 Gasometer Governing Equations

8.2.1 Assumptions

$$\frac{p_1 V_1}{T_1} = \frac{p_2 V_2}{T_2} = \frac{p_{stp} V_{stp}}{T_{stp}}$$

- Biogas acts as a perfect gas.
- The biogas once leaving the anaerobic digester quickly cools to ambient temperature. $T_1 = T_2 = T_{atm}$
- Once cooled to ambient conditions, the biogas is saturated with vapour and thus the partial pressure of the water vapour is equal to the saturated vapour pressure (SVP) at that temperature. In the case of long term storage of the biogas in the gasometer column this will always be the case except for during fast transitions of temperature. When gas sampling bags are used the gas in the bag will always be already saturated, since it comes from an anaerobic digester of higher temperature where the liquid is in equilibrium with the biogas.
- That the SVP can be modelled by the Goff-Gratch equation as shown in equation 3 (Goff and Gratch 1946) with the constants for the boiling point of water and the vapour pressure at this temperature as 373.16K and 101324.6 Pa respectively.
- The cross-sectional area in the columns is constant.

8.2.2 Weight Gasometer

Volume of gas introduced into the column at standard conditions

$$V_{stp} = V_{2stp} - V_{1stp} \qquad \text{Eq.1}$$

Perfect Gas Law $\frac{p_a V_a}{T_a} = \frac{p_b V_b}{T_b}$ Eq.2

Pressure variation with depth $p_1 = p_{atm} - p_{h2o}(T_{atm}) - \rho_b g(H - h_1)$ Eq.3

As Eq.3 but for condition 2 Eq.4

Relationship between heights $h_2 = h_1 + \frac{V_w}{A}$ Eq.5

Volume of gas in column $V_1 = A h_1$ Eq.6

As Eq.6 but for condition 2 Eq.7

Volume of water displaced $$V_w = \frac{m_w}{\rho_b}$$ Eq.8

From Eq.2 $$V_{1stp} = \frac{p_1 V_1 T_{stp}}{T_{atm} p_{stp}}$$ and the same for condition 2.

Substituting into Eq.1 $$V_{stp} = V_{2stp} - V_{1stp} = \frac{p_2 V_2 T_{stp}}{T_{atm} p_{stp}} - \frac{p_1 V_1 T_{stp}}{T_{atm} p_{stp}}$$ Eq.9

Substituting for all unknowns in Eq.9 from Eq.3,4,6,7

$$V_{stp} = \left(\frac{\left(p_{atm} - p_{h2o}(T_{atm}) - \rho_b g\left(H - h_2\right)\right)\left(Ah_2\right)T_{stp}}{T_{atm} p_{stp}} \right) - \left(\frac{\left(p_{atm} - p_{h2o}(T_{atm}) - \rho_b g\left(H - h_1\right)\right)\left(Ah_1\right)T_{stp}}{T_{atm} p_{stp}} \right)$$

Eq.10

Due to the relationship in Eq.5 it is possible to remove the need to measure the second (or first) height, or alternatively the two ways of calculating can be used as a check. For example, if only the height at condition 1 is measured, an expression for the volume can be derived by substituting Eq.5 and Eq.8 into Eq.10.

$$V_{stp} = \left(\frac{\left(p_{atm} - p_{h2o}(T_{atm}) - \rho_b g\left(H - \left(h_1 + \frac{m_w}{A\rho_b} \right) \right) \right)\left(A\left(h_1 + \frac{m_w}{A\rho_b} \right) \right)T_{stp}}{T_{atm} p_{stp}} \right) - \left(\frac{\left(p_{atm} - p_{h2o}(T_{atm}) - \rho_b g\left(H - h_1\right)\right)\left(Ah_1\right)T_{stp}}{T_{atm} p_{stp}} \right)$$

Eq. 11

Can be rewritten as

$$V_{stp} = \frac{T_{stp} A}{T_{atm} p_{stp}} \left[\left(\left(p_{atm} - p_{h2o}(T_{atm}) - \rho_b g\left(H - h_1 - \frac{m_w}{A\rho_b} \right) \right)\left(h_1 + \frac{m_w}{A\rho_b} \right) \right) - \left(p_{atm} - p_{h2o}(T_{atm}) - \rho_b g\left(H - h_1\right)\right)h_1 \right]$$

Eq. 12

8.2.3 Trough Gasometer

Equations 1 and 2 apply to this setup.

Volume of gas in column $V_1 = Ah_{c,1}$ Eq.13

As Eq.12 but for condition 2 Eq.14

Pressure variation with depth $p_1 = p_{atm} - p_{h2o}(T_{atm}) - \rho_b g(h_{t,1} - h_{c,1})$

Eq.15

As Eq.14 but for condition 2 Eq.16

Derivation

From Eq.2 $V_{1stp} = \frac{p_1 V_1 T_{stp}}{T_{atm} p_{stp}}$ and the same for condition 2.

Substituting into Eq.1 $V_{stp} = V_{2stp} - V_{1stp} = \frac{p_2 V_2 T_{stp}}{T_{atm} p_{stp}} - \frac{p_1 V_1 T_{stp}}{T_{atm} p_{stp}}$ Eq.9

Substituting for all unknowns in Eq.9 from Eq.13,14,15 and 16.

$$V_{stp} = \left(\frac{\left(p_{atm} - p_{h2o}(T_{atm}) - \rho_b g\left(h_{t,2} - h_{c,2}\right)\right)\left(Ah_{c,2}\right)T_{stp}}{T_{atm} p_{stp}} \right) - \left(\frac{\left(p_{atm} - p_{h2o}(T_{atm}) - \rho_b g\left(h_{1,t} - h_{1,c}\right)\right)\left(Ah_{c,1}\right)T_{stp}}{T_{atm} p_{stp}} \right)$$

Eq.17

Can be rewritten as

$$V_{stp} = \frac{T_{stp} A}{T_{atm} p_{stp}} \left(\left(p_{atm} - p_{h2o}(T_{atm}) - \rho_b g\left(h_{t2} - h_{c2}\right)\right)h_{c2} - \left(p_{atm} - p_{h2o}(T_{atm}) - \rho_b g\left(h_{t1} - h_{c1}\right)\right)h_{c1} \right)$$

Eq. 18

9 References

Aguilar, A., C. Casas and J. M. Lema (1995). "Degradation of volatile fatty acids by differently enriched methanogenic cultures: Kinetics and inhibition." *Water Research* **29**(2): 505-509.

Ahn, J. H. and C. F. Forster (2000). "A comparison of mesophilic and thermophilic anaerobic upflow filters." *Bioresource Technology* **73**(3): 201-205.

Akram, A. and D. C. Stuckey (2008). "Flux and performance improvement in a submerged anaerobic membrane bioreactor (SAMBR) using powdered activated carbon (PAC)." *Process Biochemistry* **43**(1): 93-102.

APHA (2005). Standard Methods for the Examination of Water and Wastewater, American Technical Publishers.

Babel, S., K. Fukushi and B. Sitanrassamee (2004). "Effect of acid speciation on solid waste liquefaction in an anaerobic acid digester." *Water Research* **38**(9): 2417-2423.

Bae, J. H., K. W. Cho, S. J. Lee, B. S. Bum and B. H. Yoon (1998). "Effects of leachate recycle and anaerobic digester sludge recycle on the methane production from solid wastes." *Water Science and Technology* **38**(2): 159-168.

Banks, C. J. and P. N. Humphreys (1998). "The anaerobic treatment of a ligno-cellulosic substrate offering little natural pH buffering capacity." *Water Science and Technology* **38**(4-5): 29-35.

Banks, C. J. and Z. Wang (1999). "Development of a two phase anaerobic digester for the treatment of mixed abattoir wastes." *Water Science and Technology* **40**(1): 69-76.

Banks, C. J. and Z. Wang (2000). "Accelerated Hydrolysis and Acidifcation of Municipal solid Waste (MSW) in an Flushing Anaerobic Bioreactor Using Treated Leachate Recycle." *Waste Management Research* **18**: 215-223.

Baronofsky, J. J., W. J. A. Schreurs and E. R. Kashket (1984). "Uncoupling by Acetic Acid Limits Growth of and Acetogenesis by Clostridium Thermoaceticum." *Applied and Envoronmental Mocrobiology* **48**(6): 1134-1139.

Beccari, M., F. Bonemazzi, M. Majone and C. Riccardi (1996). "Interaction between acidogenesis and methanogenesis in the anaerobic treatment of olive oil mill effluents." *Water Research* **30**(1): 183-189.

Bhattacharya, S. K., V. Uberoi and M. M. Dronamraju (1996). "Interaction between acetate fed sulfate reducers and methanogens." *Water Research* **30**(10): 2239-2246.

Bohdziewicz, J., E. Neczaj and A. Kwarciak (2008). "Landfill leachate treatment by means of anaerobic membrane bioreactor." *Desalination* **221**(1-3): 559-565.

Bolzonella, D., L. Innocenti, P. Pavan, P. Traverso and F. Cecchi (2003). "Semi-dry thermophilic anaerobic digestion of the organic fraction of municipal solid waste: focusing on the start-up phase." *Bioresource Technology* **86**(2): 123-129.

Boone, D. R., W. B. Whitman and P. Rouviere (1993). Diversity and Taxonomy of Methanogens. Methanogenesis. J. G. Ferry. New York, Chanpman and Hall.

Bouallagui, H., M. Torrijos, J. J. Godon, R. Moletta, R. Ben Cheikh, Y. Touhami, J. P. Delgenes and M. Hamdi (2004). "Two-phases anaerobic digestion of fruit and vegetable wastes: bioreactors performance." *Biochemical Engineering Journal* **21**(2): 193-197.

Bouallagui, H., Y. Touhami, R. Ben Cheikh and M. Hamdi (2005). "Bioreactor performance in anaerobic digestion of fruit and vegetable wastes." *Process Biochemistry* **40**(3-4): 989-995.

Bougrier, C., H. Carrere and J. P. Delgenes (2005). "Solubilisation of waste-activated sludge by ultrasonic treatment." *Chemical Engineering Journal* **106**(2): 163-169.

Broudiscou, L., Y. Papon and A. F. Broudiscou (1999). "Optimal Mineral Composition of Artificial Saliva for Fermentation and Methanogenesis in Continuous Culture of Rumen Microorgansims." *Animal Feed Science and Technology* **79**: 43-55.

Brummeler, E. t. and I. W. Koster (1990). "Enhancement of dry anaerobic batch digestion of the organic fraction of municipal solid waste by an aerobic pretreatment step." *Biological Wastes* **31**(3): 199-210.

BS (2005). BS ISO 14853:2005 Plastics - Determination of the ultimate anaerobic biodegradation of plastic materials in an aqueous system - Method by measurement of biogas production.

Calli, B., B. Mertoglu, B. Inanc and O. Yenigun (2005). "Effects of high free ammonia concentrations on the performances of anaerobic bioreactors." *Process Biochemistry* **40**(3-4): 1285-1292.

Chanakya, H. N., S. Borgaonkar, M. G. C. Rajan and M. Wahi (1992). "Two-phase anaerobic digestion of water hyacinth or urban garbage." *Bioresource Technology* **42**(2): 123-131.

Chen, L., W. Z. Jiang, Y. Kitamura and B. Li (2007). "Enhancement of hydrolysis and acidification of solid organic waste by a rotational drum fermentation system with methanogenic leachate recirculation." *Bioresource Technology* **98**(11): 2194-2200.

Chen, Y., J. J. Cheng and K. S. Creamer (2008). "Inhibition of anaerobic digestion process: A review." *Bioresource Technology* **99**(10): 4044-4064.

Chiu, Y.-C., C.-N. Chang, J.-G. Lin and S.-J. Huang (1997). "Alkaline and ultrasonic pretreatment of sludge before anaerobic digestion." *Water Science and Technology* **36**(11): 155-162.

Choo, K.-H. and C.-H. Lee (1996). "Membrane fouling mechanisms in the membrane-coupled anaerobic bioreactor." *Water Research* **30**(8): 1771-1780.

Chynoweth, D. P., R. A. Mah, P. H. Smith and A. C. Wilkie (1993a). "Ecology and Microbiology of Biogassification." *Biomass and Bioenergy* **5**: 191-202.

Chynoweth, D. P., J. Owens, D. O'Keefe, J. F. K. Earle, G. Bosch and R. Legrand (1992). "Sequential batch anaerobic composting of the organic fraction of municipal solid waste." *Water Science and Technology* **25**(7): 327-339.

Chynoweth, D. P. and P. C. Pullammanappallil (1996). Anaerobic Digestion of Municipal Solid Waste. Microbiology of Solid Wastes. A. C. Palmisano and M. A. Barlaz, CRC Press.

Chynoweth, D. P., C. E. Turick, J. M. Owens, D. E. Jerger and M. W. Peck (1993b). "Biochemical Methane Potential of Biomass and Waste Feedstocks." *Biomass & Bioenergy* **5**(1): 95-111.

Climenhaga, M. A. and C. J. Banks (2007). Anaerobic Digestion of Catering Wastes: Effect of Micronutrients and Retention Time. 11th World Congress on Anaerobic Digestion. Brisbane, Australia, IWA.

Cuetos, M. J., X. Gómez, M. Otero and A. Morán (2008). "Anaerobic digestion of solid slaughterhouse waste (SHW) at laboratory scale: Influence of co-digestion with the organic fraction of municipal solid waste (OFMSW)." *Biochemical Engineering Journal* **40**(1): 99-106.

Cysneiros, D., K. A. G. Karatzas, S. Heaven and C. J. B. Banks (2007). Anaerobic Digestion of Maize for Energy Production in Leach-bed Reactors. *11th IWA World Congress on Anaerobic Digestion*, Brisbane, Australia, IWA.

Dalhoff, R., A. Rababah, V. Sonakya, N. Raizada and P. A. Wilderer (2003). "Membrane separation to improve degradation of road side grass by rumen enhance solid incubation." *Water science and technology* **48**(4): 163-168.

Davidsson, A., C. Gruvberger, T. H. Christensen, T. L. Hansen and J. I. C. Jansen (2007). "Methane yield in source-sorted organic fraction of municipal solid waste." *Waste Management* **27**(3): 406-414.

De Baere, L. (2000). "Anaerobic digestion of solid waste: State-of-the-art." *Water Science and Technology* **41**(3): 283-290.

Defra (2007a). Municipal Waste Statistics 2006/7. Defra.

Defra (2007b). Waste Strategy 2007/2008 (WS2007). Defra.

Delgenes, J. P., V. Penaud and R. Moletta (2003). Pretreatments for the Enhancement of AD of Solid Waste. Biomethanisation of the Organic Fraction of Municipal Solid Waste. J. Mata-Alvarez, IWA Publishing.

EU (1999). "Council Directive 1999/31/EC on the Landfill of Waste." *Official Journal of the European Communities* **L182/1-19**

Fan, B. and X. Huang (2002). "Charateristics of a Self-Forming Dynamic Membrane Couple with a Bioreactor for Municiapl Wastewater Treatment." *Environmental Science and Technology* **36**: 5245-5251.

Field, R. W., D. Wu, J. A. Howell and B. B. Gupta (1995). "Critical flux concept for microfiltration fouling." *Journal of Membrane Science* **100**(3): 259-272.

Forster-Carneiro, T., M. Perez, L. I. Romero and D. Sales (2007). "Dry-thermophilic anaerobic digestion of organic fraction of the municipal solid

waste: Focusing on the inoculum sources." *Bioresource Technology* **98**(17): 3195-3205.

Fox, P. and F. G. Pohland (1994). "Anaerobic Applications and Fundamentals: Substrate Specificity During Phase Searation." *Water Environment Research* **66**(5): 716.

Fuchs, W., C. Resch, M. Kernstock, M. Mayer, P. Schoeberl and R. Braun (2005). "Influence of operational conditions on the performance of a mesh filter activated sludge process." *Water Research* **39**(5): 803-810.

Gallert, C., A. Henning and J. Winter (2003). "Scale-up of anaerobic digestion of the biowaste fraction from domestic wastes." *Water Research* **37**(6): 1433-1441.

Garcia-Heras, J. L. (2003). Reactor Sizing, Process Kinetics and Modelling of Anaerobic Digestion of Complex Wastes. Biomethanisation of the Organic Fraction of Municipal Solid Waste. J. Mata-Alvarez, IWA Publishing.

Garcia, J. B., R. G. Ibanez, G. H.A.A and S. O.C. (1991). "On the Effect of Basicity on the Kinteics of CO2 Absorbtion in Tertiary Amines." *Chemical Engineering Science* **46**(11): 2927-2931.

Gavala, H. N., I. V. Skiadas and G. Lyberatos (1999). "On the performance of a centralised digestion facility receiving seasonal agroindustrial wastewaters." *Water Science and Technology* **40**(1): 339-346.

Gerardi, M. H. (2003a). 1. Overview. Microbiology of Anaerobic Digesters. M. H. Gerardi, John Wiley and Sons.

Gerardi, M. H. (2003b). 2. Bacteria. Microbiology of Anaerobic Digesters. M. H. Gerardi, John Wiley and Sons.

Gerardi, M. H. (2003c). 3. Methane-Forming Bacteria. Microbiology of Anaerobic Digesters. M. H. Gerardi, John Wiley and Sons.

Gerardi, M. H. (2003d). 14. Temperature. Microbiology of Anaerobic Digesters. M. H. Gerardi, John Wiley and Sons.

Gerardi, M. H. (2003e). 17. Toxicity. Microbiology of Anaerobic Digesters. M. H. Gerardi, John Wiley and Sons.

Gerardi, M. H. (2003f). 19 Upsets and Unstable Digesters. Microbiology of Anaerobic Digesters. M. H. Gerardi, John Wiley and Sons.

Gerardi, M. H. (2003g). 23. Types of Anaerobic Digesters. Microbiology of Anaerobic Digesters. M. H. Gerardi, John Wiley and Sons.

Gijzen, H. J., P. J. L. Derikx and G. D. Vogels (1990). "Application of rumen microorganisms for a high rate anaerobic digestion of papermill sludge." *Biological Wastes* **32**(3): 169-179.

Gijzen, H. J., F. J. Lubberding, F. J. Verhagen, K. B. Zwart and G. D. Vogels (1987a). "Aplication of Rumen Microorganisms for an Enhanced Anaerobic Digestion of Solid Organic Waste Materials." *Biological Wastes* **22**: 81-95.

Gijzen, H. J., H. J. M. Op den Camp, G. J. M. Verkley and G. D. Vogels (1989). "Application of rumen microorganisms in the anaerobic fermentation of an organic fraction of domestic refuse." *Biological Wastes* **30**(4): 309-316.

Gijzen, H. J., K. B. Zwart, M. J. Teunissen and G. D. vogels (1987b). "Anaerobic Digestion of Cellulose Fraction of Domsetic Refuse by Means of Rumen Microorganisms." *Biotechnology and Bioengineering* **32**: 749-755.

Gijzen, H. J., K. B. Zwart, P. T. van Gelder and G. D. vogels (1986). "Continuous Cultivation of Rumen Microorganisms, a System with Possible Application to the Anaerobic Digestion of Lignocellulosic Waste Materials." *Applied Microbiology and Biotechnology* **25**: 155-162.

Gijzen, H. J., K. B. Zwart, F. J. M. Verhagen and G. D. Vogels (1988). "High-Rate 2-Phase Process for the Anaerobic Degradation of Cellulose, Employing Rumen Microorganisms for an Efficient Acidogenesis." *Biotechnology and Bioengineering* **31**(5): 418-425.

Goff, J. A. and S. Gratch (1946). "Low Pressure Properties of Water from -160 to 212 degrees F." *Transactions of the American Society of Heating and Ventilating Engineers*: 347-354.

Gunaseelan, V. N. (1997). "Anaerobic digestion of biomass for methane production: A review." *Biomass and Bioenergy* **13**(1-2): 83-114.

Hartmann, H. and B. K. Ahring (2005). "Anaerobic digestion of the organic fraction of municipal solid waste: Influence of co-digestion with manure." *Water Research* **39**(8): 1543-1552.

He, P.-J., F. Lu, L.-M. Shao, X.-J. Pan and D.-J. Lee (2007). "Kinetics of enzymatic hydrolysis of polysaccharide-rich particulates." *Journal of the Chinese Institute of Chemical Engineers* **38**(1): 21-27.

Heaven, S., C. J. Banks and M. Cornell (2008a). Effect of solid and liquid retention times on hydrolysis of maize. *5th International Symposium of*

Anaerobic Digestion of Solid Waste and Energy Crops, Hammamet, Tunisia, IWA.

Heaven, S., J. A. Siles-Lopez, C. J. Banks and M. A. Martin-Santos (2008b). Two-phase Hydraulic Flush Reactors for Maize Digestion at High Loading Rates. *5th International Symposium of Anaerobic Digestion of Solid Waste and Energy Crops*, Hammamet, Tunisia, IWA.

Hills, D. J. and K. Nakano (1984). "Effects of particle size on anaerobic digestion of tomato solid wastes." *Agricultural Wastes* **10**(4): 285-295.

Hu, A. Y.-C. (2004). Submerged Anaerobic Membrane Bioreactor for Wastewater Treatment. Departement of Chemical Engineering. London, Imperial College. **PhD**.

Hu, Z.-H., G. Wang and H.-Q. Yu (2004). "Anaerobic degradation of cellulose by rumen microorganisms at various pH values." *Biochemical Engineering Journal* **21**(1): 59-62.

Hu, Z.-H. and H.-Q. Yu (2005). "Application of rumen microorganisms for enhanced anaerobic fermentation of corn stover." *Process Biochemistry* **40**(7): 2371-2377.

Jeison, D. and J. B. van Lier (2007). "Cake formation and consolidation: Main factors governing the applicable flux in anaerobic submerged membrane bioreactors (AnSMBR) treating acidified wastewaters." *Separation and Purification Technology* **56**(1): 71-78.

Jiang, W. Z., Y. Kitamura and B. Li (2005). "Improving acidogenic performance in anaerobic degradation of solid organic waste using a rotational drum fermentation system." *Bioresource Technology* **96**(14): 1537-1543.

Jones, A., S. Nesaratnam and A. Porteous (2006). The Open University Household Waste Study. F. R. A. The Department for Environment, The Open University.

Jones, W. J., D. P. Naggle Jr and W. B. Whitman (1987). "Methanogens and the Diversity of Archaebacteria." *Microbiological Reviews* **51**: 135-177.

Kayhanian, M. (1995). "Biodegradability of the organic fraction of municipal solid waste in a high-solids anaerobic digester." *Waste Management & Research* **13**(2): 123-136.

Kayhanian, M. and S. Hardy (1994). "The Impact of 4 Design Parameters on the Performance of High-Solids Anaerobic Digestion Process of Municipal

Solid Waste for Fuel Gas Production." *Environmental Technology* **15**(6): 557-567.

Kiso, Y., Y.-J. Jung, T. Ichinari, M. Park, T. Kitao, K. Nishimura and K.-S. Min (2000). "Wastewater treatment performance of a filtration bio-reactor equipped with a mesh as a filter material." *Water Research* **34**(17): 4143-4150.

Kiso, Y., Y.-J. Jung, M.-S. Park, W. Wang, M. Shimase, T. Yamada and K.-S. Min (2005). "Coupling of sequencing batch reactor and mesh filtration: Operational parameters and wastewater treatment performance." *Water Research* **39**(20): 4887-4898.

Kivaisi, A. K. and S. Eliapenda (1995). "Application of rumen microorganisms for enhanced anaerobic degradation of bagasse and maize bran." *Biomass and Bioenergy* **8**(1): 45-50.

Kivaisi, A. K., H. J. Gijzen, H. Dencamp and G. D. Vogels (1992). "Conversion of Cereal Residues into Biogas in a Rumen-Derived Process." *World Journal of Microbiology & Biotechnology* **8**(4): 428-433.

Koseoglu, H., N. O. Yigit, V. Iversen, A. Drews, M. Kitis, B. Lesjean and M. Kraume (2008). "Effects of several different flux enhancing chemicals on filterability and fouling reduction of membrane bioreactor (MBR) mixed liquors." *Journal of Membrane Science* **320**(1-2): 57-64.

Kroeker, E. J., D. D. Schulte, A. B. Sparling and H. M. Lapp (1979). "Anaerobic treatment process stability." *Journal of Water Pollution Control Federation* **51**: 718-727.

Lai, T. E., A. Nopharatana, P. C. Pullammanappallil and W. P. Clarke (2001). "Cellulolytic activity in leachate during leach-bed anaerobic digestion of municipal solid waste." *Bioresource Technology* **80**(3): 205-210.

Lehtomaki, A. (2006). Biogas Production from Energy Crops and Crop Residues. Jyvaskyla Studies in Biological and Environmental Sciences. Jyvaskyla. **PhD**.

Lehtomäki, A., S. Huttunen, T. M. Lehtinen and J. A. Rintala (2008). "Anaerobic digestion of grass silage in batch leach bed processes for methane production." *Bioresource Technology* **99**(8): 3267-3278.

Lin, J.-G., C.-N. Chang and S.-C. Chang (1997). "Enhancement of anaerobic digestion of waste activated sludge by alkaline solubilization." *Bioresource Technology* **62**(3): 85-90.

Lissens, G., P. Vandevivere, L. De Baere, E. M. Biey and W. Verstraete (2001). "Solid waste digestors: Process performance and practice for municipal solid waste digestion " *Water Science & Technology* **44**(8): 91-102.

Liu, H. W., H. K. Walter, G. M. Vogt, H. S. Vogt and B. E. Holbein (2002). "Steam pressure disruption of municipal solid waste enhances anaerobic digestion kinetics and biogas yield." *Biotechnology And Bioengineering* **77**(2): 121-130.

Liu, T. and S. Ghosh (1997). "Phase separation during anaerobic fermentation of solid substrates in an innovative plug-flow reactor." *Water Science and Technology* **36**(6-7): 303-310.

Llabres-Luengo, P. and J. Mata-Alvarez (1988). "Hydrolytic Step in a Dry Digestion System." *Biological Wastes* **23**(1): 25-37.

Mata-Alvarez, J. (2003a). Biomethanisation of the Organic Fraction of Municipal Solid Waste.

Mata-Alvarez, J. (2003b). Fundamentals of the Anaerobic Digestion Process. Biomethanisation of the Organic Fraction of Municipal Solid Waste. J. Mata-Alvarez, IWA Publishing.

Mata-Alvarez, J., S. Mace and P. Llabres (2000). "Anaerobic digestion of organic solid wastes. An overview of research achievements and perspectives." *Bioresource Technology* **74**(1): 3-16.

Mathrani, I. M., D. R. Boone, R. A. Mah, P. Fox and P. P. Lau (1988). "Methanohalophilus Zhilinae sp. nov., an Alkaliphilic, Halophilic, Methylotrophic Methanogen." *International Journal of Systematic Bacteriology* **38**: 139-142.

McDougall, E. O. (1948). "Studies on Ruminant Saliva. I. The Composition oand Output of Sheep's Saliva." *Biochemistry Journal* **43**: 99-109.

Mshandete, A., L. Björnsson, A. K. Kivaisi, M. S. T. Rubindamayugi and B. Mattiasson (2006). "Effect of particle size on biogas yield from sisal fibre waste." *Renewable Energy* **31**(14): 2385-2392.

Neves, L., R. Oliveira and M. M. Alves (2006). "Anaerobic co-digestion of coffee waste and sewage sludge." *Waste Management* **26**(2): 176-181.

Nopharatana, A., W. P. Clarke, P. C. Pullammanappallil, P. Silvey and D. P. Chynoweth (1998). "Evaluation of methanogenic activities during anaerobic digestion of municipal solid waste." *Bioresource Technology* **64**(3): 169-174.

Nopharatana, A., P. C. Pullammanappallil and W. P. Clarke (2007). "Kinetics and dynamic modelling of batch anaerobic digestion of municipal solid waste in a stirred reactor." *Waste Management* **27**(5): 595-603.

O'Sullivan, C., P. C. Burrell, W. P. Clarke and L. L. Blackall (2008). "The effect of biomass density on cellulose solubilisation rates." *Bioresource Technology* **99**(11): 4723-4731.

O'Sullivan, C. A., P. C. Burrell, W. P. Clarke and L. L. Blackall (2005). "Structure of a Cellulose Degrading Bacterial Community During Anaerobic Digestion." *Biotechnology and Bioengineering* **92**: 871-878.

Odier, E. and I. Artaud (1992). Degradation of lignin. Microbial Degradation of Natural Products. G. Winkelmann. Weinheim, Germany, Verlag Chemie.

Palmowski, L. and J. Muller (2000). "Influence of the Size Reduction of Organic Waste on their Anaerobic Digestion." *Water Science and Technology* **43**(3): 155-162.

Parawira, W., M. Murto, J. S. Read and B. Mattiasson (2005). "Profile of hydrolases and biogas production during two-stage mesophilic anaerobic digestion of solid potato waste." *Process Biochemistry* **40**(9): 2945-2952.

Paulo, P. L., M. V. G. Vallero, R. H. M. Trevino, G. Lettinga and P. N. L. Lens (2004). "Thermophilic (55 [degree sign]C) conversion of methanol in methanogenic-UASB reactors: influence of sulphate on methanol degradation and competition." *Journal of Biotechnology* **111**(1): 79-88.

Pavan, P., P. Battistoni, F. Cecchi and J. Mata-Alvarez (1999). Two Phase Anaerobic Digestion of Source-Sorted OFMSW: Performance and Kinetic Study. *International Symposium of Anerobic Digestion, Solid Waste*, Barcellona, International Association on Water Quality.

Poll, J. (2003). The Composition of Municipal Solid Waste in Wales. W. A. Government.

Psoch, C. and S. Schiewer (2006). "Anti-fouling application of air sparging and backflushing for MBR." *Journal of Membrane Science* **283**(1-2): 273-280.

Rao, M. S. and S. P. Singh (2004). "Bioenergy conversion studies of organic fraction of MSW: kinetic studies and gas yield-organic loading relationships for process optimisation." *Bioresource Technology* **95**(2): 173-185.

Ripley, L. E., W. C. Boyle and J. C. Converse (1986). "Improved alkalimetric monitoring for anaerobic digestion of high-strength wastes." *Journal of the Water Pollution Control Federation* **58**(5): 406-411.

Rivard, C. J., W. S. Adney and M. E. Himmel (1991). Enzymes of Anaerobic Municipal Solid Waste Disposal. Enzymes in Biomass Conversion, ACS Symposium Series 460. G. F. Leatham and M. E. Himmel. Washington, DC, American Chemical Society.

RRF (2001). Assessment of Kerbside Collection Schemes of Dry Recylables, Resource Recovery Forum (RRF).

Rufener Jr, W. H., W. O. Nelson and M. J. Wolin (1962). "Maintenance of Rumen Populations in Continuous Culture." *Applied Microbiology* **11**: 196-201.

Saddoud, A., M. Ellouze, A. Dhouib and S. Sayadi (2007). "Anaerobic membrane bioreactor treatment of domestic wastewater in Tunisia." *Desalination* **207**(1-3): 205-215.

Sanders, W. M. T., A. H. M. Veeken, G. Zeeman and J. B. Van Lier (2003a). Analysis and Optimisation of Anaerobic Digestion of the Organic Fraction of Municipal Solid Waste. Biomethanisation of the Organic Fraction of Municipal Solid Waste. J. Mata-Alvarez, IWA Publishing.

Sanders, W. M. T., A. H. M. Veeken, G. Zeeman and J. B. van Lier (2003b). Analysis and optimisation of the anaerobic digestion of the organic fraction of municipal solid waste. Biomethanization of the Organic Fraction of Municipal Solid Wastes. J. Mata-Alvarez**:** pp. 67–89.

Satyawali, Y. and M. Balakrishnan (2008). "Treatment of distillery effluent in a membrane bioreactor (MBR) equipped with mesh filter." *Separation and Purification Technology* **63**(2): 278-286.

Schonberg, J. C., S. K. Bhattacharya, R. L. Madura, S. H. Mason and R. A. Conway (1997). "Evaluation of anaerobic treatment of selected petrochemical wastes." *Journal of Hazardous Materials* **54**(1-2): 47-63.

Sekiguchi, Y., Y. Kamagata and H. Harada (2001). "Recent advances in methane fermentation technology." *Current Opinion in Biotechnology* **12**(3): 277-282.

Shanmugam, P. and N. J. Horan (2009). "Simple and rapid methods to evaluate methane potential and biomass yield for a range of mixed solid wastes." *Bioresource Technology* **100**(1): 471-474.

Shin, H. S., S. K. Han, Y. C. Song and C. Y. Lee (2001). "Performance of uasb reactor treating leachate from acidogenic fermenter in the two-phase anaerobic digestion of food waste." *Water Research* **35**(14): 3441-3447.

Siegert, I. and C. Banks (2005). "The effect of volatile fatty acid additions on the anaerobic digestion of cellulose and glucose in batch reactors." *Process Biochemistry* **40**(11): 3412-3418.

Siegrist, H., D. Renggli and W. Gujer (1993). "Mathematical Modelling of Anaerobic Meshophilic Sewage Treatment." *Water Science and Technology* **27**(2): 25-36.

Song, H., W. P. Clarke and L. L. Blackall (2005). "Concurrent Microscopic Observations and Activity Measurements of Cellulose Hydrolyzing and Methanogenic Populations During the Batch Anaerobic Digestion of Crystalline Cellulose." *Biotechnology and Bioengineering* **91**: 369-378.

South, C. R., D. A. L. Hogsett and L. R. Lynd (1995). "Modeling simultaneous saccharification and fermentation of lignocellulose to ethanol in batch and continuous reactors." *Enzyme and Microbial Technology* **17**(9): 797-803.

Sponza, D. T. (2003). "Toxicity and treatability of carbontetrachloride and tetrachloroethylene in anaerobic batch cultures " *International Biodeterioration and Biodegradation* **51**(2): 119-127

Tiehm, A., K. Nickel and U. Neis (1997). "The use of ultrasound to accelerate the anaerobic digestion of sewage sludge." *Water Science and Technology* **36**(11): 121-128.

Torres-Castillo, R., P. Llabrés-Luengo and J. Mata-Alvarez (1995). "Temperature effect on anaerobic digestion of bedding straw in a one phase system at different inoculum concentration." *Agriculture, Ecosystems & Environment* **54**(1-2): 55-66.

Trzcinski, A. P. and D. C. Stuckey (2008). Continuous treatment of the Organic Fraction of Municipal Solid Waste in an anaerobic two-stage membrane process. 5th International Symposium on Anaerobic Digestion of Solid Waste and Energy Crops. Hammamet, Tunisia, IWA.

Vandevivere, P., L. De Baere and W. Verstraete (2003a). Types of Anaerobic Digester. Biomethanisation of the Organic Fraction of Municipal Solid Waste. J. Mata-Alvarez, IWA Publishing.

Vandevivere, P., L. De Baere and W. Verstraete (2003b). Types of Anaerobic Digester for Solid Waste. Biomethanisation of the Organic Fraction of Municipal Solid Waste. J. Mata-Alvarez, IWA Publishing.

Vargas, A., I. Moreno-Andrade and G. Buitrón (2008). "Controlled backwashing in a membrane sequencing batch reactor used for toxic wastewater treatment." *Journal of Membrane Science* **320**(1-2): 185-190.

Vavilin, V. A., B. Fernandez, J. Palatsi and X. Flotats (2008). "Hydrolysis kinetics in anaerobic degradation of particulate organic material: An overview." *Waste Management* **28**(6): 939-951.

Vavilin, V. A., S. V. Rytov and L. Y. Lokshina (1996). "A Description of Hydrolysis Kinetics in Anaerobic Digestions of Particulate Matter." *Bioresource Technology* **56**: 229-237.

Vaz, F., A. Torres and C. Neiva Correia (2008). Case Study: The Characteristics of the Biodegradable Waste for the Anaerobic Digestion Plant in Lisbon Area. 5th International Symposium on on Anaerobic Digestion of Solid Wastes and Energy Crops. Hammamet, Tunisia, IWA.

Veeken, A. H. M. and B. Hamelers (1999). "Effect of temperature on hydrolysis rates of selected biowaste components." *Bioresource Technology* **69**(3): 249-254.

Veeken, A. H. M., S. V. Kalyuzhnyi, H. Scharff and B. Hamelers (2000). "Effect of pH and VFA on Hydrolysis of Organic Solid Waste." *Journal of Environmental Engineering* **126**(12): 1076-1081.

Vishniac, W. and M. Santer (1957). "The Thiobacilli." *Bacteriological Reviews* **21**: 195-213.

Wang, W., Y.-J. Jung, Y. Kiso, T. Yamada and K.-S. Min (2006). "Excess sludge reduction performance of an aerobic SBR process equipped with a submerged mesh filter unit." *Process Biochemistry* **41**(4): 745-751.

Wang, Z. and C. J. Banks (2000). "Accelerated hydrolysis and acidification of municipal solid waste (MSW) in a flushing anaerobic bio-reactor using treated leachate recirculation." *Waste Management & Research* **18**(3): 215-223.

Weiland, P. (1993). "One- and two-step anaerobic digestion of solid agroindustrial residues." *Water Science and Technology* **27**(2): 145-151.

Westwood, D. (2007). The determination of chemical oxygen demandand in waters and effluents, Environment Agency, National Laboratory Service.

Williams, R. T. and R. L. Crawford (1984). "Methane Production in Minesota Peatlands." *Applied Environmental Microbiology* **47**: 1266-1271.

Wu, G., L. Cui and Y. Xu (2008). "A novel submerged rotating membrane bioreactor and reversible membrane fouling control." *Desalination* **228**(1-3): 255-262.

Yang, J. and R. E. Speece (1986). "The effects of chloroform toxicity on methane fermentation." *Water Research* **20**(10): 1273-1279.

Yang, W., N. Cicek and J. Ilg (2006). "State-of-the-art of membrane bioreactors: Worldwide research and commercial applications in North America." *Journal of Membrane Science* **270**(1-2): 201-211.

Zhang, B., L. L. Zhang, S. C. Zhang, H. Z. Shi and W. M. Cai (2005). "The influence of pH on hydrolysis and acidogenesis of kitchen wastes in two-phase anaerobic digestion." *Environmental Technology* **26**(3): 329-339.

Zhang, Y., C. J. Banks and S. Heaven (2008). The Characterisation of Municipal Waste Streams and a Preliminary Evaluation of the Co-Digestion Efficiency with Some Industrial and Agricultural Wastes. 5th International Symposium on Anaerobic Digestion of Solid Wastes and Energy Crops. Hammamet, Tunisia, IWA.

Zinder, S. H. (1993). Physiological Ecology of Methanogens. Methanogenesis. J. G. Ferry. New York, Chanpman and Hall.