UNIVERSITY OF SOUTHAMPTON

FACULTY OF ENGINEERING AND THE ENVIRONMENT

THE ANAEROBIC DIGESTION OF SUGAR BEET PULP

By

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ABSTRACT

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World-wide there are substantial quantities of sugar beet pulp, which arises as a residue after the processing of whole beet to extract sugar for refining as a foodstuff or for use in fermentation, in particular for the production of ethanol for the biofuel market. In both cases the resulting pulp residue is still rich in pentose sugars and fibre, and the research considered anaerobic digestion (AD) as a potential technology for the conversion of this material into renewable energy in the form of biogas. To determine the best operational conditions for biogas production both mesophilic and thermophilic digestion options were considered. Both were tested using 4-litre working volume mixed digesters operated with semi-continuous feed over a minimum of three hydraulic retention times (HRT). The first long term trial used mesophilic temperatures (37 ^{o}C±0.5 ^{o}C) at applied organic loading rates (OLR) from 2-5 g volatile solids (VS) l^{-1} day^{-1}. This resulted in a specific methane yield of ~0.31 l CH_4 g^{-1} VS day^{-1} with a biogas methane content of 51.05%. VS destruction was ~90% at all loadings, and increasing the loading resulted in an increase in volumetric biogas and methane production without significant loss in specific yields. The major limitation found was not in the biochemical conversion but in dewatering of the digestate, the characteristics of which were assessed using capillary suction time (CST) and frozen image centrifugation (FIC). At the higher loading there was also the appearance of a stable foam which made the digesters difficult to operate as this could block the gas outlet, leading to pressure increases and the loss of digestate by 'blow out'.

In the same digesters at mesophilic temperatures antifoam was tested to assess if this could offer a solution by suppressing foam formation. In practice this required unusually high doses of the reagent and, in continued use, these appeared to have an inhibitory effect on the digestion process. Dilution of the feedstock to the digester was also tested but showed no beneficial effects on dewaterability or foaming. As a post-treatment alternative cellulolytic enzymes were added to the digestate, but had no effect on improving dewaterability. Trace element (TE) supplementation to the digesters was, however, shown to eliminate the occurrence of foaming and also gave a slight improvement in dewaterability. TE supplementation reduced the polymer dose required for dewatering as determined by the CST test, and eliminated polymer dosing when dewatering was by centrifugation. Digestate dewaterability could also be improved in a post-digestion one- and two-stage chemical treatment with the use of chemical coagulants/flocculants alone or combined.

The second long-term trial compared mesophilic (37 ^{o}C±0.5 ^{o}C) and thermophilic (55 ^{o}C±0.5 ^{o}C) digestion over 3 HRT using duplicate digesters fed at OLR of 4 and 5 g VS l^{-1} day^{-1}. The digesters were operated without water addition. The thermophilic digesters gave higher biogas and methane productivity and were also able to operate stably at the higher OLR, whereas the mesophilic digesters showed signs of instability. Digestate dewaterability was assessed by the CST and FIC tests and the likelihood of stable foam forming was assessed using a foaming potential test. The results showed thermophilic operation performed better even at the higher loading and gave a digestate with superior dewatering characteristics and with very little foaming potential. Using a combination of CST tests, filtration tests, Frozen Image Centrifugation, SEM and grading centrifugation it was concluded that the poor dewaterability seen in mesophilic digestate was due to the presence of extracellular polymer substance (EPS) leading to blinding of the filter by fine particulate materials.

The carbon, energy and nutrient (CEN) footprint was estimated for mesophilic and thermophilic digestion in which the process was coupled with combined heat and power (CHP) and biogas upgrading to biomethane. The results showed that the energy input for thermophilic digestion was higher than for mesophilic although this could be compensated for by the increased specific methane yield at the higher loadings modelled. There was also no significant difference in the emissions savings or in the quantities of nutrients recycled in the digestate. The model indicated that the use of CHP gave a higher net energy yield compared to biogas upgrading, but this of course is dependent on there being an economic use for the heat produced.

Keywords: anaerobic digestion, sugar beet pulp, biogas, trace elements; digestate, dewatering, foam formation, carbon footprint, energy footprint, nutrient footprint

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DECLARATION OF AUTHORSHIP

I, Sri Suhartini

declare that the thesis entitled

ANAEROBIC DIGESTION OF SUGAR BEET PULP

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:

- this work was done wholly or mainly while in candidature for a research degree at this University;
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- 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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Signed: ……..

Date:…….

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DEFINITIONS AND ABBREVIATIONS

AD	Anaerobic Digestion
BMP	Biochemical Methane Potential
CEN	Carbon Energy Nutrient (footprint)
COD	Chemical Oxygen Demand
CSTR	Continuously Stirred Tank Reactor
CST	Capillary Suction Time
DM	Dry Matter
EPS	Extracellular Polymeric Substances
EU	European Union
FIC	Frozen Image Centrifuge
FVSW	Fruits and Vegetable Waste
GC	Gas Chromatography
HCV	Higher Calorific Value
HHV	Higher Heating Value
HRT	Hydraulic Retention Time
IA	Intermediate Alkalinity
MFW	Mixed Food Waste
OLR	Organic Loading Rate
PA	Partial Alkalinity
PAC	Powered Activated Carbon
PS	Primary Sludge
PVC	Poly Vinyl Chloride
SBP	Sugar Beet Pulp
SEM	Scanning Electron Microscope
SMP	Specific Methane Production
SRF	Specific Resistance to Filtration
SRT	Solid Retention Time
SS	Surplus Sludge
STP	Standard Temperature and Pressure
TA	Total Alkalinity
TE	Trace Element
TKN	Total Kjedhal Nitrogen
uPVC	unplasticated Poly Vinyl Chloride
VFA	Volatile Fatty Acid
TS	Total Solids
VS	Volatile Solids
WAS	Waste Activated Sludge
WW	Wet Weight

CHAPTER 1. INTRODUCTION

1.1. Background

Sugar Beet Pulp (SBP) is the solid organic residue that arises from sugar beet processing and is generated worldwide, in particular in Europe and the USA. It has a number of potential uses, such as the production of soluble neutral- and acid-sugars by enzymatic hydrolysis (Spagnuolo et al., 1997); non-food grade xanthan gum using *Xanthomonas campestris* (Yoo and Harcum, 1999); biodegradable composites for lightweight construction materials (Liu et al., 2005); and as a soil fertiliser (Medina et al., 2007). In the UK, which has an annual SBP production of around of 500,000 tonnes $year^{-1}$ (British Sugar, 2011), it is mainly used as cattle feed as it contains all the basic constituents of forage including fibre and amino acids.

There is also the potential to use SBP as a feedstock for anaerobic digestion providing biogas as a renewable energy source: this has an energy value in the range 22 to 26 MJ m^{-3} depending on the fraction of methane in the biogas (Lapp et al., 1978). Anaerobic digestion (AD) is capable of processing large tonnage quantities and in addition to biogas production its use allows nutrient and organic carbon recycle back to agriculture through the spreading of digestate. The process also gives a reduction in biological risks and the potential for malodour (Berglund and Börjesson, 2006). There may also be economic benefits through reduced material handling and transport costs and better revenue returns from energy production compared to the animal feed market (Mata-Alvarez et al., 2000; Parawira et al., 2008; Koppar and Pullammanappallil, 2008).

Several experimental studies have looked at the anaerobic digestion of SBP in single-stage and two-stage processes under a wide range of operational parameters conditions, including different organic loading rates, temperature and pH. Although substrate conversion was generally successful, one of the main concerns in SBP digestion is the dewaterability of the digestate, due to the requirement to transport the material to land for agricultural re-use. Another problem in the anaerobic digestion of SBP is foaming which may be due to the presence of fine particles causing entrainment of gas bubbles. Stoppok and Buchholz (1985) showed that digestion of SBP at high loading rate (15 g l^{-1} day^{-1}) led to several technical problems due to highly viscous fluid-substrate mixture, and suggested that this was due to the high cellulosic content of SBP. Similarly, work

by Brooks et al. (2008) found that foaming occurred at loading rates higher than 5.5 kg COD m^{-3} d^{-1} (lab-scale) and 10 kg COD m^{-3} d^{-1} (pilot-scale), possibly as a result of the high sugar content in fresh SBP.

Optimising the positive effects of applying AD to SBP is crucial to the achievement of economic savings and environmental-friendly performance. Mitigating digestate dewaterability problems could also be beneficial since the digestate still contains organic materials that represent valuable products, such as compost (soil conditioner) or bio-fertiliser. These products can be applied as an alternative fertiliser for agriculture. Improved dewaterability could reduce digestate volume and handling costs, and improve the ease of transportation. Furthermore, minimising foaming problems in the anaerobic digestion of SBP may also improve its dewaterability.

Although anaerobic digestion plays an important role in providing a source of renewable energy, it also consumes resources and creates by-products such as gaseous emissions (CO_2). These products may cause environmental impacts including global warming or greenhouse effects. There are also impacts from the materials used in construction and operation of the AD plant and from fugitive methane emissions during normal operation. The possible environmental impact from GHG emissions is a function of the materials going into the biogas plant and the efficiency of biogas utilisation (Djatkov et al., 2011), as well as the transportation distance (Chevalier and Meunier, 2005). Other environmental impacts may arise from nitrogen emissions during digestate treatment, storage and field application (Rehl and Müller, 2011).

Knowing the net energy balance of an AD plant, and hence the efficiency of the digestion process, is vital for assessing whether AD is a feasible and profitable option for treating SBP. This is supported by Salter and Banks (2009), who stated that an energy balance can be used to determine whether AD is ‘renewable’ and ‘sustainable’ option as a source of energy. In addition, this type of analysis establishes all energy inputs and outputs and other resources used in AD, thus both the process economy (i.e. energy footprint) and its environmental impacts (i.e. carbon and nutrient footprint) can be identified; and these ‘footprints’ are currently one of the key parameters to be considered in designing and implementing AD systems. Therefore this study, anaerobic

digestion of SBP will be assessed by measuring and analysing the carbon, energy and nutrient footprints.

1.2. Research Aims and Objectives

The research had three main aims:

Aim 1. To quantify the potential of SBP for biogas production through anaerobic digestion and to determine the optimum operating parameters for a stable high-performance process

Aim 2. To identify methods of reducing foaming and improving dewaterability of digestate from the AD of SBP

Aim 3. To assess the feasibility of anaerobic digestion of SBP in terms of its Carbon, Energy, and Nutrient (CEN) footprint.

The work was divided into a number of experiments and activities, with specific objectives that developed sequentially throughout the course of the research. These objectives were:

- To determine the physico-chemical characteristics of SBP, including its biochemical methane yield as a baseline for comparison with performance in semi-continuous digestion.
- To determine the long-term digestion performance of SBP under semi-continuous fed conditions in laboratory-scale digesters, in terms of biogas and methane production and operational stability.
- To ascertain whether there were factors that might adversely affect this performance and if so to identify possible solutions.
- To identify any effects from trace element supplementation in terms of process performance, foaming or dewaterability.
- To assess whether measures such as addition of water to the feedstock or the use of chemical antifoam preparation could reduce foaming and enhance digestate dewaterability in mesophilic conditions.

- To determine whether thermophilic digestion could improve the specific methane production, maximum organic loading rates and the physical properties of the digestate, by reducing the tendency to foam and improving the dewaterability.
- To identify the optimum dilution to be used in chemical treatment to improve digestate dewaterability.
- To compare the effect of commonly-used chemical coagulants on digestate from mesophilic and thermophilic digesters.
- To evaluate the effect of two-stage chemical conditioning on dewaterability of digestates from a range of operating conditions (with and without TE addition, at different OLR, at mesophilic or thermophilic temperature).
- To assess whether freeze/thaw treatment improved digestate dewaterability.
- To assess whether ageing of digestate altered its dewatering properties, at a range temperatures.
- To assess the effect of treatment by cellulolytic enzymes on digestate dewaterability
- To evaluate the effect of antifoam on biogas production and to estimate any threshold concentration for antifoam inhibition of the digestion process.
- To compare the effectiveness of antifoams used in this study.
- To identify the centrifugation force(s) required for separating the liquid–solid fractions in SBP digestate.
- To compare layer formation in mesophilic and thermophilic digestates at different OLR, and to isolate and identify a non-cellular light fraction in the supernatant, with a view to suggesting causes for differences in dewaterability characteristics.
- To assess whether heating mesophilic digestate to 55 ^{o}C, 60 ^{o}C and 65 ^{o}C improved dewaterability.
- To confirm that the non-cellular light fraction affected digestate dewaterability.
- To determine the nutrient content and other properties of whole digestate and liquid and solid digestate fractions from thermophilic and mesophilic digesters with respect to their potential for utilisation in agriculture.
- To evaluate quantitatively the carbon, energy and nutrient (CEN) footprints of the AD process for SBP.

CHAPTER 2. LITERATURE REVIEWS

2.1. Sugar Beet Pulp

Sugar Beet Pulp (SBP) is a by-product resulting from the processing of sugar beet for sugar production (Figure 2.1). Sugar beet is planted commercially throughout the world, particularly in cooler and temperate climates areas such the European Union, the United States, the Russian Federation and the United Kingdom. In the UK, the production of sugar beet in 2012 was 7.3 million tonnes from 120,000 hectares, 14% lower than that in 2011, due to the high rainfall and lack of sunshine which caused some difficulties and delays in the development and harvesting of the sugar beet crop (DEFRA, 2012). The area and tonnage of sugar beet production in the UK from 1998 to 2012 can be seen in Table 2.1.

Table 2.1. Agricultural area and production of sugar beet in the UK

Year	Area (thousand hectares)	Yield (adjusted tonnes per hectare)	Volume of harvested production (million tonnes)	Value of production (£ million)	Sugar content (%)
1998	181	53.0	10.002	298	17.34
1999	183	58.0	10.584	280	17.16
2000	173	52.5	9.079	252	17.10
2001	177	47.0	8.335	256	17.16
2002	169	56.5	9.557	283	17.38
2003	162	57.3	9.296	283	18.46
2004	154	58.7	9.042	278	17.20
2005	148	58.5	8.687	279	17.40
2006	130	56.6	7.400	178	16.63
2007	125	53.8	6.733	162	17.96
2008	120	63.8	7.641	208	17.65
2009	119	69.9	8.330	241	18.00
2010	118	55.3	6.527	197	16.87
2011	113	75.4	8.504	251	18.44
2012	120	60.7	7.291	227	17.02

Source: (DEFRA, 2005, 2009, 2012)

Sugar beet is mainly used for sugar production, which involves several activities, including cleaning, slicing, filtering, evaporation, crystallisation, and centrifuging (British Sugar, 2010, 2011, 2012). Sugar beet processing in a British Sugar factory is illustrated in more detail in Figure 2.2. It can be seen that SBP is generated from the diffuser or filtering process where the thin strips of beet (cossettes) are mixed with hot water to extract the juice which is further processed via purification and crystallization

to produce sugar, while the remaining fibre or pulp is often used for animal feedstock. In 2011 in the UK, British Sugar processed approximately 7.5 million tonnes of sugar beet, supplied between September and March from over 4,000 growers around the UK, and produced 2.3 million of refined sugar (British Sugar, 2011). A typical factory, such as the British Sugar factory in Wissington, processes 3 million tonnes of sugar beet annually which generates 400,000 tonnes of refined sugar and over 100,000 tonnes of dried SBP (British Sugar, 2012). The large amount of SBP resulting from this process thus represents a major material handling and/or disposal operation.

2.2. Composition of SBP

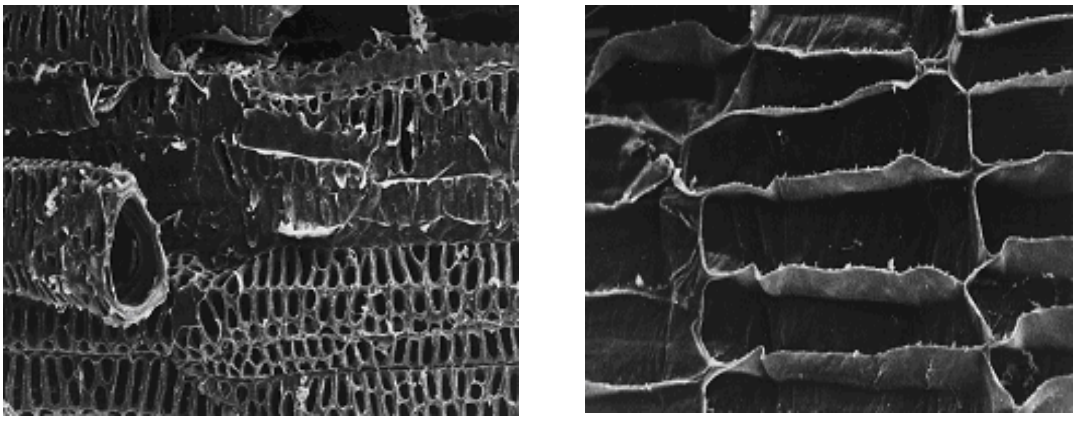
Many studies have been conducted to identify the composition of SBP. McCready (1966) reported that SBP contained 26% cellulose and 25% pectin, while Kelly (1983) found 22% cellulose and 3.2% lignin. Weibel (1989) identified that SBP contains 40% cellulose, 30% arabinogalactan and 30% pectin. Micard et al. (1996) concluded that the main component of SBP was sugars (~74% of dry matter), notably arabinose, glucose, and galacturonic acid, as well as pectins. Spagnuolo et al. (1997) also investigated the composition of SBP and reported that it mainly consisted of cellulose (22-30%), hemicelluloses (24-32%), pectin (24-32%) and lignin (3-4%), respectively. Rouilly et al. (2006) found that the main components of SBP are hemicelluloses, pectins and parietal cellulose, which has similar rheological behaviour to starch; while Šimkovic et al. (2009) reported that the predominant component of the SBP cell wall is arabinose.

According to Dinand et al. (1999), carbohydrates constitute the main part of SBP before and after disencrustation. The initial composition of SBP was 22% cellulose, 32% hemicellulose, 27% pectin, 9% minerals, 7% proteins, 2% lignin and 2% fat. After disencrustation, the composition became 88% cellulose, 7% hemicellulose, 3% pectin and 2% minerals (Dinand et al., 1996). The cellulose from SBP, or parenchyma cell cellulose (PCC), can be traced back to the particular morphology of the sugar beet root where most of the tissue is parenchymal, known as the non-woody structures or the soft parts of plants, as shown in Figure 2.3.

Several studies have investigated the concentration of trace elements (TE) found in sugar beet. According to Draycott and Christenson (2003), TE found in in sugar beet roots include (on a dry matter (DM) basis): B (15 mg kg^{-1}), Cu (1 mg kg^{-1}), Fe (100 mg kg^{-1}), Mn (30 mg kg^{-1}), Mo (5 mg kg^{-1}), and Zn (10 mg kg^{-1}). Another study conducted by Škrbić et al. (2010) found that on a DM basis SBP contained K (2650±369.2 mg kg^{-1}), Na (1025±197.2 mg kg^{-1}), Ca (5220±189.7 mg kg^{-1}), Mg (2400±122.5 mg kg^{-1}), Zn (10.10±0.26 mg kg^{-1}), Fe (156.2 ± 20.2 mg kg^{-1}), and Cu (6.32 ± 0.37 mg kg^{-1}).

Figure 2.1. Sugar Beet Pulp (SBP) resulted from sugar processing in British Sugar Factory

Figure 2.2. Operations in production of sugar from sugar beet in a British Sugar Factory (British Sugar, 2010, 2011, 2012)



(a) (b)

Figure 2.3. Scanning electron micrographs (SEM) of sugar beet sections cut parallel to the central axis of the sugar beet: (a) Section cut in one of the vascular bundles and (b) Section cut through the phloem parenchyma (Dinand et al., 1999)

2.3. Potential Uses of SBP

Several studies have reported potential uses for SBP, in addition to use as an animal feedstock. For example, Dufresne et al. (1997) found that the cellulose microfibril from SBP can be used to make paper as the sole fibre ingredient and improves the strength and stiffness of the paper sheets. In addition, dried SBP has been studied for its potential as biosorbent for Gemazol turquoise blue-G, a copper-pthalocyanine reactive dye commonly used in dyeing of cotton (Aksu and Isoglu, 2006). Cellulose microfibril from SBP can also be used as polymer composites (Leitner et al., 2007; Habibi and Vignon, 2008). According to Chen et al. (2008a) and Liu et al. (2011a), SBP alone or mixed with polylactic acid (PLA) can be used to make the bioplastic strips after extrusion compounding using a twin screw extruder. The resulting thermoplastic (bioplastic) composites retain mechanical properties similar to those of polystyrene and polypropylene, which are the compounds used to make white, spongy food packages. Another study found that SBP can be used to produce oligomeric compounds, such as oligogalacturonides (OGaU), arabinooligosaccharides (AOS), and galactooligosaccharides (GaOS), which are promising candidates for prebiotic properties (Martínez et al., 2009). Prebiotic is a non-digestible food ingredient that stimulates and enhances the growth and/or activity of the gastrointestinal microorganism thus providing benefits on health and well-being (Roberfroind, 2007).

The potential uses of SBP as a feedstock for renewable energy production have also been investigated in several studies, such as for bioethanol (Doran et al., 2000; Hinková and Bubník, 2001; Mahro and Timm, 2007; Zheng et al., 2012), hydrogen (Hussy et al., 2005; Urbaniec and Grabarczyk, 2009; Ozkan et al., 2011) and biogas (Labat et al., 1984; Ghanem et al., 1992; Hutnan et al., 2000; Polematidis et al., 2007; Koppar and Pullammanappallil, 2008). Although SBP has many potential uses, biogas production through the AD process can offer better revenue returns from energy production compared to the animal feed market, while providing a direct means of recycling nutrients to arable land (Parawira et al., 2008).

2.4. Anaerobic Digestion

2.4.1. Review of Anaerobic Digestion

Anaerobic digestion is a biological process that occurs in the absence of oxygen, converting complex organic materials into biogas: a mixture of methane (CH_4) and carbon dioxide (CO_2). Methane is a natural flammable gas that can be utilised to produce heat and electricity, or directly as a vehicle fuel. The digestate produced from the AD process can be used to produce valuable by-products, such as soil conditioner or organic fertiliser.

Anaerobic digestion is often divided into the four stages of hydrolysis, acidogenesis, acetogenesis and methanogenesis (see Figure 2.4). During the hydrolysis stage, the fermentative bacteria hydrolyse complex organic compounds (i.e. cellulose, protein, lipids) into simple soluble compounds such as sugars, amino acids and fatty acids. The fermentative bacteria then ferment the resulting products from the previous stage into volatile fatty acids (VFA) (e.g. propionic, butyric acid, etc.) in the acidogenesis stage. In the next stage, acetogenic bacteria (also known as hydrogen-producing acetogenic bacteria) convert VFA along with ethanol into acetic acid, H_2 and CO_2. This step is commonly known as acetogenesis. The final stage is methanogenesis and involves two groups of microorganisms known as acetoclastic methanogens and hydrogenotrophic methanogens (Hobson and Wheatley, 1993; Wheatley et al., 1997; Ahring, 2003; Gavala et al., 2003; Garcia-Heras, 2003; Khanal, 2008).

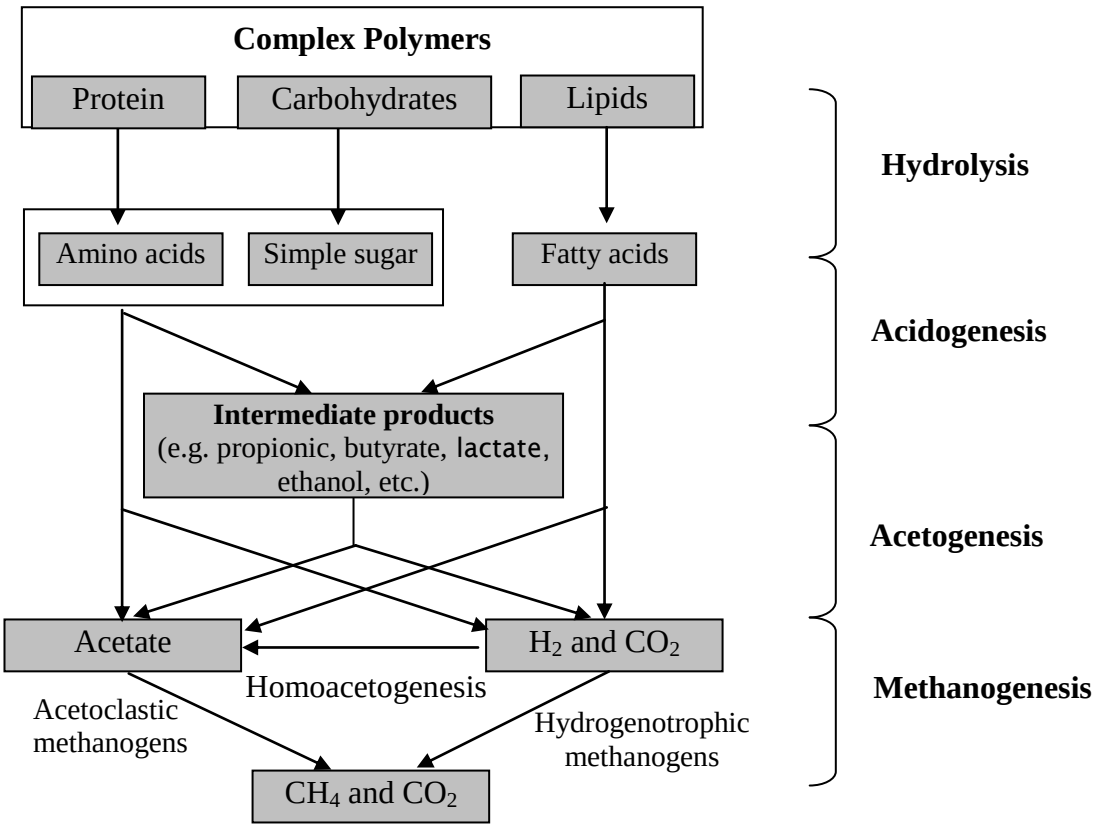


Figure 2.4. Stages of anaerobic digestion (adapted from Hobson and Wheatley, 1993; Wheatley et al., 1997; Ahring, 2003; Gavala et al., 2003; Garcia-Heras, 2003; Khanal, 2008)

2.4.2. Factors Influencing Anaerobic Digestion

Many factors can influence the stability of the anaerobic digestion process, including those outlined below.

2.4.2.1. Temperature

Angelidaki et al. (2003) stated that temperature in the AD process not only influenced microbial growth, but also physical parameters such as viscosity, surface tension and mass transfer properties. Other studies have also observed that temperature has a significant effect on the AD process, including accumulation of VFAs, biogas and methane production, and methanogenic activity (Lyberatos and Skiadas, 1999; Sánchez et al., 2001; Sanders et al., 2003; Appels et al., 2008). Methanogens are particularly affected by sharp and/or frequent fluctuations in temperature, and sustaining a stable temperature in an AD system is therefore critical (Appels et al., 2008; Ward et al., 2008). AD is normally carried out in one of two temperature ranges: mesophilic (30-45 ^{o}C) and thermophilic (50-60 ^{o}C) (Angelidaki et al., 2003; Speece, 2008; Weiland,

2010). Operating AD at higher temperature (thermophilic digestion) may increase the rate of processing in line with a general increase in biochemical reaction rates (Mackie and Bryant, 1995; de la Rubia et al., 2002, 2006; Appels et al., 2008). For certain types of substrate, however, such as manure and foodwaste, operation at mesophilic temperature is favourable because it gives more stable digestion and with no sign of inhibition from VFA, as indicated by an increase in biogas yield (Angelidaki and Ahring, 1994; Hansen et al., 1998; Banks et al., 2008)

2.4.2.2. pH

The optimum pH for the AD process is between pH 6.7 and 7.6, which is favourable for methane-producing archaea (Parkin and Owen, 1986; Speece, 1996, 2008). Methanogens grow very slowly at pH lower than 6.6 (Angelidaki et al., 2003). Low pH can be caused by an imbalance of conditions in the digester due to domination of the acid-producing to the acid-consuming bacteria (Speece, 2008). Therefore, in an AD system, pH is usually maintained between methanogenic limits to inhibit the predominance of acid-forming bacteria and avoid VFA accumulation (Rajeshwari et al., 2000; Khalid et al., 2011). Veeken et al. (2000) found that pH influenced the hydrolysis rate in AD of organic solid waste, and an accumulation of VFA may result in a decrease of pH and/or vice versa, but this depended on the composition of the waste or substrates. Degradation of protein, for example, may increase buffer capacity through ammonia production, which may increase the pH.

2.4.2.3. Substrate Composition and Concentration

The effect of substrate composition and concentration in AD processes has been the subject of several studies. Angelidaki and Sanders (2004) considered the effect substrate composition in terms of its carbohydrate, protein and lipid content with respect to anaerobic biodegradation and methane production. In addition, the composition of energy crops and agro-waste substrates, in terms of the proportion of cellulose, hemicellulose, crude fat and crude protein, influences methane production since some of these components are readily degradable (e.g. carbohydrates, lipids and proteins) (Amon et al., 2007a, 2007b); while others are not (e.g. cellulose, lignin) (Hobson, 1983). Wang et al. (2010) also noted that the composition and characteristics of the fibre component affect the anaerobic digestibility of lignocellulosic substrates. Sánchez et al.

(2001) observed that substrate concentration affected AD performance, as increasing the initial concentration of organic matter caused a reduction of the COD removal rate. This was supported by Mahnert and Linke (2009), who mentioned that the biogas yield depends on the concentration of VS in the input feedstock.

2.4.2.4. Toxicity

Methanogens are considered to be the most sensitive microorganisms in the anaerobic consortium. One of the most common sources of toxicity in anaerobic digestion is due to accumulation of free ammonia in the digesters, where un-ionized ammonia (NH_3) is highly toxic compared with the ionized ammonia (NH_4^+) (Angelidaki and Ahring, 1993; Kayhanian, 1994; Sung and Liu, 2003; Speece, 2008; Chen et al., 2008b). Free ammonia inhibition results in an accumulation of VFA and a decrease in pH (Angelidaki et al., 2003; Chen et al., 2008b). Critical concentrations of free ammonia where inhibition of the digestion process starts to occur are reported by (Yenigün and Demirel, 2013). For example, the critical free ammonia concentration for mesophilic AD of sewage sludge and cattle manure was 400 mg l^{-1} and 700 mg l^{-1}, while for thermophilic AD of cattle manure the value was 900 mg l^{-1}. This study highlighted that the free ammonia threshold for inhibition in an AD system may vary depending on the type of substrate and the operating temperature. It was further stated that excessive free ammonia concentrations above the critical values cause process instability, indicated by a decrease in both biogas and methane yields, and leads to digestion failure.

The presence of heavy metals (Hickey et al., 1989; Chen et al., 2008b); surfactants, antibiotics, sulphide, detergents, etc. (Chen et al., 2008b) above an acceptable limit can also inhibit the AD process.

2.4.2.5. Organic Loading Rate (OLR)

OLR is an important parameter due to its effect on the performance of AD. Overloading or shock loading in feeding may cause instability in AD due to an accumulation of VFA and finally to a pH breakdown (Wheatley et al., 1997; Lyberatos and Skiadas, 1999); or biomass washout may lead to process failure (Rajeshwari et al., 2000), as indicated by a reduction in biogas and methane yields (Wheatley et al., 1997; Gómez et al., 2006). Demirer and Chen (2004) stated that in AD of cattle manure, a high OLR of 20-30 kg

VS m^{-3} day^{-1} contributed to a decrease in specific methane production (0.066 m^3 kg^{-1} VS_{added} day^{-1}) and in pH value (< pH 6), possibly through wash-out of acidifiers due to low retention times. Rincón et al. (2008) also found that in AD of olive mill residues, an increase in OLR resulted in an increase in effluent VFA and COD concentration. At an OLR of 11 g COD l^{-1} day^{-1}, the VFA reached ~6.1 g l^{-1} following a sharp decline in pH to 5.3, indicating that the digestion process had failed.

2.4.2.6. Retention Time

Zhang and Noike (1994) observed that retention time affects both the microbial population in the AD process (e.g. methanogens, homoacetogens and sulphate reducing-bacteria) and the composition of fermentation products. Sanders et al. (2003) concluded that hydraulic retention time (HRT) and solids retention time (SRT) should be monitored since these parameters influence the AD process, as they indicate the time available for organic matter to degrade. A decrease in the SRT is linked to a decrease in the extent of the reactions, and Appels et al. (2008) stated that SRT is therefore an essential parameter to be considered in designing and operating an AD system. They further added that there is a relationship between biogas production and retention time (SRT) in a CSTR: stable digestion is difficult to achieve at SRT shorter than 10 days, but at SRT > 10 days the breakdown of compounds starts to improve as indicated by more stable biogas production. Furthermore, Sánchez et al. (2005) reported that an increase in SRT may allow better adaptation of microorganisms to the substrate, thus supporting methanogenesis and improving process performance as slower-growing organisms will not be washed out. Increasing SRT can be achieved through increasing the HRT by using greater reactor volume, reducing the influent flow or recycling the sludge (biomass).

2.4.2.7. Mixing intensity

Mixing is necessary in AD to ensure that microorganisms and substrate are in contact, and to enhance the ability of microorganisms to consume the nutrient in the medium, as well as reducing the dead zone of scum in digester (Hobson and Wheatley, 1993; Speece, 2008; Chen et al., 2008b). For example, in AD of primary sludge (PS) or a mixture of PS and the fruit and vegetable fraction of the municipal solid wastes, the absence of mixing resulted in a reduction in specific biogas production (0.3 and 0.5 l g^{-1}

VS) due to reduction of the contact between the substrate and the microorganisms, while a low mixing rate (80 rpm) gave high specific biogas productions of 0.5 and 0.6 l g^{-1} VS, respectively (Gómez et al., 2006). Excessive mixing can reduce biogas production (Gómez et al., 2006; Ward et al., 2008), causing a decrease in the oxidation rate of fatty acids and leading to instability in digesters (McMahon et al., 2001).

2.4.2.8. Nutrients

Macro-nutrients

In the AD process, the macro-nutrients required in highest concentration are nitrogen and phosphorus, where the phosphorus requirement for bacterial growth is about 14-20% of the nitrogen requirement (Parkin and Owen, 1986). A deficiency in macro-nutrients may cause inadequate microbiological activity, indicated by a reduction in biogas production and yield (Vintiloiu et al., 2012). Therefore, nutrients must be present in sufficient quantities to ensure an efficient digestion. Macro-nutrients for the growth and survival of microorganisms in anaerobic digestion are generally required in the ratio of 600:15:5:3 for C:N:P:S (Fricke et al., 2007) or 600:15:5:1 for C:N:P:S (Weiland, 2010). Some authors have reported an improvement in methane production from addition of macro-nutrients to specific substrates, such as cellulose, rice straw, maize/sugar beet silage, energy crops (i.e. maize, sugar beet and triticale), etc. (Khan et al., 1979; Lei et al., 2010; Nges et al., 2012; Nges and Björnsson, 2012).

Stability of the digestion process is also influenced by the C/N ratio. A lower C/N ratio can result in inhibition due to an excess of nitrogen; while a very high C/N ratio can lead to nitrogen deficiency for biomass synthesis, or trigger the production of EPS (Miqueleto et al., 2010; Yenigün and Demirel, 2013). In AD of manure, a C/N ratio in the range of 25-32 has been reported to improve methane yield (Hills and Roberts, 1981; Angelidaki et al., 2003); while for a mixture of manure and various agricultural waste, higher methane production was achieved at C/N ratio of 25 or more (Hills, 1979). For AD of dairy manure, chicken manure and wheat straw C/N ratios of 25, 30, and 35 provided low and stable ammonia and free ammonia concentrations (Wang et al., 2012). The optimal C/N ratio for AD of organic waste (e.g. fruit and vegetable waste, etc.) has been reported to be in the range of 25-30 according to Hartmann and Ahring (2006) or 20–35 (Khalid et al., 2011). The composition of the organic fractions

in MSW (such as food waste, yard waste, paper, newspaper, etc.) affects both the nutrient values and C/N ratio (Kayhanian, 1995; Plaza et al., 1996). From these findings, although the C/N ratio varies according to the substrate used, it can be concluded that a C/N ratio of 20-35 is in general a good range for stable AD processes.

Micro-nutrients

Micro-nutrients or trace elements (TE) are known to be important to anaerobic microorganisms and may improve specific gas production or process stability. According to Oleszkiewicz and Sharma (1990), TE requirements depend not only on their role in certain enzymes and as cofactors in the microorganisms' metabolic pathway, but also on concentration, type of metal and speciation. In their review of TE in anaerobic granular sludge reactors Zandvoort et al. (2006) noted that enzymes used in methanogenesis required certain TE at various dosages. A review by Jiang (2006) identified the different roles and functions of TE in the enzymes of several microorganisms involved in the anaerobic digestion process, such as methanogenic archaea, homoacetogenic bacteria and sulphate-reducing bacteria (SRB). TE and their functions were also reviewed by Schattauer et al. (2011), who identified the recommended concentrations for anaerobic digestion and those that might prevent or inhibit the microbial metabolic process.

Some TE (e.g. Co, Mo, Ni, Se, W, etc.) are part of the essential enzymes involved in anaerobic reactions (Oleszkiewicz and Sharma, 1990; Hinken et al., 2008; Worm et al., 2011; Facchin et al., 2013; Brulé et al., 2013). Zhang et al. (2003) found that Fe, Cu, Ni and Zn are the essential elements required by methanogens to achieve high methane productivity.

Florencio et al. (1993) studied the effect of the addition of cobalt with and without other TE on anaerobic digestion of methanol in upflow anaerobic sludge blanket reactors (UASB). The results showed that the addition of cobalt alone dramatically stimulated methanogenic activity to the same extent as the full TE mixture. At an OLR of 8 g COD l^{-1} day^{-1}, 87% of the COD was converted to methane in the digester with cobalt supplementation and the methane productivity was almost three times that without cobalt addition. Zhang et al. (2011) suggested that TE supplementation enhanced the performance of the anaerobic co-digestion of food waste with piggery wastewater

resulting in a high methane yield of 0.396 m^3 kg^{-1} VS, 75.6% VS destruction, and no VFA accumulation.

A review by Demirel and Scherer (2011) concluded that in the anaerobic digestion of energy crops it is important to pay attention to the requirement for trace elements, as this can help to improve the overall process performance. Pobeheim et al. (2010) showed that methane yields could be increased by up to 30% when supplementing a synthetic model substrate for maize silage with cobalt, nickel and molybdenum. When used as single elements both cobalt and nickel showed enhancement of methane production, by up to 15%, whereas molybdenum alone showed no effect. Jarvis et al. (1997) demonstrated that the addition of cobalt at concentrations of 0.2 and 2.2 mg $l^{-1}_{digestate}$ could improve anaerobic digestion of a grass-clover silage-feed. No benefit was shown from the addition of molybdenum or nickel, and when increasing the nickel concentration to 23 mg l^{-1} inhibited methane production. Banks et al. (2012) studied TE supplementation in AD of food waste at elevated ammonia concentration. Four different TE mixtures were tested: (1) Se and Mo; (2) Se, Mo, Co and W; (3) Se, Mo, So, W, Fe and Ni; (4) Se, Mo, Co, W, Fe, Ni, Zn, Cu, Mn, Al, and B. The results indicated that addition of TE improved digestion performance and the operation stability. Critical Se and Co concentrations were found to be 0.16 and 0.22 mg kg^{-1} WW, which allowed the OLR to be raised to 5 g VS l^{-1} day^{-1} with specific and volumetric biogas productions of 0.75 m^3 kg^{-1} VS and 3.75 STP m^3 m^{-3} day^{-1}.

Although many studies have considered the AD of SBP, little research or scientific information is available regarding the effect of TE supplementation on the performance of AD of SBP. Therefore, in this study, to gain fundamental information, laboratory-scale digesters were fed semi-continuously on SBP on a daily basis and the treatment performance including biogas/methane production and the stability of the operation was evaluated.

2.4.3. Performance of AD in a Single- and Two-stage System

Anaerobic digestion can be carried out using single-stage or two-stage systems. Single-stage AD in continuously stirred tank reactors (CSTR) is a commonly used technology for waste fractions with high moisture content (e.g. manure, sewage sludge, food waste

(FW), waste activated sludge (WAS), SB silage, etc.) (Angelidaki and Ahring, 1994; Gunaseelan, 1997; Hansen et al., 1998; Heo et al., 2004; Demirel and Scherer, 2008). In a single-stage AD, all phases of the process (hydrolysis, acidogenesis, acetogenesis, and methanogenesis) occur simultaneously in one reactor; optimum reaction conditions for the overall process may thus be difficult to achieve due to differences in the environmental requirements of each stage, resulting in a lower degradation rate and the need for a higher retention time (Ghosh, 1987; Forster-Carneiro et al., 2008; Ward et al., 2008). However, the biological performance of this system can be enhanced if the reactor design and the operational conditions are carefully planned and selected (Weiland, 1993; Forster-Carneiro et al., 2008).

The pioneering work on two-stage AD to improve the AD process was carried out by Pohland and Ghosh (1971). The results showed that two-stage AD of sewage sludge could provide a significant improvement in sludge treatment efficiencies, enhance the conversion process of organic materials in the acidification stage, prevent possible inhibitors and ensure uniformity of feedstock for the methanogens. Gosh and Klas (1977) also found that two-phase AD of wastewater sludges improved methane yields and solid destructions. Gunaseelan (1997) noted that two-stage AD basically involves using two digesters with different retention times optimised for acidification (1st digester) and methanogenesis (2nd digester), thus the digestion time may be shorter than the single-stage digester. A comparison of single- and two-stage AD process is given in Table 2.2.

Table 2.2. Comparison of single- and two-stage processes

Process Operation	Single-Stage	Two-Stage
Operational reliability	in the same range	
Technical equipment	relatively simple	very complex
Process control	compromise solution	optimal
Risk of process instability	high	minimal
Retention time	long	short
Degradation rate	reduced	increased

Source: Rilling (2005)

Several studies have found that two-phase AD provides a better digestion performance than single-phase (Liu, 1998; Ghosh et al., 2000; Nielsen et al., 2004; Dearman et al., 2006; Lim et al., 2013; Shahriari et al., 2013). The two-phase system requires more

sophisticated operation and control, however, and may have higher capital costs (De Baere, 2000; von Sachs et al., 2003; Bouallagui et al., 2005; Rapport et al., 2008; Ward et al., 2008); thus it is seldom applied at full-scale. For many materials a single-stage system is preferred as it has many positive aspects, such as reduced complexity and lower capital costs combined with effective degradation of organic materials at typical retention times. Single stage systems are generally cheaper to build and simple to operate and control (Mtz-Viturtia et al., 1995; Speece et al., 1997; Ong et al., 2000; Bouallagui et al., 2005; Forster-Carneiro et al., 2008; Park et al., 2008).

From these practical perspectives, several approaches or strategies have been tested to improve the performance and efficiency of single-stage AD. For example, Ong et al. (2000) studied biogas production from cattle slurry using a single-stage system and found that strategies that enhanced the process efficiency included digesting without mixing and withdrawing the digestate from the middle layer rather than from the bottom or top layers. Jiang et al. (2012) studied single-stage CSTR of food waste digestion and found supplementing with TE, adding with yeast extract or co-digesting with other substrates (i.e. card packaging and cattle slurry) resulted in a more stable digestion. Wan et al. (2013) found that a single-stage AD system effectively co-digested a mixture of food waste, wastepaper, and non-biodegradable plastic at a ratio 2:1:1, resulted in relatively high biogas and methane production of 0.592 and 0.370 m^3 kg^{-1} VS. More generally, kinetic studies of single-stage CSTR systems can be performed to determine the optimal operational parameters to achieve high biogas production and stable and/or efficient operation.

2.5. Biochemical Methane Potential Test

The Biochemical Methane Potential (BMP) test is used to determine the biodegradability of a substrate under anaerobic conditions by monitoring the cumulative methane production during the test period (Owen et al., 1979; Jensen et al., 2011). This measurement can provide important information including the anaerobic digestibility and potential biogas (methane) production from substrates which is useful for evaluating, designing, and optimising the AD process (Lim and Fox, 2013). Besides measuring the conversion of organic matter to methane, the BMP test can also be used to determine the residual organic material amenable to further anaerobic treatment, the

non-biodegradable fraction remaining after treatment, and the potential efficiency of anaerobic digestion for a particular substrate (Speece, 1996). Angelidaki et al. (2009) developed guidelines to standardise the techniques and units used in BMP tests. Important aspects to be considered when conducting a BMP test include: the substrate and its characterisation, substrate particle size, inoculum and its activity, nutrients/micronutrient/vitamins and mixing. These guidelines also present information on a typical basic medium used in the test.

Cho et al. (1995) studied the effect of the initial organic loading in the BMP of Korean mixed food waste (MFW). The BMP test was performed in duplicate for 28 days on waste samples, blank control, and positive controls of α-cellulose. The initial loadings used in this study were 2, 4, 10 and 50 kg VS m^{-3}. The results suggested that the rate of methane production differs significantly with initial loading, being higher and faster at a low initial loading (2 kg VS m^{-3}) compared to higher loadings, as indicated by a short lag time before the onset of gas production and achievement of the maximum cumulative methane yield of 0.472 m^3 kg^{-1} VS within a 5-day period. For initial loadings of 4 and 10 kg VS m^{-3} the initial rate of methane production was quite slow compared to the net gas production rate at loading 2 kg VS m^{-3} characterized by a lag phase to day 2 and 5 respectively. Initial loading 50 kg VS m^{-3} required much longer (50 days) before net methane production became positive and showed a failure in the digestion process due to excessive acidification as indicated by a decline in pH value to ~3.

Brulé et al. (2013) performed a BMP test which focused on the effect of TE on methane production from energy crops (green cuttings and grass silage). Three different sources of TE (a solid powder constituting clay particles (A), a solid powder containing trace metal (B) and a liquid solution of trace metals (C) that are commonly applied in full-scale biogas plants across Germany were used in this study. Fresh substrate was fed at an initial loading of 0.4 g VS l^{-1} to an inoculum obtained from a laboratory-scale digester fed with a mixture of digested manure, maize silage, cereals, rapeseed oil and soybean extract at OLR of 0.5 kg VS m^{-3} day^{-1}. The test was carried out with undiluted and diluted inoculum (3-fold and 10-fold dilution) and included samples with TE supplementation and controls without TE addition). TE was added at doses of: 240 mg

of TE-A, 10 mg of TE-B and 1.6 µl of TE-C. Four replicates per variant were employed. The results suggested that for undiluted inoculum, none of the TE supplementation methods had any significant effect on methane production, giving approximately the same values at ~0.30 m^3 kg^{-1} VS (grass silage) and ~0.35 m^3 kg^{-1} VS (green cutting). With 10-fold diluted inoculum, the addition of TE help to maintain net methane production to the same value as that produced in undiluted inoculum, while samples without TE addition gave values of ~0.25 m^3 kg^{-1} VS for both substrates. This study confirmed that TE is an important factor in enhancing microbial activity. For instance, in the diluted inoculum, despite the smaller amount of microbial biomass, the addition of TE helped to improve the growth or the metabolism of the microbial consortia as indicated by a stable or increased methane production.

Other studies have investigated factors that may affect biogas (or methane) production in the BMP test. For instance, three different factors (inoculum source, inoculum to substrate ratio $R_{I/S}$, particle size) were evaluated by Chynoweth et al. (1993) who found that the conversion rate for biomass and waste feedstock to methane was higher at a $R_{I/S}$ of 2:1 on VS basis than at $R_{I/S}$ of 1:1 or 1.5:1 and was not influenced by the particle size (1-8 mm) or type of inoculum (i.e. rumen or digestate). Neves et al. (2004) measured the BMP of kitchen waste using $R_{I/S}$ of 1:1, 1:1.35, 1:2.3 and 5:1 on a VS basis. Two different inoculums were used: suspended and granular sludges. The BMP test was performed in two consecutive runs with initial ratio of alkalinity/COD in inoculum set at 37 mg $NaHCO_3$ g^{-1} COD (run 1) and 2 mg $NaHCO_3$ g^{-1} COD (run 2). The findings confirmed that $R_{I/S}$ is an important factor in conducting a BMP test as it affects the methane production rates and the biodegradability of substrates. At $R_{I/S}$ of 5:1, methane production and biodegradability were much higher than at the other $R_{I/S}$ used, with values of ~0.30 - 0.35 m^3 CH_4 (STP) kg^{-1} COD (both runs). On the other hand, at $R_{I/S}$ of 1:2.3, the methane production and biodegradability reduced significantly, probably due to the high ratio of substrate to inoculum. This led to an accumulation of VFA indicated by a decline in pH to below 6.5 caused an inhibition to the digestion process. Other research examined the effect of inoculum to substrate ratios ($R_{I/S}$) in the range of 3:1, 2:1, 1.5:1 and 1:1 on a VS basis in maize digestion, which gave maximum specific methane productions of 196, 211, 210, and 233 l CH_4 (STP) kg^{-1} VSS day^{-1}, respectively. At $R_{I/S}$ 1:1 there was an accumulation of VFA (~6000 g COD

l^{-1}) and a high IA/PA ratio (~0.4) at 72 - 96 hours of the digestion compared to other $R_{I/S}$ (< 3000 g COD l^{-1}) with IA/PA ratio of < 0.2. All tested $R_{I/S}$ were able to recover, however, as indicated by a reduction in VFA concentration to < 24 mg l^{-1} at the end of the test (Raposo et al., 2006).

Kryvoruchko et al. (2009) carried out a BMP study on SBP silage using 1 l eudiometer batch digesters at 37.5^{o}C and $R_{I/S}$ of 3:1 on a DM basis operated for 28 - 38 days. Each eudiometer, consisting of six digesters connected to equilibrium vessels, was placed on magnetic stirrers in a temperature-controlled water bath. Biogas was collected in gas-collection cylinders connected to the digesters and gas composition was analysed using a nondispersive infrared (NDIR) analyser. The SBP had a specific methane yield of 0.43 m^3 kg^{-1} VS, was higher than typical values for other organic solid wastes. For example, BMP tests of fruit and vegetable waste (FVSW) samples showed that most of the FVSW had methane yields between 0.3 and 0.7 m^3 kg^{-1} VS (Gunaseelan, 2004). Owen and Chynoweth (1993) tested the BMP of MSW and several of its components and showed methane yields of 0.20 m^3 kg^{-1} VS for MSW and 0.21 m kg^{-1} VS, for yard waste, while paper waste varied within a range of 0.08 – 0.37 m^3 kg^{-1} VS for different classes of paper.

A BMP test was conducted over a 28-day period on pressure treated and untreated SBP from the same source as that used in the current study (Banks, 2009). The test was carried out in triplicate in static batch reactors at 35 ^{o}C with initial loadings of 7-8 g VS l^{-1} and $R_{I/S}$ of 3.1 - 3.3:1 on a VS basis. The treated material was subjected to CO_2 pressurisation to 650 kPa for a period of 3 hours before the start of the BMP test. The biogas was collected under acidified saline solution in Perspex cylinders and measured its composition using gas chromatography (GP-3800, Varian, USA). The biogas yields were 0.645 and 0.664 m^3 kg^{-1} VS and the methane potentials 0.350 and 0.364 m^3 kg^{-1} VS for untreated and treated SBP.

Furthermore, the above findings also indicate that a $R_{I/S}$ of 2:1 or more is best for BMP test, as according to Tong et al. (1990) the increase of $R_{I/S}$ is probably needed for certain types of substrates to achieve a better estimation of the maximum methane production. With respect to BMPs the conclusion from the study above seems to be that addition of

TE may be helpful when using dilute inoculum but not necessarily essential with inoculum from a typical source.

2.6. Previous Work on Anaerobic Digestion of SBP

2.6.1. Single-Stage Anaerobic Digestion of SBP

2.6.1.1. Mesophilic Anaerobic Digestion of SBP

Brooks et al. (2008) performed laboratory and pilot-scale studies on single-stage anaerobic digestion of SBP. The feedstock was crushed in the laboratory-scale study, but not in the pilot scale. OLR in the laboratory-scale experiments varied from 1.4 to 10.3 kg COD m^{-3} day^{-1}, while at pilot scale it ranged from 0.13 to 12.8 kg COD m^{-3} day^{-1}. The results showed that for stable biogas production OLR should be maintained at less than 8.5 kg COD m^{-3} day^{-1} (laboratory-scale) and 10 kg COD m^{-3} day^{-1} (pilot-scale). The average specific biogas yield even at these high OLR was 610 Nl kg^{-1} VS with CH_4 concentration between 50-53%, equating to a specific methane yield of ~314 Nl kg^{-1} VS; VS degradation was in the range of 70-81%, and COD degradation was 72-77%. These results indicated that the single-stage process can provide an efficient and effective process with regards to cost and simplicity.

Studies have also been carried out on AD of SBP after different feedstock pre-treatments. Labat et al. (1984) studied anaerobic digestion of SBP with enzymatic hydrolysis pre-treatment by *Trichoderma harzianum* at OLR in the range of 0.2 – 1 kg VS m^{-3} day^{-1} over an 18-week period. In this work, the optimum performance was achieved at an average OLR of 1 kg VS m^{-3} day^{-1} resulting in biogas production of 1 m^3 m^{-3} day^{-1} and a biogas yield of 0.74 m^3 kg^{-1} VS at a HRT of 17 days, achieved by dilution of the feedstock. Ghanem et al. (1992) carried out laboratory-scale AD of untreated and alkali-treated SBP under mesophilic conditions (37 ^oC) at 15-day HRT over 3 HRT, using duplicate 100 ml wide-mouth flasks with a 64-ml working volume. The digesters were fed on a daily basis at OLR 5 g WW l^{-1} day^{-1}. In the first HRT biogas yields from both samples were almost the same at 0.863 l l^{-1} day^{-1} with the same methane concentration for alkali-treated SBP (50.6%) and untreated SBP (50.9%), giving methane yields at the values of 0.436 l l^{-1} day^{-1} and 0.437 l l^{-1} day^{-1}, respectively. After 3 HRT, however, the biogas yield for both samples decreased continually to 0.209 l l^{-1} day^{-1} (treated SBP) and 0.259 l l^{-1} day^{-1} (untreated SBP). Although biogas yields for

both samples showed the same trend, the methane concentration for treated SBP remained stable (50.97%) whereas for untreated SBP it decreased to 39.40%, resulting in methane yields of 0.107 l^{-1} day^{-1} and 0.102 l^{-1} day^{-1}.

2.6.1.2. Thermophilic Single-stage Anaerobic Digestion of SBP

Polematidis et al. (2007) studied anaerobic digestion of SBP in a batch system under thermophilic conditions (55 ^{o}C) using 5-litre digesters with a 4-litre working volume, repeating the test three consecutive times. They reported that more than 90% of dry matter of the spent pulp was degraded. The cumulative methane yield was 350, 350 and 374 l STP kg^{-1} VS for 10 days digestion with daily rates peaking at 1.75, 4.5 and 2.5 l l^{-1} day^{-1} for run 1, 2 and 3, respectively. A study by Koppar and Pullammanappallil (2008) used a single-stage leach-bed process under thermophilic conditions in batch mode. Three digesters (5 litre) with a working volume of 4 litre, made of a modified Pyrex glass jars, were used. The inoculum was taken from a thermophilic digester that had been or fed on sugar beet tailings (e.g. leaves and small beets removed during processing of sugar beet) for over a year. To prevent compaction of the solids, 2 kg of a bulking material (lava rock) was added to the digester, and 10 g l^{-1} of sodium bicarbonate was also added to buffer against pH changes. The digesters were fed with 0.450 kg WW of spent SBP. The average final methane yield was 0.336 m^3 CH_4 at STP kg^{-1} VS with a maximum methane production rate of 0.087 m^3 CH_4 at STP kg^{-1} VS day^{-1} and 96% VS reduction. The authors stated that this system improved the digestion process performance, was simple to operate (single-stage system) and did not need feedstock pre-treatment or mixing.

Other than these studies there appears to have been little or no work carried out on single-stage AD of SBP in thermophilic conditions, and no extended studies of thermophilic operation in conventional CSTR systems have been reported in the literature.

2.6.2. Two-Stage Anaerobic Digestion of SBP

Stoppok and Buchholz (1985) conducted continuous two-stage anaerobic digestion of SBP with OLR of 5, 10 and 15 g DM l^{-1} day^{-1} and a total HRT of 2.4, 3.6 and 7 days. The results indicated that 90% of COD from the SBP was successfully converted into

methane with biogas yields in the range 591 to 670 l kg^{-1} DM and specific methane yields of 325 - 369 l kg^{-1} DM.

Laboratory-scale two-stage semi-continuous anaerobic digestion of SBP under mesophilic conditions (35 ^{o}C) conducted by Hutnan et al. (2000) showed very good digestion performance with a specific methane production of 0.36 m^3 kg^{-1} VS and methane content of 71.9%. Hutnan et al. (2001) carried out two-stage mesophilic anaerobic digestion of SBP at a pilot scale. The feedstock was diluted with water at a ratio of 0.8:10 (SBP: water) and Na_2CO_3 was added to adjust the pH to 4.0 - 4.4 before loading into an acidogenic reactor. Na_2CO_3 was also added to the feedstock in the methanogenic reactor to maintain pH 7. The maximum OLR for the acidogenic reactor was 20 kg COD m^{-3} day^{-1} with 4 days HRT and the DM concentration of the feedstock (acidified SBP) was at maximum 6-7%; while for the methanogenic reactor, the maximum OLR was 21 kg COD m^{-3} day^{-1} with 3.9 days HRT. Specific biogas production was 0.391 m^3 kg^{-1} VS, in reasonably good agreement with the values found by Hutnan et al. (2000). Brooks et al. (2008) also performed two-stage anaerobic digestion of SBP. The SRT was set to 5 days at an OLR of 14 kg COD m^{-3} day^{-1} in the pre-acidification reactor and 20 days at an OLR of 3 kg COD m^{-3} day^{-1} in the methanogenic reactor. Total biogas production was 520 Nl kg^{-1} VS with a CH_4 concentration of 60% and a VS and COD degradation of approximately 84% and 74%, respectively.

Table 2.3 summarises the process parameters for anaerobic digestion of SBP from several studies. These confirm that SBP is an excellent substrate for AD with a specific methane yield similar to that for many energy crops (Tong et al., 1990; Chynoweth et al., 1993, 2001; Amon et al., 2007a; Brulé et al., 2013) or complex organic substrates (i.e. dairy manure, cheese whey, used vegetable oil, corn silage, etc.) (Labatut et al., 2011); and that it was concluded it is not worthwhile to adopt the more complex two-phase operation, whilst retention time are longer and overall OLR not improved.

Table 2.3. Process parameters from studies on single- and two- stage anaerobic digestion of sugar beet pulp

Authors	Operation	T (^oC)	Loading rate		HRT (days)	Biogas Production	Methane Yield	CH_4
			Acidogenesis	Methanogenesis				
Single-stage								
Labat et al. (1984)*	Continuous	35		1 kg VS m^{-3}	17	0.74 m^3 kg^{-1} VS	0.429 m^3 kg^{-1} VS	58%
Weiland (1993)	Semi-continuous	35		8 kg COD m^{-3} day^{-1} (6.18 kg VS m^{-3} day^{-1})	10	0.365 m^3 kg^{-1} COD (0.271m^3 kg^{-1} VS)	0.21 m^3 kg^{-1} COD (0.16 m^3 kg^{-1} VS)	59%
Polematidis et al. (2007)	Batch	55		225 kg WW m^{-3} (0.45 kg WW)	5-10	-	0.350-0.374 m^3 kg^{-1} VS	-
Koppar and Pullammanappallil (2008)	Batch, leach-bed	55		4 kg COD m^{-3} day^{-1} (3.09 kg VS m^{-3} day^{-1})	7	-	0.336 m^3 (STP) kg^{-1} VS	50% (2 day) 95% (8 day)
Brooks et al. (2008)	Semi-continuous	37		1.4 -10.3 kg COD m^{-3} day^{-1}/ 1.08 – 7.95 kg VS m^{-3} day^{-1} (laboratory-scale)	25	0.530 m^3 kg^{-1} COD (0.409 m^3 kg^{-1} VS)		50-53%
				0.13-12.8 kg COD m^{-3} day^{-1}/ 0.1 – 9.88 kg VS m^{-3} day^{-1} (pilot scale)	22	0.510 m^3 kg^{-1} COD (0.394 m^3 kg^{-1} VS)		
Two-stage								
Stoppok and Buchholz (1985)**	Continuous	35	5 – 15VS kg m^{-3} day^{-1} (3.86-11.58 kg COD m^{-3} day^{-1})	5 - 15 kg VS m^{-3} day^{-1} (3.86-11.58 kg COD m^{-3} day^{-1})	2-8	0.591 – 0.670 m^3 kg^{-1}	-	70%
Weiland (1993)	Semi-continuous	35	10 kg COD m^{-3} day^{-1} (7.72 kg VS m^{-3} day^{-1})	6 kg COD m^{-3} day^{-1} (4.63 kg VS m^{-3} day^{-1})	13	-	0.230 m^3 kg^{-1} COD (0.178 m^3 kg^{-1} VS)	61%
Hutnan et al. (2000)	Semi-continuous	35	10.8-21.6 kg m^{-3} day^{-1} (8.34-16.68 kg VS m^{-3} day^{-1})	3.24-9.71 kg m^{-3} day^{-1} (2.5-7.5 kg VS m^{-3} day^{-1})	13-17	0.504 m^3 kg^{-1} VS	0.380 m^3 kg^{-1} COD (0.216 m^3 kg^{-1} VS)	71.9%
Hutnan et al. (2001)***	Semi-continuous	35	20 kg COD m^{-3} day^{-1} (15.44 kg VS m^{-3} day^{-1})	10-12 kg COD m^{-3} day^{-1} (7.72-9.27 kg VS m^{-3} day^{-1})	13	0.4 – 0.5 m^3 kg^{-1} VS	0.235 m^3 kg^{-1} VS	60-70%
Brooks et al. (2008)	Semi-continuous	37	14 kg COD m^{-3} day^{-1} (10.81 kg VS m^{-3} day^{-1})	3 kg COD m^{-3} day^{-1} (2.32 kg VS m^{-3} day^{-1})	-	0.520 m^3 kg^{-1} VS	0.312 m^3 kg^{-1} VS	60%

Note: :* enzymatic hydrolysis applied as a pre-treatment before digestion, ** all values are reported for the methane reactor; *** pilot plant study; T=Temperature; assume that 1 kg VS of dry SBP equal to 1.295 kg COD (Hutnan et al., 2001)

2.7. Potential Advantages of Thermophilic Anaerobic Digestion

The main potential advantage of thermophilic AD is an increase in specific methane production due to higher conversion of organic matter present in the feedstock. Chi et al. (2010) studied AD of thickened waste activated sludge (TWAS) at both mesophilic and thermophilic temperatures, and found that the methane yield in thermophilic AD was higher than in mesophilic; similar results were observed by Kim et al. (2002) and de la Rubia et al. (2006). When digesting wastewater biosolids at 55 ^{o}C, Ferrer et al. (2010) found that increasing the OLR from 0.5 to 2.5–6 kg VS m^{-3} day^{-1} resulted in: an increase in methane production from 0.2 to 0.4–0.8 m^3 CH_4 $m^{-3}_{reactor}$ day^{-1}, efficient pathogen destruction and a reduction in the capillary suction time (CST) from 437 s to 60–160 s. The increase in methane production was proportional to the increase in OLR. When the OLR was increased to > 6 kg VS m^{-3} day^{-1}, however, methanogenic activity was severely affected as indicated by: decline in biogas production, decrease in methane content (< 50%), accumulation of VFA (> 6 g l^{-1}), and poor digestate dewaterability (CST ~630–1370 s). Ahn and Forster (2002) suggested the improved methane yields and greater degradation capacity and in thermophilic digesters could be due to improved microbial growth rates.

Umetsu et al. (2006) showed that thermophilic AD can operate successfully with a sugar beet and dairy manure mixture, with the ultimate methane production increasing as the sugar beet ratio increased. Koppar and Pullammanappallil (2008) claimed that use of a single-stage batch thermophilic leach bed system improved biogas and methane production as it allowed reuse of the inoculum from the previous run, triggering the growth of a robust microbial population. The higher temperature (55 ^{o}C) also enhanced the VS destruction to 96%, indicating an excellent degradation of substrate to biogas or methane.

Chi et al. (2010) found that thermophilic AD improved dewaterability of TWAS due to a higher reduction (48%) in the amount of particulate organic matter present in the digestate compared to that of in mesophilic digesters (36%). Amani et al. (2011) concluded that WAS digested under thermophilic conditions showed better dewaterability characteristics than with mesophilic digestion, giving a CST value of less than 20 s. Kim et al. (2002) found that thermophilic AD gave superior performance compared to mesophilic with respect to higher volatile solids removal.

Other studies have shown less positive results for thermophilic conditions. For instance, Kugelman and Guida (1989a, b) found that thermophilic AD of PS from municipal wastewater led to poor dewaterability, indicated by a high CST value of 500 - 800 s compared to 350 - 450 s in mesophilic conditions. Similar results have been reported by other researchers (Reusser and Zelinka, 2004; Marneri et al., 2009; Ferrer et al., 2010). Bivins and Novak (2001) confirmed that thermophilic AD creates colloidal materials which adversely affect the dewatering properties of digested sludge.

In term of foaming inhibition, Marneri et al. (2009) found that thermophilic AD resulted in a higher destruction of filamentous bacteria (*Micothrix parvicella* and *Gordona amarae*) with an average removal of 97%, possibly leading to a reduction in foaming occurrence. Rimkus et al. (1982) and Zábranská et al. (2002) observed that sewage sludge digested in thermophilic conditions was more resistant to foaming.

2.8. Dewaterability

2.8.1. Review of Dewaterability Test

Dewaterability is a key parameter in wastewater treatment. The results from dewaterability tests can be used to assess the viscosity of digestate, which is important for designing industrial dewatering facilities and equipment (Sawalha and Scholz, 2007). As noted above, anaerobic digestion can improve the dewaterability of a material to different extents. Lawler et al. (1986) claimed that a good digestion process can help to enhance dewaterability, while poor digestion leads to poor dewaterability. This was supported by Houghton et al. (2000a) who claimed that anaerobic digestion alters sewage sludge dewaterability as the AD process can minimize the presence of microbial extracellular polymer substances (EPS) by 25% compared to that of the raw sludge.

Dewaterability can be measured by Specific Resistance to Filtration (SRF) and CST. The CST test is a rapid, easy, and practical means to assess the filterability and the ease of removing moisture from slurry and sludge, with the result defined in units of time (seconds) (EEA, 1997; Dentel and Abu-Orf, 1995; Scholz, 2005; Sawalha and Scholz, 2007). The typical range of CST times for unconditioned organic wastewater

sludge is from 100 to 200 seconds. However, a CST of 10 seconds or less is required for dewatering in a filter press (USEPA, 1987). The CST method is based on the varying pressure applied by the movement of water through the filter paper and the result is known to be a good indicator of sludge filterability (Scholz, 2005).

2.8.2. Factors Affecting Dewaterability

Many factors can influence dewaterability. These include particle size of substrates (Zhou et al., 2002; Zhou, 2003; Liming et al., 2009); digestion temperature and the presence of EPS (Mikkelsen and Keiding, 2002; Zhou et al., 2002); feed sludge or feedstock composition (i.e protein, polysaccharide) (Joyce et al., 1978; Lovett et al., 1983; Houghton and Stephenson, 2002; Liming et al., 2009); sludge or digestate age (Lovett et al., 1983); as well as variation in digester design and operation, and particle size distribution (Lawler et al., 1986). Some studies have indicated, however, that dewaterability is not affected by the pH, volatile solids, ammonia or phosphate concentrations in wastewater biosolids (Zhou, 2003); or by digestion time (residence time) (Yan et al., 1987).

Previous studies have demonstrated that dewaterability is significantly influenced by particle size distribution (PSD) (Karr and Keinath, 1978; Lawler et al., 1986). According to Olboter and Vogelphol (1993), a larger amount of fine materials in sludge is related to poor dewaterability. In contrast, large and dense particles are desirable for good settling and dewatering. For example, Liming et al. (2009) carried out an experiment on the effect of protein, polysaccharides and particle size on dewaterability of AS from wastewater treatment during hydrolysis and acidification processes. Four batch vessels were used and adjusted the pH 5.5 or 10.0 on a daily basis. Two sets of vessels were maintained at 37 °C; while the others were at 55 °C. After 20 days incubation, all set of experiments resulted in a decrease in the mean of PSD from ~12 μm to 3 – 9 μm, giving an increase in CST from 9.7 s to 300 - 450 s (pH 10) and to ~13 - 28 s (pH 5.5).

Several studies have been reported that the distributions of proteins and polysaccharides in sludges (e.g. digested sludge, raw WAS and raw wastewater biosolids) affected the dewaterability (Murthy and Novak, 1999; Yu et al., 2008; Liming et al., 2009). A high amount of protein in the slime fraction (or non-cellular

light fraction) was associated with deterioration in dewaterability. This was because the protein in the slime fraction, released from hydrolysis and acidification, has a stronger water binding capability and made a greater contribution to poor dewaterability than the polysaccharide (Yu et al., 2008)

The amount of EPS in sludge plays a big role in affecting dewaterability compared to the composition of EPS, as reported in several studies (Urbain et al., 1993; Poxon and Darby, 1997; Liao et al., 2001; Houghton et al., 2000a, 2000b, 2001; Houghton and Stephenson, 2002; Sponza, 2003; Jin et al., 2004; Li and Yang, 2007; Hosnani et al., 2010). For example, Houghton et al. (2000b) found that the amount of EPS in the digested sludge affected the dewaterability characteristics, where an increase from ~11 to ~23 mg EPS g^{-1} SS gave an increase in CST values from ~11 to 23 s. Houghton and Stephenson (2002) also found that the amount of EPS influenced the degree of dewaterability: an increase in EPS in digested PS with glucose from 19.6 to 24.4 – 25.3 mg EPS g^{-1} SS increased CST from 2.1 s to 14.0 – 16.3 s.

Ramesh et al. (2006) and Li and Yang (2007) confirmed that within the EPS structure that surrounds the cells, loosely bound EPS (LB-EPS) has a greater influence on sludge dewaterability than tightly bound (TB-EPS). For instance, an increase in LB-EPS from ~1.5 to ~7 mg TOC g^{-1} SS led to an increase in SRF from 1 x 10^{10} to 1 x 10^{13} mg kg^{-1} (Yang and Li, 2009). A high concentration of EPS raised the viscosity of the sludge flocs, leading to poor dewaterability (Li and Yang, 2007; Niu et al., 2013).

According to several studies, viscosity significantly contributed to sludge dewaterability (Christensen et al., 1993; Dentel and Abu-Orf, 1995; Chen and Yang, 2012). A study by Jin et al. (2004) also found that viscosity and bound water content influenced the dewaterability of activated sludge (AS). An increase in viscosity from ~3.8 to ~11.0 mPa s or in bound water content from ~13% to ~27% increased CST of AS from ~14 s to ~20 s. These findings reveal that viscosity and bound water may be a good indicator to assess dewaterability together with normalised PSD.

2.8.3. Treatments for Improving Dewaterability

Improving digestate dewaterability may be useful to reduce the volume and increase the solids content of a digestate, thus reducing the transport costs and potentially improving its value as a biofertiliser. Dewatering by itself does not resolve all issues, however, as the liquid fraction which remains must be treated or disposed of; and it may contain a proportion of the valuable nutrients e.g. ammonia. Dewatering also requires energy and chemicals. Therefore, it is necessary to strike a balance between the economic and environmental costs and benefits.

Numerous studies have been conducted to find ways of improving dewaterability, including chemical, thermal, mechanical, microbial and enzymatic treatments, and some of these are discussed in the following sections.

2.8.3.1. Chemical Treatments

a. Mechanism of Particle Aggregation due to Chemical Treatments

The basic aim of chemical treatment in a dewatering process is by conditioning the sludge or digestate with the addition of chemical coagulants/flocculants to promote particle aggregation, favouring floc formation and release of absorbed water (Dentel, 2001; Tchobanoglous et al., 2003), and thus reducing the moisture content of the treated sludge/digestate. In chemical dewatering, two processes are involved known as coagulation and flocculation process. Coagulation is the process of neutralising the surface charge of particle to promote interparticle collision to form microfloc. Flocculation is the process of bridging the microflocs to build a larger particle (Bratby, 1980; Tchobanoglous et al., 2003). In general, according to Gregory (2009), particle aggregation in chemical treatment involves the following steps: destabilisation of particles by the addition of coagulants/flocculants or possibly by adjusting chemical conditions (e.g. pH); and collision of destabilised particles to form flocs. These most commonly occur by Brownian diffusion (perikinetic aggregation) or by induced fluid motion (orthokinetic aggregation) (see Figure 2.5).

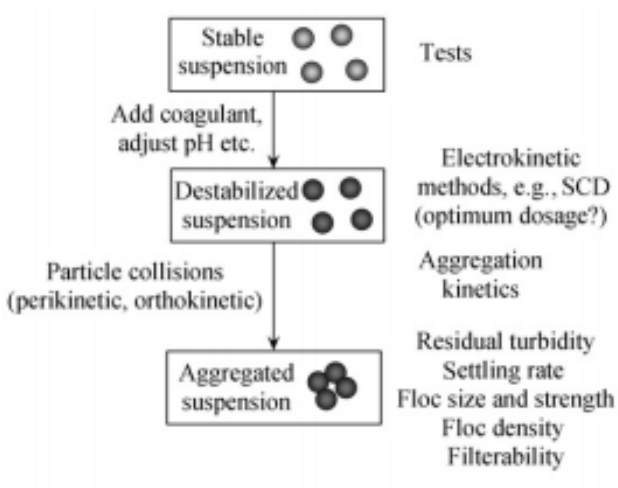


Figure 2.5. Particle aggregation and test methods (Gregory, 2009)

b. Examples of Chemical Treatments

Chemical coagulants are widely used to enhance the dewaterability of raw or digested sludge especially in the water and wastewater treatment industry. The addition of chemical coagulants causes particle aggregation and leads to an increase in the rate of water removal and the efficiency of the dewatering process. Common chemical coagulants originally used in dewatering were iron salts (i.e. ferric chloride/$FeCl_3$, ferrous sulphate/$FeSO_4$), aluminium salts (i.e. alum) and lime ($Ca(OH)_2$) (Hwa and Jeyaseelan, 1997; Novak et al., 1999; Deneux-Mustin et al., 2001; Lo et al., 2001; Novak, 2006; Verrelli et al., 2009).

Novak et al. (1999) and Buyukkamaci (2004) stated that in dewatering processes, chemical treatment was preferable as it is more economical than mechanical treatment. They further added that the use of chemical conditioning agents is an important aspect to consider in the operation of mechanical dewatering and vice versa, because the selection of chemical conditioning agents is usually subject to the type of mechanical equipment and the biosolids characteristics.

A study on the addition of ferric chloride alone or combined with lime showed an improvement in dewaterability of digestate from mesophilic and thermophilic AD of PS (Kugelman and Guida, 1989a, b). It was found that ferric chloride alone performed better than when combined with lime. To reduce CST time to < 20 s, thermophilic digestate required 10.4 g l^{-1} of ferric chloride ($FeCl_3.6H_2O$) while

mesophilic digestate needed a much lower dose of 7 g l^{-1}. Agarwal et al. (2005) studied the addition of ferric chloride (25% (w/v)) at a dose of 0.036 – 0.177 g g^{-1} TS for conditioning WAS from autothermal thermophilic aerobic digestion (ATAD) and found that it improved sludge dewaterability, giving CST values in the range of 69-54 s. These values, however, were still above the desired level for effective sludge dewatering of CST < 20 s (EPA, 1987). When combined with addition of anionic polymer at a dose of ~0.016 g g^{-1} TS, the required dose of ferric chloride was reduced to 0.093 g g^{-1} TS and the CST decreased to 12 s, potentially leading to a more economical dewatering process.

Others have investigated the use of chemical coagulants such as Fenton's reagent (Fe^{2+}/H_2O_2), and found an improvement of dewaterability (Neyens et al., 2003; Tony et al., 2008). Buyukkamaci (2004) studied the effect of Fenton's reagent (Fe^{2+}/H_2O_2) in conditioning of biosolids from municipal wastewater treatment. Various doses of Fe^{2+} (1000–6000 mg l^{-1}) and H_2O_2 (2000–6000 mg l^{-1}) were evaluated, with the effect on dewaterability measured by SRF and CST. The greatest improvement in dewaterability was at dose of 5000 mg l^{-1} Fe^{2+} and 6000 mg l^{-1} H_2O_2, as indicated by a decrease in SRF from 9.2×10^{10} m kg^{-1} to 6.2×10^{9} m kg^{-1} and in CST from 30.2 s to 15.7 s. Chen et al. (2001) studied the effect of acid and surfactant addition on dewaterability performance. The experiment was performed in duplicate using a jar test method. Sulphuric acid at a concentration of 16 mol l^{-1} was added to 100 ml of sample to achieve pH 2.5 and the surfactant betaine was added in the concentration range of 0 – 0.2 g l^{-1}. The addition of sulphuric acid alone or with 0.1 g surfactant improved sludge dewaterability and settleability, indicated by a reduction in water content from ~84% to ~74%. This was due to removal of polymers from the sludge surface and a decrease in EPS.

Work by Lin et al. (2001) focused on the use of chemical coagulants and physical conditioners and their effect on the dewaterability of sludge from a sludge thickener at laboratory-scale. The chemical coagulants used were alum and ferric chloride at concentrations of 10 and 15 % on a DM basis; while the physical conditioners were wheat pulp (particle size range 3 to 5 mm) and wood chips (between 0.5 and 3 mm) at doses of 0, 30, 60, 90 % on a DM basis, respectively. The combinations used were ferric chloride-wood chips, alum-wood chips and alum-wheat pulp. The results

showed that all of these combinations improved dewaterability, shown by a reduction in SRF value to < 1 x 10^{12} m kg^{-1} and a high cake solid yield, particularly at the highest dose for both chemical coagulants and physical conditioners. Çeçen et al. (2003) studied the effect of powdered activated carbon (PAC) on dewaterability of activated sludge (AS) from an AS system fed with a mixture of leachate and domestic wastewater. The results showed that the addition of PAC significantly improved sludge dewaterability as well as reducing persistent COD.

Synthetic organic polymers have also been widely used as chemical coagulants in dewatering processes (Chang et al., 1998; Özacar and Şengil, 2000; Chu and Lee, 2001; Lo et al., 2001; Novak, 2006). Type of polymers used include cationic (Hou and Li, 2003; Chu and Lee, 2005), anionic (Zhao and Bache, 2002; Glover et al., 2004) and non-ionic (Lee and Liu, 2000; Mpofu et al., 2004). A study by Dentel and Abu-Orf (1995) investigated the effect of cationic polymer (Poilu-Treat C420) on the dewaterability of anaerobically digested sludge from the Warminster Wastewater Treatment Plant, Pennsylvania, at both laboratory and full-scale. The laboratory-scale results demonstrated that the greatest improvement in dewaterability was achieved at the maximum polymer dose tested, which was 250-350 $mg_{polymer}\ l^{-1}_{sludge}$, and which reduced CST from ~220s to ~12 s. When the polymer dose was increased to > 350 $mg_{polymer}\ l^{-1}_{sludge}$, the CST gradually increased. In full-scale application, due to the limited flow rate of the polymer feed pump, the doses used were in the range of 40 - 170 $mg_{polymer}\ l^{-1}_{sludge}$. In general, at those rates the CST decreased to less than 10 s, with the greatest improvement at dose of 140 $mg_{polymer}\ l^{-1}_{sludge}$ giving a CST value of ~4 s. Experiments by Chang et al. (2001) demonstrated the addition of cationic polymer (Oya, CN60) to WAS significantly improved its dewaterability, resulting in a CST of 36 s at the optimal dosage of 100 mg l^{-1}.

A great deal of work has been done on the use of dual polymers to improve dewaterability (Lee and Liu, 2000; Glover et al., 2004; Ayol et al., 2005; Kuglarz et al., 2008). Lee and Liu (2001) carried out a laboratory-scale experiment on the effect of single or dual polymer addition on the dewaterability and floc structure of WAS from a synthetic fibre plant. The polymers added were cationic polymer (KP-201C) and non-ionic (NP-800) with a dose range from 0 to 2.5 kg $tonne^{-1}$ DM. The results showed that polymer addition significantly improved the dewaterability of WAS, as

indicated by a decrease in CST from 52 s to less than 20 s. The non-ionic polymer resulted in a CST of 14 s where doses ranged from 5.0 to 10.0 kg tonne^{-1} DM; while cationic polymer gave a CST of 17 s with an optimal dosage of 15 kg tonne^{-1} DM. The dual polymer addition, however, gave better dewaterability characteristics, resulting in a CST of 10 s at the optimal dose of 2.5 kg tonne^{-1} DM (non-ionic polymer) and 5.0 kg tonne^{-1} DM (cationic polymer), with less chance of overdosing and a much stronger floc structure. Agarwal et al. (2005) found that the use of dual polymer, cationic (BC 650, 2% (w/v)) and anionic (superfloc A 1820, 1% (v/v)) at dose of 0.04 g g^{-1} TS and 0.135 g g^{-1} TS, reduced the CST of WAS from 8000 s to 29 s, much lower than the 172 s achieved by the use of cationic polymer alone at a dose of 0.05 g g^{-1} TS.

In work by Huang et al. (2010), the effect of addition of H_2SO_4 followed by polymer on the dewaterability of alum sludge from wastewater treatment in central Taiwan was investigated. For acidification, the sample was placed in a water bath at 25 ^{o}C and concentrated H_2SO_4 (98%) was added to achieve target pH values from 2 to 7 followed by stirring for 2 hours. The results showed that reducing the pH from 7.0 (original sludge) to less than 4.0 gave a significant improvement in dewaterability, shown by a decline in SRF from 4.89 x 10^{11} m kg^{-1} to 1.69 x10^{10} – 5.09 x 10^{9} m kg^{-1}. Further addition of cationic polymer (PC-320) to the mixture resulted in an additional improvement to the alum sludge dewaterability, reducing water content by 2% and CST time from 200 s to 50 s.

A great deal of expertise is available on chemical dewatering of raw and digested sludges from the wastewater industry, but relatively little research has been applied to digestates from other feedstocks, and there is thus considerable potential for investigating this approach with respect to SBP digestate.

2.8.3.2. Thermal Treatments

Various studies have demonstrated that thermal treatments, such as heating in an autoclave at 689.5 kPa (Kovacs, 1992) and thermophilic aerobic digestion (Attar et al., 2005), can improve dewaterability. Neyens and Baeyens (2003) noted in a review of thermal pre-treatment processes to improve dewaterability that both thermal and thermochemical (e.g. combined with acid or alkali) pre-treatments improved the

dewaterability of undigested and digested WAS. Neyens et al. (2004) concluded that advanced sludge treatment methods such as thermal hydrolysis, acid thermal hydrolysis and peroxidation, improved AS dewaterability. The mechanisms of these techniques were mainly by degradation of EPS proteins and polysaccharides, resulting in a decrease of EPS water retention properties; and by promoting flocculation, thus leading to a reduction in the amount of fine floc.

Thermal processes, both pre- and post-treatments, seem to offer considerable potential for dewatering; however the cost can be high due to the energy demand for operation (Vaxelaire et al., 1999; Neyens and Baeyens, 2003; Carrère et al., 2010). If thermal treatment can be integrated with the treatment process for wastewater biosolids or organic biowaste, however, it may offer the possibility not only to enhance the dewatering rates but also to minimise cost.

2.8.3.3. Mechanical Treatments

Mechanical dewatering treatments, such as filtration and compression, centrifugation, grinding etc., have been widely used in different areas (e.g. wastewater biosolids treatment, cattle slurry treatment, etc) due to their low energy requirement (Covington and Ekiner, 1979; Maffet, 1983; Berktay, 1998; Vaxelaire et al., 1999; Lo et al., 2001; Chen et al., 2002; Novak, 2006; Wakeman, 2007; Carrère et al., 2010). Typically, mechanical dewatering processes consist of two steps: filtration and compression. Filtration removes the water in the biosolids to the point of cake solids formation. Compression squeezes the cake by the application of a mechanical pressure or force to take out the remaining water (Novak et al., 1999; Vaxelaire et al., 1999; Novak, 2006; Mahmoud et al., 2010; Qi et al., 2011).

Qi et al. (2011) reported that the use of physical conditioners, such as carbon-based materials (e.g. char, coal fines, wood chips, wheat dregs, bagasse, etc.) and minerals (e.g. fly ash, cement kiln dust, gypsum, etc.), improved the performance of mechanical dewatering processes. They further added that carbon-based materials are better than minerals as physical conditioners because of their low ash content, high calorific value and generally high porosity, which are good properties for filter aids. In some cases mineral conditioners may have hazardous effects on the environment which limit the routes for application or disposal of the dewatered products.

Several studies have investigated ultrasonic treatment as an alternative to mechanical dewatering processes. The basic principle of this treatment is to mechanically disrupt the cell structure and floc matrix through cavitation (formation of gas bubbles due to rapid changes in pressure) and chemical reactions due to the formation of $OH^{\bullet}$, $HO_2^{\bullet}$, $H^{\bullet}$ radicals, which improves the dewaterability (Carrère et al., 2010). For example, Kim and Kim (2003) investigated the effect of ultrasound treatment on dewaterability of sludge from municipal sewage treatment, using an ultrasonic processor (Chosun, Model CS-1000). The treatment significantly enhanced dewaterability, indicated by a decrease in CST time, an increase in drying efficiency, and an increase in particle size. Feng et al. (2009) looked at effect of ultrasound treatment alone or combined with polymer addition on the dewaterability of WAS. The doses of ultrasonic energy varied from 0 to 35,000 kJ kg^{-1} TS. For sludge subjected to ultrasound treatment the resulting CST and SRF value depended on the energy dose, with 800 kJ kg^{-1} TS being the optimal dose for enhancing dewaterability and while also contributing to a slight decrease in EPS concentration and in floc size. The addition of polymer prevented ultrasound giving any improvement in dewaterability.

Freeze/thaw (F/T) treatment represents another form of mechanical treatment. Hu et al. (2011) carried out freeze/thaw (F/T) experiments to enhance the dewaterability of primary sludge (PS) and WAS. The samples used were raw WAS and a mixture of PS and WAS at a ratio of 1:4 (v/v). The samples were then placed into polyethylene terephthalate bottles sealed with polyethylene lids and frozen at -18 ^{o}C for 0, 1, 3 and 72 hours followed by thawing at 29 ^{o}C and 47-56% of relative humidity for 3 hours. The results demonstrated that F/T treatment enhanced dewaterability, particularly freezing for 72 hours which gave a reduction in the sedimentation volume by 31.2 - 31.3% (mixed sludge) and by 33.3 - 44.7% (WAS).

Advanced mechanical dewatering methods have also been developed. For example, Aziz et al. (2006) tested electrically enhanced dewatering (EED) using a single-ended pressure filtration rig connected to a regulated direct current power supply (Good Will Instrument GPS 3060). The samples were coagulated and dispersed kaolinite suspensions, and sludge from a potable water treatment plant. The pressure

was in the range 0.1– 400 kPa, while the selected electrical field strengths were 250, 500, 750, 1000 and 1250 V m^{-1}. The results for all applied pressures indicated that, as the applied electrical field strength increased from 250 to 1250 V m^{-1}, the dewatering rate improved. The best performance for the coagulated kaolinite suspensions, however, was found at the lowest applied pressure. The results for the sludge showed that better dewatering performances occurred at all pressures above 10 kPa. An experiment on EED treatment of wastewater biosolids was carried out by Mahmoud et al. (2011b), and showed that the combination of EED with a conventional mechanical dewatering technology at 200 - 1200 kPa and 10 - 50 V improved the removal of the water remaining after mechanical treatment. The energy consumption required for this process dropped by 10-25%. This treatment looks like a promising technology but is unlikely to be widely adopted because of its sophisticated system, a high capital cost to build and/or problems arising during its application, such as corrosion and high energy consumption.

2.8.3.4. Microbial Treatments

A recent study focusing on secondary sludge from wastewater treatment showed that the addition of microbial supplements such as 'effective microorganisms' (EM) in combination with chemical coagulant (i.e. lime, alum and ferrous sulphate) had a significant effect on enhancing dewaterability. This study also claimed that conditioning sludge by adding 60 mg l^{-1} of alum at 1% EM gave the best performance in improving dewaterability with a minimum SRF of 0.98348 $x10^{12}$ mg kg^{-1} (Shihab, 2010). In work by Zhang et al. (2010), a microbial flocculant (MBF) *P. mirabilis* TJ-F1 was used as a novel conditioner for wastewater sludge. 2 ml of $CaCl_2$ (1%, w/v) and various volumes of TJ-F1 (0, 0.5, 1.0, 2.0, 4.0, and 6.0 ml) were added to 50 ml of wastewater sludge, then the pH was adjusted to 7.5. Optimal conditions were observed at a MBF TJ-F1 dose of 2 ml (or 0.17% w/w) with 2 ml of $CaCl_2$ (1.3% w/w) and pH 7, resulting in the highest filtrate volume (~40 ml).

2.8.3.5. Enzymatic Treatments

Many studies have been carried out to assess the effect of enzymes on the dewaterability of digestate. An experiment by Barjenbruch and Kopplow (2003) showed better dewaterability for a mixture of surplus sludge (SS) and PS after enzymatic pre-treatment using carbohydrase enzyme. Dewaterability improved

because the enzyme was able to degrade the microbiological slime/cells. However, the destruction of cells was much lower than that under high pressure or high temperature pre-treatment.

Ayol (2005) investigated the effect of enzyme addition on dewaterability of pre-conditioned anaerobically digested biosolids collected from two municipal wastewater treatment facilities in New York City (NYC) and Wilmington (WIL), USA. The samples were conditioned with Percol 757 polymer stock solution at dosages of up to 500 mg l^{-1} and then enzyme was added. The addition of enzymes mixtures containing protease, lipidase, *Aspergillus oryzae* and hydrolytic enzyme (at a dosage of 0 to 100 mg l^{-1}) improved the dewaterability of the samples, based on the CST, solids content of the final product, protein and polysaccharide concentrations, filtrate turbidity and suspended solids. These results demonstrated that enzymatic pre-treatment with polymer conditioning can enhance the dewaterability of anaerobically digested biosolids, and showed a high potential for full-scale application. SEM images (Figure 2.6) showed that after the addition of both polymer and enzyme, the biosolid structures formed into much larger flocs creating a gel-like biocolloidal matrix.

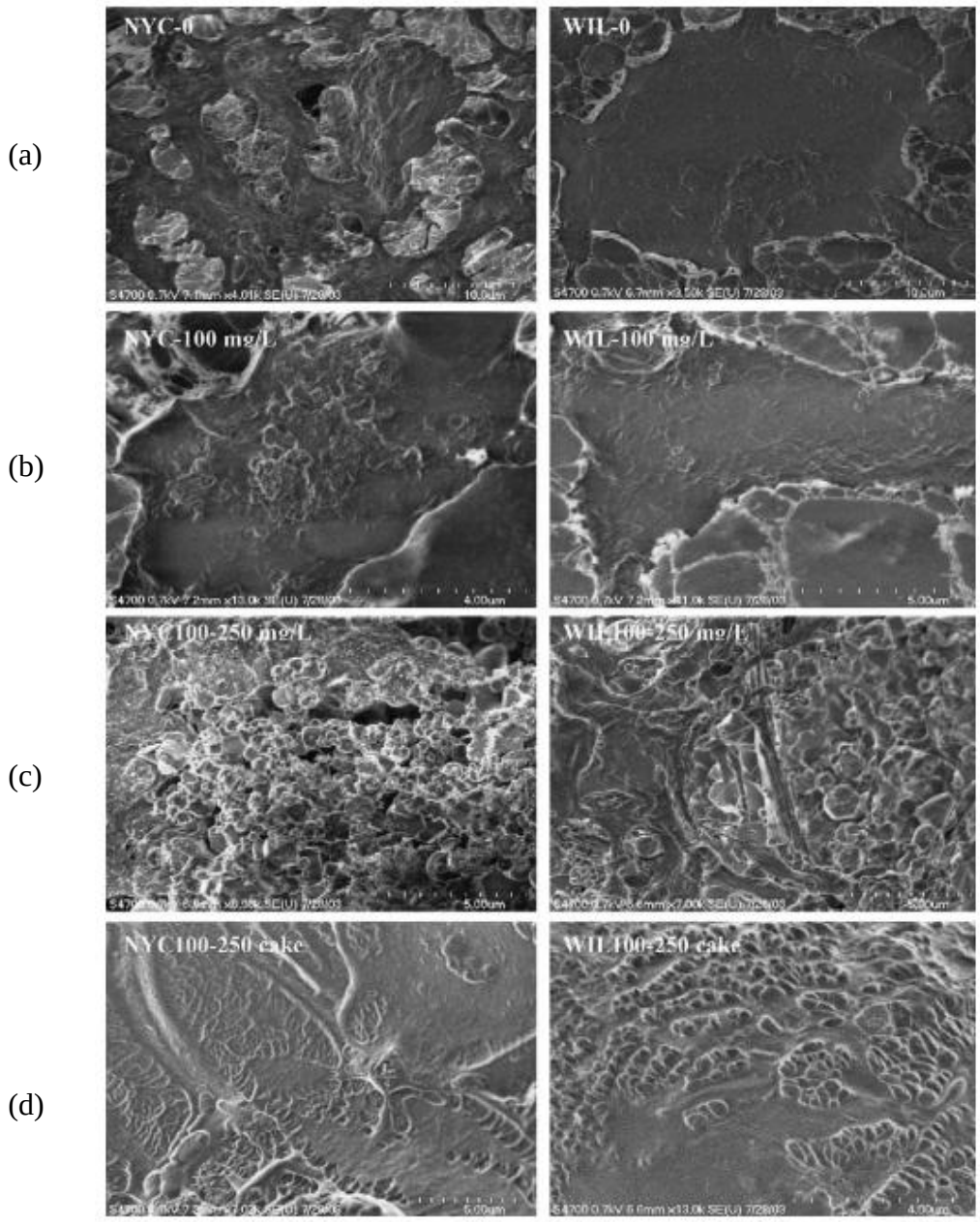


Figure 2.6. SEM images for NYC (left) and WIL (right) biosolids samples: (a) raw, (b) enzyme treated (100 mg l^{-1}), (c) enzyme treated and polymer flocculated (250 mg l^{-1}), and (d) final cake product (Ayol, 2005)

Dursun et al. (2006) conducted a study of enzymatic pre-treatment followed by centrifugation of an anaerobically digested mixture of PS and AS. At a laboratory scale, using enzymes (e.g. commercial mixtures, amylase, cellulose, and protease enzymes) a dose of 20 mg l^{-1} improved the cake solids content after centrifugation to 27%. In a pilot-scale experiment, however, the cake solids content was less than 20%, with the difference being due to the higher shear applied in centrifugation. Rheological measurements in the sludge further suggested that the gel structure of

the flocs was weakened by the enzymatic pre-treatment, through the hydrolysis of EPS. Thus, they further added that a better dewatering process could be achieved using filtration processes rather than centrifugation due to the deterioration of flocs when centrifuged at high shear.

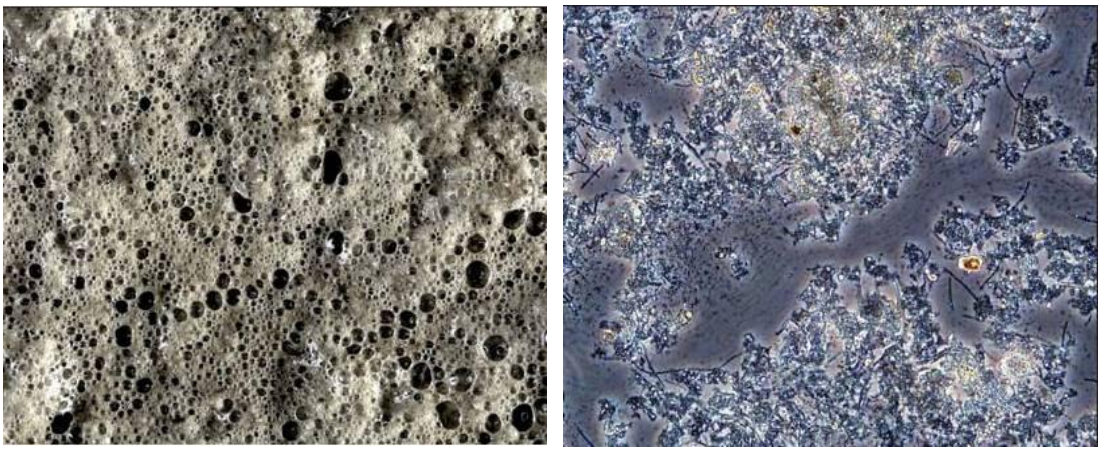
DeLozier and Holmes (2008) developed methods for enzymatic treatment using an alpha-amylase enzyme of *Geobacillus stearothermophillus* on various sludges: WAS, municipal PS, municipal WAS and pulp and paper-mill WAS. The enzymes were added to 400 ml of inoculum sludge at dosages of 3.5 – 69.7 g protein kg^{-1} TSS. Dewaterability was measured based on the cake solids and cake volume obtained after a filtration process. In general sludge dewaterability improved significantly, as indicated by an increase of ~0.6-7% in cake solids and a decrease in cake volume by ~3.4 – 40% from the original values. Pei et al. (2010) observed the effect of the enzyme protease (from *Aspergillus orzae*) and cellulase (from *Aspergillus* sp.) on the dewaterability of AS. The enzymes were added at dose of 500 activity units per 100 ml into 250 ml AS samples and placed in a water bath at 35 ^{o}C. After 26 hours, treatment with protease increased the CST from ~ 10 s to 26.7 s; while cellulase increased CST from ~ 10 to 16.2 s after 24 hours. This was due to an increase in the number of smaller particles and a slight decrease in EPS concentration.

From the studies reported above, it is obvious that there are several alternative treatments or technologies that can be applied in the dewatering process, but when implemented alone the water removal efficiency may be low. This has prompted further developments in or combinations of treatments and technologies, such as mechanical-chemical treatments (Yoon and Basilio, 1997; Lo et al., 2001; Wakeman, 2007), thermal-chemical treatments (Kang et al., 1990; Smith and Göransson, 1992; Takashima and Tanaka, 2008) and thermal-mechanical treatments (Clayton et al., 2006; Mahmoud et al., 2011a), to give greater improvements in dewatering rates and filtrate clarity. In practice, however, economic aspects and environmental safety must be considered when selecting the appropriate treatment. Chemical-mechanical or thermal dewatering treatments may provide more economic and less harmful options, but this depends on the selection of chemical conditioners and mechanical methods.

2.9. Foaming

2.9.1. Review of Foaming

Foam is defined as a dispersion of gas in liquid, which is usually composed of thousands of tiny bubbles (Vardar-Sukan, 1998) or can be defined as the accumulation of gas bubbles surrounded by a liquid film on the sludge surface (Ganidi et al., 2009) as shown in Figure 2.7. Foaming has been recorded in many anaerobic digestion plants, sometimes with severe impacts on the process which may also lead to serious economic consequences.



(a) (b)

Figure 2.7. Foam image (a) in biogas plant and (b) under microscope (Moeller et al., 2010)

2.9.2. Factors Causing Foaming

Foaming appears to have a complex set of causes. It can be due to the presence of excessive numbers of filamentous bacteria (such as *Gordonia* and *Microthrix*) (Westlund et al., 1998; Massart et al., 2006); excessive surface active agents (oils and grease) (Wheatley et al., 1988; Massart et al., 2006); air entrainment and solids concentration (Wheatley et al., 1988); feed sludge composition, and inconsistent feed to the digester (Massart et al., 2006); unstable conditions because of shock load or overloading (Speece, 2008; Moeller et al., 2012); temperature fluctuation (Barber, 2005); hydrophobic substances, inadequate mixing or accumulation of acetic acid (Pagilla et al., 1997); protein-rich and easily degradable substrates (Moeller et al., 2012). The causes of foaming are summarised in Figure 2.8. Ganidi et al. (2009) stated that egg-shaped digesters have a smaller open surface area above the bulk

phase of the digester which may reduce the potency of foam accumulation, but Tchobanoglous et al. (2003) found no evidence to suggest that egg-shaped digesters prevent foam formation.

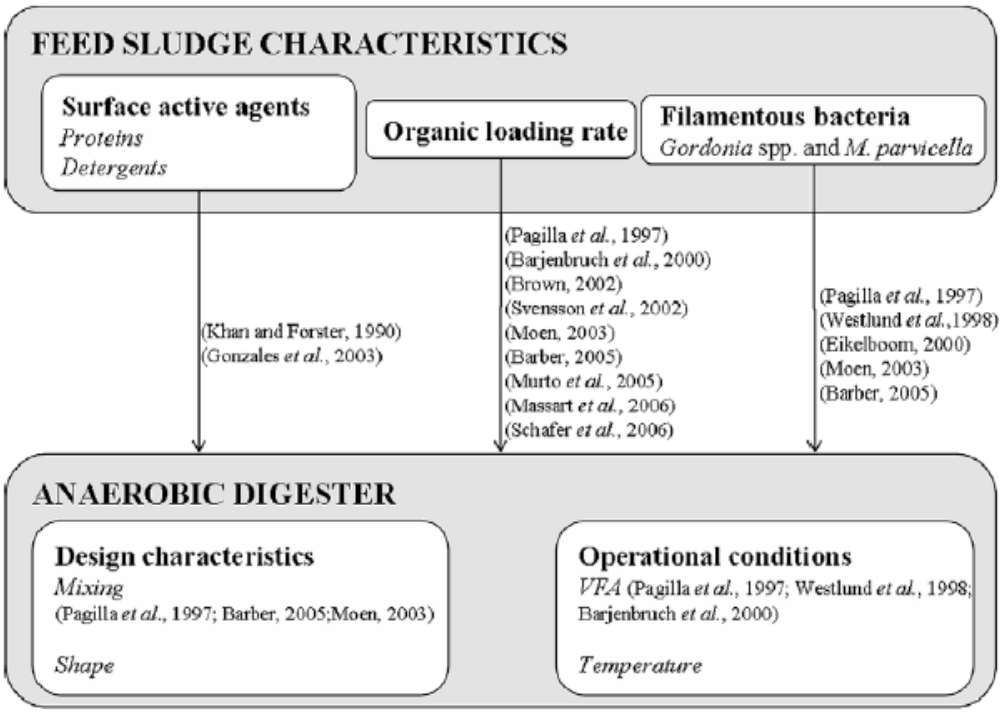


Figure 2.8. Schematic representation of the possible causes for foaming in anaerobic digestion (Dalmau et al., 2010)

A high OLR can cause foaming because the excess feedstock is not fully degraded by microorganisms in the digester, resulting in the accumulation of hydrophobic or surface-active by-products (Ganidi et al., 2009). In digestion of municipal wastewater biosolids, foaming usually occurs in digesters operating at OLR higher than 4.5 kg VS m^{-3} day^{-1} (Brown and Sale, 2002). In several biogas plants, foam formation problems have occurred during the start-up process or when a low OLR is followed by a rapid increase in OLR (Moeller et al., 2010).

2.9.3. Problems Related to Foaming

Foaming causes many problems which may lead to a drop in biogas or methane production, as well as a decrease in organic matter degradation (Barjenbruch and Kopplow, 2003). Table 2.4 provides examples of the various consequences of the occurrence of foaming in the anaerobic digestion process (Vardar-Sukan, 1998).

Table 2.4. Problems created by foaming in bioprocess

Physical effects	- Increased heterogeneity of broth - Enhancement of gas-liquid transfer - Increased effective reactor volume - Reduction in the working volume - Enhanced gas hold-up - Changes in air bubbles size and composition - Decreased power dissipation - Changed pattern of dissolved gases due to heterogeneous dispersion - Reduction in apparent viscosity - Lower mass and heat transfer rates - Invalid process data due to interference at the electrodes - Decreased circulation rate - Incorrect monitoring and control - Reduction in aeration and mixing - Blockage of inlet and exit gas filters
Biological effects	- Enrichment of cells in the stagnant liquid film around the air bubbles - Deposition of cells on upper parts of the bioreactor - Loss of culture fluid from exit lines causing product and biocatalyst loss - Microbial lysis - Changes in microbial metabolism due to nutrient limitations - Froth flotation and foam separation causing preferential removal of surface active agents - Proteins denaturation in foam layer - Problems in sterile operation - Risk of environmental contamination due to aerosol formation

Source: Vardar-Sukan (1998)

2.9.4. Previous Studies on Reducing Foaming

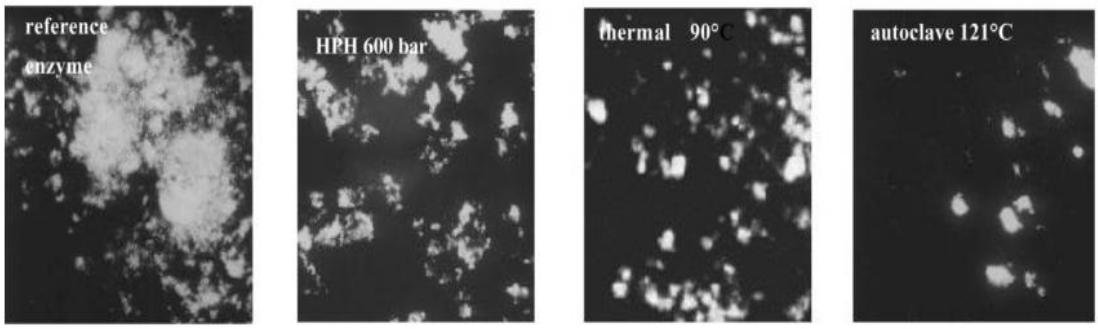
Westlund et al. (1998) carried out a study on foaming in anaerobic digestion of a mixture of PS and WAS from three wastewater plants in Sweden and suggested several methods to reduce foaming. The first was by increasing the OLR, which resulted in a significant inhibition on the growth of *M. parvicella*. The second was by decreasing the sludge age in the aeration tank, which resulted in a reduction of the proportion of filamentous bacteria entering the digester, causing a greater reduction in foaming; these methods could not be implemented, however, if the plant had nitrification or limited dewatering capacity. A third method was by adding anti-foam such as a poly-aluminum salt. A fourth was by installing a top stirrer in the digester

which was effective in breaking up foam and preventing gas tube blockage. The last method trialled was heating the sludge mixture (70 ^{o}C, 5 min) prior to loading into the digester, which damaged the cell wall of *M. parvicella* but did not cause a significant reduction in foaming.

Barjenbruch and Kopplow (2003) studied different pre-treatment methods for reducing the occurrence of foaming, including mechanical, thermal (thermal decomposition) and enzymatic treatment, at a laboratory scale. In this experiment, four double-walled 10-litre digesters were fed with a mixture of 60% SS and 40% PS from a municipal wastewater treatment plant. The mechanical treatment used a high pressure homogenizer (HPH) with a high pressure-pump (up to 60 MPa) to destroy cell membranes. Thermal destruction was performed in an autoclave at 80, 90 and 121 ^{o}C for 1 hour. The c*arbohydrase* enzyme was used for enzymatic pre-treatment. Thermal pre-treatment at 121 ^{o}C performed better than the other treatments as it completely prevented foaming (see Figure 2.9a). The mechanical pre-treatment led to a slight decrease in foaming; however the application of enzymatic pre-treatment had no effect on foaming. It was concluded that in this case the foam was caused by the existence of EPS rather than the structure of the digestate (see Figure 2.9b).

The use of antifoams to control and reduce the occurrence of foaming has been widely reported in several studies (van Niekerk et al., 1987; Wheatley et al., 1997; Westlund et al., 1998; Junker, 2007). For instance, Brown and Sale (2002) stated that, in the case of Southern Water's sludge treatment with high-rate digestion, the use of oil-based antifoams at shock dose of 20 mg $l^{-1}_{digester}$ was able to control foaming in the digesters. Yamamoto et al. (2006) conducted an experiment in anaerobic digestion using swim-bed technology for treatment of swine wastewater with influent suspended solid and total COD values of 8000–17,000 mg l^{-1} and 5000–11,000 mg l^{-1} , respectively. During the process, severe foaming occurred in the digester, but adding silicone antifoam (KM72; Shinetsu, Tokyo) as needed, overcame the problems. Cooney et al. (2007) who studied two-phase anaerobic digestion of a mixture of glucose, yeast extract and peptone at OLR of 2 - 4 l day^{-1} and at C:N ratio of 13.4 - 19.1 with the addition of 0.02 ml l^{-1} silicone antifoams (antifoam A, Sigma) to prevent foaming occurrence, did not report any disturbance in digestion performance, with biogas production at 1.58 – 2.19 l day^{-1}.

(a)



(b)

Figure 2.9. (a) Foam production in test reactors and (b) Micrographs of surplus sludge for the determination of EPS (Indian ink reverse stain) (Barjenbruch and Kopplow, 2003)

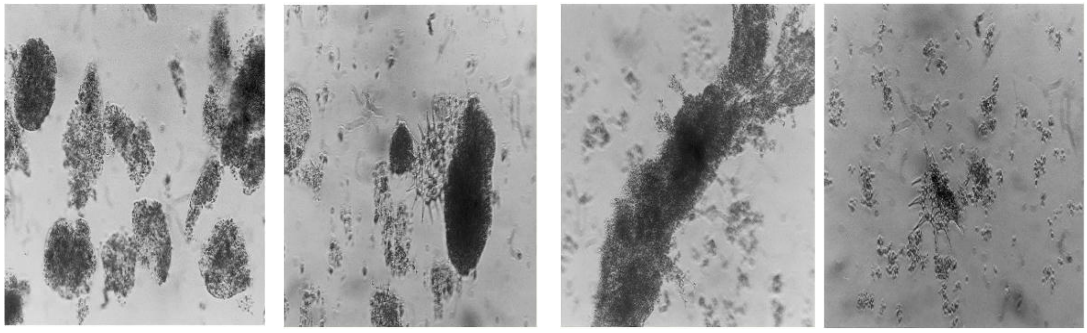
2.10. Sludge Ageing

Some reports have indicated that ageing can affect the dewaterability of digestates but only a few detailed studies have been carried out. A study by Rasmussen et al. (1994) demonstrated that anaerobic storage of AS had a decrease in dewaterability after 10 days storage. Stepkowska et al. (1997) studied the effect of ageing on dredged sludge collected from a canal in Holland (*Oude Maas-Botlek*). The experiment was performed on four different sludge samples: fresh sludge, after three months of storage (at < 10 ^{o}C), after storing for an additional month (at room temperature) and after four years (at ambient temperature). The analysis included drying rate, water sorption and thermal mass loss, microstructure by Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD) study. For calculation of the drying rate, samples were placed in cylindrical glass beakers with a base area of 20 cm^2 and a height of 5 cm, then were dried using an air-drier at atmospheric pressure and constant temperature (30 or 45 ^{o}C). The mass changed with time was

measured. The drying rate was calculated based on the formula $-\Delta G/\Delta t$, where $-\Delta G$ is the value of water content determined by mass loss. The drying rate increased with the age of sludge as did the water sorption rate. Based on the SEM and XRD study, ageing caused the formation of aggregates and of macropores between them which changed the sludge particle thickness and specific surface, opening water escape channels. With ageing the aggregates were more compact and easier to dry.

Pan et al. (1999) studied the effect of the ageing process on the characteristics of algae-containing residues from potable water treatment at Ming-Der water treatment plant in Taichung, Taiwan. The sludge was stored at room temperature and covered with plastic to avoid direct sunlight, and monitored every 2-3 days. The results demonstrated that during the ageing process up to 5 days, the surface charge of the sludge became more negative as the zeta potential became positive, thus the total organic carbon (TOC) content decreased and the dewaterability was enhanced by algal exudates. Microphotographs of the algae-containing sludge suggested that algae and their exudates cause the bio-flocculation phenomenon which caused the formation of larger flocs (see Figure 2.10).

Other work has shown that, after 100 days incubation of three mixtures of biosolid and sand, the hot-water-extricable carbohydrate decreased as the age increased and carbon groups were changed in association with organic matter degradation (Stacey et al., 2001). This implies that the degradation of organic matter in sludge ageing may be linked to the improvement of dewaterability or filterability of the sludge. However, Yang et al. (2008), who studied the effect of ageing on dewatered alum sludge from municipal wastewater treatment in Ireland for periods of 0 to 18 months, found that ageing had no effect on either the structure (i.e. surface area, porous size distribution and pore type) or the chemical (i.e.-OH and/or humic substances) characteristics. These differences could be explained by the relative inorganic contents of the wastewater being treated, compared to wastewater biosolids.



(a) (b) (c) (d)

Figure 2.10. Photomicrograph (250 x) of (a)1st-day sludge particles; (b)5th-day sludge particles showing bio-flocculation with Pediastrum; (c) 5th-day sludge particles showing enlargement of flocs through bio-flocculation; (d) 10th-day sludge particles showing degradation of algae and the breakdown of flocs (Pan et al., 1999)

These findings suggest that ageing may be a possible technique to improve dewaterability but presents problems when when very large volumes are involved, as in wastewater industry and for SBP digestates. It is clear, however that the effect of different treatments on dewaterability may vary depending on the composition or characteristics of the sludge or digestate, and that finding the optimum procedure is likely to require an approach which is at least partly empirical.

2.11. Carbon, Energy and Nutrient Footprint

Current studies suggest climate change is mainly caused by anthropogenic greenhouse gas (GHG) emissions due to processes and activities undertaken to provide consumer goods and services (Hertwich and Peters, 2009). With the growth in consumption, although the use of waste processing technologies is critical, in a certain way these also affect the environment as they emit carbon and consumes energy (Oh et al., 2010). Quantification of the environmental effects or 'footprint' of waste management technologies is therefore essential in order to minimise the impacts, to assess potential methods to prevent negative effects and to obtain more cost-effective methods which can give significant economic savings (Aiyuk et al., 2004; Pandey et al., 2011; Hermann et al., 2011).

Carbon, energy and nutrient footprints are used to analyse the amount of carbon, energy and nutrient required or produced for a given process or product. Measuring

carbon footprint is important to identify the contribution of a process or product to climate change, as well as for the design of cost-effective strategies to mitigate carbon emissions (Matthews et al., 2008). The use of energy also contributes to carbon footprint, for instance through the release of CO_2 from the burning of fossil fuels: thus reducing energy use will eventually decrease both CO_2 levels and operational costs. The energy footprint can be used for these purposes, as well as to provide information on other potential uses of energy (Perry et al., 2008). In terms of nutrients, organic waste or residues of biological waste treatment still contain certain amounts of nitrogen (N), phosphorus (P) and potassium (K) which represent resources, such as fertiliser substitutes, that need to be recovered and not simply removed (Hu et al., 2012). The nutrient footprint can therefore provide a means of evaluation for removal and usage of nutrients. Thus, there is considerable interest in establishing carbon, energy and nutrient footprints of waste treatment processes, such as AD.

2.11.1. Review on Carbon, Energy and Nutrient (CEN) Footprint

2.11.1.1. Carbon Footprint

The carbon footprint is a term used to describe the amount of GHG emissions generated from a particular activity or entity, to allow assessment of its contribution to climate change (British Standards, 2008). Another definition of carbon footprint is '...a measure of the exclusive total amount of carbon dioxide emissions that is directly and indirectly caused by an activity or is accumulated over the life stages of a product' (Wiedmann and Minx, 2008). Alternatively carbon footprint can be defined as 'a measure of the impact that human activities have on the environment in terms of the amount of GHG emitted over the full life cycle of a process or product measured in units of carbon dioxide (CO_2)' (Frijns, 2012, p. 64).

The global warming potential (GWP) of GHG is expressed as CO_2 equivalent (CO_2-eq). The three most important GHGs (CO_2, CH_4 and N_2O) should be included in a GHG balance, which is then quantified for time horizons of 20, 100 or 500 years with different equivalence factors for each period (Table 2.5).

Table 2.5. Lifetime and global warming potentials (GWPs) for different time horizons of the three most important GHGs

GHG	Lifetime (years)	GWP 20 years	GWP 100 years	GWP 500 years
Carbon dioxide (CO_2)	n/a	1	1	1
Methane (CH_4)	12	72	25	7.6
Nitrous oxide (N_2O)	114	289	296	153

Note: n/a, not available.
Source: IPCC (2007)

2.11.1.2. Energy Footprint

Energy footprint is a measure of the energy inputs or outputs in a system or a process. The energy footprint is the amount of energy demand and recovery in a process, and can be used to evaluate its performance (e.g. potential energy savings or energy deficits) (Gori et al., 2010). Ferng (2002) stated that the calculation of energy footprints should be based on the direct and indirect use of primary energy in the system. Stöglehner (2003) developed a modified calculation model for the footprint of energy supplies based on the model of Wackernagel and Rees (Rees, 1996), to evaluate energy saving potentials of substituting fossil with renewable energy carriers, so that effects on users or environment could be identified. In this modified model, the type of energy used was calculated based on energy demand categories; while its footprint was calculated by multiplying the final energy use of the different energy carriers with their land need index. The demands categories were such as for fossil energy carriers; or renewable energy carriers; or as electricity, in units of MJ m^{-2} $year^{-1}$.

Rodríguez et al. (2011) proposed an LCA-based indicator to evaluate energy, based on the transformations occurring when processing a primary natural resource into a final product or service. This included direct and indirect primary resources used in a system or process during the transformation of a primary resource; as well as its environmental impact. Cherubini et al. (2009) suggested that the unit of energy can be categorised, for example bioenergy from dedicated crops should be expressed on a per hectare basis (MJ ha^{-1}); bioenergy from systems based on biomass residues should be expressed on per unit output basis (MJ kg^{-1}_{output}); and bioenergy from transportation biofuel production should be expressed per km basis (MJ km^{-1}).

The energy balance in AD is calculated based on energy inputs and outputs (Berglund and Börjesson, 2006). In anaerobic digestion of any crop-based material, for example, it is important to consider the energy needs in the three stages of crop production, biomass conversion and final use of the biogas (Salter and Banks, 2009). The energy input to be taken into account includes the energy needed for feeding, pumping, heating and stirring, while the energy output includes energy (biogas) production during the observation period (Bohn et al., 2007). Pöschl et al. (2010) stated that energy input, particularly in AD, was significantly influenced by the characteristics of the feedstock used in association with the energy needs for pre-treatment purposes, while the energy balance was influenced by several factors: biogas yield, utilization efficiency, and energy value of intended fossil fuel substitution. Davis (1996) and Barber (2010) suggested that the energy balance of AD was affected by dewatering and digestate treatment.

Calculation of the theoretical calorific values (or energy potential)

Various approaches can be used to estimate the energy content of biomass or solid waste through the calculation of calorific value (CV) based on: physical composition, proximate analysis, or ultimate analysis/elemental content (C, N, H, S, O) (Liu et al., 1996; Sheng and Azevedo, 2005; Komilis et al., 2012). The basic equations used to estimate the energy content from physical composition (i.e. paper, water, etc) and proximate analysis (i.e. moisture, ash, fixed carbon, etc) can be seen in equations [2.1] and [2.2] (Liu et al., 1996). Other equations based on the proximate analysis have been reported in different studies (Sheng and Azevedo, 2005; Erol et al., 2010), which indicated that these formulae result in acceptable values for CV and are easy and simple to use.

$$Hn = 88.2\,R + 40.5(G+P) - 6\,W \qquad [2.1]$$

Where,
Hn is Net calorific value (Kcal kg^{-1} dry solids).
R is Plastics, % weight on dry basis.
G is Garbage, % weight on dry basis.
P is Paper, % weight on dry basis.
W is Water, % weight on dry basis

$$Hn = 45\,B - 6\,W\ (traditional\ equation) \qquad [2.2]$$

Where:
B is combustible volatile matter in MSW (%)

Of the models based on elemental composition, the most commonly used is the Du Long formula, which was originally used to estimate the combustion heat of coal or fossil fuels from its chemical composition (equation [2.3]), and assumes that the heat of formation of the organic matter is negligible compared with the heat of combustion of the elements (Chang et al., 1997; Niessen, 2002).

$$HHV = 81\,C + 342.5(H - \frac{1}{8}O) + 22.5\,S \qquad [2.3]$$

Where,
HHV is higher heating value or calorific value of dry solids (in MJ kg^{-1} dry solids)
C, H, O, and S are represented as the weight of each proportion

A modified version of Du Long formula is reported in (Tchobanoglous et al., 1993; Pichtel, 2005):

$$HHV = 80.5\,C + 338.6\,H - 42.3\,O + 22.2\,S + 5.55\,N \qquad [2.4]$$

Where,
HHV is higher heating value or calorific value of dry solids (Kcal kg^{-1} dry solids)
C, H, O, and S are represented as the weight (%) of each fraction in the samples

Another modified Du Long equation is given by International Flame Research Foundation (IFRF, 2013a):

$$HHV = (34.1\,C + 102\,H + 6.3\,N + 19.1\,S - 9.85\,O)/100 \qquad [2.5]$$

Where,
HCV is higher calorific value in MJ kg^{-1}
C, H, N, S and O are the weight (%) of each fraction in the samples

Another formula based on the ultimate analysis is the Boie formula, which was originally developed for the estimation of the heat of combustion of mixed wastes, particularly especially high cellulosic materials, such as refuse or wood (Niessen, 2002). The Boie formula is the most widely used for biomass as follows (Mason and Gandhi, 1983; Annamalai et al., 1987; IFRF, 2013b):

$$HCV = 35.160\,C + 116.225\,H - 11.090\,O + 6.280\,N + 10.465\,S \qquad [2.6]$$

Where,
HCV is higher calorific value in MJ kg^{-1}
C, H, N, S and O are expressed as a mass fraction

The use of other formulae based on the ultimate analysis to predict CV has been reported in several studies, and includes the Steuer's, the Scheurer-Kestner, Mott-Spooner formulae and others (Mason and Gandhi, 1983; Niessen, 2002; Sheng and Azevedo, 2005; Komilis et al., 2012). Sheng and Azevedo (2005) and Friedl et al. (2005) found that calculating CV of biomass using the ultimate analysis or the elemental composition gave the highest accuracy and was generally more accurate compared to alternatives. Hence in this study, modified Du Long and Boie formulae are used to calculate the theoretical CV of SBP. This value can then be used to estimate the potential energy content from AD of SBP and compare it with the results obtained in experiments.

2.11.1.3. Nutrient Footprint

According to Hanafiah et al. (2010) the nutrient footprint is defined as the amount of nutrient released that impacts the total GWP of a system or production process, such as agriculture, landfill, wastewater treatment, etc. Halleux et al. (2008), for instance, included nutrient emission in assessing the feasibility of bioethanol production. Hospido et al. (2010) mentioned that nutrient-related direct emissions, such as those from an AD system, can potentially have negative environmental impacts including eutrophication, as well as human and terrestrial toxicity effects from heavy metals on soil. Skjøth et al. (2008) stated that the ammonia footprint has been acknowledged in Europe since 1994, as ammonia (NH_3) releases to the atmosphere contribute significant negative effects on both terrestrial and aquatic ecosystems due to nitrogen

deposition. Hu et al. (2012) suggested that the ability of a waste treatment to recover nutrients as fertilizer is as valuable as its ability to reduce carbon emissions and energy requirements.

2.11.2. Previous Studies on CEN Footprint in AD System

Many studies have been performed to analyse the CEN footprint of AD systems. Bachmaier et al. (2010) calculated the GHG balance on ten agricultural AD plants in Germany using an LCA approach. The selected system boundary covered raw material acquisition, transportation, operation of the AD plant, management of the digested residue, construction of the biogas plant and upstream processes for the supply of electricity, fuel and mineral fertilizer. The results showed that GHG emissions from electricity production in the AD plants ranged from 85 to 251 g CO_{2eq} kWh_{el}^{-1}; which represented a saving in GHG emissions from fossil resources of 573 to 910 g CO_{2eq} kWh_{el}^{-1}. The savings in cumulative energy demand (CED), which represents 'the primary energy demand of all fossil energy carriers that were supplied from outside of the system boundaries', were between 2.3 and 3.2 kWh_{fossil} kWh_{el}^{-1} generated by AD.

A study by Adelt et al. (2011) on biomethane production from energy crops using an LCA method showed that the specific GHG emissions were lower at 44.6 g CO_{2eq} kWh^{-1} than Bachmaier et al. (2010); and reduced the overall GHG emission by 82% compared with natural gas. Capponi et al. (2011) investigated CO_2 emission from AD plants fed with maize silage and co-generating electrical power of 1000 kW_{el}. This resulted in total avoided emissions at 4095 tonnes of CO_2 per year, equivalent to a 35% saving in CO_2 emissions, as well giving savings from avoided use of fertilisers at 1112 tonnes of CO_2 per year. Hermann et al. (2011) concluded that anaerobic digestion has the lowest footprint and the most favourable for biodegradable materials compared to other treatments such as composting or incineration, as AD treatment combines energy recovery with the production of digestate, which can be used as a soil conditioner or biofertiliser.

The above findings confirm that performing a carbon, energy and nutrient balance in an AD system provides benefits not only for estimating the potential energy

produced but also the potential savings that can be obtained or emissions can be avoided.

2.11.3. Application of digestate to land: Regulations

Besides producing renewable energy, AD of biomass also produces digestate which may be rich in organic materials and nutrients such as N, P and K, and can be used as a soil conditioner or bio-fertiliser (Abdullahi et al., 2008; Holm-Nielsen et al., 2009; Tambone et al., 2009). The use of digestate as alternative soil conditioner or biofertiliser can reduce nitrogen losses to groundwater, surface water and the atmosphere and/or minimise the carbon footprint through a reduction in GHG emissions (Al Seadi, 2000). Thus, it is likely to make a beneficial contribution in reducing negative impact on the environment as opposed to the use of synthetic fertilisers (Chen et al., 2012).

Although digestate may contain useful nutrients, it can also contain potentially toxic elements (PTE) (e.g. Cd, Ni, etc.) or pathogens (Matthews, 2001; Vesilind and Spinosa, 2001; Andreoli et al., 2005; da Silva et al., 2005). Therefore, application of digestate to agricultural land is normally regulated to fulfil the requirements of both agricultural best practice and environmental protection. For instance, in the UK, The publicly-available specification (PAS) 110:2010 provides guidance on how to treat, manage and control digestate prior to application to land through a set of standard parameters or processes (British Standards, 2010).

Standards for digested materials in England and Wales are presented in Table 2.6, as a reference to safe utilisation of digestate from AD system as fertiliser and soil conditioner. The EU Nitrates directive (91/676/EEC) established Nitrate Vulnerable Zones (NVZ) which provides guidance and the standard value for applying digestate as biofertiliser, where the maximum application rate for N is 170 kg N ha^{-1} (European Commission, online).

Table 2.6. Parameters and upper limit values for digested materials for land application in England and Wales (PAS 110:2010)

Parameters	Upper limit and units
Pathogens (human and animal indicator species) in WD/SL/SF	
E. coli	1000 CFU g^{-1} WW
Salmonella sp.	Absent in 25 g WW
Potentially Toxic Elements in WD / SL / SF	
Cadmium (Cd)	1.5 mg kg^{-1} DM
Chromium (Cr)	100 mg kg^{-1} DM
Copper (Cu)	200 mg kg^{-1} DM
Lead (Pb)	200 mg kg^{-1} DM
Mercury (Hg)	1.0 mg kg^{-1} DM
Nickel (Ni)	50 mg kg^{-1} DM
Zinc (Zn)	400 mg kg^{-1} DM
Stability of WD / SL / SF	
Volatile Fatty Acids	Screening value: 0.43 g COD g^{-1} VS
Residual Biogas Potential	0.25 l g^{-1} VS
Physical contaminants in WD / SL / SF	
Total glass, metal, plastic and any 'other' non-stone, man-made fragments > 2 mm	0.5 % m m^{-1} DM, of which none are 'sharps'
Stones > 5 mm	8 % m m^{-1} DM
*Nutrient loading**	
N	17 g m^{-2} Total Nitrogen
P	6 g m^{-2} P_2O_5
K	12 g m^{-2} K_2O

Note : WD= whole digestate, SL= supernatant liquid, SF= solid fraction, *Criteria for European Eco-label for soil improvers

Source: British Standards (2010), *Nordberg (1999)

2.12. Conclusions

Several studies have found that SBP, which is highly abundant particularly in the UK, has a great potential as feedstock for renewable energy production in the form of biogas from AD, which then can be converted into heat and electricity or used directly as a fuel with or without gas upgrading to biomethane. The valorisation of SBP through anaerobic digestion technology has other advantages, such providing a source of bio-fertiliser that can be returned to agricultural land, making this a favourable option for dealing with this material.

Experimental studies on anaerobic digestion of SBP indicate that is not necessary to adopt complex two-phase systems to get ultimate biogas and methane yield. Major difficulties encountered when using SBP as a substrate for AD, however, are poor dewaterability of the digestate and the appearance of stable foam in the digesters especially when they are highly loaded and operated at mesophilic temperatures (~37 ^{o}C).

Several studies on anaerobic digestion of various substrates have shown that, although mesophilic digestion is often considered more stable, thermophilic AD may give a higher rate of organic matter degradation, produce more biogas, and offer a shorter retention time and the possibility of feeding at a higher OLR. Other benefits from thermophilic AD may include improved dewatering characteristics and preventing the occurrence of foam. Although foaming is a known problem in SBP digestion, however, there are no published studies on ways of dealing with this in mesophilic or thermophilic conditions.

Foaming can lead to severe operational problems at large scale. Therefore, in utilising AD system it is important to consider and/or modify other elements (i.e. design, operational parameters) or to combine AD with other treatments, which can reduce or eliminate the occurrence of foaming and at the same time can provide stable, efficient, and low cost operation. Experimental studies have also shown that the addition of anti-foaming agents can help to remove foaming, however little information has been found on applications in AD of SBP.

Without an effective dewatering step the logistics of applying the digestate to agricultural land poses a significant economic and environmental challenge due to the costs of digestate transport and the high tonnages involved. The quantities of SBP generated in one place at one time are large enough that digestate will have to be transported over large distances in order to find sufficient area for spreading in accordance with the regulations governing application to land. This difficulty might be reduced or overcome if effective dewatering techniques could be found, or the digestate characteristics improved through process manipulation to allow the use of conventional dewatering technologies. Reducing the water retention properties of the

digestate through process manipulation may also contribute towards minimising the tendency for stable foam formation in the digester itself.

Increasing environmental awareness initiated the current practice of calculating and assessing the CEN footprint of AD operations. This is important to mitigate negative effects on the environment (such as GHG, nitrogen emission), as AD deals with large amounts of carbonaceous organic matter on a daily basis. At the same time, performing an energy footprint analysis can add benefits in evaluating and selecting appropriate options for pre- and post-treatment in AD systems, in terms of the potential for renewable energy (electricity/heat) produced, the reduction of fossil fuels used, and the reduction of CO_2 emissions.

The research thus focuses not only on optimising the biogas production from SBP but also on minimising the difficulties arising in single-stage CSTR systems, particularly in mesophilic conditions, by improving the digestate dewaterability and reduce foaming occurrence. From the literature it appears that single-stage thermophilic AD of SBP may have the potential to provide an enhanced anaerobic digestion process and dewaterability characteristics, and for foam control; however further research is required to confirm this and identify the key factors and reasons for any improvement. In addition, a CEN footprint analysis should be performed to determine the most appropriate scenarios for the AD of SBP, under both mesophilic and thermophilic conditions.

The following chapters of this thesis address these points through a series of laboratory experiments and discussion of the results obtained.

CHAPTER 3. MATERIALS AND METHODS

3.1. Materials

3.1.1. Feedstocks

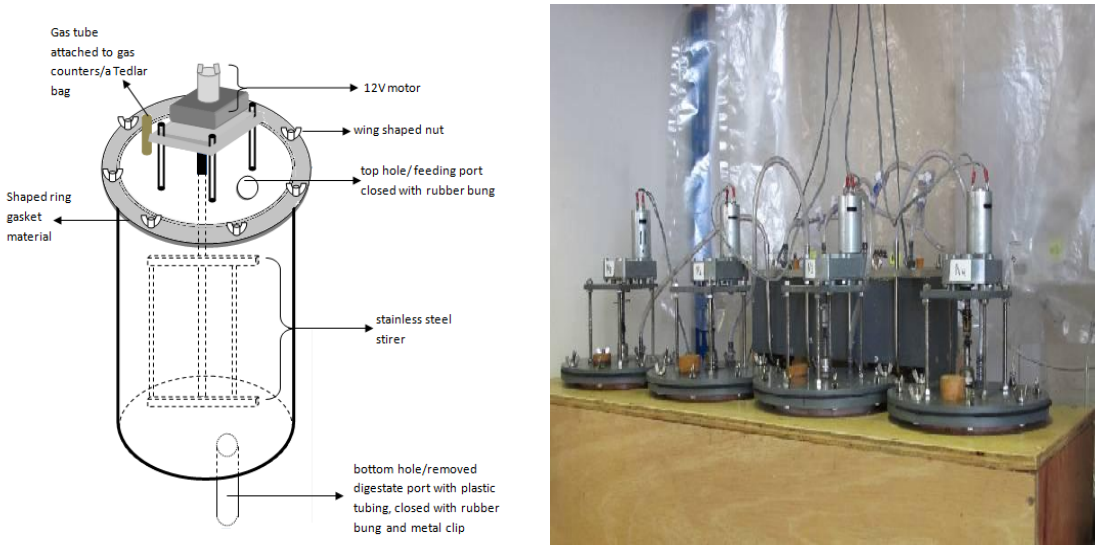
Sugar beet pulp was collected fresh from British Sugar's Wissington Factory, Kings Lynn, UK. It was then packed into 4-litre containers and frozen at approximately -20 ^{o}C. Before feeding to the digesters the material was thawed for at least 24 hours at room temperature, then stored in a refrigerator at ~4 ^{o}C for use within a few days.

3.1.2. Digester inoculums

The inoculum for the mesophilic digesters was prepared by mixing 1 part of digestate taken from an anaerobic digester treating sugar beet pulp (British Sugar, Wissington, UK) with 1 part of digestate taken from a mesophilic digester treating municipal wastewater biosolids (Millbrook Wastewater Treatment Works, Southampton, UK). The thermophilic digesters used an inoculum taken from this mesophilic municipal wastewater biosolids digester, which was then acclimated to thermophilic conditions.

3.1.3. Digester design

The digesters used were stirred tank reactors (STR). These were constructed of PVC tube with gas-tight top and bottom plates (see Figure 3.1). The top plate was fitted with a gas outlet, a feed port sealed with a rubber bung, and a draught-tube liquid seal through which an asymmetric bar stirrer was inserted with a 40 rpm motor mounted directly on the top plate. Temperature was maintained by circulating water through an external heating coil, at 37 ^{o}C ± 0.5 ^{o}C (for mesophilic) or at 55 ^{o}C ± 0.5 ^{o}C (for thermophilic), depending on the study. The digesters were connected to tipping-bucket gas counters with continuous data logging. Calibration of gas counters was checked weekly by collecting the gas in a Tedlar bag (SKC Ltd, Blandford Forum, UK). The volume and composition was then measured accurately using the procedure described in the section on gas analysis (section 3.2.3). Semi-continuous operation was achieved by removing digestate through an outlet port in the base plate before adding feed via the hole in the top plate. Digesters of two different volumes were used depending on the study: one set had a 5-litre capacity and were operated at 4-litre working volume (unless noted), and the second set had a 2-litre capacity and were operated with a 1-litre working volume.



(a) (b)

Figure 3.1. Drawing (a) and photo (b) of the 5 litre anaerobic digesters

3.1.4. Trace element solution

The two trace element (TE) solutions used, one composed of cations and the other oxyanions (see Table 3.1.) were based on a modified TE recipe developed by University of Southampton. TE were supplemented by weekly addition of the two solutions either at a rate of 1 ml of each solution for every 1 litre of digestate removed or based on the amount of feedstock added to give a steady state minimum concentration of TE in the digester.

Table 3.1. Concentration of trace elements in stock solution

Trace element as	Compound used	Element concentration in the working condition (after diluted by 1000 times) (mg l^{-1})	Compound concentration in stock solution, (g l^{-1})
Cation			
Aluminium (Al)	$AlCl_3 \cdot 6H_2O$	0.1	0.895
Boron (B)	H_3BO_3	0.1	0.572
Cobalt (Co)	$CoCl_2 \cdot 6H_2O$	1.0	4.038
Copper (Cu)	$CuCl_2 \cdot 2H_2O$	0.1	0.268
Iron (Fe)	$FeCl_2 \cdot 4H_2O$	10.0	35.597
Manganese (Mn)	$MnCl_2 \cdot 4H_2O$	1.0	3.602
Nickel (Ni)	$NiCl_2 \cdot 6H_2O$	1.0	4.050
Zinc (Zn)	$ZnCl_2$	1.0	2.084
Oxyanion			
Molybdenum (Mo)	$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$	0.1	0.184
Selenium (Se)	Na_2SeO_3	0.1	0.219
Tungsten (W)	$Na_2WO_4 \cdot 2H_2O$	0.1	0.179

3.2. Analytical Methods

3.2.1. General

Reagents

Except where otherwise stated all chemicals used were of laboratory grade and were obtained from Fisher Scientific (Loughborough, UK).

Water

Solutions and standards were prepared using ultra-pure deionised water (DI water) obtained from a Barnstead Nanopure ultrapure water purification system (Thermo Scientific, UK).

Diluted feedstocks for digester experiments were prepared using tap water.

Laboratory practice

All laboratory operations were carried out using good laboratory practice, having first carried out the appropriate risk assessments and, where necessary, COSSH assessments. Equipment, laboratory apparatus, and analytical instruments were operated in accordance with the manufacturers' instructions. All glassware was washed using washing detergent followed by rinsing with tap water and deionised water. The glassware used for acid digestion was soaked in a 10% nitric acid bath for a 24-hour period after which the glassware was rinsed with Milli-Q water.

3.2.2. Gravimetric Analysis

3.2.2.1. Total Solid and Volatile Solid (TS/VS)

TS and VS determination was based on Standard Method 2540 G (APHA, 2005). After thorough agitation, approximately 10 g of sample was transferred into a weighed crucible by pipetting (digestate samples) or spatula (substrate samples). Samples were weighed to an accuracy of 10±0.001 g (Sartorius LC6215 balance, Sartorius AG, Gottingen Germany) and placed in an oven (LTE Scientific Ltd., Oldham UK) for drying overnight at 105 ^{o}C ± 1 ^{o}C. After drying the samples were transferred to a desiccator to cool for at least 40 minutes. Samples were then weighed again with the same balance, transferred to a muffle furnace (Carbolite Furnace 201, Carbolite UK, Hope Valley UK) and heated to 550 ^{o}C ± 10 ^{o}C for two hours. After this ashing step, samples were again cooled in a desiccator for at least one hour before weighing a third time.

After all analyses, crucibles were washed with detergent, rinsed with deionised water, and stored in an oven until required for the next analysis. Crucibles were transferred from the oven to a desiccator for cooling to room temperature before each analysis. Total and volatile solids were calculated according to the following formulae:

$$\% TS = \frac{W_3 - W_1}{W_2 - W_1} x\,100 \qquad [3.1]$$

$$\% VS\ (based\ on\ total\ \ weight) = \frac{W_3 - W_4}{W_2 - W_1} x\,100 \qquad [3.2]$$

$$\% VS\ (based\ on\ total\ \ solid) = \frac{W_3 - W_4}{W_3 - W_1} x\,100 \qquad [3.3]$$

Where,
W_1 is the weight of the empty crucible
W_2 is the weight of the crucible containing fresh sample
W_3 is the weight of the crucible and sample after drying at 105 ^oC
W_4 is the weight of the crucible and sample after heating to 550 ^oC

3.2.3. Chemical and electrochemical analysis

3.2.3.1. pH

pH was measured using a Jenway 3010 meter (Bibby Scientific Ltd, UK) with a combination glass electrode, calibrated in buffers at pH 7 and 9.2. The pH meter was temperature-adjusted and had a sensitivity of ± 0.01 pH unit and accuracy of 0.01 ± 0.005 pH units. Buffer solutions used for calibration were prepared from pH 7 and 9 buffer tablets (Fisher Scientific, UK) according to the supplier's instructions. During measurements, the sample was stirred to ensure homogeneity. The pH probe was rinsed with DI water in between measurements and placed into a mild acid solution to avoid cross-contamination. Digestate samples were measured immediately after sampling to prevent changes in pH due to the loss of dissolved CO_2.

3.2.3.2. Alkalinity

Alkalinity measurement was based on Standard Method 2320B (APHA, 2005). Digestate was sieved to obtain a homogenous sample and 2-5 g of this was made to a volume of 40 ml with DI water. Titration was carried out using an automatic digital titration burette system (Schott Titroline, Germany), with the samples magnetically

stirred during titration. A 0.25 N H_2SO_4 titrant was used to determine endpoints of pH 5.7, 4.3 and 4.0, allowing calculation of total (TA), partial (PA) and intermediate alkalinity (IA) (Ripley *et al.*, 1986). PA is a measurement of bicarbonate buffering while IA is attributed to the buffering capacity of Volatile Fatty Acids (VFA).

The pH probe was calibrated before titration using buffers as described in section 3.2.3.1 and washed with DI water between subsequent samples to avoid cross contamination. Alkalinity was calculated according to equations [3.4-3.6]:

$$TA = \frac{(V_{4.0} + V_{4.3} + V_{5.7})\ x\ N\ x\ 50000}{V} \quad [3.4]$$

$$PA = \frac{V_{5.7}\ x\ N\ x\ 50000}{V} \quad [3.5]$$

$$IA = \frac{V_{4.3}\ x\ N\ x\ 50000}{V} \quad [3.6]$$

Where,
TA is the total alkalinity (mg $CaCO_3$ l^{-1})
PA is the partial alkalinity or bicarbonate alkalinity (mg $CaCO_3$ l^{-1})
IA is the intermediate alkalinity or volatile fatty acid alkalinity (mg $CaCO_3$ l^{-1})
N is the normality of H_2SO_4
V is the volume of sample (ml), based on the assumption that 1 g of sample = 1 ml

3.2.3.3. Total Ammonia Nitrogen

Total ammonia nitrogen (TAN) analysis was based on Standard Method 4500-NH3 B and C (APHA, 2005). A sample aliquot of between 2-3 g was weighed (i201, My Weigh Europe, Huckelhoven Germany) into a digestion tube and the volume made up to 50 ml with DI water. Blanks (50 ml DI water) and standards (containing 10 ml of 1000 mg l^{-1} NH_4Cl with 40 ml DI water) were also prepared in digestion tubes. 5 ml of 10 M sodium hydroxide (NaOH) was added to each digestion tube to raise the pH above 9.5 and the samples were distilled using either a Foss Tecator Kjeltec system 1002 distillation unit (Foss Tecator A-B, Hoganas, Sweden) or a Büchi distillation unit K-350 (Büchi, UK). Erlenmeyer flasks previously filled with 25 ml of boric acid as an indicator were used to collect the distillate. The distillate was titrated manually with H_2SO_4 (0.25N) using a digital titration burette system (Schott Titroline, Germany) until an endpoint was reached as indicated by a colour change from green to purple, at which

point the volume of H_2SO_4 added was recorded. The percentage of nitrogen in sample was calculated using equation [3.7]:

$$NH_3 - N = \frac{(A-B)\ x\ 14.0\ \ x\ 0.25\ x\ 100}{V_{sample}} \qquad [3.7]$$

Where,
A is the volume of 0.25 N H_2SO_4 used to titrate the sample (ml)
B is the volume of 0.25 N H_2SO_4 used to titrate the blank (ml)
V_{sample} is the volume of sample (ml)

3.2.3.4. Total Kjeldhal Nitrogen

Total Kjeldhal Nitrogen (TKN) analysis was carried out on duplicate samples alongside blanks and controls as follows: 3-5 g of fresh sample or 0.1-1 g 1 mg of dry sample was placed into a glass digestion tube. Two Kjeltab Cu 3.5 catalyst tablets were added to facilitate acid digestion by lowering the activation energy of the reaction. 12 ml of concentrated H_2SO_4 was carefully added to each digestion tube, and the tubes were gently agitated to ensure that the entire sample was completely exposed to acid. The digestion tubes were then placed in a heating block with an exhaust system using either a Foss Tecator 1007 Digestion System 6 (Foss Analytical, Hoganas, Sweden) or a Büchi distillation unit K-350 (Büchi, UK), for approximately two hours until the solution colour became a clear blue-green. Both systems operated at 420 $^oC \pm 5\ ^oC$, and once the reaction was completed the tubes were cooled to around 50 ^oC then 40 ml of DI water was slowly added to the digestion tube to prevent crystallization. Each sample, blank and standard was then distilled and titrated as described in section 3.2.3.3. The percentage of nitrogen was calculated according to equation [3.8]:

$$\%\ N = \frac{(A-B)\ x\ 14.0\ \ x\ 0.25\ x\ 100}{m} \qquad [3.8]$$

Where,
A is the volume of 0.25 N H_2SO_4 used to titrate the sample (ml)
B is the volume of 0.25 N H_2SO_4 used to titrate the blank (ml)
m is the mass of the original sample (mg)

3.2.3.5. Gas Chromatograph (GC) determination of volatile fatty acid (VFA)

The method used was based on SCA (1979): Determination of Volatile Fatty Acids in Sewage sludge. Samples were prepared for analysis by centrifugation at 14,000 g (micro-centrifuge, various manufacturers) for 15 minutes. 0.9 ml of the supernatant was transferred by pipette to vials with 0.1 ml formic acid, to give a final formic acid concentration of 10%. Where dilution was necessary, deionised water was used and formic acid was added to give a concentration of 10% of the total volume for analysis. If the samples were turbid at this point they were centrifuged again at 14,000 rpm to obtain a clearer supernatant. The supernatant after acidification and centrifugation was transferred into the vials and loaded onto the GC autosampler ready for the VFA measurement. A standard solution containing acetic, propionic, iso-butyric, n-butyric, iso-valeric, valeric, hexanoic and heptanoic acids, at three dilutions to give individual acid concentrations of 50, 250 and 500 mg l^{-1} respectively, was used for calibration and also loaded onto the GC.

Quantification of VFA was by a Shimazdu GC-2010 gas chromatograph (Shimadzu, Milton Keynes, UK), using a flame ionization detector and a capillary column type SGE BP-21. The carrier gas was helium at a flow of 190.8 ml min^{-1} and a split ratio of 100 to give 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 minutes with a final hold time of 3 minutes. The temperatures of injector and detector were 200 and 250 ^{o}C, respectively.

3.2.3.6. Trace Element Analysis

Analysis was carried out using duplicate samples and blanks. Samples of digestate and SBP were air dried to constant weight and then homogenised by grinding in a centrifugal grinder Glen Creston type ZM1 (Glen Creston, Stanmore, UK). Approximately 1.5 g of sample was place in a test tube, and 15 ml HCl was added. After ~5 minutes 5 ml HNO_3 was added, and the tubes were agitated to mix the contents. The tubes were then placed into a digestion block (Gerhardt Kjeldatherm, Germany) and connected to the condenser system and left for 24 hours prior to heating. The acid digestion involved gradually increasing the temperature first to 100 ^{o}C and then to the final temperature of ~180 ^{o}C which was maintained for about 2 hours ± 10 min. After cooling, the mixtures were filtered (Filter paper Whatman No. 1 Qualitative 11 cm) into

a 50-ml volumetric flask. Any remaining residue in the tube was washed out with ~5 ml of warm Nitric acid (HNO_3) (12.5% v/v) and transferred to the 50 ml flask: up to 5 washes were performed. The volume was then made up to 50 ml with HNO_3 (12.5% v/v). The filtrate was transferred into a PET plastic bottle and sent for analysis using ICP-MS (Severn Trent Laboratory Limited, UK).

3.2.3.7. Fibre analysis

Neutral detergent fibre (NDF), Acid detergent fibre (ADF) and Acid detergent lignin (ADL) were analysed using the Fibertec™ 2021/2023 FiberCap™ system (FOSS Analytical AB, Sweden) according to the manufacturer's instructions with modifications adapted from Goering and Van Soest (1970) and Kitcherside et al. (2000). These acid solubilisation and gravimetric analysis were used for determination of cellulose, hemicellulose and lignin of SBP. The calculation formulae are as follows:

$$NDF = \frac{W_3\,(W_1\,x\,C) - (W_5 - W_4 - D)}{W_2}\,x\,100 \qquad [3.9]$$

$$ADF = \frac{(W_0 - W_t)\,x\,100}{S} \qquad [3.10]$$

$$ADL = \frac{(L\,x\,100)}{S} \qquad [3.11]$$

Where,
W_o is weight of oven dry crucible including fibre
W_t is tared weight of oven dry crucible
W_1 is initial capsule weight (mg)
W_2 is sample weight (mg)
W_3 is capsule + residue weight (mg)
W_4 is empty ashing crucible
W_5 is total ash (mg)
C is blank correction for capsule solubility
D is capsule ash (mg)
L is loss upon ignition after 72 % H_2SO_4 treatment
S is oven dry sample weight

3.2.3.8. Elemental Composition

C, H, and N were analysed using a FlashEA 1112 Elemental Analyser (Thermo Finnigan, Italy) according to the manufacturer's instructions. The standards used in this analysis were methionine, nicotinamide and birch leaf.

3.2.3.9. Calorific value (CV)

CV was measured using a ballistic bomb calorimeter (CAL2k, Digital Data Systems Ltd, South Africa) according to the manufacturer's instructions. Benzoic acid was used as a standard, with a higher heating value (HHV) of 26.454 kJ g^{-1}. Theoretical CV was calculated from the elemental composition assuming 0.5% S content, using the Du Long equation according to the method in Combustion File 24 (IFRF, 2013a) and the Boie equation (IFRF, 2013b). The equations of Du Long and the Boie are given in Chapter 2.

3.2.3.10. Sugar analysis

Sugar analysis was conducted for the non-cellular light fraction (liquid) resulting from the centrifugation of digestate from AD of SBP. Mono-sugars were analysed using a Dionex HPLC (DX500-Sys1) in accordance with the manufacturer's instructions (*HPAEC – PAD).* Samples for sugars determination were placed on ice as soon as they were taken, and if not analysed immediately were frozen. Before analysis both fresh and defrosted samples were centrifuged at 13000 *g* for 7 minutes in a Galaxy 16DH centrifuge (VWR, UK). The supernatant was diluted and placed in a 5 ml sample vial with a 0.45 µm nylon filter cap. Sugar analysis was carried out on a Dionex DX-500 system using a method adapted from that of Davis (1998). In this glucose, xylose, galactose, arabinose, mannose and cellobiose were separated at 30 ^{o}C on a CarboPac PA1 column (250 x 4 mm) in combination with a CarboPac guard column (25 x 4 mm) (Dionex, Sunnyvale, CA, USA). The mobile phase components were 200 mM sodium hydroxide (A), distilled water (B) and 170 mM sodium acetate in 200 mM sodium hydroxide (C). The system set up used a 2.5 µL sample loop and 300mM sodium hydroxide post column eluent at a pressure of 2.76 bar to aid sugar detection.

3.2.3.11. COD

COD was measured by the closed tube reflux method with titrometric determination of the end point (Environment Agency, 2007). If the sample COD was more than 400 mg l^{-1} pre-dilution was carried out. 2 ml of sample (or 2 ml deionised water for blanks) was

placed into the reflux tubes followed by the addition of 3.8 ml of FICODOX-plus reagent (Fisher Scientific Ltd, UK), the composition of which is shown in Table 3.2. The tube was sealed with a PTFE screw cap and the mixture refluxed at 150 ^{o}C for 2 hours. After cooling, a few drops of ferroin indicator (Table 3.3) were added (Fisher Scientific Ltd, UK) and the mixture titrated with acidified (2% Sulphuric acid) 0.025N ferrous ammonium sulphate solution, the normality of which was calculated using equation [3.12]. The end point was a colour change from blue to red. The COD value of the sample was calculated using equation [3.13]. Dilutions of a standard solution containing 3.8 g l^{-1} of potassium hydrogen phthalate with a COD of 4 g COD l^{-1} were used as a standard to check calculated values of COD.

$$Normality_{FAS} = \frac{0.12887}{Titrant\ volume_{FAS\ Std}} \quad [3.12]$$

$$COD\,(mgO_2\,l^{-1}) = \frac{8000\,x\,(Titrant\ volume_{Blank} - Titrant\ volume_{Sample})\,x\,Normality_{FAS}}{2\,x\,Dilution} \quad [3.13]$$

Table 3.2. 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 3.3. Ferroin Indicator Composition

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

3.2.3.12. Potassium

Samples of SBP and digestate were prepared for potassium (K) analysis by acid digestion as described in section 3.2.3.6. Calibration solutions were prepared from a

stock solution for atomic spectroscopy of potassium standard at 1000 mg l^{-1} in HNO_3 (Fisher Scientific, UK). Cesium Chloride (CsCl) solution was prepared and added to eliminate any interference during potassium measurement. 1.0 ml of sample was transferred into a 100 ml volumetric flask and 20 ml of Cesium Chloride solution was added, then the volume was made up to 100 ml with 12.5% (v/v) of HNO_3. The amount of sample and Cesium Chloride solution, if necessary, was adjusted according to the dilution factor used. The potassium was measured using a Spectr AA-200 Atomic Absorption Spectrometer (Varian, Australia) operated according to the manufacturer's instructions, with a hollow cathode lamp. The wave length used was 766.5 nm with a slit setting of 1.0 nm. The sample concentration was calculated using equation [3.14]:

$$Concentration\ (mg\ kg^{-1}\ TS) = \frac{\beta_1\ x\ V}{1000\ x\ m}\ x\ DF \qquad [3.14]$$

Where,
β_1 is the concentration of potassium (ppm)
m is the mass of sample (g TS)
V is total volume (ml)
DF is dilution factor

3.2.3.13. Phosphorus

SBP and digestate samples were prepared for phosphorus analysis by acid digestion as described in section 3.2.3.6. The phosphorus content of the digested sample was determined using a UV-Visible scanning spectrophotometer (Cecil 3000 series, Cecil Instruments). 2.5 ml of the acid digested sample was added to a 10 ml volumetric flask and a drop of phenolphthalein was added; this was followed by addition of several drops of 40% sodium hydroxide to produce a colour change to pink. When a pink colour was achieved 12.5% nitric acid was added to discharge the colour; sodium hydroxide (1M) was again added to reintroduce the pink colour followed by 1 drop of 0.1M nitric acid to discharge the colour. After all of the additions the solution was made up to 10 ml with DI water and from this a suitable dilution for the determination was made. The samples were measured against standards prepared at concentrations of 0.25, 0.5, 0.75 and 1.0 mg P l^{-1}. To all samples and standards 1.5 ml of colour reagent was added and left for

20 minutes to allow colour formation. The composition of the colour reagent is given in Table 3.4.

Table 3.4. Composition of colour reagent for phosphorus determination

Component	Quantity (ml)
Sulphuric acid 2.5M	25
Potassium antimonyl	2.5
Ammonium molybdate	7.5
Ascorbic acid solution	15

The UV spectrophotometer was used at a wavelength of 880 nm and sample concentration was determined against a calibration graph using equation [3.15]:

$$Concentration = \frac{Absorbance\ x\ Dilution\ factor}{Slope\ of\ calibration\ curve} \quad [3.15]$$

3.2.3.14. Turbidity Analysis

Turbidity was measured according to Bruus et al. (1992) with a modification to the centrifuge speed of 21475 *g* in a Sorvall Legend XTR Centrifuge (Fisher Scientific Ltd, UK) equipped with a rotor (Fiberlite F15-6x100) (Fisher Scientific Ltd., UK). The turbidity of the supernatant was measured as absorption at a wavelength of 650 nm using a spectrophotometer (Cecil 3000 series, Cecil Instruments Ltd., UK). Measured turbidity was expressed in nephelometric turbidity units (NTU) (APHA, 2005).

3.2.3.15. Conductivity

This was measured according to Standard Method 2510 using a LF330 conductivity meter (WTW, Germany). Readings were measured in mS cm^{-1}.

3.2.4. Gas Analysis

3.2.4.1. Gas Composition

Biogas composition was quantified using a Varian Star 3400 CX gas chromatograph (GC), (Varian Ltd, Oxford, UK). The device was fitted with a Hayesep C column and used either argon or helium as the carrier gas at a flow of 50 ml min^{-1} with a thermal conductivity detector. The biogas composition was compared with a standard gas

containing 65 % CH_4 and 35% CO_2 (v/v) for calibration. A sample of 10 ml was taken from a Tedlar bag used for sample collection and was injected into a gas sampling loop.

3.2.4.2. Gas Volume

Biogas volume was measured using a weight-type water displacement gasometer (Walker et al., 2009). The measurement procedure was as follows: the initial height of solution in the gasometer (h_1) was recorded before the gas collected with the Tedlar bag was introduced into the column through the top valve. After the bag was empty, the final height (h_2) and the weight of water (m) were recorded, as well as the temperature (T) and pressure (P) in the room, and the measurement time. All gas volumes reported are corrected to standard temperature and pressure of 0 ^{o}C, 101.325 kPa as described by Walker et al. (2009) using equations [3.16-3.17] below:

a. Height Gasometer Governing Equation

$$V_{stp} = \frac{T_{stp} A}{T_{atm} p_{stp}} \left(\left(p_{atm} - p_{H_2O}(T_{atm}) - \rho_b g \left(h_{t2} - h_{c2} \right) \right) h_{c2} - \left(p_{atm} - p_{H_2O}(T_{atm}) - \rho_b g \left(h_{t1} - h_{c1} \right) \right) h_{c1} \right) \quad [3.16]$$

b. Weight Gasometer Governing Equation

$$V_{stp} = \frac{T_{stp} A}{T_{atm} p_{stp}} \left[\left(\left(P_{atn} - p_{H_2O}(T_{atm}) + \rho_b g \left(H - h_1 - \frac{m_b}{A \rho_b} \right) \right) \left(h_1 + \frac{m_b}{A \rho_b} \right) \right) - \left(p_{atn} - p_{H_2O}(T_{atm}) + \rho_b g \left(H - h_1 \right) \right) h_1 \right] \quad [3.17]$$

Where,
V is volume (m^3)
P is pressure (Pa)
T is temperature (K)
H is total height of column (m)
h is distance to liquid surface from a datum (m)
A is X-section of gasometer (m^2)
m_b is mass of barrier solution (kg)
ρ is density (kg m^{-3})
g is gravitational acceleration (m s^{-2})
1, 2, stp, atm, b, t, c is subscripts refer to condition 1, condition 2, standard temperature and pressure, atmospheric, barrier solution, trough and column respectively.

c. Gas volume in serum bottle test

For measuring gas volumes in a serum bottle test, the pressure reading is converted to gas volume in the headspace at STP using the ideal gas law, as shown in equation [3.18]:

$$PV = nRT \qquad [3.18]$$

Where,
P is the pressure of the gas (kPa)
V is the volume of the gas (which in this case is the fixed headspace volume) (ml)
n is the amount of substance of gas (moles)
T is the temperature of the gas (^{o}C)
R is the ideal gas constant (8.314 J·K^{-1}·mol^{-1})

The corrected pressure (P in the formula above) for water vapour pressure is obtained using the Goff-Gratch equation [3.19]:

$$\begin{aligned} Log_{10}e_w = {} & -7.90298\,(373.16/T - 1) + 5.02808\,Log_{10}(373.16/T) \\ & - 1.3816\,x10^{-7}\,(10^{11.344\ (1-T/373.16)} - 1) + 8.1328\,x\,10^{-3}(10^{-3.49149\ (373.16/T-1)} - 1) \\ & + Log_{10}\,(1013.246) \end{aligned} \qquad [3.19]$$

Where,
e_w is the saturation water vapour pressure (kPa)
T is the absolute air temperature in (K)

3.2.5. Dewaterability Characteristics Analysis

Digestate dewaterability characteristics were analysed using the following tests:

3.2.5.1. Capillary Suction Time (CST) Test

The CST test was carried out using a Triton-WRPL type 130 single CST, a Triton type 319 Multi CST apparatus and paper (Triton Electronics Ltd, UK). 5 ml of digestate was poured into the sample tube, which is pressed down on a piece of CST filter paper placed on the lower perspex block of the apparatus. Two electrodes placed at a fixed distance from the sample tube detect the presence of water in the CST filter paper. The CST is the time taken for the water to travel through the paper from the first to the second electrodes. The time interval depends on the resistance of the digestate material to giving up its water (Scholz, 2005). A digestate with a CST lower than 10 s is

considered to have a good dewaterability (USEPA, 1987) and consistent with 20 s elsewhere (IWPC, 1981).

3.2.5.2. Filtration Test

The filtration test used a Büchner Funnel (diameter 7 cm) with a hardened ashless filter paper (Whatman 540). 100 g of digestate was poured into the Büchner funnel and filtered with a vacuum pump at 10 kPa for 10 minutes until the surface of the cake was visible, at which point the vacuum pressure was increased to 50 kPa for 5 minutes followed by 100 kPa for 5 minutes, with the weight of filtrate recorded at the end of each period. The results were reported on a volume basis assuming a density of 1 kg l^{-1} for digestate.

3.2.5.3. Frozen Image Centrifuge (FIC) Test

The FIC test was carried out using a Triton WRC model I6I centrifuge (Triton Electronics Ltd, UK) at 10 – 100 *g*, with supernatant height recorded against time. The time observations ranged from 10 min to 1 hour. This test uses a stroboscopic technique in which a 'frozen image' of the sample is generated that allows changes in the solid liquid interface to be observed and measured in real time without stopping the centrifuge. The mechanism operates by matching the frequency of the strobe light to the rotor speed of the centrifuge (see Figure 3.2.).

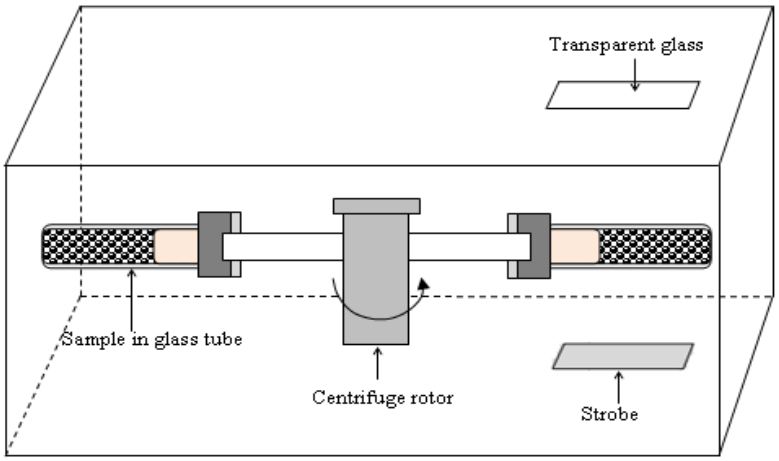


Figure 3.2. Visualisation of FIC test

3.2.5.4. Foaming potential test

Foaming potential was determined based on the aeration foaming potential test (Zábranská et al., 2002), in which 100 ml of digestate was sparged with an air flow rate of 1 l min^{-1} for 5 minutes. The foam height was then recorded every minute until the foam subsided or for 33 min, and the result calculated using equation [3.20]. Foaming tendency, foam stability and foaming propensity were calculated based on Ganidi et al. (2011) and Zábranská et al. (2002), as shown in equations [3.21-3.23]:

$$Foaming\ Potential = \frac{foam\ volume\ after\ 33\ minutes\ (ml)}{digestate\ volume\ (ml)} \quad [3.20]$$

$$Foaming\ Tendency = \frac{foam\ volume\ produce\ (ml)}{air\ flow\ rate\ (ml\ min^{-1})} \quad [3.21]$$

$$Foam\ Stability = \frac{foam\ volume\ after\ 33\ minutes\ (ml)}{air\ flow\ rate\ (ml\ min^{-1})} \quad [3.22]$$

$$Foaming\ Propensity = \frac{level\ of\ foam\ volume\ after\ aeration\ (mm)}{weight\ of\ digestate\ (g\ TS)} \quad [3.23]$$

3.2.6. Scanning Electron Microscopy

Samples were observed using an FEI Quanta 200 Scanning Electron Microscope (SEM). The procedure for sample preparation was as follows: samples were placed into fixative solution (3% glutaraldehyde and 4% formaldehyde in 0.1 M PIPES buffer pH 7.2) to preserve and maintain their original structure. The sample was rinsed in buffer solution **(**0.1 M PIPES**)** initially for 1 hour and then again for 10 minutes. This was followed by multiple rinsing in ethanol solutions according to the following order: 30%, 50%, 70%, 95% ethanol for 10 minutes each and then absolute ethanol for 20 minutes. The specimen was dried using a critical point drier (Balzers - CPD 030) for 20 minutes in order to preserve its initial structure without damage. The specimen was then placed on a stub and coated using a SEM E 5100 coating unit sputter coater (Polaron Equipment Ltd, UK) for 5 minutes, followed by mounting on an aluminium stub which was coated with gold palladium. Finally, the specimen was placed into the SEM to examine the structure and record this as a photomicrograph.

3.3. Biochemical Methane Potential (BMP)

The BMP test in this work was performed in 550 ml sealed bottles placed in a temperature controlled water bath at 37 ^{o}C. The inoculum-to-substrate (I/S) ratio used was 4:1 on a VS basis and the test was run over a period of 28 days. No supplements were added, as the inoculum used was known to be sufficiently rich in the required nutrients. Biogas was collected in perspex cylinders by displacement of a 75% saturated sodium chloride solution acidified to pH 2, in order to reduce losses of methane by dissolution. The height of the solution in the collection cylinder was recorded manually for a certain interval on a daily basis. Vapour pressure and salt solution density were taken into account in correction of gas volumes to a standard temperature and pressure (STP) of 0 ^{o}C and 101.325 kPa (Walker et al., 2009). Samples for gas composition analysis were taken from the cylinders each time they were refilled, at intervals of no more than 7 days to avoid the risk of overfilling or losses of methane. The bottles were shaken each day before the gas level measurement was taken to provide mixing. Samples were run alongside blanks (inoculums only) and positive controls (cellulose powder from Sigma-Aldrich, Dorset-UK), all in triplicate. TS/VS, pH, and VFA were measured at the beginning and end of the BMP test.

3.4. Anaerobic digestion trials on SBP

3.4.1. Digester operational parameters

The digesters were operated in a semi continuous mode i.e. fed daily with a specific amount of feedstock and digestate removed to maintain a constant volume in the digesters. The organic loading rate (OLR) was determined according to equation [3.24]:

$$OLR = \frac{m \ x \ VS_{substrate}}{V_{reactor}} \qquad [3.24]$$

Where,
m is the mass of substrate daily added to the reactor (g day^{-1})
$VS_{substrate}$ is the volatile solid content of feedstock (% WW)
$V_{reactor}$ is the volume of reactor (l)

The Hydraulic Retention Time (HRT) of the digester is expressed in equation [3.25]:

$$HRT = \frac{V_{reactor}}{Q} \quad [3.25]$$

Where:
$V_{reactor}$ is the working volume of each reactor (ml)
Q is the daily flow of material (substrate added and digestate removed) through the reactor (ml day^{-1})

Amounts of substrate and digestate were measured on a weight basis and it was assumed that both the substrate and digestate had a specific gravity of 1.0. The performance of bioreactors was monitored in terms of specific biogas and methane production and VS destruction which were calculated using equations [3.26] and [3.27].

$$Specific\ biogas\ production = \frac{V_{biogas}}{OLR\ x\ V_{reactor}} \quad [3.26]$$

$$Specific\ methane\ production = \frac{V_{CH4}}{OLR\ x\ V_{reactor}} \quad [3.27]$$

Where,
V_{biogas} is the volume of biogas produced daily (l day^{-1})
V_{CH4} is the volume of methane produced daily (l day^{-1})
OLR is the organic loading rate (g VS l^{-1} day^{-1})
$V_{reactor}$ is the volume of reactor (l)

VS destruction was calculated on a weekly basis using a mass balance approach based on the mass and VS content of the feed added, the VS of the digestate removed, and the mass of biogas produced in the digester (equation [3.28]). The mass of biogas removed was calculated from the average gas volume (after deduction of the calculated volume of water vapour), assuming a composition of 50% CH_4 and 50% CO_2 (i.e. other gases were not considered) with the weight of 1 mole of biogas taken as 1.34 g l^{-1}.

$$VS\ destruction = \frac{VS_{SBP\ in}\ x\ Mass_{SBP\ in} - VS_{digestate out}\ x\ (Mass_{SBP\ in} - Mass_{biogas out})}{VS_{SBP\ in}\ x\ Mass_{SBP\ in}} x100 \quad [3.28]$$

Where,
VS $_{SBP\ in}$ is SBP added to the digester (g VS kg^{-1} WW)
VS $_{SBP\ out}$ is digestate removed from digester (g VS kg^{-1} WW)
VS $_{Biogas\ out}$ is biogas removed from digester (g VS kg^{-1} WW)

3.5. Digester experimental runs

In all studies the digesters were monitored for biogas and methane production, dewaterability characteristics and foaming occurrence. Their stability and performance assessed by reference to stable digesters in both mesophilic and thermophilic conditions.

3.5.1. Kinetic study under mesophilic conditions and influence of trace elements

The study used 8 x 5 litre digesters (N1 to N8) run as pairs and operated over at least three hydraulic retention times (HRT) at each applied organic loading rate (OLR) (see Table 3.5). A further pair of digesters (N9 and N10) was operated at an OLR of 3 g VS l^{-1} day^{-1} and received a trace element (TE) addition at a rate of 1 ml of each solution (section 3.1.4) for every 1 litre of digestate removed on a weekly basis. All the other digesters had no TE addition.

Table 3.5. Operational parameters for AD of SBP

Reactor ID	OLR (g VS l^{-1} day^{-1})	Working volume (l)	3HRT (days)
N_1 and N_2	2	4	411
N_3 and N_4	3	4	274
N_5 and N_6	4	4*	206
N_7 and N_8	5	4*	165
N_9 and N_{10}	3 with TE addition	3	274

Note: * starting on day 1 to 164 then reduced to 3-litre working volume due to foaming problem

3.5.2. Digestion of SBP with water dilution

This used two of the digesters (N5 and N6) from the previous experiment which had been operated at a working volume of 3 litres and an OLR of 4 g VS l^{-1} day^{-1}. Operation of these digesters continued for a further 123 days with the SBP feedstock diluted 1:1 with tap water on a mass basis. On a daily basis, the digesters were fed with 44 g WW of SBP, 44 g tap water and 0.044 ml (44 µl) of the two TE solutions.

3.5.3. Residual Biogas Production

The experiment was conducted by stopping the feed to a digester that had previously been fed at OLR 5 g VS l^{-1} day^{-1} and had reached 3 HRT, connecting the gas outlet directly to a Tedlar bag (SKC Ltd, Blandford Forum, UK), and keeping the digester otherwise sealed for the duration of the test. The volume of gas accumulated in the sampling bag was then measured accurately in a weight-type water displacement gasometer and biogas composition was analysed by GC as detailed in section 3.2.4.

3.5.4. Assessment of digestion with antifoam addition

The aim of this experiment was to see if the problem of foaming could be eliminated by the use of an anti-foaming agent (J-QUELL 19, J1 Technologies, Manchester, UK). The experiment used a pair of 2-litre CSTR digesters operated at a 1-litre working volume to provide headspace for foam accumulation. The digesters were inoculated with digestate taken from digesters N7 and N8 which had been operated at an OLR of 5 g VS l^{-1} day^{-1}. The test digesters were also maintained at this OLR and one of the pair received antifoam whilst the other did not. The digesters were run for 147 days and 0.1 ml antifoam was added on day one with periodic further dosing to reduce the foam volume once the level had reached a pre-defined height above the liquid surface.

3.5.5. Comparison of mesophilic and thermophilic digestion

The experiments were carried out in four mesophilic (37 ^{o}C) and four thermophilic (55 ^{o}C) digesters with a 4-litre working volume. These were all seeded with an inoculum as described in section 3.1.2. Duplicate digesters were run at each temperature at OLR of 4 and 5 g VS l^{-1} day^{-1} for 3 HRT, equivalent to 206 and 165 days. Each digester received trace element (TE) supplementation based on the amount of feedstock added.

3.6. Carbon, Energy, and Nutrient Footprint Analysis

Calculation of the CEN balance was carried out based on a model developed at the University of Southampton (Salter and Banks, 2009; VALORGAS, online) Required data and parameters for model operation are shown in Table 3.6 and were obtained from literature reviews, the British Sugar company website, personal communications with British sugar staff and experimental results.

Table 3.6. Parameters data required in CEN footprint

Parameters data	Unit
Input OLR	kg VS m^{-3} day^{-1}
Digester temperature (mesophilic or thermophilic)	^{o}C
HRT digester	days
Volume of gas head space	m^3
Biogas losses	%
Parasitic	kWh t^{-1} WW
CHP electrical efficiency	%
CHP heat efficiency	%
Heat utilization	%
Heat energy source	-
Construction materials	-
Ambient temperature	^{o}C
TS	% WW
VS	% TS
Specific CH_4 potential	m^3 kg^{-1} VS
CH_4 composition	%
Elemental composition (C, H, O, N)	% TS
Proportion converted	%
Residual TS	% WW
Macro nutrient (N,P,K)	g kg^{-1} WW
Calorific value	MJ kg^{-1} TS
Biochemical Methane Potential	1 CH_4 g^{-1} VS
Type of transportation	-
Distance of transportation	km

CHAPTER 4. SUGAR BEET PULP DIGESTION

4.1. Research aim

The aim of the work carried out in this chapter was to determine the physico-chemical characteristics of the SBP, its biochemical methane potential (BMP) and its long term digestion performance under semi-continuous fed conditions in laboratory scale digesters. It was also to ascertain whether there were factors that might adversely affect this performance and if so to identify possible solutions.

4.2. Characterisation of SBP

SBP was characterised using physico-chemical analysis as described in section 3.2.2 and 3.2.3 in Chapter 3 and the results are given in Table 4.1. The TS was 242.1 g kg^{-1} WW indicating a high moisture content and the VS was 225.5 g kg^{-1} WW giving a VS content of 93.14 % of TS, very similar to the value of 94% found by Alkaya and Demirer (2011). The high VS content indicated a high potential for biodegradation and possible conversion to biogas. The C/N value was favourable for digestion at ~25 on a TS basis, with most of the carbon available as either cellulose or hemicellulose. In addition, the SBP had a high calorific value of 16.86 MJ kg^{-1} TS which was slightly greater than the theoretical value of 16.12 MJ kg^{-1} TS calculated using the modified Du Long formula, but very similar to the value of 16.79 MJ kg^{-1} TS calculated using the Boie formula (Mason and Gandhi, 1983; Buckley and Domalski, 1988). This difference is probably due to the organic material make up of SBP: as stated by Niessen (2002) the Du Long formula is most accurate when applied to high carbon/hydrogen materials (e.g. coal, peat, or lignite), whilst the Boie equation is more suited to high cellulosic materials of which SBP is an example.

Table 4.1. Characteristics of sugar beet pulp

Parameters	Values
TS (% of WW)	24.2
VS (% of WW)	22.6
VS (% of TS)	93.2
Biochemical composition (g kg^{-1} WW)	
Hemicellulose	70.2
Cellulose	32.2
Lignin	20.0
Crude protein (TKN x 6.25)	21.8
Elemental analysis (% TS)	
C	42.64
H	5.47
N	1.79
O (by difference)	42.58
S	0.46
Macro nutrients (g kg^{-1} WW)	
TKN (N)	3.48
Phosphorus (P)	0.41
Potassium (K)	0.84
Trace elements (mg kg^{-1} WW)	
Cobalt (Co)	0.008
Iron (Fe)	17.5
Molybdenum (Mo)	0.008
Nickel (Ni)	0.036
Selenium (Se)	0.003
Other substrate parameters	
Measured Calorific value (*MJ kg^{-1} TS*)	16.8
Theoretical CV (*MJ kg^{-1} TS*) (Du Long)	16.1
Theoretical CV (*MJ kg^{-1} TS*) (Boie)	16.8

Note: WW = wet weight, TS = total solid

About 50% of the solids in the SBP were present as fibre which is comprised mainly of hemicelluloses, with cellulose and lignin also present to a lesser extent. The hemicellulose concentration was 29% (on a TS basis), which is within the range of 24-32% found by Spagnuolo et al. (1997); and very similar to the value of 30% found by Kelly (1983) and Weibel (1989). The value for cellulose (13.29%) obtained in this study was lower than the reported values of 22-40% (McCready, 1966; Kelly, 1983; Weibel, 1989; Spagnuolo et al., 1997). The SBP also contained a high lignin content at 8.25% compared to previously reported values of 2-4% (Kelly, 1983; Spagnuolo et al., 1997). The lignin content, however, is still below that of other agro-crop residues such as unused stalks and straw from variety of crops (Chen et al., 2008b) and agricultural biomass sources such as corn stover wheat straw, napier grass and wood grass (Tong et al., 1990). Lignin is a complex organic material that is resistant to chemical breakdown

by microorganisms (Jimenez et al., 1990; Yin et al., 2000). Klimiuk et al. (2010) and Chen et al. (2008b) both point out that there is a correlation between the concentration of fibrous materials, particularly lignin, and biogas production and state that low lignin content substrates have a greater potential. It is also known that the biogas potential of cellulose is reduced when found in association with lignin (Noike et al., 1985; Adney et al., 1991; Leschine, 1995; Lynd et al., 2002). Previous experiments by Arntz et al. (1985) on anaerobic hydrolysis of beet pulp reported that the hydrolysis of cellulose was the limiting step in the AD process since this was slower than that of more readily degradable compounds (e.g. hemicellulose and sugars). This is supported by Fadel et al. (2000) who reported that the rapid digestibility of the SBP was due to the high arabinose content of hemicellulose because it is easily biodegradable compared to cellulose and lignin (Ghosh et al., 1985). The lignin and hemicellulose content of the SBP used in this study are thus indicative that it is likely to have a rapid rate of hydrolysis and a high biogas productivity.

The SBP had relatively low concentrations of cobalt, molybdenum, selenium and nickel at 0.008, 0.008, 0.036 and 0.003 mg kg^{-1} WW, respectively, compared to the values found by Draycott and Christenson (2003), and that in other materials such as manure, dung, or sewage sludge (Sager, 2007). The concentration of TE in SBP may affect its performance in biogas production, as TE is important to anaerobic microorganisms in the AD process. This is supported by Banks et al. (2011), who stated that digestion was likely to be limited by the low concentrations of Co, Se, Mo and Ni.

4.3. Biochemical methane and biogas potential of SBP

Objective. The purpose of the BMP test is to obtain a value that represents the maximum possible methane yield which can be obtained under non-limiting conditions: this value provides a baseline against which the methane yield in semi-continuous digestion trials can be compared.

Summary method.

The test was run against blank and positive controls over a period of 28 days with gas volume and composition measured at regular intervals. The TS/VS and VFA concentration were measured at the beginning and end of the test. Although steps were

taken to minimise dissolution of gases by using acidified saline water, a proportion of the CO_2 is lost in this way resulting in an apparently higher methane concentration than that actually generated.

Results

Figure 4.1 shows the specific biogas and methane production of the SBP and the cellulose positive controls against the blank samples. The blank samples (inoculum only) reached a stable value after approximately 14 days with 28-day specific biogas productions of 0.110, 0.092, and 0.092 l g^{-1} VS, giving an average value of 0.098 l g^{-1} VS. The cellulose positive controls had a rapid biogas production after a short lag time and reached a plateau after 10 days: the specific biogas yields were 0.566, 0.568 and 0.581 l g^{-1} VS with an average value of 0.572 l g^{-1} VS (Figure 4.1a).

SBP with fresh inoculum demonstrated the same trend as the positive control, showing a short lag time and reaching a plateau after 10 days with net specific biogas productions of 0.577, 0.584, and 0.653 l g^{-1} VS, respectively, thus giving an average value of 0.605 l g^{-1} VS (Figure 4.1b). This value was slightly lower than the values of 0.664 l g^{-1} VS reported in a previous study (Banks, 2009) for a different batch of material from the same source.

Figure 4.1c shows the specific methane production of the SBP and the cellulose positive controls against the blank sample. The blank samples had specific methane productions of 0.084, 0.071, and 0.071 l CH_4 g^{-1} VS, respectively; with the average value 0.075 l CH_4 g^{-1} VS. The cellulose positive controls had a specific methane yield of 0.296, 0.295 and 0.303 l CH_4 g^{-1} VS with an average of 0.298 l CH_4 g^{-1} VS. The [positive] controls showed reasonable agreement and the average specific methane yields were typical of values obtained for these materials (unpublished data, University of Southampton), indicating the suitability of the assay conditions.

Using fresh inoculum gave values of 0.302, 0.307 and 0.355 l CH_4 g^{-1} VS with an average BMP of 0.321 l CH_4 g^{-1} VS (Figure 4.1d). This was slightly lower than the value of 0.35 l CH_4 g^{-1} VS found by Banks (2009), but within the range found in the literature: Alkaya and Demirer (2011) reported a BMP for SBP of 0.294 – 0.385 l CH_4 g^{-1} VS. This BMP value is fairly typical for crop and agro-waste substrates which have

high cellulose content and relatively low lignin (Wang et al., 1994; Chynoweth et al., 2001; Labatut et al., 2011; Brulé et al., 2013).

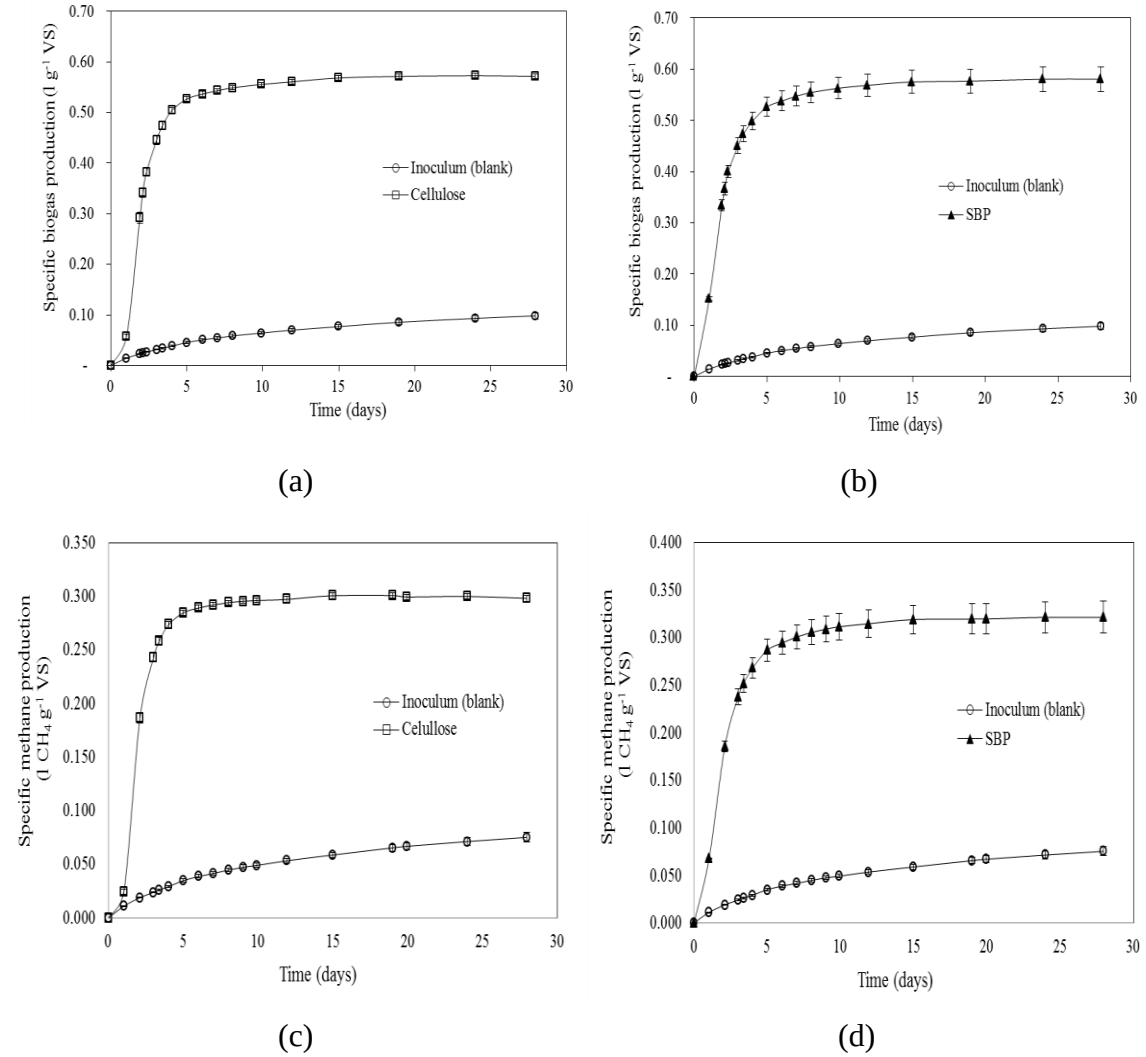


Figure 4.1. Specific biogas and methane production from the BMP test: the cellulose positive control and blank samples (a & c); and SBP with fresh inoculum and blank samples (b & d). Data are expressed as means of triplicate samples and bars represent range

Table 4.2 shows the average values for pH and VFA concentration at the end of the BMP test. The pH was well within the acceptable value for the growth of anaerobic microorganisms and similar to the value found by Kryvoruchko et al. (2009); and only slightly lower than that found by Alkaya and Demirel (2011) at pH 7.43 – 7.48. The average VFA concentration was ~79, ~32 and ~33 mg l^{-1} for blank, cellulose positive control and SBP respectively: this was lower than the values obtained for SBP by

Kryvoruchko et al. (2009). Low VFA concentrations at the end of the BMP test indicate that the intermediate products such as propionic and butyric acid have been converted into acetate, H_2, and CO_2 which are then further used by methanogenic archaea to produce methane. No accumulation of VFA also indicated no inhibition of the degradation process and that the methanogenic microorganisms were successfully using acetate for biogas production. This is supported by Kryvoruchko et al. (2009), who stated that a low concentration of VFA at the end of BMP test of SBP silage indicated no inhibition of the digestion process.

Table 4.2. VFA concentration and pH value at end of BMP test (average value)

Sample ID	VFA concentration (mg l^{-1})								pH
	HAC	PRO	i-BUT	n-BUT	i-VAL	n-VAL	HEX	HEP	
Blank	23.04	2.16	2.61	4.28	8.26	7.48	13.19	17.51	7.36 ± 0.0
Cellulose	10.69	0.34	n.d	0.82	2.01	2.61	5.36	9.97	7.33 ± 0.0
SBP	12.15	n.d	0.79	1.20	4.02	2.53	4.68	7.54	7.34 ± 0.0

HAC = acetic acid, PRO = propionic acid, i-BUT = iso butyric acid, n-BUT = butiryc acid, i-VAL = iso valeric acid, n-VAL = valeric acid, HEX = hexanoic acid, HEP = heptanoic acid, n.d. = not detected

4.3.1. Kinetics of the BMP

Two different kinetic models were fitted to the BMP data to estimate the performance of AD process: a first-order model (equation [4.1])

$$Y = Y_m (1 - e^{-kt}) \quad [4.1]$$

Where:
Y is the cumulative methane yield at time t
Y_m is the ultimate methane yield
k is the first order rate constant

and a pseudo-parallel first order model (equation [4.2]).

$$Y = Y_m (1 - Pe^{-k_1 t} - (1-P) e^{-k_2 t}) \quad [4.2]$$

Where:
Y is the cumulative methane yield at time t
Y_m is the ultimate methane yield
k_1 is the first order rate constant for the proportion of readily degradable material
k_2 is the first order rate constant for the proportion of less readily degradable material
P is the proportion of readily degradable material

The results for the two models are shown in Figure 4.2 and the kinetic constants obtained are given in Table 4.3. The first order model overestimated methane production at the beginning of the experiment; but then fitted well from day 6 up to day 28 ($R^2 \approx 0.9881$). The pseudo-parallel first order model gave a marginally better fit ($R^2 \approx 0.9888$), and then fitted well from day 2 to end of the BMP test (day 28). Rincón et al. (2011) reported the pseudo-parallel first order model gave the best fit for biogas production from solid organic substrates. From Figure 4.2, it can be seen that about 90% of the methane had been produced after 7 days and 97% after 10 days indicating a rapid biodegradation rate. According to Gunaseelan (2009) a fast biodegradation rate of substrate in a BMP test indicates that a relatively small reactor will be required giving an economical digestion process. In practice the size of the reactor is governed by the organic loading that can be applied and this has to be estimated in a continuous (or batch fed) kinetic study (Banks and Heaven, 2013). The results from the BMP test do, however, confirm that SBP is a very promising feedstock for AD.

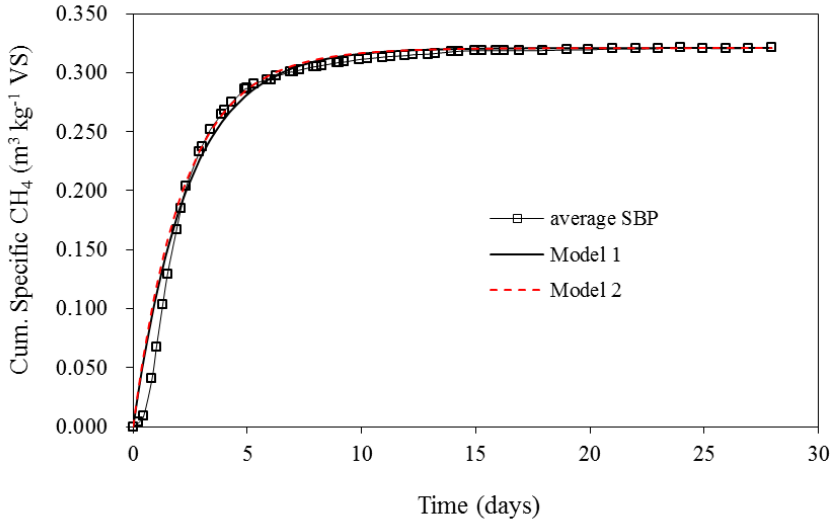


Figure 4.2. Kinetics of methane production for first order (model 1), pseudo-parallel first order (model 2) and experimental data for SBP

Table 4.3. Kinetic constants from modelling

Parameter values		Y_m	P	k_1	k_2	Correlation coefficient R^2
SBP	Model 1	0.321	1	0.42	0	0.9881
	Model 2	0.321	0.74	0.49	0.35	0.9888

4.3.2 Effect of inoculum age on the BMP test

Many factors can affect the results of a BMP test (see section 2.5 in Chapter 2), and the objective of this part of the work was to contribute towards the overall understanding of these by assessing one element: the effect of inoculum age as shown by the final BMP values and the BMP kinetics.

Summary method

Triplicate samples and blank controls were set up using the single inoculum sample, taken from Millbrook WWTW, while fresh and after ageing for 1, 3, 4, 6 and 7 days. All the BMP reactors were filled with inoculum at the same time and maintained in the same water bath with the gas collection cylinders sharing a common bath of barrier solution: the only difference was the length of time the inoculum was left without food. Each test was run over 31 days in a total experimental period of 39 days.

Results

The results of the six tests are shown in Table 4.4, with each individual test at a different inoculum age compared to the result using fresh inoculum. The specific biogas production determined was between 0.580 - 0.637 l g^{-1} VS and this was within the reported range in the literature (Amon et al., 2007a; Banks, 2009). There was no clear pattern of decreasing or increasing cumulative gas production values with inoculum age. The major difference observed was that, for older inoculums, the initial rate of biogas production was quite slow compared to that for the fresh inoculum, with an obvious lag phase. Within two days of addition of the feedstock sample all inoculums had responded, and had an equal or higher gas production rate to that of the fresh material; and all tests reached a plateau after a period of 10 days. The same was true for methane potential, with the start of each test using an aged inoculum showing a similar lag phase to that seen in biogas production. The specific methane potentials were in the range of 0.292 - 0.348 l CH_4 g^{-1} VS. Again the values all lie within the range reported in the literature; and the variability between results at different inoculum ages was similar to that between the samples tested with fresh inoculum, suggesting that it was no greater than that caused by inhomogeneity in a natural feedstock. At the end of all the tests, irrespective of age of the inoculum the pH was between 7.28 and 7.40, total VFA concentration was in the range of ~11–18 mg l^{-1} (Table 4.4).

Table 4.4. Biogas and methane production, VFA concentration, and pH value at the end of BMP test of SBP (average value for triplicates)

Parameters	Unit	D-1	D-3	D-4	D-6	D-7
Spec. biogas production	l g^{-1} VS	0.603	0.580	0.610	0.637	0.607
Spec. methane production	l CH_4 g^{-1} VS	0.325	0.292	0.348	0.341	0.320
pH	-	7.28	7.32	7.35	7.32	7.40
VFA	mg l^{-1}	18	14	16	15	11
HAC	mg l^{-1}	7.84	8.98	8.21	8.36	7.76
PRO	mg l^{-1}	n.d	n.d	n.d	n.d	n.d
i-BUT	mg l^{-1}	n.d	n.d	n.d	n.d	n.d
n-BUT	mg l^{-1}	0.30	n.d	n.d	0.35	n.d
i-VAL	mg l^{-1}	1.48	0.49	0.20	1. 11	0.21
n-VAL	mg l^{-1}	1.33	n.d	4.88	0.08	0.30
HEX	mg l^{-1}	1.94	1.11	0.66	1.67	0.94
HEP	mg l^{-1}	4.79	2.59	1.99	3.96	1.75

HAC = acetic acid, PRO = propionic acid, i-BUT = iso butyric acid, n-BUT = butiryc acid, i-VAL = iso valeric acid, n-VAL = valeric acid, HEX = hexanoic acid, HEP = heptanoic acid, n.d. = not detected, D-1= 1-day old inoculum, D-3 = 3-day old inoculum, D-4 = 4-day old inoculum, D-6 = 6-day old inoculum, D-7 = 7-day old inoculum

Conclusion. The age of the inoculum appeared to have no significant effect on the final values of physico-chemical parameters such as pH and VFA, which supports the view that there was no effect on specific biogas or methane yields. The only difference resulting from ageing of the inoculum was a lag in the onset of biogas and methane production.

4.4. Kinetic study under mesophilic conditions and influence of trace elements

4.4.1. Effect of different OLR on digestion performance

Objective

The objective of this experiment was to determine the methane potential of SBP under mesophilic conditions at different loading rates in a semi-continuous experiment run over at least 3HRT. The experiment also aimed to assess the operational stability of the digesters and identify any effect from trace element supplementation.

Summary method

The study used 8 x 5 litre digesters (N1 to N8) run in duplicate and sequentially loaded to achieve a pre-determined applied organic loading rate (OLR) (see Table 3.5 in Chapter 3). All the digesters started operation at an OLR of 2 g VS l^{-1} day^{-1}. This was

increased to 3 g VS l^{-1} day^{-1} in digesters N3 to N8 between days 25-40; to 4 g VS l^{-1} day^{-1} in digesters N5 to N8 between days 60 to 75; and to 5 g VS l^{-1} day^{-1} in digesters N7 and N8 between days 140-155. There were operational problems between days 80-90 due to a heater failure and during this time feeding was stopped. When the heater had been repaired feeding was gradually restored to the previous OLR over a period of 8 days so as not to cause a shock to the digesters. By day 164 all of the digesters had reached their target loading rates, but operational difficulties were apparent in the higher loaded digesters as a stable foam was forming and occupying the head space of these digesters. A few days after reaching an OLR 5 g VS l^{-1} day^{-1}, this resulted in a gas tube blockage in digester N7, causing an increase in the pressure and explosive loss of digestate. The digester explosion caused a decrease in the volume of inoculum of around one third, established by weighing of the digester. The digester was maintained in operation, however, at a working volume of 3 litres and an OLR of 5 g VS l^{-1} day^{-1}. As a precaution the working volumes in digesters N5, N6 and N8 were also reduced to 3 litres.

A further pair of digesters (N9 and N10) were operated at an OLR of 3 g VS l^{-1} day^{-1} and received a trace element (TE) addition at a rate of 1 ml of each TE solution (section 3.1.4 in Chapter 3) for every 1 litre of digestate removed, on a weekly basis. All the other digesters had no TE addition.

The digesters were monitored for biogas composition, biogas and methane volume, pH, total ammonia nitrogen (TAN), total VFA, alkalinity (ALK), pH, and VS destruction.

Results

Results for the experimental digesters leading up to, and at, their target organic loading rates are shown in Figure 4.3 to 4.6 and average values for key parameters are given in Table 4.5: these are taken from the 30-day period at the end of 3 HRT (OLR 2 (days 381 – 411), OLR 3 (days 244-274), OLR 4 (days 176-206), and OLR 5 (days 130-165), when the digesters were considered to be close to steady state conditions. All values for digestion at OLR 5 g VS l^{-1} day^{-1} are for N8 only, as N7 failed before 3 HRT due to foaming problems, as discussed in section 4.4.2.

From Table 4.5 and Figure 4.3, it can be seen that the specific biogas and methane production of 0.621 l g^{-1} VS day^{-1} and 0.316 l CH_4 g^{-1} VS day^{-1}, respectively, at an OLR of 2 g VS l^{-1} day^{-1} was similar to that obtained in the BMP test. At OLRs higher than 2 g VS l^{-1} day^{-1} specific biogas and methane production reduced to ~0.57 l g^{-1} VS day^{-1} and 0.29 l CH_4 g^{-1} VS day^{-1}, respectively. As expected the volumetric biogas and methane yield increased with increasing OLR (Figure 4.4) and at the highest loading was 2.8 l biogas l^{-1} day^{-1} and 1.4 l CH_4 l^{-1} day^{-1}. The lower values between days 84-98 were due to the temperature drop caused by technical problems with the water heater pump and the reduced loading in the subsequent period (days 105-147) to allow the digesters to recover from this shock.

Table 4.5. Average steady state values of performance indicators for duplicate digesters at different OLR (between days 130 and 411)

	Unit	OLR (g VS l^{-1} day^{-1})			
		2	3	4	5*
Specific biogas Production	l g^{-1} VS day^{-1}	0.621	0.572	0.565	0.579
Specific methane production	l g^{-1} VS day^{-1}	0.316	0.293	0.286	0.294
Vol. biogas production	l l^{-1} day^{-1}	1.24	1.69	2.17	2.83
Vol. methane production	l l^{-1} day^{-1}	0.63	0.87	1.12	1.44
Digestate TS	g l^{-1}	56.1	63.3	67.6	75.3
Digestate VS	g l^{-1}	38.6	43.0	46.6	54.8
VS destruction	%	90.9	87.9	86.9	83.5
pH	-	7.56	7.45	7.37	7.12
TAN	mg N kg^{-1} WW	2060	1647	1442	1022
Total alkalinity	mg $CaCO_3$ kg^{-1} WW	18909	16355	16007	13357
IA/PA ratio	-	0.28	0.43	0.42	0.57
Total VFA	mg l^{-1}	82	232	219	376

Note:*values for mesophilic digestion at OLR 5 g VS l^{-1} day^{-1} are for N8 only, as N7 failed before 3HRT due to foaming problem

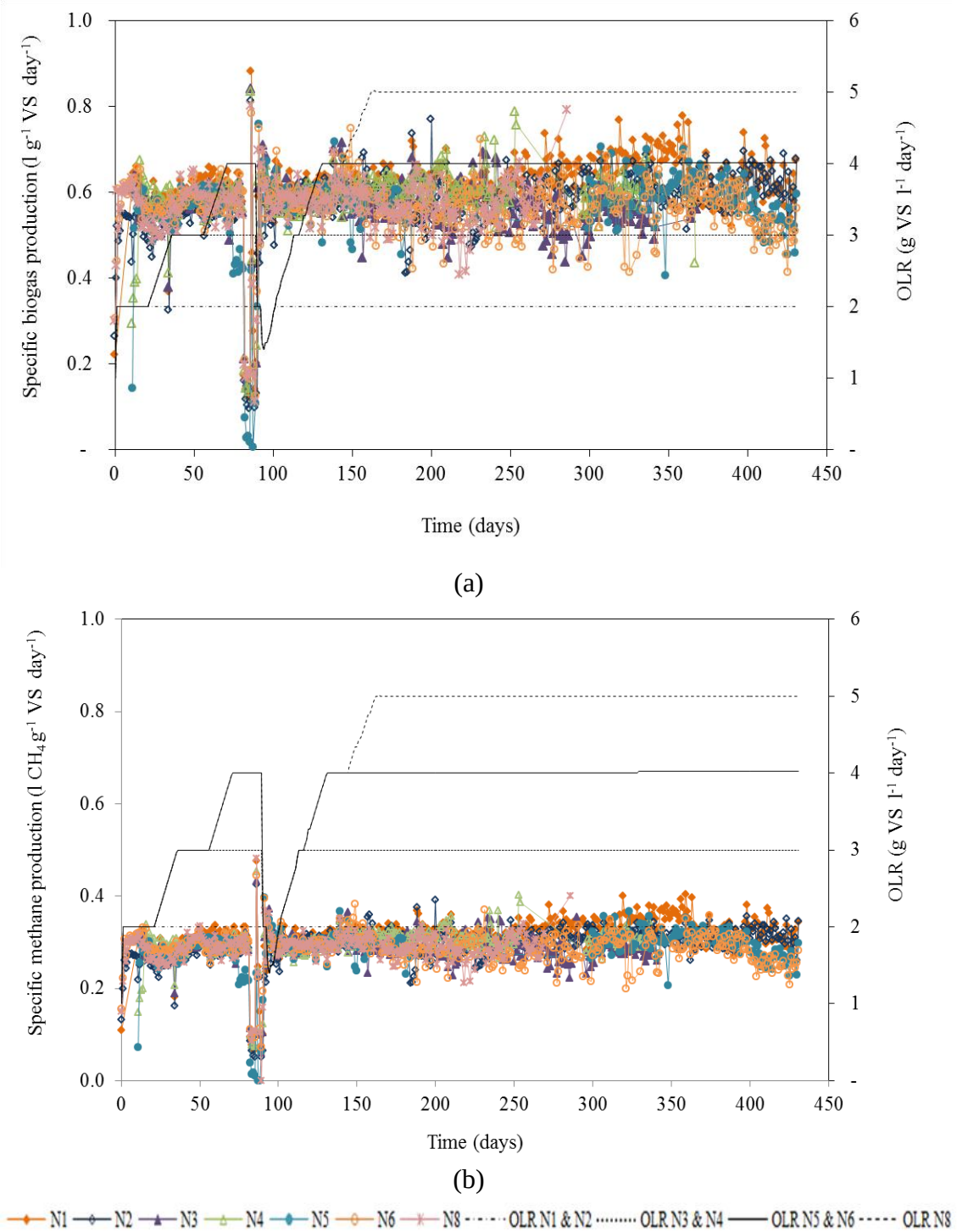


Figure 4.3. Specific biogas (a) and methane (b) production at different OLR

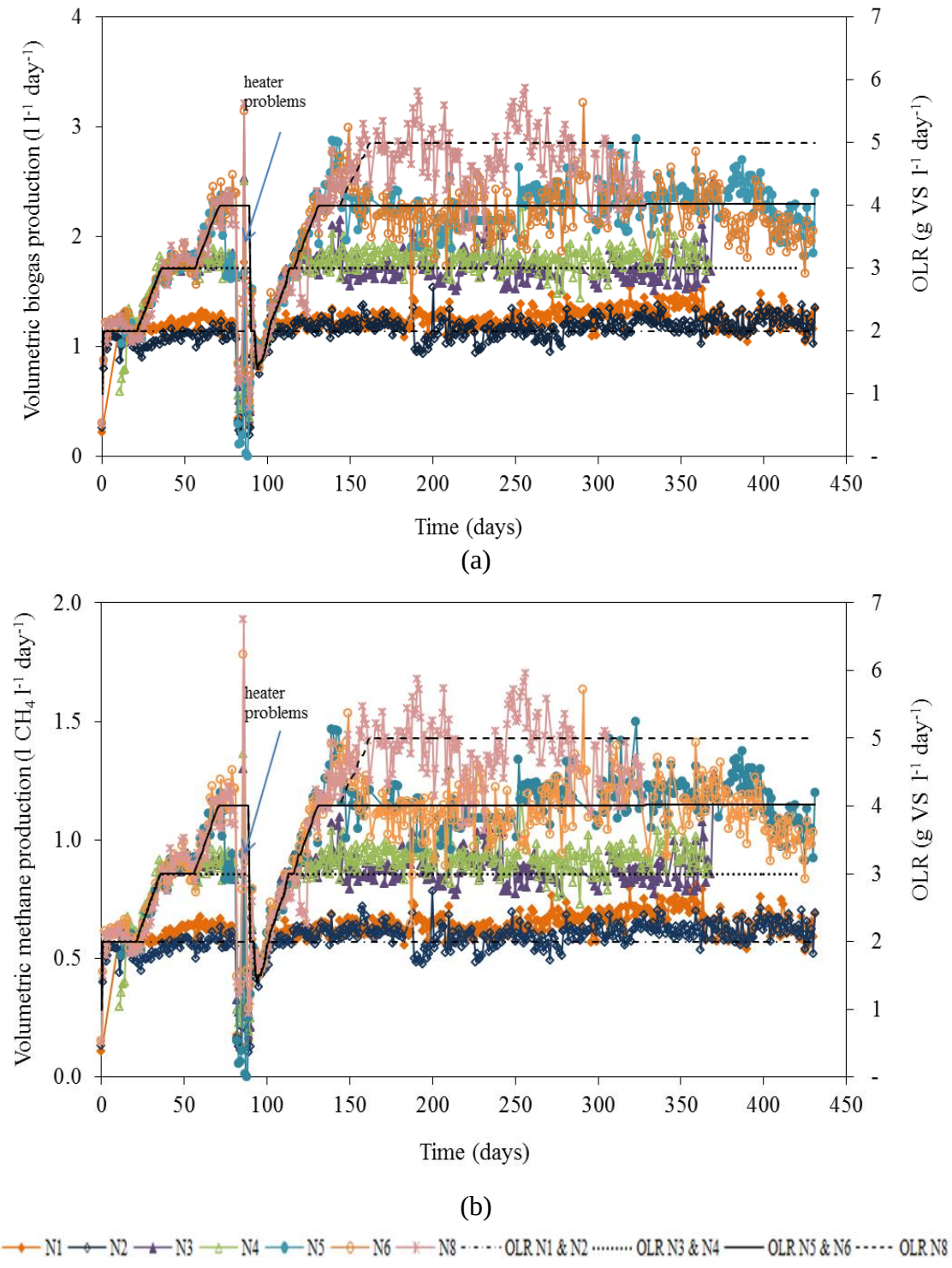


Figure 4.4. Daily volumetric biogas (a) and methane (b) production at different OLR

The average VS destruction was approximately 91%, 88%, 87%, and 84% for the digesters fed at OLR 2, 3, 4, and 5 g VS l^{-1} day^{-1}, respectively equivalent to a 2.35% reduction in VS destruction per g VS l^{-1} day^{-1} of OLR (Figure 4.5). If this reduction in VS destruction is taken into account then the specific methane production was 0.348, 0.333, 0.329 and 0.309 l CH_4 g^{-1} VS $_{destroyed}$ for the four loadings used. As well as leading to a decrease in VS destruction, increases in OLR corresponded to a decrease in both specific biogas and methane production.

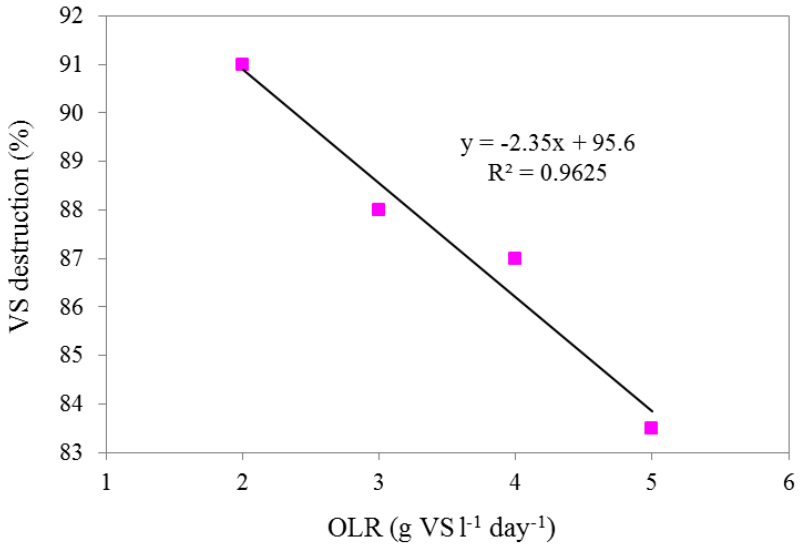


Figure 4.5. Correlation between OLR and percentage of VS destruction

As can be seen from Table 4.5 and Figure 4.6 the total VFA concentration in all sets of digesters varied slightly during the experimental period; in steady state conditions, however, values remained below 500 mg l^{-1}, well within the acceptable range of 200-2000 mg l^{-1} suggested by Cecchi et al. (2003). VFA concentration increased slightly with OLR giving a very slight decrease in the pH value but this did not drop below 7.2 and did not exceed 7.5 which is within the acceptable range of 6.5 to 7.5 for stable operation quoted by van Haandel and van der Lubbe (2007).

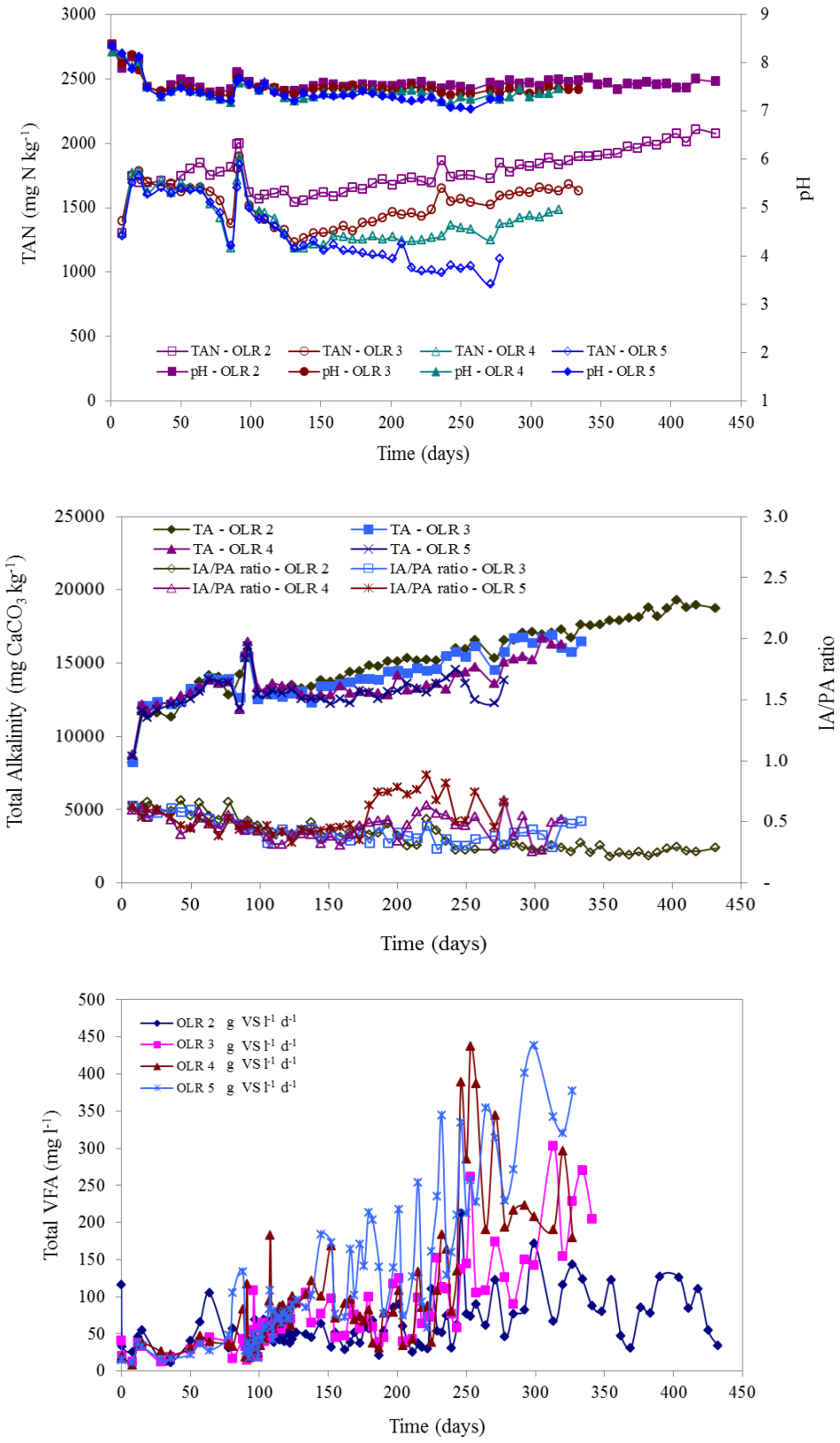


Figure 4.6. Trend of pH, TAN, total alkalinity, IA/PA ratio and total VFA at different OLR

Figure 4.6 shows TAN and alkalinity over time at all OLR. At an OLR of 2 g VS l^{-1} day^{-1}, there was a gradual increase in total alkalinity from ~12 g $CaCO_3$ kg^{-1} WW to a steady state value of ~17-18 g $CaCO_3$ kg^{-1} WW. This corresponded to an increase in TAN, which reached a concentration of ~1.9-2 g N kg^{-1} WW. For the digesters fed at OLR 3 g VS l^{-1} day^{-1}, the alkalinity and TAN concentrations were slightly lower than at OLR 2 g VS l^{-1} day^{-1}, at average values of ~16.3 g $CaCO_3$ kg^{-1} WW and ~1.6 mg N kg^{-1} WW, respectively. Increasing the OLR to 4 and 5 g VS l^{-1} day^{-1} resulted in a decrease in alkalinity to ~16.2 and ~14.8 g $CaCO_3$ kg^{-1} WW. Similarly, TAN concentration decreased to an average value of ~1.5 g N kg^{-1} WW (OLR 4 g VS l^{-1} day^{-1}) and ~1.1 mg N kg^{-1} WW (OLR 5 g VS l^{-1} day^{-1}). These results are in agreement with those for VS destruction, as TAN results from the degradation of proteins and other organic nitrogen-containing compounds and alkalinity is closely tied to the TAN concentration. The relationship between OLR, total alkalinity and TAN is shown in Figure 4.7 and was equivalent to ~1.7 g $CaCO_3$ kg^{-1} WW and ~0.3 g N kg^{-1} WW per g VS l^{-1} day^{-1}, respectively.

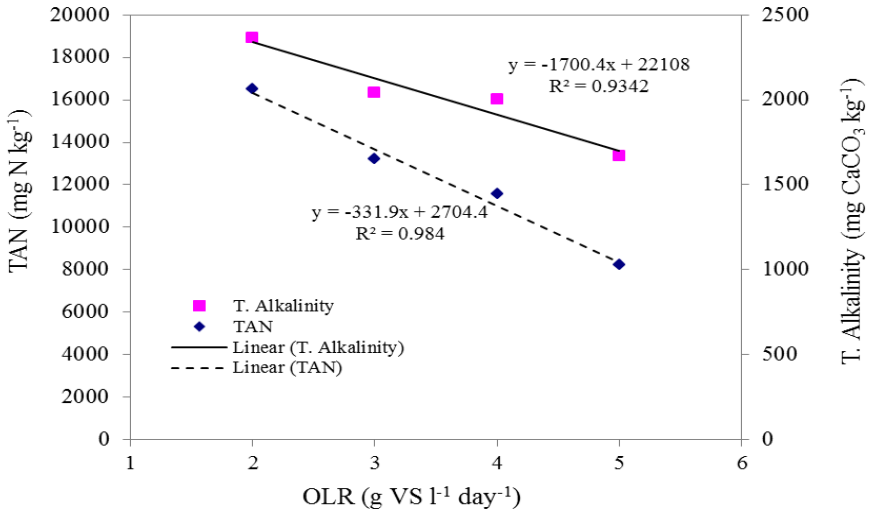


Figure 4.7. Correlation between OLR and TAN and alkalinity

The ratio of intermediate to partial alkalinity (IA/PA) for the digesters fed at OLR 2 g VS l^{-1} day^{-1} was 0.28, very close to the value of 0.3 originally suggested by Ripley et al. (1986) as indicating good process stability. The IA/PA ratio for the other OLRs was slightly higher, in the range 0.44 – 0.54, indicating a minor change in process stability with increased OLR as also suggested by the pH and VFA values.

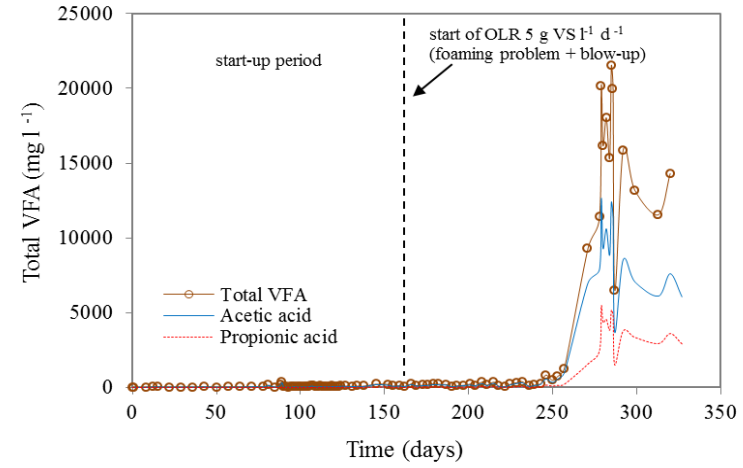
Conclusion. Overall the results indicated that increasing the OLR influenced process stability parameters such as pH, total and partial alkalinity, total VFA and IA/PA ratio, as well as slightly reducing organic matter degradation, biogas and methane production.

4.4.2. Effect of loss of digestate on AD of SBP

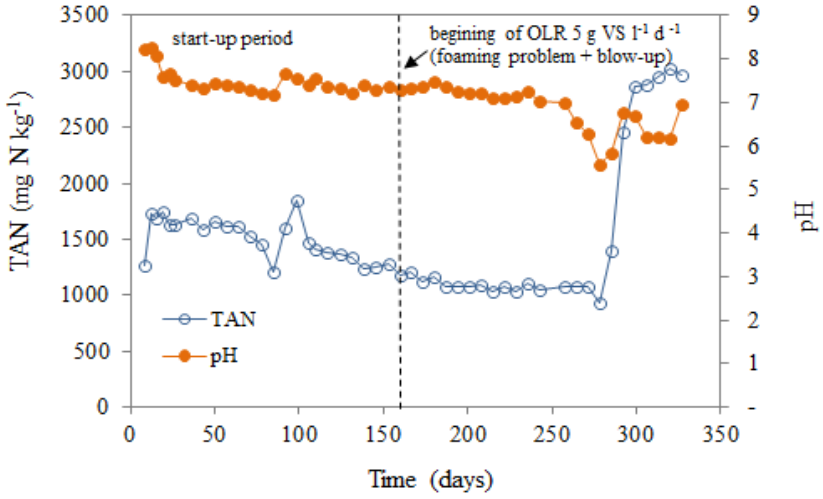
As explained above, digester N7 suffered severe foaming as the loading increased to 5 g VS l^{-1} day^{-1}, leading to a loss of digestate from the reactor. Even though the daily feed weight was reduced in proportion to the reduced working volume to maintain the same OLR, this digester showed signs of imbalance with an increase in VFA concentration and a decline in pH. Another possible cause was due to overloading as loss of microbial population in digestate limited the degradation rate of organic materials in SBP. As the revised OLR was based on digestate volume but this could have led to an over-estimate of the mass of digestate present, and therefore the microbial population, due to the amount of entrained gas. It has been noted on several occasions that digester performance is impaired after an explosion (unpublished data, Southampton), and the reasons for this are unknown but may be related to changes in physical and/or chemical conditions in the digester during pressurisation (e.g. increased dissolution of CO_2). This effect was apparently irreversible and despite the addition of TE, the digestion performances continued to decline and eventually fail. The results for this digester were excluded from the overall analysis of results above and are shown separately in Figure 4.8.

From Figure 4.8, it can be seen that the loss of digestate and/or onset of foaming appeared to have a negative effect on the stability of the AD process, indicated by an increase in VFA concentration from ~day 260 with an accompanying reduction in pH to below 6.5. In an attempt to correct this, 20 ml of ammonium bicarbonate solution (1 M) was added on day 279, 282 and 291 in an attempt to raise the pH. This was only partially successful (Figure 4.8), despite the increase in alkalinity to ~19 g $CaCO_3$ kg^{-1} WW brought about by a rise in TAN to ~3 g l^{-1}. The process imbalance was also reflected by the dramatic increase in IA/PA ratio to 5.13, much higher than the acceptable value suggested by Ripley et al. (1986). By the end of experiment (day 327) the VFA concentration was > 22 g l^{-1} indicating a kinetic uncoupling between acid producers and consumers (Ahring et al., 1995). This is supported by the concurrent

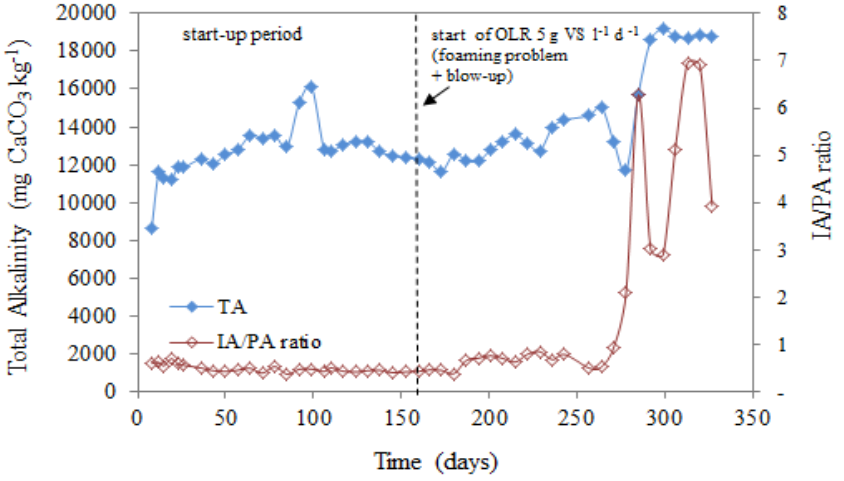
reduction of biogas and methane production, which fell to 0.077 l biogas g^{-1} VS day^{-1} and 0.032 l CH_4 g^{-1} VS day^{-1} by day 327 (Figure 4.9).



(a)



(b)



(c)

Figure 4.8. Trends in total VFA, pH, total ammonia nitrogen (TAN), total alkalinity, and IA/PA ratio in digester N7 after loss of digestate on day 164

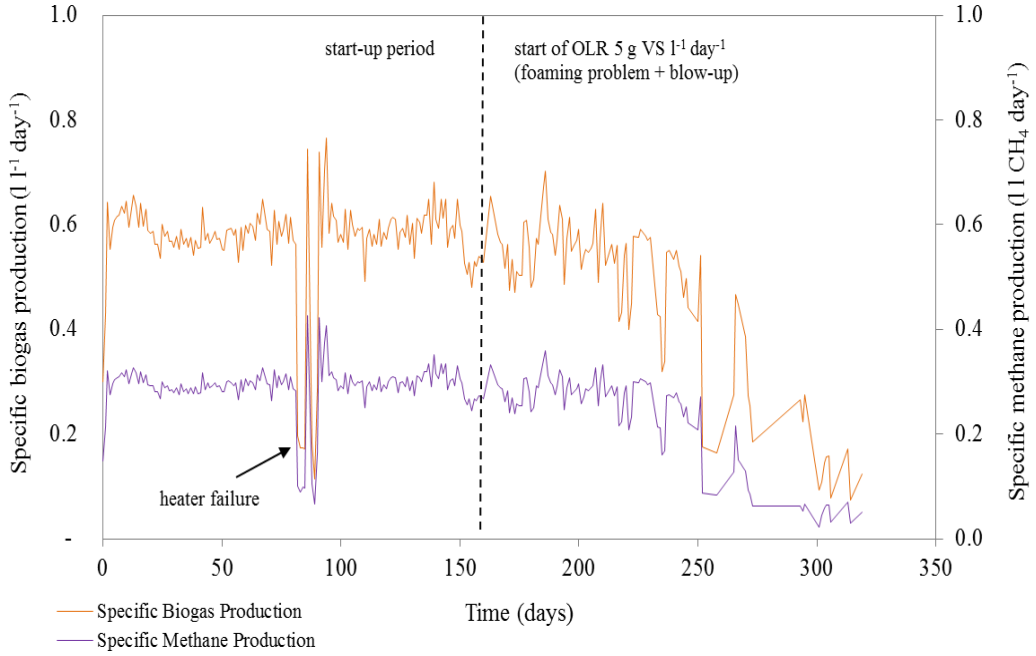


Figure 4.9. The biogas and methane production digester N7 at an OLR of 5 g VS l^{-1} day^{-1}

4.4.3. Effect of TE supplementation

TE solution was added to digesters N9 and N10 which were fed at OLR 3 g VS l^{-1} day^{-1}. These digesters were started with the same mixed inoculum were operated for a period of 240 days to allow full acclimatisation. The pair of digesters with TE supplementation appeared to show a slightly higher gas yield and VS destruction than those without TE; but these differences were very minor, and could not be shown to be statistically significant because of the small number of replicates (Table 4.6 and Figure 4.10). As can be seen in Figure 4.11 there was very little difference between the digesters with and without TE supplementation with respect to TAN, pH, or total alkalinity. With respect to VFA and IA/PA ratio the average values for these two parameters were slightly lower in the digesters with TE supplementation (Table 4.6), but both sets were clearly in the stable operational range.

The only noticeable difference between the two sets of digesters was that those without TE addition experienced foaming starting several weeks before the completion of 3 HRT, while no foaming was observed in the supplemented digesters.

Table 4.6. Average steady state values of performance indicators for duplicate digesters with and without TE additions (average over last 30 days of operation)

	Unit	With TE	Without TE
Specific biogas production	$l\ g^{-1}$ VS day^{-1}	0.608	0.572
Specific methane production	$l\ g^{-1}$ VS day^{-1}	0.313	0.293
Volumetric biogas production	$l\ l^{-1}$ day^{-1}	1.83	1.69
Volumetric methane production	$l\ l^{-1}$ day^{-1}	0.94	0.88
Digestate TS	$g\ l^{-1}$	65.1	63.3
Digestate VS	$g\ l^{-1}$	44.5	43.0
VS destruction	%	88.6	87.9
pH	-	7.46	7.45
Ammonia N	mg N kg^{-1} WW	1711	1647
Total alkalinity	g $CaCO_3$ kg^{-1} WW	17.5	16.3
IA/PA ratio	-	0.28	0.44
Total VFA	$mg\ l^{-1}$	155	233

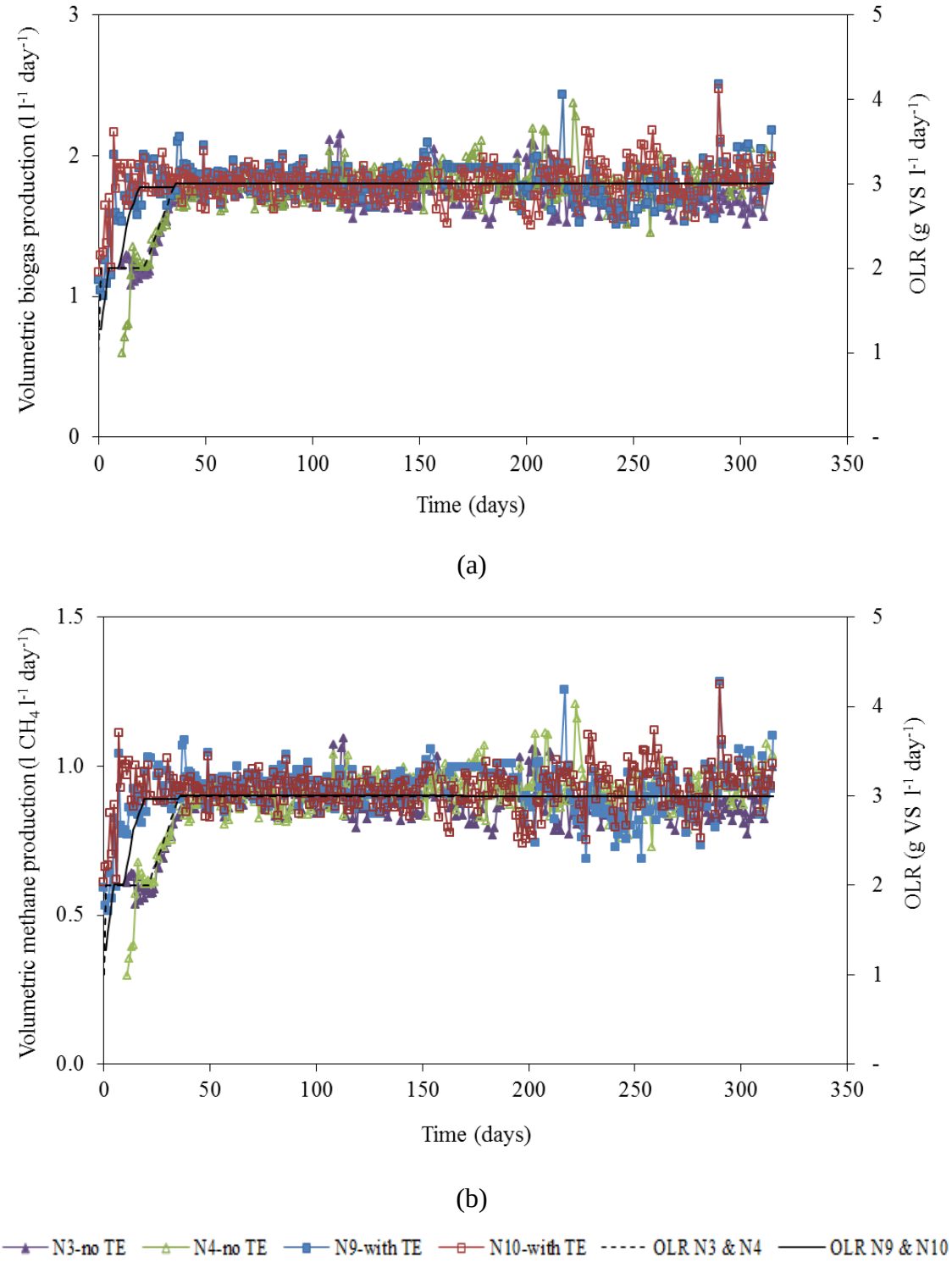


Figure 4.10. Daily volumetric biogas (a) and methane (b) production from digester with and without TE supplementation fed at OLR 3 g VS l^{-1} day^{-1}

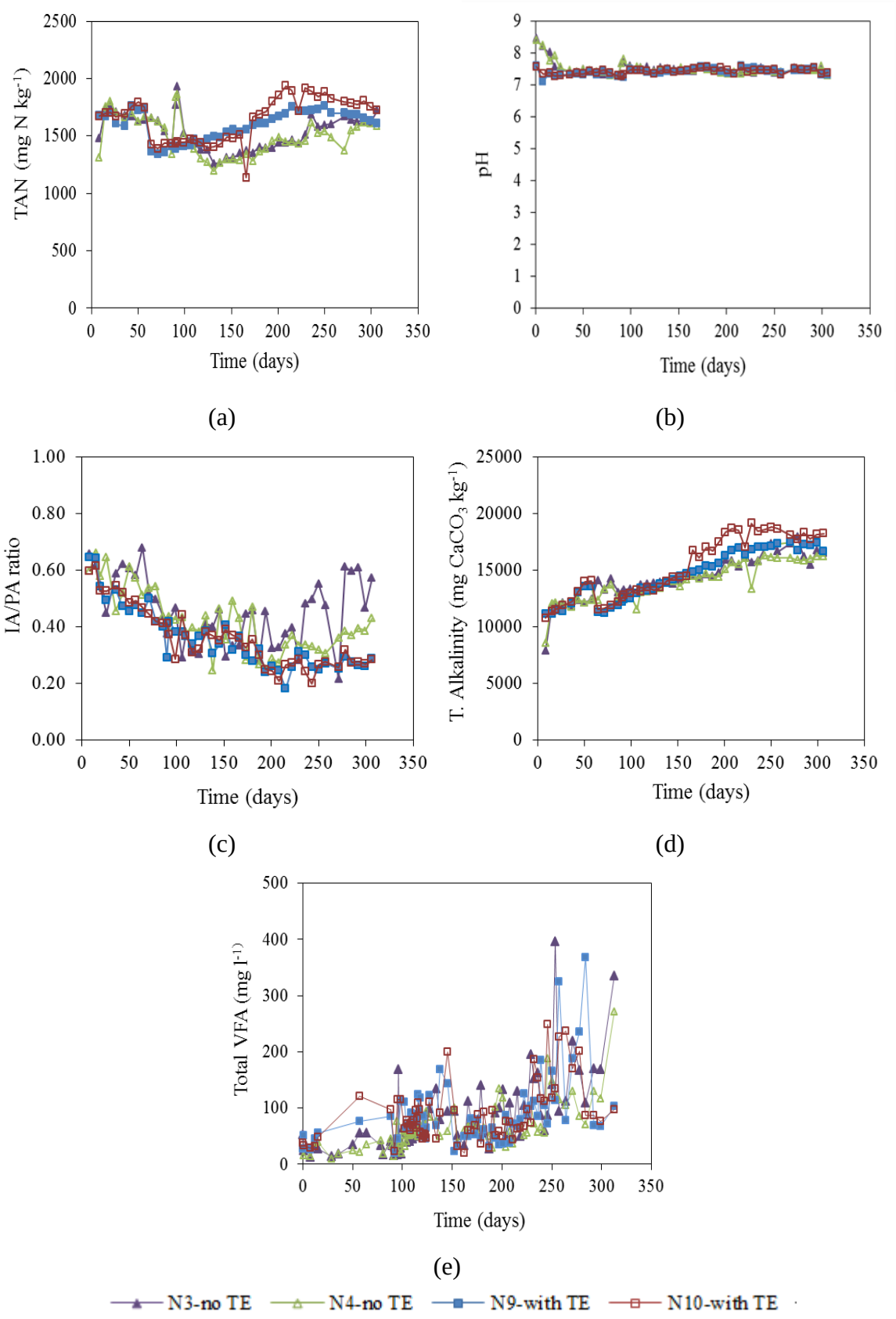


Figure 4.11. Trends in total ammonia nitrogen (TAN), pH, IA/PA ratio, total alkalinity, and total VFA in the mesophilic digester fed at OLR 3 g VS l^{-1} day^{-1} with and without TE supplementation

4.4.4. Residual Biogas Production

The residual biogas production (RBP) was measured for a digester (N8) fed at an OLR 5 g VS l^{-1} day^{-1} .This was achieved by stopping the feed and connecting the gas outlet to a gas sampling bag, and taking gas volume measurements over the following 222 days. Figure 4.12 shows the daily volumetric biogas and methane yields corrected to STP. There was a rapid decrease in volumetric biogas and methane production within 4 days of stopping feeding after which time the rate fell to low levels but production continued for around 50 days before the cumulative value does start to tail off. At the end of the trial production was ~0.008 l biogas l^{-1} day^{-1} and ~0.006 l CH_4 l^{-1} day^{-1}. In total, the residual biogas and methane production were 37.31 l biogas and 23.55 l CH_4 over the period of 222 days (Figure 4.13), giving a specific residual biogas and methane production of 0.015 l biogas g^{-1} VS and 0.010 l CH_4 biogas g^{-1} VS based on a mass balance of materials over the whole experimental period. Adding these values to the average specific biogas and methane production obtained from the digesters fed at 5 g VS l^{-1} day^{-1} gave a total of 0.594 l biogas g^{-1} VS and 0.319 l CH_4 biogas g^{-1} VS, close to the values found in the static BMP test in section 4.3 (Table 4.7).

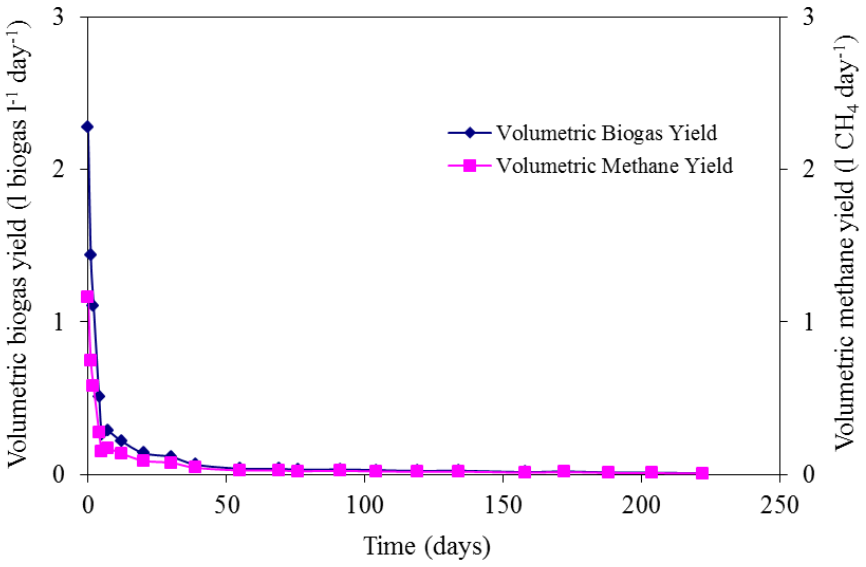


Figure 4.12. Trends in volumetric biogas and methane yields on the residual biogas production test

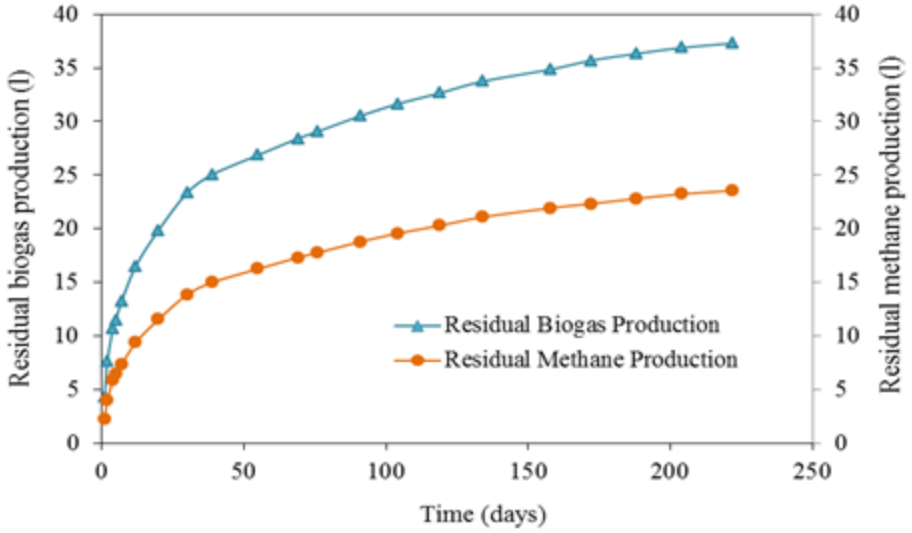


Figure 4.13. Cumulative residual biogas and methane production

Table 4.7. Calculation of specific residual biogas and methane production

	Unit	Value	Note
CSTR Trials OLR 5 g VS l^{-1} day^{-1} (291 days)			
Total VS added	g VS kg^{-1} WW	14577.99	A
Total VS digestate removed	g VS kg^{-1} WW	12003.95	B
Biogas equivalence of VFA concentration	g VS kg^{-1} WW	122.50	C
Total VS in digester	g VS kg^{-1} WW	2451.54	D = A – B - C
Specific biogas production	l g^{-1} VS	0.579	E
Specific methane production	l g^{-1} VS	0.309	F
Residual Biogas Production			
Volumetric biogas production	l biogas	37.31	G
Volumetric methane production	l CH_4	23.55	H
Specific residual biogas production	l biogas g^{-1} VS	0.015	I = G/D
Specific residual methane production	l CH_4 g^{-1} VS	0.010	J = H/D
BMP value			
Specific biogas production	l g^{-1} VS	0.605	K
Specific methane production	l g^{-1} VS	0.321	L
CSTR + RBP			
Specific biogas production	l g^{-1} VS	0.594	M = E +I
Specific methane production	l g^{-1} VS	0.319	N = F + J

4.5. Digester-based studies aimed at reducing foaming

The appearance of a stable foam caused operational problems which were more severe at higher OLRs. The objective of the experimental work described below was to assess whether this tendency to foaming could be controlled, firstly by water addition, and secondly by using chemical antifoam preparations.

4.5.1. Effect of water dilution on foaming in digestion of SBP

Summary method. The experiment used the pair of digesters (N5 and N6) that had previously been operated at a working volume of 3 litres with OLR of 4 g VS l^{-1} day^{-1}. Operation of these digesters continued for a further 123 days with the SBP feedstock diluted 1:1 with water on a mass ratio. On a daily basis, the digesters were fed with 44 g WW of SBP, 44 g water and 0.044 ml (44 µl) of each TE solution.

Results

Digestion performance. Specific biogas production in the digesters with water addition decreased during the experiment from an average of 0.565 l g^{-1} VS day^{-1} for the 206-day period before the start of dilution to around 0.497 l g^{-1} VS day^{-1} over the last 123 days. A similar trend was observed for the specific methane production which fell from 0.286 to 0.234 l CH_4 g^{-1} VS day^{-1} (Figure 4.14). Volumetric biogas and methane yields, although varying slightly during the experimental period, also showed a decrease to around 1.81 l biogas l^{-1} day^{-1} and 1.00 l CH_4 l^{-1} day^{-1}, respectively. These results were well below those for the digesters fed at the same OLR without dilution, which had average volumetric biogas and methane productions of 2.17 l l^{-1} day^{-1} and 1.12 l CH_4 l^{-1} day^{-1}, respectively.

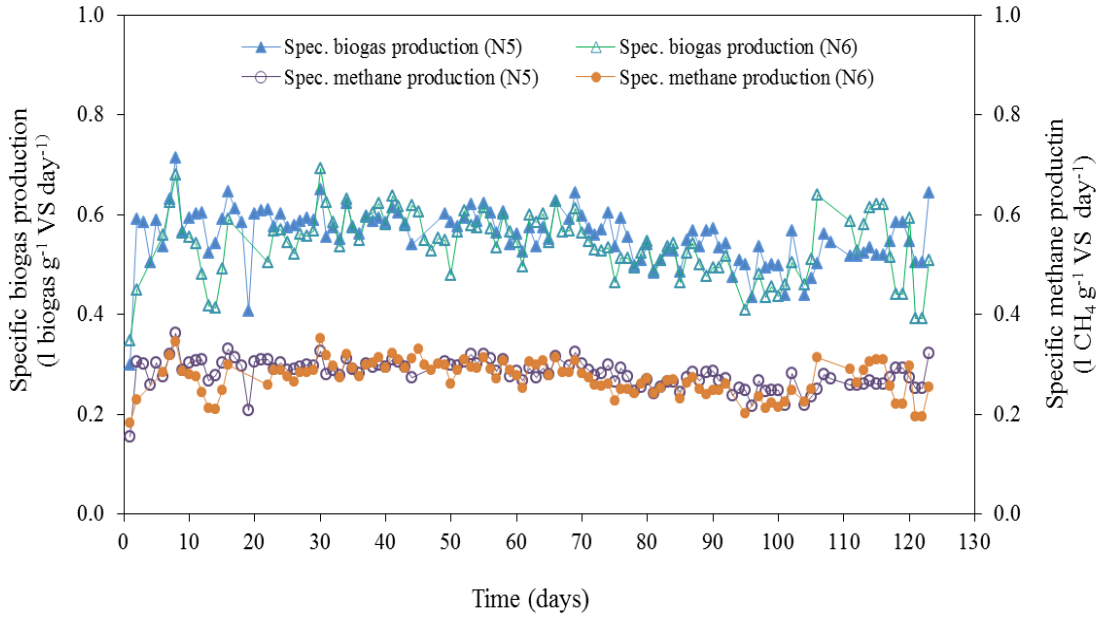


Figure 4.14. Specific biogas and methane production at OLR 4 g VS l^{-1} day^{-1} with feedstock dilution ratio 1:1 (SBP: water)

Figure 4.15 illustrates the trends in TAN, pH, total alkalinity, IA/PA ratio and total VFA in digesters N5 and N6. The concentration of TAN and total alkalinity continuously decreased during the experimental period, from ~1500 to ~400 mg N kg^{-1} WW and from ~16,000 to ~6,500 mg $CaCO_3$ kg^{-1} WW, respectively. Although these values remained well within recommended ranges (Cecchi et al., 2003), the reduction of total alkalinity showed that the addition of water reduces the buffer capacity of the digestate due to wash-out. This corresponded to a decrease in pH value throughout the experimental period from 7.44 to 7.06, although again the values remained in an acceptable range. The total VFA concentration varied slightly, with an average of less than 100 mg l^{-1}. At start-up the IA/PA ratio was relatively high at 0.5 and continuously increased reaching a value above 1 at the end of experiment, indicating process imbalance.

These findings indicated that feedstock dilution affected the stability and performance of the digesters treating SBP, probably due to a change in the buffering capacity as shown by a decline in TAN and total alkalinity, leading to a fall in pH and a rise in IA/PA ratio. From the start-up period to day 107 no excessive foaming was noted in either reactor. Starting on day 108, however, foaming occurred and continued up to the

end of the experiment (day 123), particularly in digester N6. These results showed that dilution of the feedstock did not prevent the occurrence of foaming in digesters using SBP.

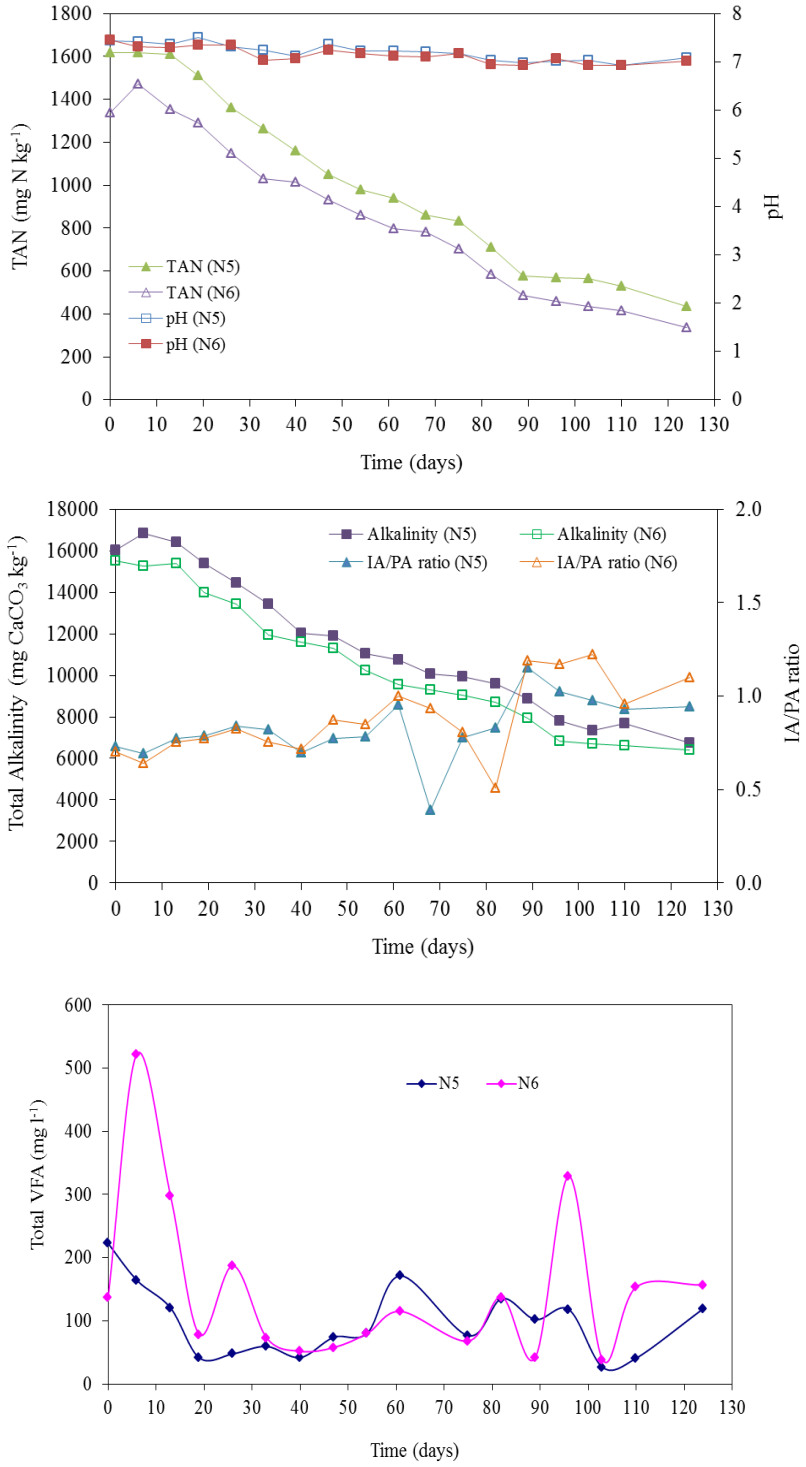


Figure 4.15. Changes in TAN, pH, total alkalinity, IA/PA ratio, and total VFA during the anaerobic digestion of SBP at OLR 4 g VS l^{-1} day^{-1} with feedstock dilution at ratio 1:1 (w/w)

4.5.2. Trials using anti-foam

Summary method. Two 2-litre STR digesters were operated at 1-litre working volume to see if foaming could be eliminated by the use of an anti-foaming agent (J-QUELL 19, J1 Technologies, Manchester, UK). Each digester was fed at OLR 5 g VS l^{-1} day^{-1}, one with the addition of antifoam and another without antifoam. This experiment ran for 147 days.

Results

The results showed that a very high dose of antifoam was required (1 ml l^{-1} compared to typical values of $\leq$ 0.1 ml l^{-1} in industrial practice) to reduce the level of foam in the digester. This dose had to be repeated at intervals of 2 weeks. After four treatments (8 weeks in total) no further addition of antifoam was needed, but over the following one-month period the digester supplemented with anti-foam showed a decline in both volumetric biogas and methane yields. VFA concentrations rose and the pH fell to 6.2, indicating failure of the digestion process.

Figure 4.16 illustrates the effect of antifoam addition on the performance of the digesters.

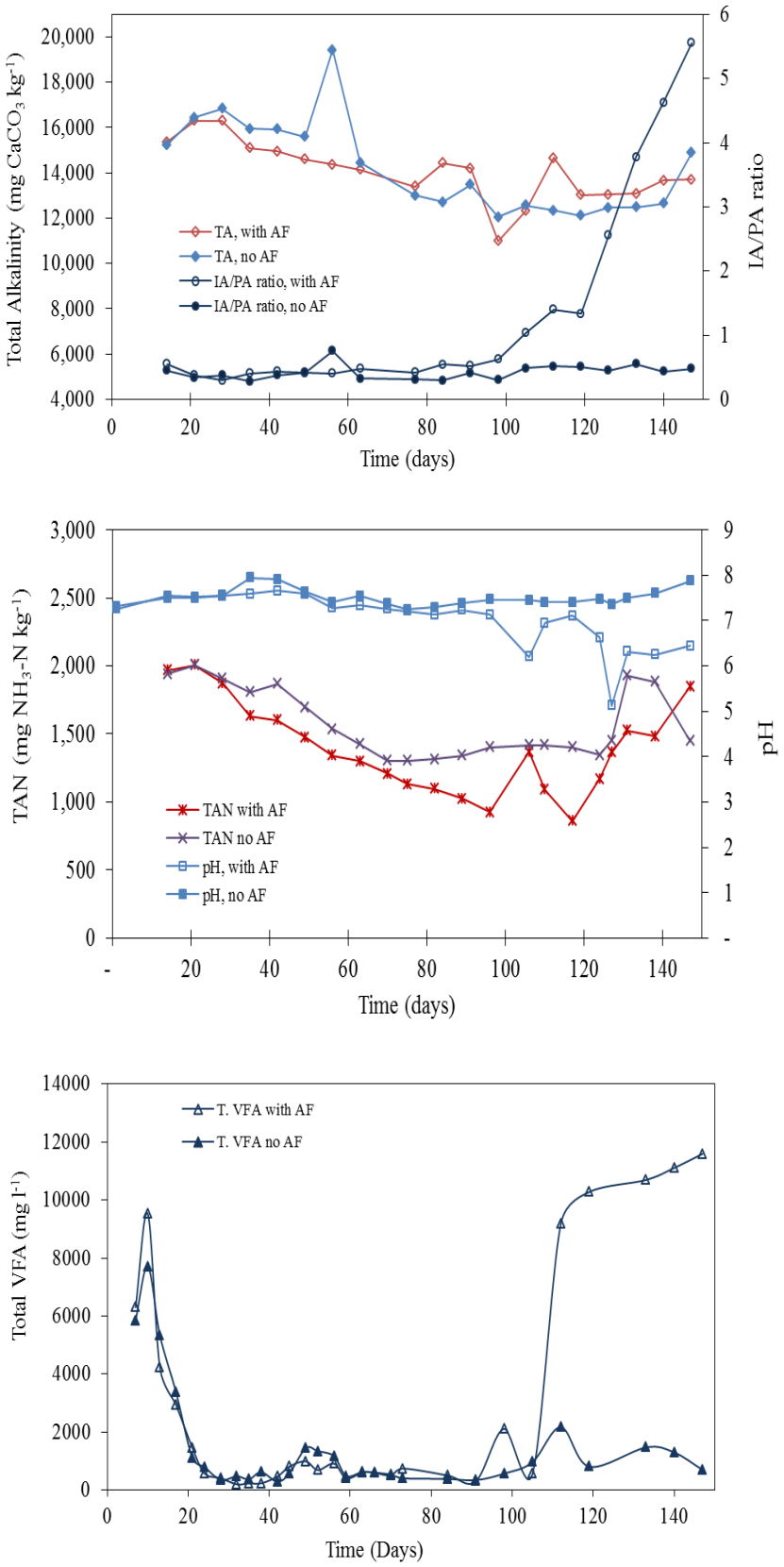


Figure 4.16. Trends in total alkalinity, IA/PA ratio, pH, TAN and total VFA on the foaming trials

In an attempt to recover the failed digester 20 ml of an alkaline solution (NaOH) was added on day 106 to boost the buffering capacity and increase the pH; but this did not solve the problem and the pH again fell below 6.5. The digester without antifoam addition showed no sign of VFA accumulation or other instability in performance, and volumetric biogas and methane yields remained constant at 2.66 l l^{-1} day^{-1} and 1.37 l CH_4 l^{-1} day^{-1} (Figure 4.17).

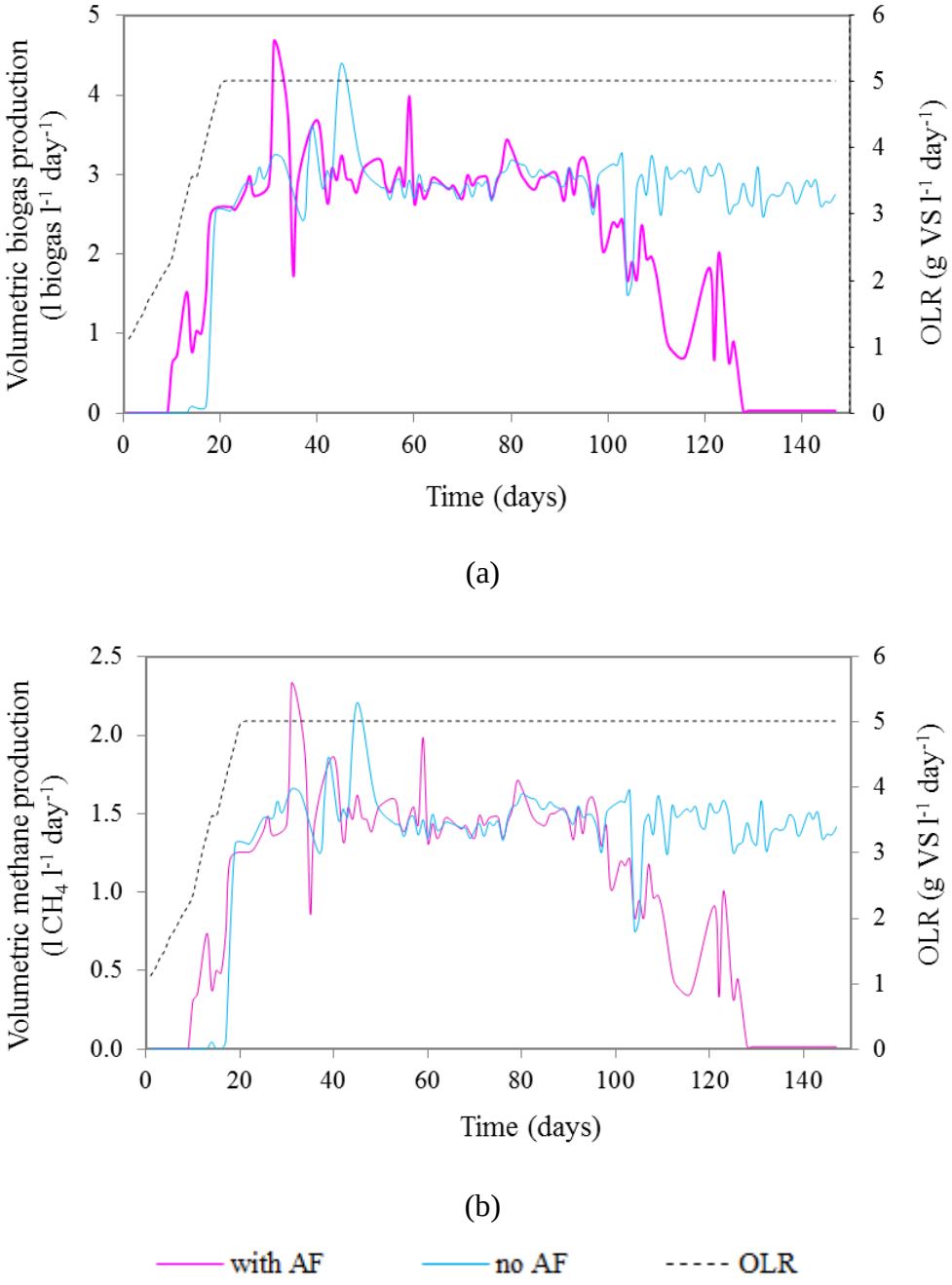


Figure 4.17. Daily volumetric biogas (a) and methane (b) production from digester with and without antifoams addition fed at OLR 5 g VS l^{-1} day^{-1}

The antifoam addition was very effective in reducing the foam, but this benefit was only short term with longer exposure having a toxic effect. Moeller et al. (2010) noted that antifoaming agents can influence the anaerobic biological process, while Vardar-Sukan (1998) stated that antifoam can be toxic to the microorganisms and also change operational parameters such as digester pH.

Figure 4.18 shows foaming at the surface of the SBP digestate in the untreated digester sampled on day 180. As can be seen, the foam consists of gas bubbles entrapped in the digestate; as OLR increases the rate of biogas generation may exceed the rate of escape causing expansion of the matrix.

Figure 4.18. Image of fresh foam in the digester

4.6. Comparison of mesophilic and thermophilic digestion of SBP

Objective

The objective of this experiment was to see whether thermophilic digestion could improve the specific methane production at higher organic loading rates and also improve the physical properties of the digestate, by reducing the tendency to foam and improving the dewaterability. Thermophilic digesters were run with mesophilic digesters as controls.

Summary method

The inoculum for the mesophilic digesters was prepared by mixing at a 1:1 mass ratio digestates taken from an anaerobic digester treating sugar beet pulp (British Sugar, Wissington, UK), and one treating municipal wastewater biosolids (Millbrook Wastewater Treatment Works, Southampton, UK). The mesophilic digesters (M1-M4) were initially fed at an OLR of 3 g VS l^{-1} day^{-1} which was then steadily raised to the target OLRs of 4 g VS l^{-1} day^{-1} by day 34 in M1 and M2, and 5 g VS l^{-1} day^{-1} by day 62 in M3 and M4.

The thermophilic digesters (T1 - T4) used inoculum from the municipal wastewater biosolids digester which was acclimated to thermophilic conditions by raising the inoculum temperature from 35 ^{o}C to 55 ^{o}C in one step and then not feeding for 12 days. After this the OLR was steadily increased from 0.5 g VS l^{-1} day^{-1} to 4 g VS l^{-1} day^{-1} by day 120 in T1 and T2, and to 5 g VS l^{-1} day^{-1} by day 150 in T3 and T4. Digesters were then operated for at least 3 HRT, with one HRT being equal to 68.5 and 54.8 days at OLR 4 and 5 g VS l^{-1} day^{-1}, respectively.

Each digester received trace element (TE) supplementation based on the amount of feedstock added to maintain a cation concentration (in addition to that naturally present from the feedstock) of 10 mg l^{-1} Fe (as $FeCl_2 \cdot 4H_2O$); 1 mg l^{-1} of Co ($CoCl_2 \cdot 6H_2O$), Mn ($MnCl_2 \cdot 4H_2O$), Ni ($NiCl_2 \cdot 6H_2O$), Zn ($ZnCl_2$); and 0.1 mg l^{-1} of Cu ($CuCl_2 \cdot H_2O$), B (H_3BO_3) and Al ($AlCl_3 \cdot 6H_2O$). Likewise, oxyanion concentrations were maintained at a minimum of 0.1 mg l^{-1} of Mo (as $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$), Se ($Na_2SeO_3$) and W ($Na_2WO_4 \cdot 2H_2O$). This formulation was based on a preliminary version of that subsequently recommended by Banks et al. (2012). In both cases the TE were supplied by weekly addition of concentrated solutions of these two mixes.

Results

Results of monitoring are shown in Figure 4.19 to 4.23, and average values for key parameters over a 30-day period at the end of 3 HRT for both mesophilic and thermophilic digesters are given in Table 4.8.

Table 4.8. Steady state values of performance and stability indicators for duplicate digesters in mesophilic and thermophilic condition (average over last 30 days of operation)

	Unit	Mesophilic		Thermophilic	
		OLR 4	OLR 5*	OLR 4	OLR 5
Specific Biogas production	l g^{-1} VS day^{-1}	0.554	0.549	0.664	0.681
Specific Methane Production	l CH_4 g^{-1} VS day^{-1}	0.292	0.283	0.345	0.355
Vol. Biogas Production	l l^{-1} day^{-1}	2.22	2.67	2.64	3.41
Vol. Methane Production	l CH_4 l^{-1} day^{-1}	1.17	1.38	1.37	1.78
Spec Methane (VS destroyed)	l CH_4 g VS^{-1} $_{destroyed}$	0.358	0.369	0.391	0.402
VS destruction	%	85.2	76.6	88.3	88.2
pH	-	7.49	7.13	7.73	7.72
Total Ammonia Nitrogen	mg N kg^{-1} WW	1395	803	2094	1977
Total Alkalinity	g $CaCO_3$ kg^{-1} WW	16.1	11.6	16.9	16.7
Digestate VS	g l^{-1}	43.1	67.6	33.8	33.8
Digestate TS	g l^{-1}	61.5	89.6	47.6	47.8
Total VFA	mg l^{-1}	23	222	503	796
IA/PA ratio	-	0.35	0.66	0.32	0.32

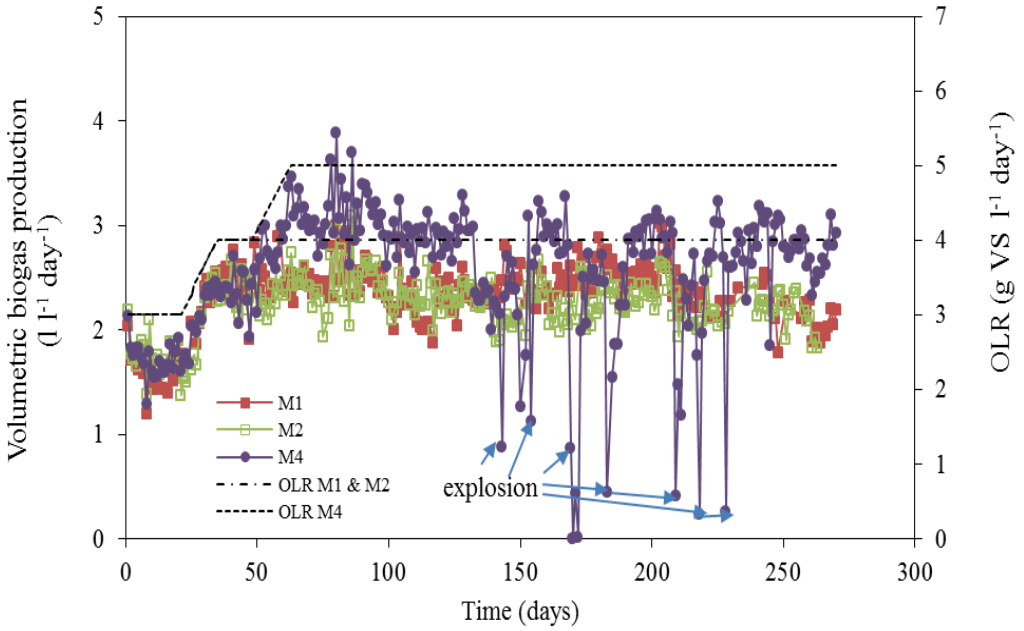
Note:*values for mesophilic digestion at OLR 5 g VS l^{-1} day^{-1} are for M4 only, as M3 failed before 3HRT due to foaming problem

The specific biogas and methane production was higher in thermophilic than in mesophilic conditions at both OLR tested. As had been observed in the previous mesophilic trial (section 4.4) specific biogas and methane production reduced slightly as the OLR increased. In both mesophilic and thermophilic conditions the increased OLR gave an increase in volumetric biogas production, as expected and previously demonstrated in the mesophilic trial. As in the previous trial, operating in mesophilic conditions at an OLR > 4 g VS l^{-1} day^{-1} led to severe foaming in the digester and this had a tendency to block the gas outlet line. This led to quite wide variations in values for volumetric biogas and methane production, in particular in digester M4 fed at OLR 5 g VS l^{-1} day^{-1}, due to uncontrollable 'blow outs' which led to unstable conditions (Figure 4.19a and 4.20a).

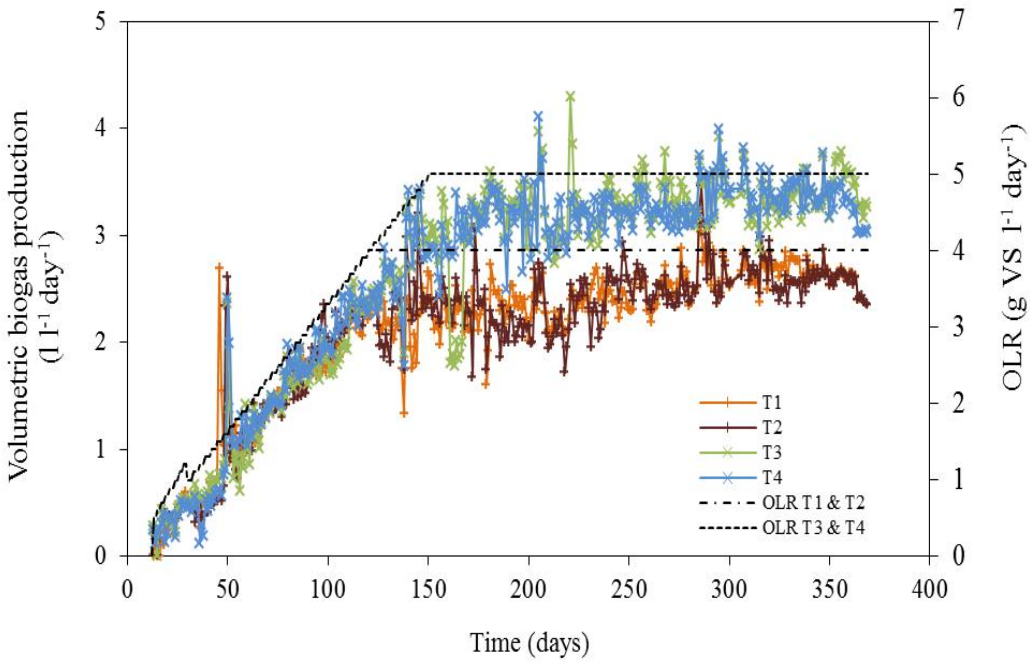
In the thermophilic digesters specific and volumetric biogas and methane production at both OLR 4 and 5 g VS l^{-1} day^{-1} were stable (Figure 4.19b and 4.20b) and higher than for the mesophilic digesters. The results are in agreement with those observed by Nges and Liu (2010) and more generally with those of Ferrer et al. (2010) who found that in thermophilic AD, the increase in methane production rate remained linear with increasing OLR. This is probably as a result of the greater degradation capacity in thermophilic digesters (Ahn and Forster, 2002), and can be attributed to the growth rates

of methanogens in thermophilic digesters which can be 1.6 - 3 times higher than those in mesophilic digesters (Kiyohara et al., 2000).

The total VFA concentration in the mesophilic digesters increased during transitional increases in OLR but under steady state conditions remained below 250 mg l^{-1} (Figure 4.21). Increasing the OLR also caused a very slight decrease in the pH although fluctuations remained within the range 7.2-7.5 (Figure 4.23a). Similar trends occurred in the thermophilic digesters, but the average pH was slightly higher at ~pH 7.7 (Figure 4.24a) as a result of the higher alkalinity. The total VFA concentration, however, was more than double that in the mesophilic digesters with an average of ~503 mg l^{-1} and ~796 mg l^{-1} at the end of 3 HRT for the two OLR applied. Thermophilic digestion showed a gradual increase in VFA concentration over the 200 days of operation, but at the end of the experiment (day 364) values were still below 1000 mg l^{-1} (Figure 4.22). The results agree with other reports that indicate VFA concentrations in thermophilic digesters are higher than in comparable mesophilic digesters (Kim et al., 2002; Moen et al., 2003; Song et al., 2004; de la Rubia et al., 2006; Nges and Liu, 2010).

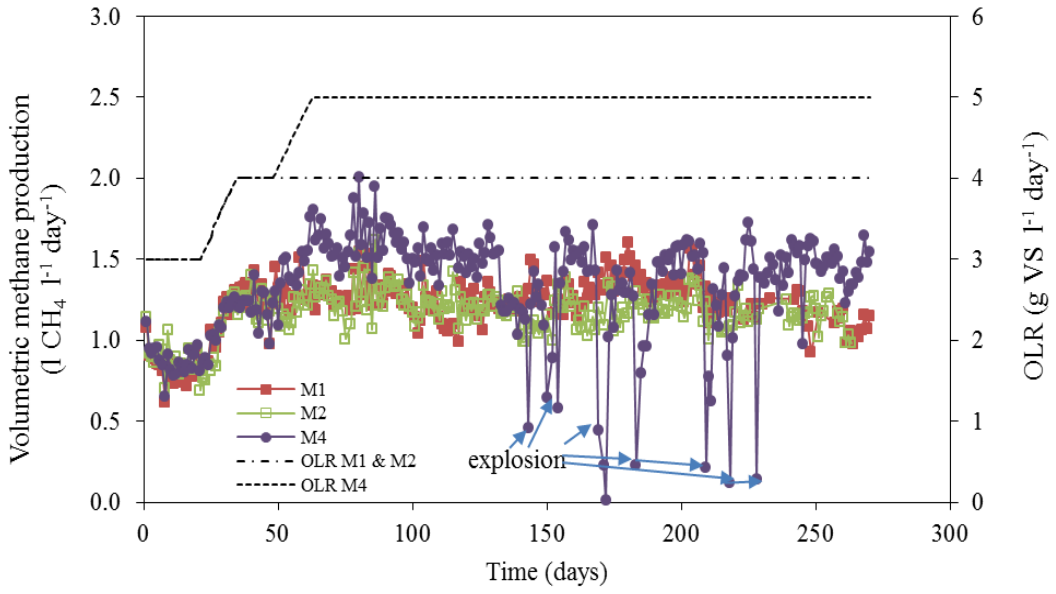


(a)

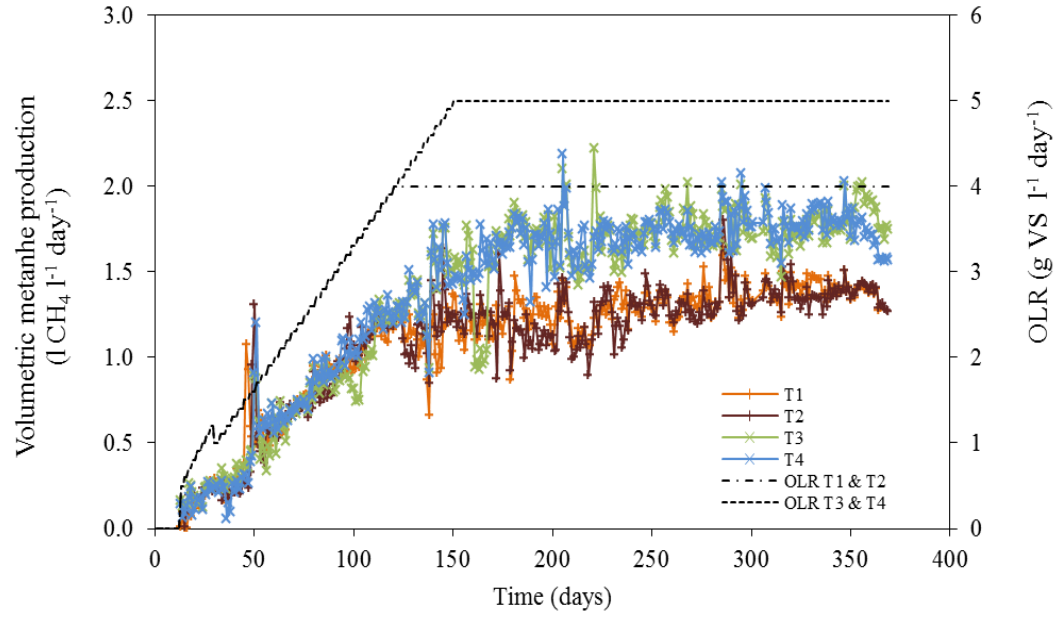


(b)

Figure 4.19. Daily volumetric biogas production in mesophilic (a) and thermophilic (b) digesters at OLR of 4 and 5 g VS l^{-1} day^{-1}

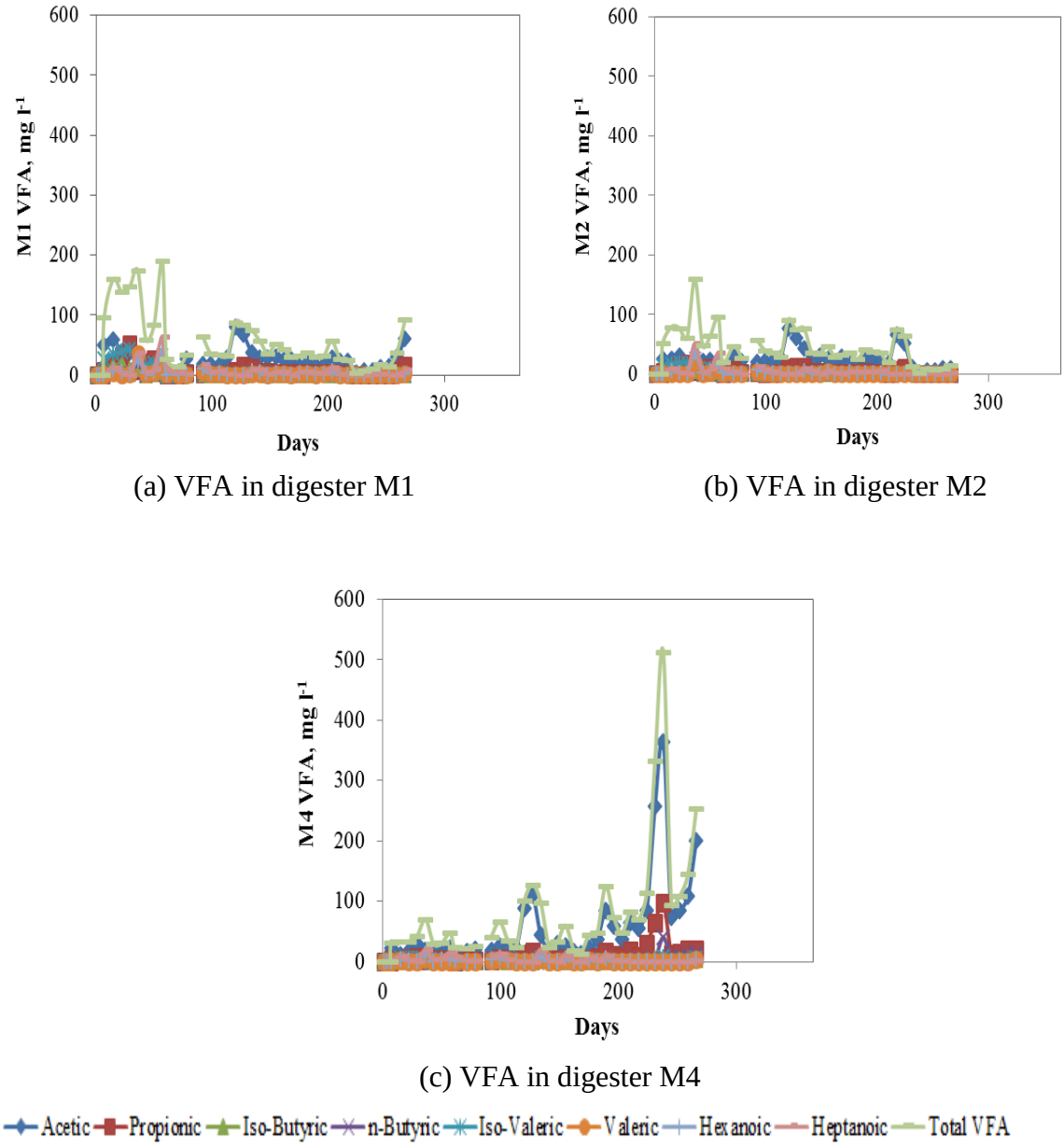


(a)



(b)

Figure 4.20. Daily volumetric methane production in mesophilic (a) and thermophilic (b) digesters at OLR of 4 and 5 g VS l^{-1} day^{-1}

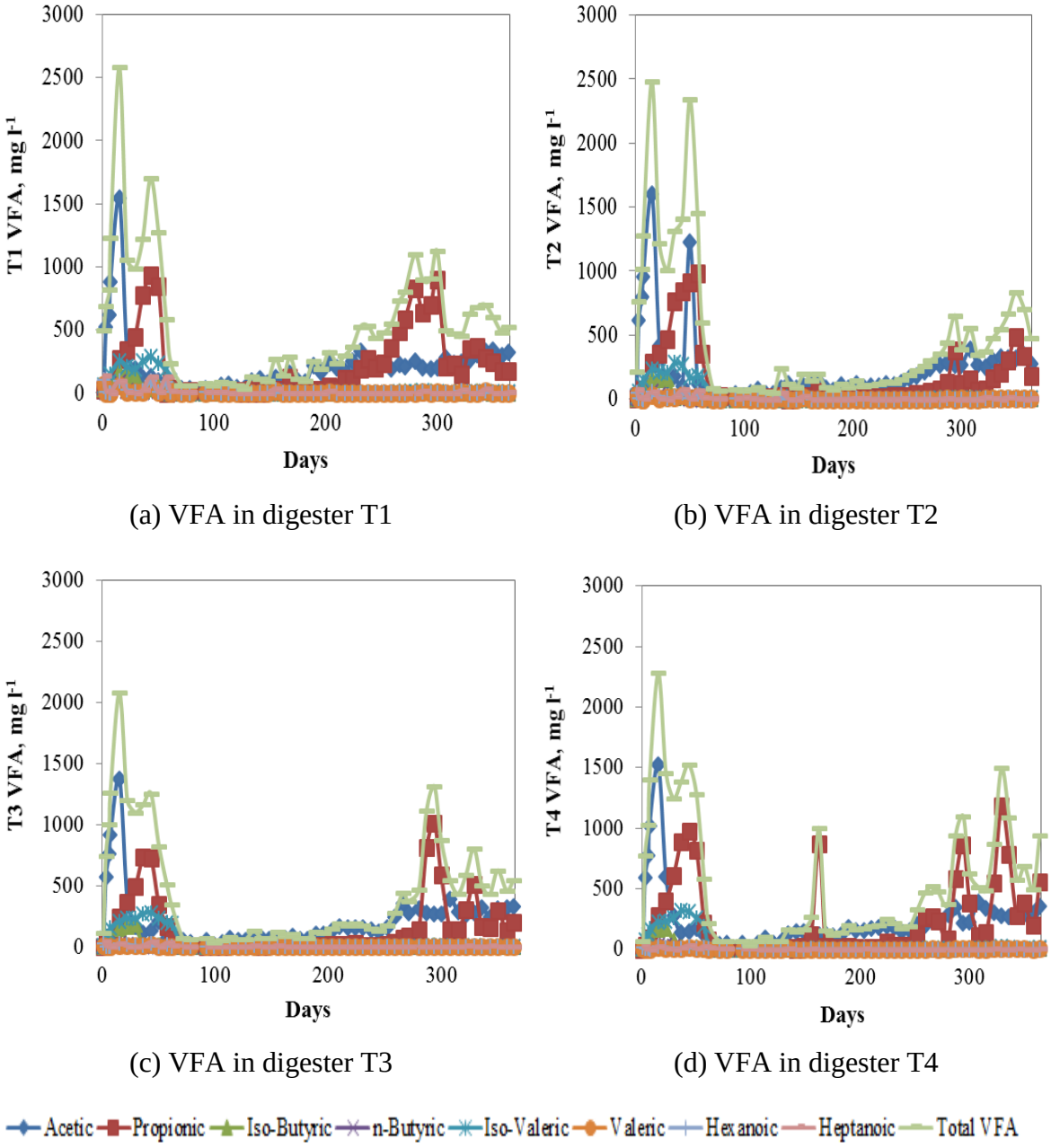


(a) VFA in digester M1

(b) VFA in digester M2

(c) VFA in digester M4

Figure 4.21. VFA profiles in mesophilic digester at different OLR: 4 g VS l^{-1} day^{-1} (a & b); 5 g VS l^{-1} day^{-1} (c)



(a) VFA in digester T1

(b) VFA in digester T2

(c) VFA in digester T3

(d) VFA in digester T4

Figure 4.22.VFA profiles in thermophilic digester at different OLR: 4 g VS l^{-1} day^{-1} (a & b); 5 g VS l^{-1} day^{-1} (c & d)

As in the previous trial (section 4.4), there was an initial increase in total alkalinity and TAN concentrations: but under mesophilic conditions the steady state TAN concentration was ~1.4-1.5 g N l^{-1} which is well below values considered inhibitory (Yenigün and Demirel, 2013). At an OLR of 5 g VS l^{-1} day^{-1} there was a decrease in total alkalinity and TAN concentration (Figure 4.23b and c), but there was still sufficient alkalinity to prevent any drop in pH. The reduced TAN concentration at OLR 5 g VS l^{-1} day^{-1} could have been due to a reduction in protein hydrolysis (Gallert and Winter, 1997) and washout as a result of a shortened HRT and overloading (Miron et al., 2000), as ammonia is mainly produced from nitrogenous materials such as proteins (Chen et al., 2008b). In contrast, thermophilic AD at both OLRs tested showed a gradual increase over time in both TAN and alkalinity reflecting greater hydrolysis. In the case of thermophilic digestion there was, however, a danger that the TAN concentration of 2.5 g N kg^{-1} WW at end of experiment on day 364 (Figure 4.24b and c) could be on the threshold of toxicity at this temperature (Hashimoto, 1986; Angelidaki and Ahring, 1993; Gallert and Winter, 1997; Liu and Sung, 2002; Yirong et al., 2013). This in turn may account for the slightly higher VFA concentrations seen in the thermpohilic digesters in this period. In general, pH, total alkalinity and ammonia nitrogen values were higher for the thermophilic digester at all OLRs tested, similar to the finding by Moen et al (2003).

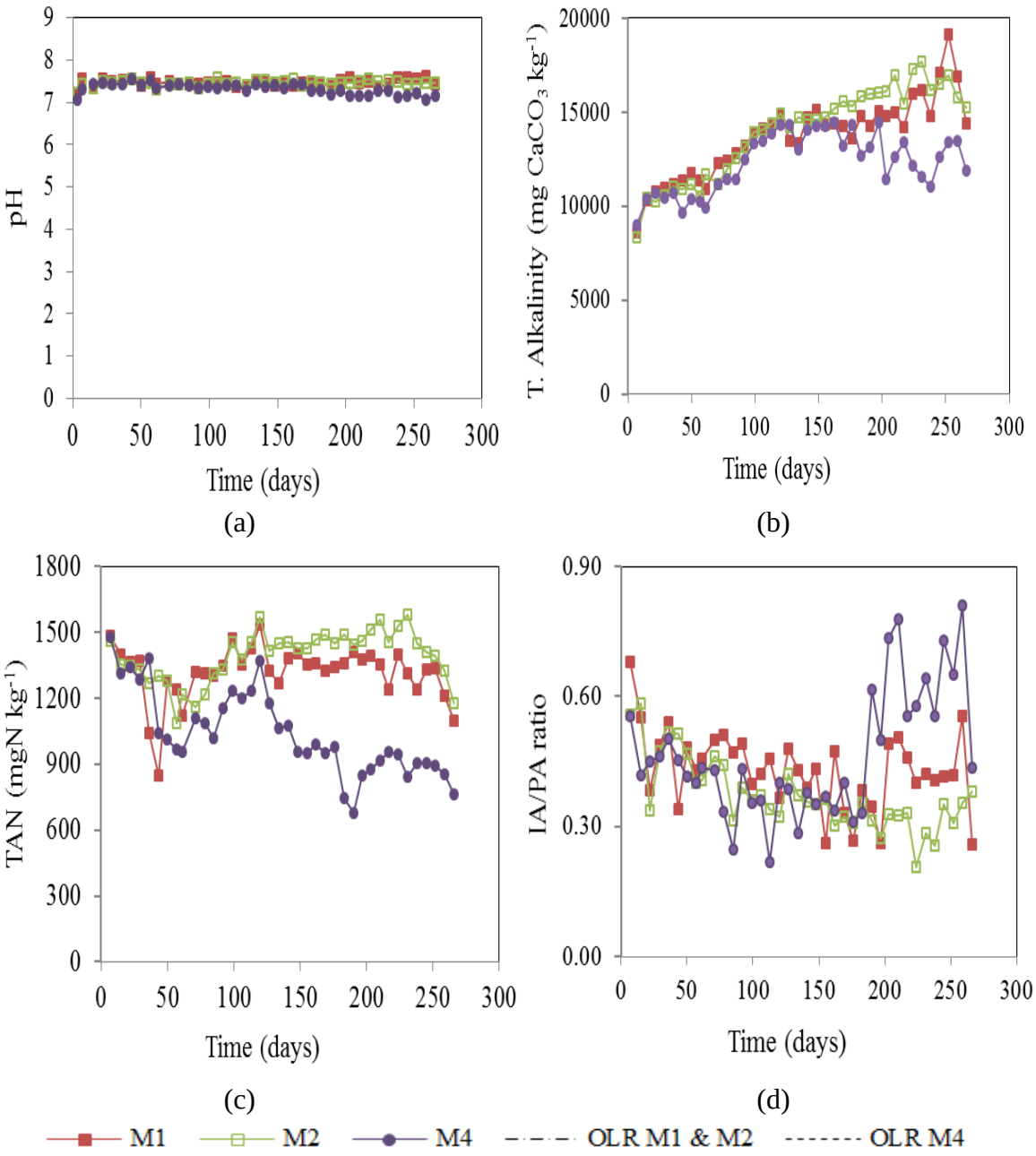


Figure 4.23. Trends in pH, total alkalinity, total ammonia nitrogen (TAN) and IA/PA ratio, in the mesophilic digester fed at OLR of 4 and 5 g VS l^{-1} day^{-1}

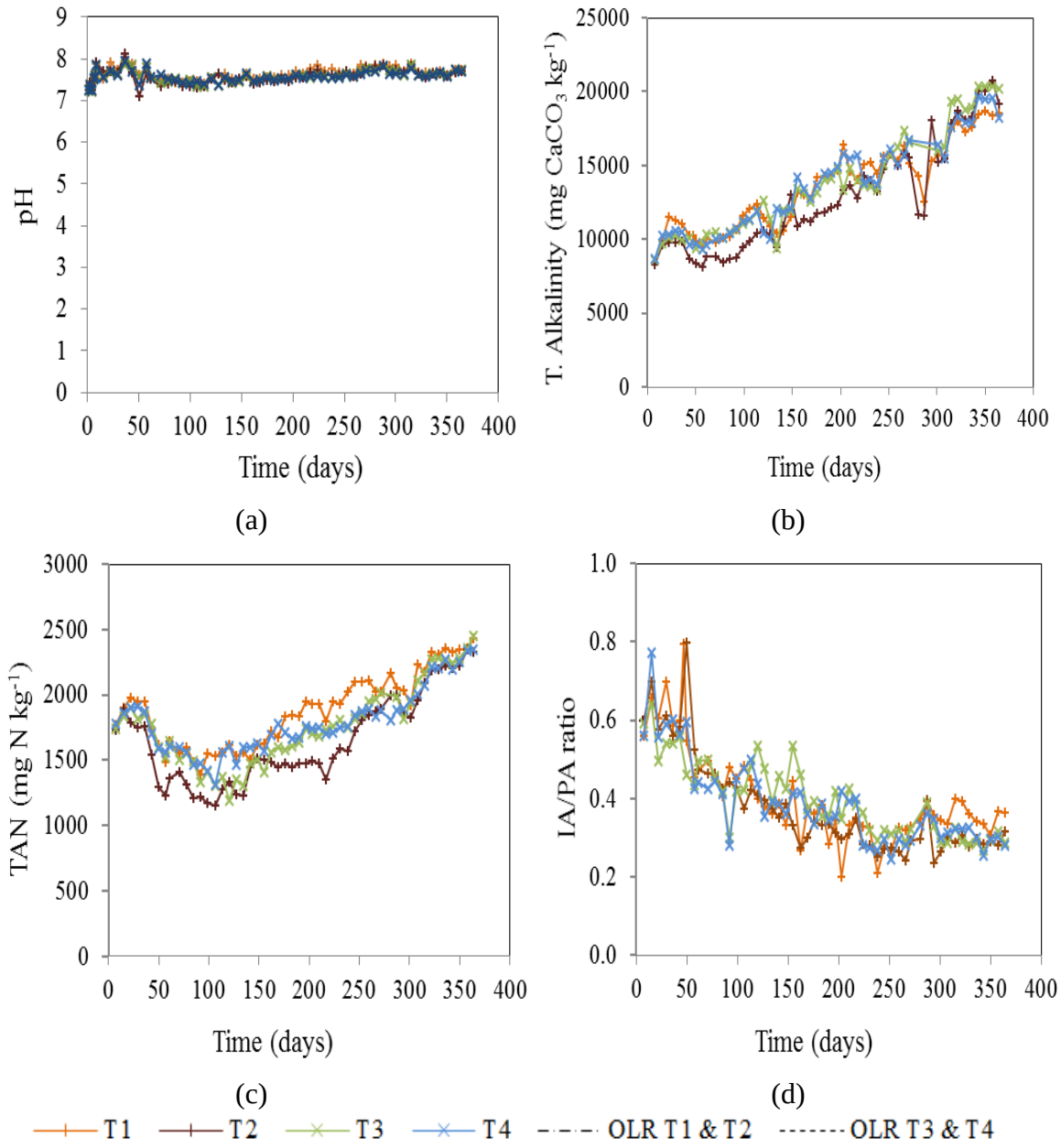
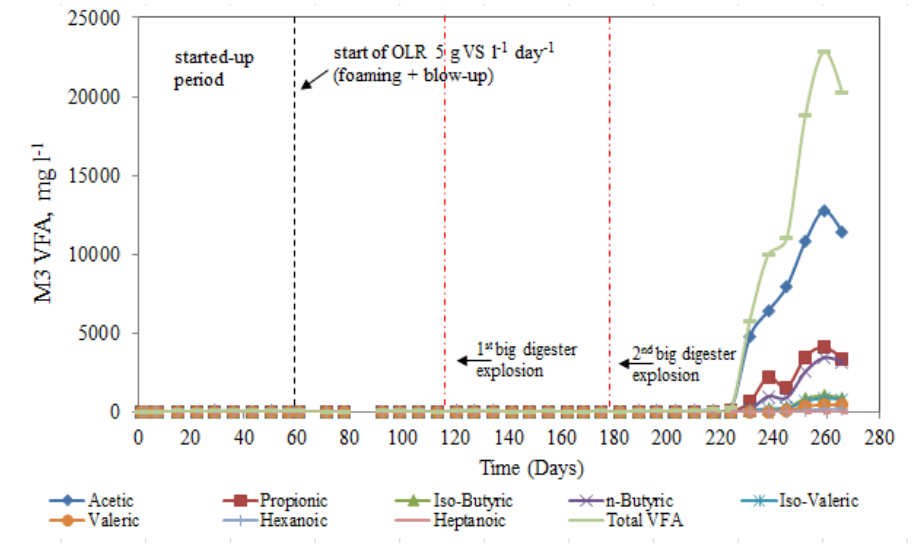


Figure 4.24. Trends in pH, total alkalinity, total ammonia nitrogen (TAN) and IA/PA ratio, in the thermophilic digesters fed at OLR of 4 and 5 g VS l^{-1} day^{-1}

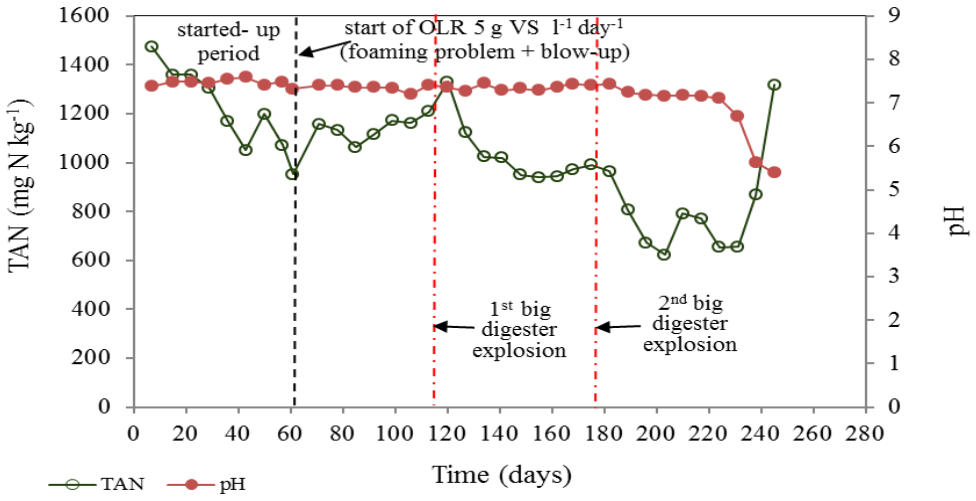
The IA/PA ratio for all digesters under thermophilic conditions was 0.32 (Figure 4.23d), indicating good process stability (Ripley et al., 1986). Zhao and Kugel (1996) also noted that an IA/PA ratio below 0.4 indicated sufficient buffering capacity. In the mesophilic digesters, however, the IA/PA ratio was slightly higher with values in the range 0.35-0.66 indicating a potential decrease in process stability with increasing OLR (Figure 4.22d); this was also reflected in the lower pH and higher VFA concentrations observed. Under mesophilic conditions the average VS destruction was 85% at an OLR

of 4 g VS l^{-1} day^{-1} and 77% at an OLR of 5 g VS l^{-1} day^{-1}. This result confirmed that previously seen for mesophilic digesters (section 4.4) and is in contrast to the behaviour of the thermophilic digesters which showed no loss in VS destruction from 88.3% as the OLR was increased. Other studies have also found that VS destruction in thermophilic digestion is higher than that in mesophilic digesters using the same substrate (Borghi et al., 1999; Song et al., 2004)). The higher VS destruction is also related to the higher biogas and methane productions as previously stated.

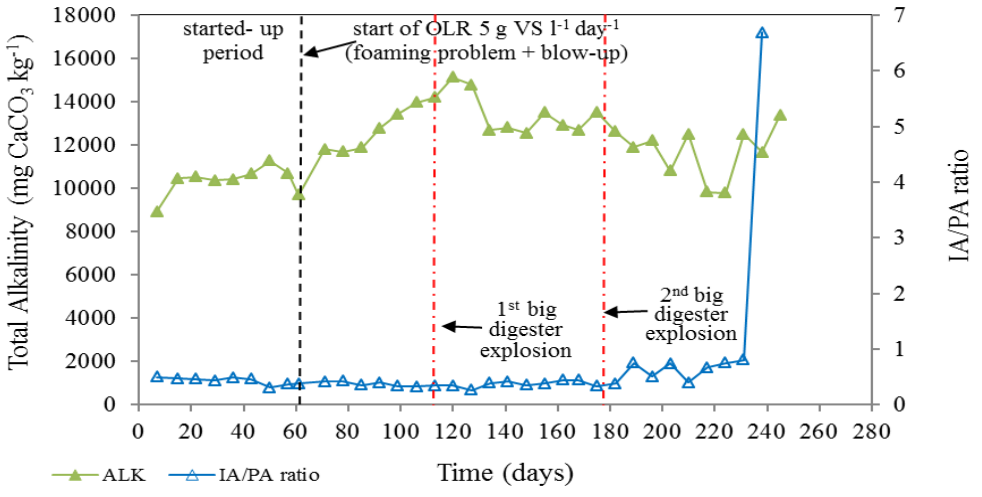
Excluded from the above results are those for digester M3 in which the OLR was increased to 5 g VS l^{-1} day^{-1}, in line with that for digester M4. Digester M3 suffered extreme foaming which was first noted on day 100. Despite a reduction in the working volume there was an explosive loss of digestate on days 115 and 179, with smaller losses on other occasions. Although attempts were made to recover the digester by adding waste digestate from digester M4, this was not entirely successful. These losses led to process imbalance accompanied by a large increase in total VFA and a decrease in pH. The series of events that led to the eventual failure of this digester are shown in Figure 4.25 and were brought about by the high total VFA concentration, mainly as a result of acetic and propionic acids, which reached ~20.3 g l^{-1} on day 266. This was reflected in the IA/PA ratio increasing to 6.68, the pH falling to <6 and methane production falling to 0.044 l CH_4 g^{-1} VS day^{-1} as shown in Figure 4.26.



(a)



(b)



(c)

Figure 4.25. Total VFA (a); total ammonia nitrogen (TAN) and pH (b); and , IA/PA ratio and total alkalinity as seen in digester M3 operated unsuccessfully at an OLR 5 g VS l^{-1} day^{-1}

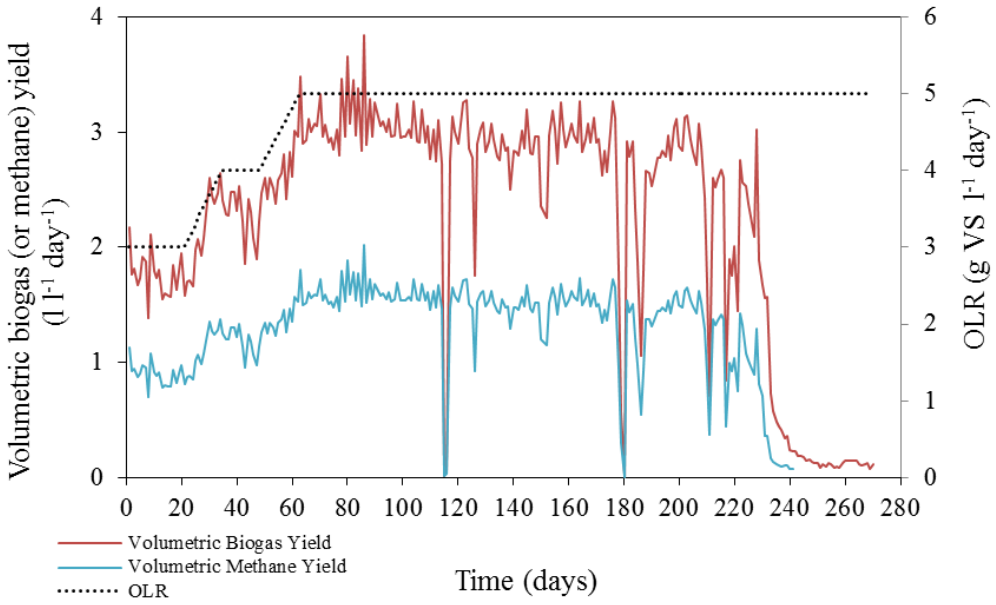


Figure 4.26. Daily volumetric biogas and methane production in the mesophilic digester fed at OLR 5 g VS l^{-1} day^{-1} after experiencing loss of digestate due to foaming occurrence (2^{nd} trial)

4.7. Overall discussion and conclusions from digester studies

Although SBP was shown to be easily degraded under ideal conditions in the BMP test, with a good gas yield and VS destruction, in practice semi-continuous digestion under mesophilic conditions proved to be much more difficult, particularly at high OLR. An increase in OLR to above 3 g VS l^{-1} day^{-1} led to symptoms of process imbalance, with a reduction in specific methane production and in VS destruction. The effects of a high OLR were also shown by a substantial increase in IA/PA ratio to above 0.4, indicating deterioration in process stability. Further confirmation of the negative effect of high OLR on AD of SBP was obtained from the behaviour of M1 - M4 in section 4.6., where all digesters experienced disturbances in the digestion process, as shown by a high IA/PA ratio and low biogas and methane production. The addition of trace elements did not appear to give any major improvement in process stability.

The major problem encountered under mesophilic conditions was that of stable foam formation, which was more evident in highly loaded digesters. A study by Brook et al. (2008) found foaming problems at loading rates above 5.5 kg COD m^{-3} day^{-1} (laboratory scale) and 10 kg COD m^{-3} day^{-1} (pilot scale), and attributed this to the high sugar content of fresh SBP. Stoyanova et al. (2013) also observed stable foam production in single-stage mesophilic AD of sugar beet pressed pulp fed at a high loading rate of 8.45

kg VS m^{-3} day^{-1}. Ganidi et al. (2011) were able to identify that, in bench-scale batch anaerobic digestion, an organic loading of 2.5 kg VS m^{-3} was a critical threshold for foam initiation for sludge obtained from a non-foaming full scale digester while 5 kg VS m^{-3} resulted in persistent foaming.

Stoppok and Buchholz (1985) observed foaming in mesophilic SBP digesters and attributed this to the high viscosity of the fluid-substrate mixture and the high cellulosic composition of SBP. Ganidi et al. (2009) stated that high viscosity limits the escape of small biogas bubbles from the digestate into the gas phase, causing foam formation. Stoyanova et al. (2013) found that the digestate viscosity in single-stage AD of sugar beet pressed pulp was much higher than in a two-stage system, leading to more foam formation in the single-stage system. Although no measurements of digestate viscosity were undertaken in the current study, based on visual inspection the mesophilic digestates appeared to be considerably more viscous than the thermophilic and in mesophilic conditions the higher OLR digestate more viscous than the lower OLR.

Several studies have found that high viscosity is also linked to poor dewaterability of sludges and digestate (Christensen et al., 1993; Dentel and Abu-Orf, 1995; Jin et al., 2004; Chen and Yang, 2012). As noted in Chapter 2, many studies have reported that thermophilic digestion produced a better quality and dewaterability of digestate compared to mesophilic (Buhr and Andrews, 1977; Kim et al., 2002; Bouallagui et al., 2004; Chi et al., 2010; Amani et al., 2011). In the current study, the differences between mesophilic and thermophilic digestates may be due to a higher destruction of ligno-cellulosic materials in thermophilic conditions.

In thermophilic conditions the higher OLR did not have a negative effect on digestion performance, in contrast to what happened in mesophilic AD. There was also evidence that thermophilic digestion had a higher degradation efficiency and degradation rate compared to mesophilic. This is indicated by, for example, the high VS destruction (~89%) and an increase in SMP (~0.355 l CH_4 g^{-1} VS day^{-1}) compared to the equivalent values for mesophilic digestion of ~77% VS destruction and ~0.283 l CH_4 g^{-1} VS day^{-1} at the same OLR of 5 g VS l^{-1} day^{-1}. VS destruction is one of the best parameters for determining the degree of degradation in an anaerobic digestion process, as it indicates the amount of organic material that has been broken down. The results for SBP

therefore support those from other studies that have reported a better performance from thermophilic digestion than mesophilic in terms of: organic matter destruction, process stability, and specific biogas and methane production (Buhr and Andrews, 1977; Cecchi et al., 1991; Converti et al., 1999; Kim et al., 2002; Bouallagui et al., 2004; Kim et al., 2006; Vindis et al., 2009).

The increase in degradation rate in thermophilic digestion may be due to the enhanced growth rate and metabolism of microorganisms at the higher temperature. Several studies have found that the hydrolytic activity or hydrolysis coefficient, which defines the degradation rate of organic materials, was higher in thermophilic digestion than in mesophilic (Kim et al., 2003; Song et al., 2004; Bouallagui et al., 2004). This is matched by an increase in the growth rate and activity of methanogens (Ahn and Forster, 2002; Moen et al., 2003; Appels et al., 2008; Ho et al., 2013), to approximately double that in in mesophilic methanogens (Zinder et al., 1984; Clarens and Moletta, 1990; Kiyohara et al., 2000; Siegrist et al., 2002), giving an overall improvement in digestion performance indicated by an increase in biogas production and in degradation rates.

The most obvious advantage obtained from thermophilic AD of SBP besides the above parameters was elimination of foaming. From the experimental results, no foaming was observed during the digestion process at either of the OLRs tested.

Conclusion

Thermophilic digestion was superior and better suited to the digestion of SBP when compared to mesophilic digestion. It could operate at higher organic loading rates, produced a higher specific methane yield, and offered better solids destruction. The mesophilic process when operated at a high organic loading rate suffered further disadvantages in that it showed a relatively high residual methane production, indicating a potential for methane emissions during any digestate storage phase. The major finding, however, was that thermophilic digestion was unlikely to result in foaming problems in the digester, and possible causes and reasons for the foaming behaviour of both systems are investigated in the following chapter.

CHAPTER 5. DEWATERABILITY CHARACTERISTICS AND FOAMING OCCURRENCE IN AD OF SBP

5.1. Chapter Summary

The major problems encountered in the semi-continuous anaerobic digestion of SBP were the difficulty of dewatering the digestate and the appearance of a stable foam, especially in more highly-loaded digesters. The dewatering behaviour of digestate from AD of SBP and the effects of antifoam addition were therefore studied. Several treatments were performed to improve digestate dewaterability, including: physical, chemical, sludge ageing, enzyme, thermal treatment and the use of centrifugation. Techniques used to measure the degree of dewaterability and digestate condition included: capillary suction time (CST), frozen image centrifuge (FIC), Buchner funnel test and scanning electron microscopy (SEM).

The CST value and the filtration test indicate the required suction or pressure needed to separate water from the digestate solids. The test is therefore affected by filter blinding, particularly if the digestate contains fine particles that can block filter pores. When this occurs, the results are often interpreted as indicating a hydrophilic digestate where water is strongly bound between discrete particles (Colin and Gazbar, 1995; Jin et al., 2004). The FIC test, on the other hand, is based on the movement of solids through the liquid phase by centrifugation and, provided the water is not strongly bound to the particles, larger particles will sediment more rapidly than fine ones. The FIC test thus provides a useful means of differentiating between a hydrophilic digestate and one containing fine particulates, as well as giving a direct indication of the relative dewaterability under centrifugation.

Some of the treatments above, such as chemical and thermal treatment, aimed to reduce or control foaming occurrence. Chemical treatment involved the use of antifoam and addition of TE into the digesters, while thermal treatment employed thermophilic AD of SBP at high OLR where the foaming mostly occurred. Foaming was visually observed and the foaming potential was measured as described in section 3.2. The effect of antifoam addition on digestion performance was also evaluated.

5.2. Dewaterability characteristics of digestate

5.2.1. Dewaterability of fresh mesophilic digestate

Objective. To determine the dewaterability characteristics of fresh digestate obtained from AD of SBP at mesophilic temperature, and to examine the effect of TE supplementation on digestate dewaterability.

Method. Digestate samples were taken from digesters run at OLR of 2 to 5 g VS l^{-1} day^{-1} (without TE addition) and OLR 3 g VS l^{-1} day^{-1} (with TE addition) (see section 4.4.3). The samples were taken on days 210 and 154, and were tested for CST, filtration and FIC according to the methods in section 3.2.5.

Results

CST and Filtration. The CST of fresh digestates from all digesters trialled (with and without TE supplementation) was in excess of 2 days, making this test unsuitable for identifying any differences between them. The filtration test failed to produce an identifiable filter cake within 20 minutes.

These extremely high CST values and the failure to produce a filter cake are usually interpreted to mean that the water is strongly bound to the digestate, and a suction or filtration method without any pre-treatment is unlikely to be successful if no supernatant liquid can be obtained in these periods.

FIC. After one hour at 100 *g* the FIC test was able to show a clear difference in the dewaterability of the digestates (Figure 5.1). The results showed that approximately 40%, 30%, 12% and 0% of supernatant could be separated in digestate from digesters fed at OLR 2, 3, 4, and 5 g VS l^{-1} day^{-1} without TE addition, and 40% in digestate from a digester fed at OLR 3 g VS l^{-1} day^{-1} with TE addition.

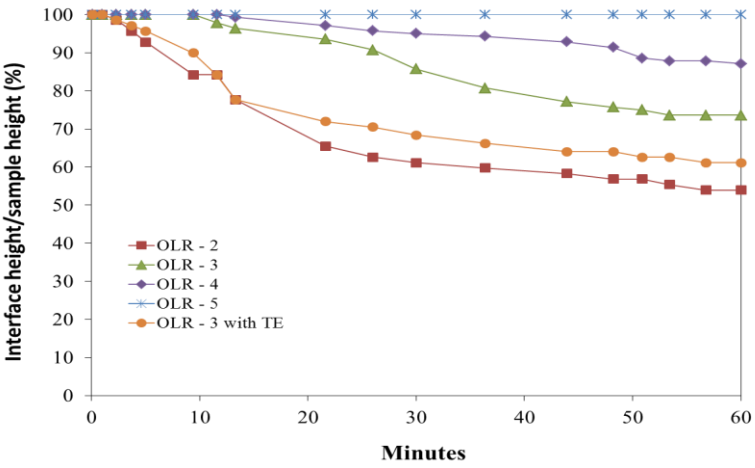


Figure 5.1. Supernatant interface height as a % of original height during FIC analysis for SBP digestates from digesters operating at different OLR at 100 *g*

Conclusions. From the above results it is clear that the untreated digestates can be dewatered to a limited extent without chemical destabilisation of the matrix to alter the floc structure and reduce hydrophilicity, and that addition of trace elements during digestion makes this easier. The fact that filtration and CST tests did not show any appreciable dewatering, while centrifugation did, suggests that the difficulty in dewatering SBP digestate may be associated with the presence of very fine particles. The results also demonstrated that increasing OLR corresponded to a decrease in dewaterability.

5.2.2. Effect of physical pre-treatments on dewaterability of digestate

5.2.2.1. Digestate dilution

The overall aim of the experiments was to see whether addition of water to SBP digestate could improve its dewaterability. Two digestates were investigated: one from the pilot-scale digester at British Sugar's Wissington Factory and one from the laboratory-scale digesters.

Experiment 1 – Dilution of British Sugar Wissington digestate

Objective. To identify the optimum dilution to be used in chemical treatment to improve digestate dewaterability.

Method. Fresh digestate collected from British Sugar's Wissington Factory was diluted with tap water as shown in Table 5.1. Undiluted digestate was used as a control sample.

Samples were stirred for 5 minutes using a magnetic stirrer at ~100 rpm to ensure uniformity. The samples were then tested for CST and FIC according to the methods in section 3.2.5.

Results

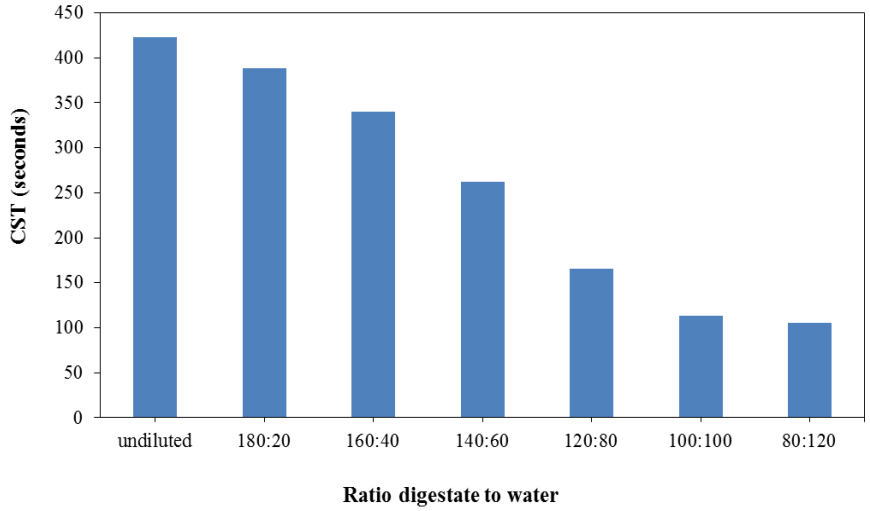
CST. CST decreased greatly with increasing dilution, from ~423 s for undiluted samples to 105 at for a digestate:water ratio of 80:120 (see Table 5.1 and Figure 5.2a), equivalent to a ~75% improvement. Unfortunately, these CST values still did not meet the commonly recommended value for dewatering purposes of < 10 s.

Table 5.1. Results of CST and FIC tests on digestate samples at different dilutions

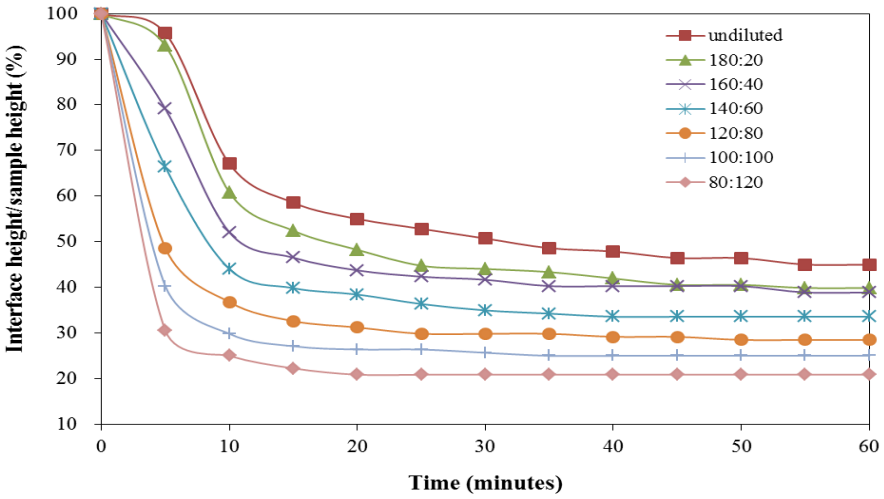
Ratio of digestate to water	CST (seconds)	Solid cake (% WW)	Initial TS (% WW)	Final TS (% WW)	Time* (minutes)
200:0 (undiluted)	422.8	47.8	4.1	8.1	55
180:20	388.1	42.2	3.6	7.6	50
160:40	340.6	37.7	3.3	7.8	50
140:60	261.8	29.0	2.8	8.0	30
120:80	165.4	28.4	2.5	7.9	25
100:100	113.4	24.6	2.1	8.0	20
80:120	105.1	19.6	1.6	7.8	20

Note: all values are averages of duplicate samples, * refers to time required to achieve the final interface height in FIC test

FIC. The FIC profiles showed the same trend as the CST results, with increasing dilution ratio giving increased supernatant separation. Approximately 50% of supernatant could be separated in the undiluted digestate. Adding water at ratio of 180:20 and 160:40 had only a small effect on separation, which increased to ~55%. As the ratio of digestate to water increased to 140:60, 120:80, 100:100, and 80:120, however, the separation increased to approximately 61%, 70%, 72%, and 80% (Figure 5.2b). The time needed in the FIC test to achieve the final interface height improved sharply with water addition, from ~55 mins with no dilution to ~20 mins with max dilution.



(a)



(b)

Figure 5.2. Results of dewaterability testing on digestates from British Sugar's Wissington Factory at different dilutions: (a) CST; (b) Supernatant interface height as a % of original height of SBP digestate during FIC run

Conclusions. These results indicated that diluting the digestate generally had a positive impact on dewaterability. On the basis of the experimental results, the most appropriate dilution ratio appeared to be 120:80 since it gave a 61% reduction in CST and reached the final interface height after ~25 mins of centrifugation, while ratios of 100:100 and 80:120 only improved this time by ~5 mins. The results for higher dilutions were not significantly better, and these have operational disadvantages (e.g. an increase in water consumption, greater volumes of supernatant for treatment or disposal etc.). Therefore, a 120:80 dilution ratio was used in the following dewaterability experiments.

Experiment 2 – Dilution of digestate from mesophilic and thermophilic AD of SBP

Objective: To compare the effect of dilution on digestates from mesophilic and thermophilic digestion of SBP.

Method. Digestate samples were taken from laboratory-scale mesophilic and thermophilic digesters fed at OLR 4 and 5 g VS l^{-1} day^{-1}, as described in section 4.6, in each case after 3 HRT of operation. The mesophilic digestate was sampled from digesters M1 and M4 on day 226, and the thermophilic digestate from digester T1 and T3 on day 237. The samples were tested 200:0 (no dilution) and 120:80 ratio of digestate to water. All diluted samples were stirred for 5 minutes to ensure homogeneity, using magnetic stirrer at ~100 rpm. The samples were measured for CST and FIC according to the methods in section 3.2.5.

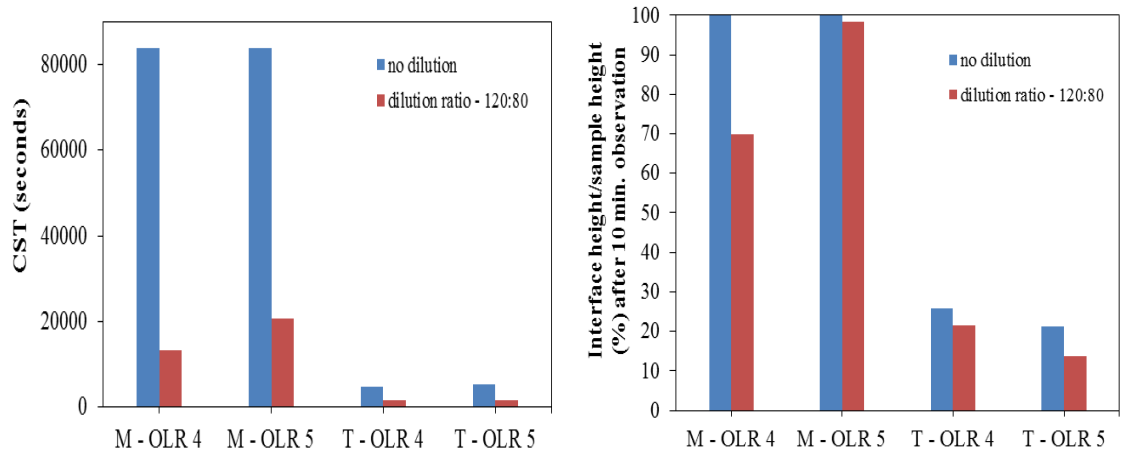
Results

CST. The results demonstrated a great difference in dewaterability characteristics between mesophilic and thermophilic digestate. Without dilution, the CST for mesophilic digestate was > 84000 s; after dilution this reduced to ~14000 s and ~21000 s at OLR 4 and 5 g VS l^{-1} day^{-1}, respectively (Table 5.2 and Figure 5.3a). For thermophilic digestate the CST without dilution was ~5000-6000 s, and after dilution this reduced to ~1500 s at both OLR tested.

Table 5.2. Results of CST and FIC test on digestate samples from mesophilic and thermophilic digesters at different dilutions

Parameters	undiluted				120:80 ratio of digestate to water			
	M-OLR4	M-OLR5	T-OLR4	T-OLR5	M-OLR4	M-OLR5	T-OLR4	T-OLR5
CST (s)	> 84000	> 84000	4592	5255	13227	20516	1455	1587
Interface height/sample height (%)	100	100	25.7	21.2	70.0	98.5	21.4	13.8
Solid-liquid separation (%)	0	0	74.3	78.8	30.0	1.54	78.6	86.2
Solid cake (% WW)	100	100	29.4	24.8	90.6	99.1	20.6	16.6
Initial TS (% WW)	8.5	8.7	4.1	4.6	6.5	6.4	4.1	3.6
Final TS (% WW)	8.5	8.7	10.5	11.3	7.8	5.5	9.1	9.9
Initial VS (% WW)	6.5	6.9	3.1	3.3	3.5	3.9	3.0	3.5
Final VS (% WW)	6.8	6.9	7.5	7.4	3.9	3.6	6.6	6.7

Note: all values are averages of duplicate samples



(a) (b)

Figure 5.3. Results of dewaterability testing on digestates from mesophilic and thermophilic AD of SBP with and without dilution: (a) CST; (b) Final supernatant interface height as a % of original height of SBP digestate in FIC test

FIC. FIC testing showed a considerable difference in supernatant separation for each type of digestate (Figure 5.3b). For mesophilic digestate at both OLR without dilution, no supernatant separation had occurred after 10 minutes centrifugation. After dilution this increased to 30% for at OLR 4 g VS l^{-1} day^{-1}, but only 1.54% at OLR 5 g VS l^{-1} day^{-1}. For thermophilic digestates much greater separation was possible, with values of ~75% and ~79% at OLR 4 and 5 g VS l^{-1} day^{-1} without dilution, rising to ~79% and ~87% respectively with dilution. Furthermore, the addition of water to thermophilic digestate reduced the time needed in the FIC test to achieve the final interface height, from ~7 mins with no dilution to ~4-5 mins with dilution, compared to that in mesophilic digestate from > 10 mins with no dilution to ~8-9 min with dilution.

The VS content of the mesophilic digestate at OLR 5 was slightly higher than at OLR 4; whereas VS concentrations in the two thermophilic digestates were very similar and much lower than in the mesophilic digestates (Table 5.2). VS content is associated with dewaterability (Houghton and Stephenson, 2002) and this may account for the difference between the digestates tested.

Conclusions. The results from this experiment confirmed that addition of water to the digestate improved the dewaterability of the SBP digestates. The extent of the effect, however, is likely to depend on the digestate type or composition.

5.2.2.2. Effect of feedstock dilution

Objective. To identify whether addition of water to the feedstock in AD of SBP under mesophilic conditions could enhance the dewaterability of the digestate.

Method. The digestates were obtained from digesters fed at OLR 4 g VS l^{-1} day^{-1} with the addition of tap water to the feed, as described in section 4.5.1. The samples were taken on day 131 and were analysed immediately using CST and FIC according to the methods in section 3.2.5.

Results

CST and FIC. The CST of fresh digestates from the digesters with dilution was in excess of 24 hours, similar to values obtained from digesters without dilution. Filtration tests showed that no liquid supernatant was produced after 20 minutes. FIC tests for the digestate were conducted at different centrifuge speeds in the range 264 – 1100 rpm (20-100 *g*) for 10 min. The results showed no separation at < 100 *g*; while a small amount of supernatant could be separated at 100 *g* with a final reduction of 2.82% from the initial height (Figure 5.4).

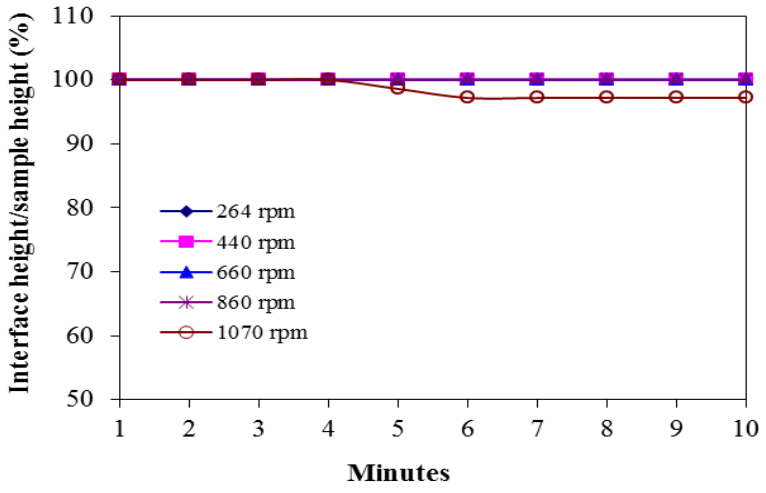


Figure 5.4. Supernatant interface height as a % of original height during FIC run (digestate originated from digesters with feedstock dilution) (average of 2 runs)

Conclusions. These results indicated that addition of water to the feedstock did not improve the digestate liquid-solid separation.

5.2.2.3. Digestate ageing study

The overall objective of these experiments was to assess whether ageing of the digestate altered its dewatering properties.

Experiment 1 – Sludge ageing of digestates at different temperatures

Objective: To assess the effect of temperature during the ageing process on digestate dewatering properties.

Method: The digestates used were from the pair of anaerobic digesters fed at OLR 3 g VS l^{-1} day^{-1} with and without TE supplementation (N3 and N9; see section 3.5.1). The samples were taken at the end of the experimental run (day 325) when the digesters had operated for ≥3 HRT. 150 g aliquots of digestate were placed in duplicate 250 ml Erlenmeyer flasks sealed with rubber bungs and maintained at 4 ^{o}C, room temperature and 35 ^{o}C. The experiment was carried out in duplicate and samples were taken at intervals of 2 - 4 weeks over a period of 9 months; the headspace was not flushed after sampling but left as air. Prior to undertaking any measurements, the sample was stirred manually (± 2 minutes) to ensure homogeneity. The digestate was immediately tested using CST and FIC tests according to the methods in section 3.2.5. Details of the experimental design are given in Table 5.3.

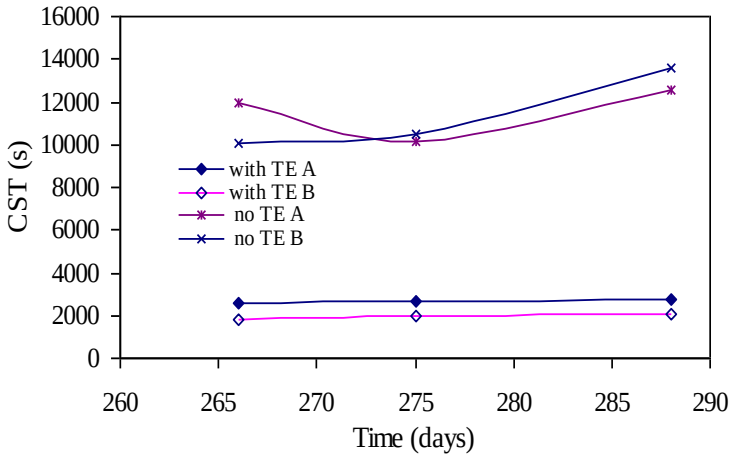
Table 5.3. Experimental design for the ageing study flask trial

Flask ID	OLR (g VS l^{-1} day^{-1})	Temperature (^{o}C)
S_1 and S_2	3 without TE	4
S_3 and S_4		Room temperature
S_5 and S_6		35
TE_1 and TE_2	3 with TE	4
TE_3 and TE_4		Room temperature
TE_5 and TE_6		35

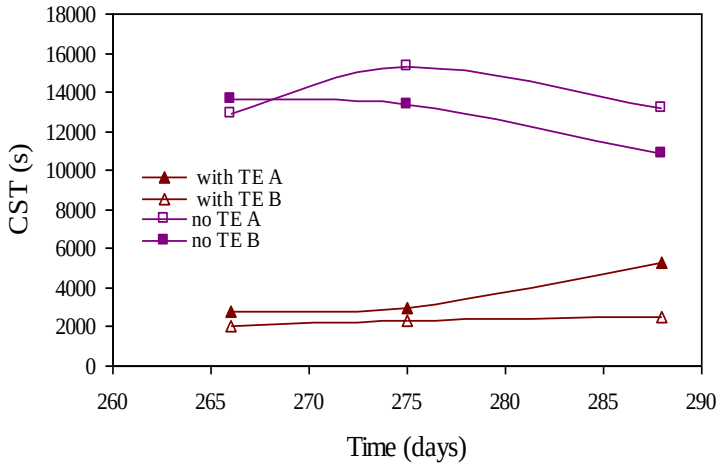
Results

Storage of the samples at 4 ^{o}C gave no significant improvement in CST value, which was in excess of 86,400 s for the first 7 months (day 202). After a further 2 months (day 288), the CST showed a considerable improvement, with values of ~11,000 s for digestate without TE and ~2,200 s with TE; these values, however, are still much higher than the recommended CST (Figure 5.5a). A similar result was found for samples at

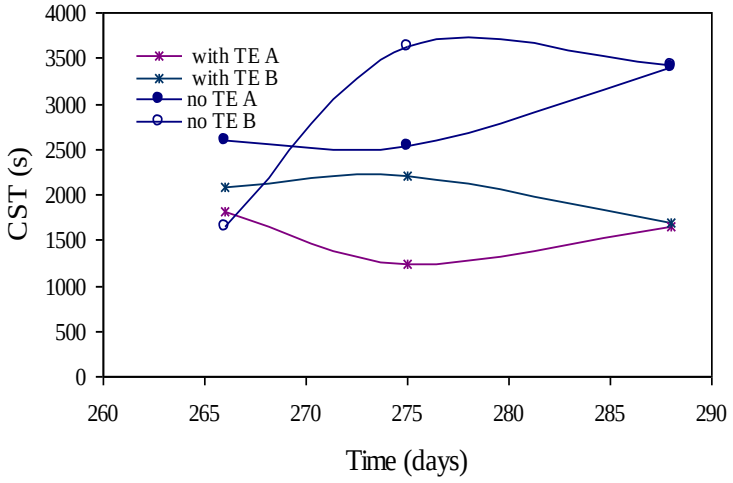
room temperature, where after 7 months (day 243) no significant effect was observed, with CST > 86,400 s; but after 9 months' storage the CST improved to ~2,000-3,300 s for digestate with TE and ~13,000 – 16,000 s without TE (Figure 5.5b). The samples stored at 35 ^{o}C showed larger changes, with CST of less than 10,000 s after 7 months; this decreased further over the next 2 months to ~1,000 – 2,500 s (with TE) and ~2,500 – 3,700 s (without TE) (Figure 5.5c). These results indicated that storage of digestate over a long period can reduce the CST, with the effect substantially enhanced at 35 ^{o}C. It is therefore likely that this is due to microbial activity in the digestate. On storage the digestate became more liquid especially when kept at 35 ^{o}C.



(a) CST of digestate stored at 4 ^{o}C



(b) CST of digestate stored at room temperature



(c) CST of digestate stored at 35 ^{o}C

Figure 5.5. CST at different temperatures for digestates with and without TE addition after storage for up to 9-month

Conclusions. It can be concluded that long-term storage is an option to improve the dewaterability of digestate from AD of SBP; but because of the length of time required it is unlikely to be economic due to the large storage volumes.

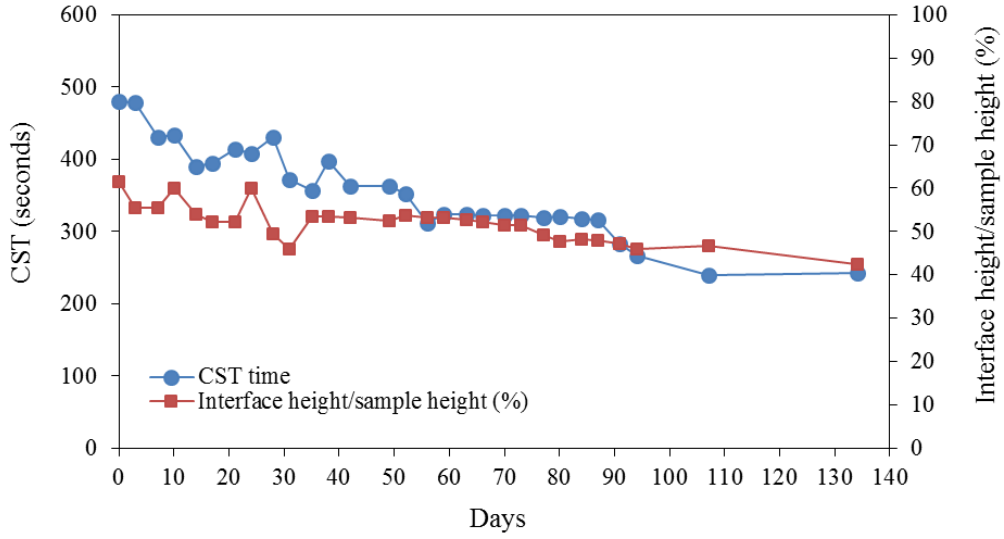
Experiment 2 – Sludge ageing of British Sugar Wissington digestate

Objective: To determine the effect of ageing of digestate at room temperature (~20 ^{o}C) on dewaterability characteristics.

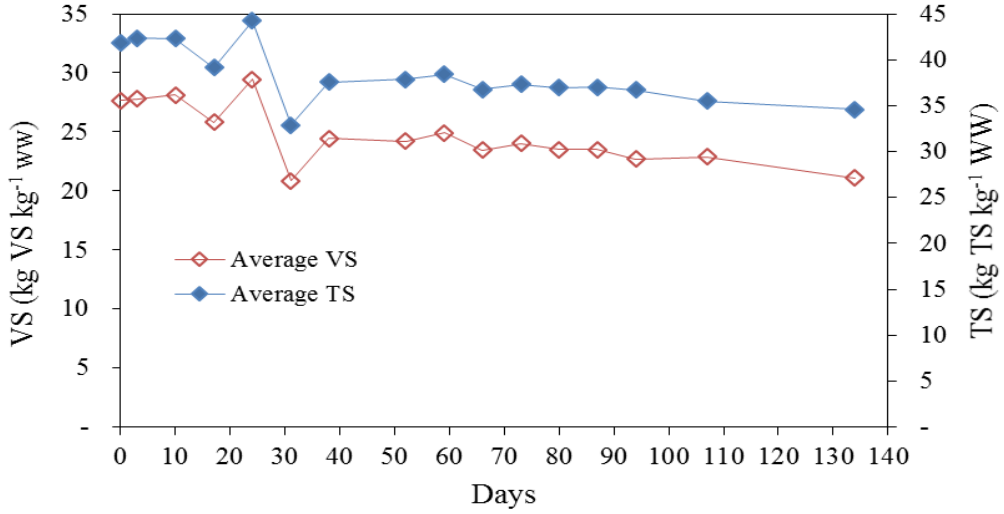
Method. This experiment used fresh digestate collected from a pilot plant operated by British Sugar Company at the Wissington Factory. Fresh digestate was placed in 10-litre open-top drum and stored at 20 ^{o}C in an incubator to maintain a stable temperature. Samples were analysed weekly for CST and FIC of the liquid supernatant. The ageing study ran for 134 days (8 May 2012 to 19 October 2012). The 10-litre sample was mixed for about 5 minutes before testing to ensure homogeneity. After mixing samples were immediately analysed by the CST and FIC tests according to the methods in section 3.2.5.

Results

Storage of the samples at 20 ^{o}C gave a slight improvement in supernatant dewaterability according to the CST and FIC test (Figure 5.6a). The initial CST was ~481 s and after 2 months (day 59) this had improved to ~325 s. The CST continued to decrease to ~243 s at the end of experiment (day 134). Separation of solids in the FIC test increased from an initial ~39% to ~58% by day 134. From the results in Figure 5.20, it can be seen that the reduction in CST was in line with the decrease in interface height. TS and VS values also slightly decreased over the storage period, from ~42 to ~36 g TS kg^{-1} WW (14% reduction) and from ~28 to ~23 g VS kg^{-1} WW (18% reduction) (Figure 5.6b).



(a)



(b)

Figure 5.6. Results of CST and FIC testing (a) and TS/VS (b) on ageing digestates from Sugar's Wissington Factory (in average value)

Conclusions. In the case of the sample from British Sugar Company, ageing of the digestate at room temperature improved dewaterability of the digestate and could offer a means of reducing the energy requirement for dewatering part of the material. The process may still not be feasible, however, due to time and volume constraints.

5.2.2.4. Freeze/Thaw treatment

The overall objective of this experiment was to assess whether freezing and then thawing digestate improved its dewaterability characteristics.

Experiment 1 – Freeze/thaw treatment of British Sugar Wissington digestate

Objective. To determine the effect of freeze/thaw (F/T) treatment on fresh digestate from the pilot-scale digester at British Sugar's Wissington Factory.

Method. Samples of fresh digestate from British Sugar's Wissington Factory were put into 500 ml PET plastic bottles (in duplicate) and placed in a freezer at -20 °C. Samples were frozen for 24 hours, then left to thaw for ~6 hours at room temperature, and mixed thoroughly. The dewaterability characteristics of the digestate were measured by the CST and FIC tests according to the methods in section 3.2.5.

Results

CST and FIC. After the F/T treatment there was a 30% reduction in CST from ~423 s to ~298 s (Table 5.4). The FIC test results also showed a significant improvement, indicated by an increase in supernatant separation after 10 minutes centrifugation from ~39% for fresh digestate to ~73% after F/T treatment (Figure 5.7). F/T treatment greatly reduced the time required to achieve the final separation percentage of the liquid fraction from ~10 mins to 1 mins, giving a difference in final interface height of ~34% after 10 minutes centrifugation. This big difference in the final separation percentage and time required implied that F/T conditioning prior to dewatering may be a possible option, especially in cold climate areas, with the added advantage of no requirement for chemicals.

Table 5.4. Results of CST and FIC tests before and after F/T treatment

Parameters	Initial	After F/T
CST (s)	423	298
Interface height/sample height (%)	61.1	24.7
Solid-liquid separation (%)	38.9	72.6
Solid cake (% WW)	26.7	25.1
Initial TS (% WW)	3.5	6.4
Final TS (% WW)	8.1	10.9
Initial VS (% WW)	2.3	4.4
Final VS (% WW)	5.7	7.8

Note: all values are averages of duplicate samples

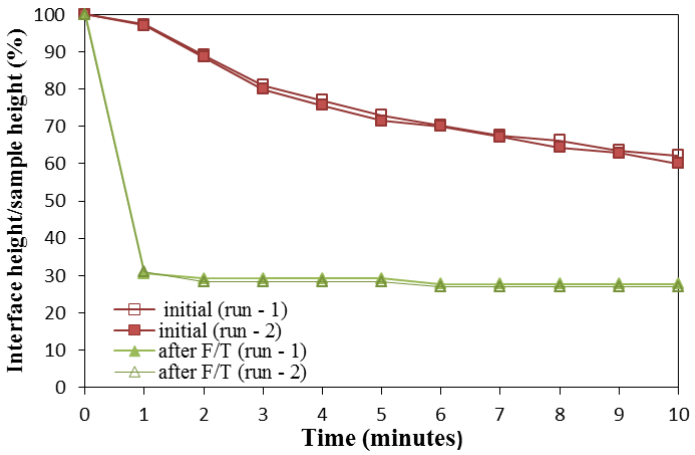


Figure 5.7. Supernatant interface height as a % of original height of SBP digestate during FIC run

The results showed that freeze/thaw treatment improved the dewaterability of the digestate samples without the need for sophisticated mechanical equipment or processing. Similar findings have been reported in other studies (Lee and Hsu, 1994; Wang et al., 2001; Vesilind and Martel, 1990; Hu et al., 2011). Previous studies found that formation of ice crystals during freezing may promote particle aggregation and break up colloids, and may also disrupt cells etc, thus changing the floc structure of the digestate into a denser form and freeing bound water enabling it to drain away (Vesilind and Martel, 1990; Lee and Hsu, 1994).

Conclusions. Freeze/thaw treatment enhanced the dewaterability of digestate; the CST time, however, was still less than that required for an effective dewatering process.

Experiment 2 – Freeze/thaw treatment of digestate from mesophilic and thermophilic AD of SBP

Objective. To determine the effect of F/T treatment on digestate from mesophilic and thermophilic digesters fed at OLR 4 and 5 g VS l^{-1} day^{-1}.

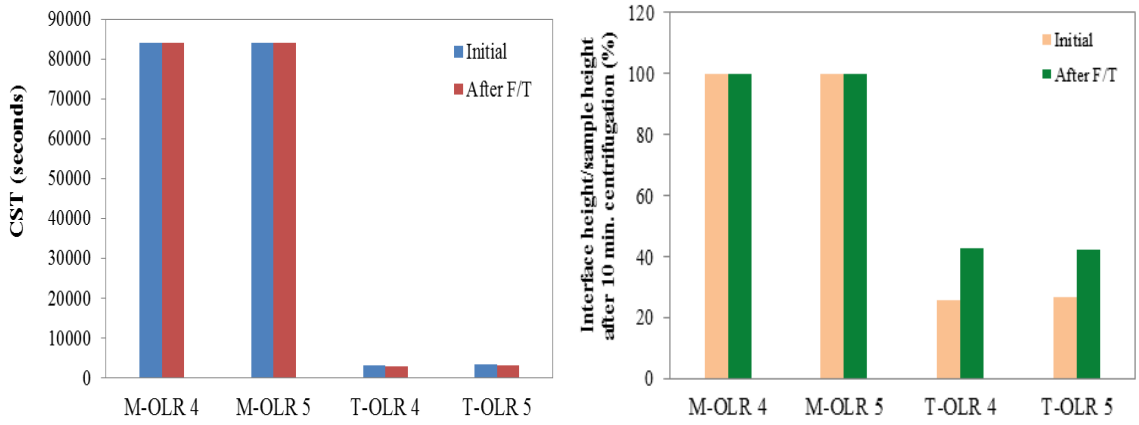
Method. The procedure used was the same as for the first F/T experiment. The digestate samples were taken from digesters M1, M4, T1 and T3 as described in section 4.6 on day 137. Changes in structure were assessed using SEM according to the method described in section 3.2.6.

Results

CST and FIC. Results for CST and FIC test are presented in Table 5.5 and Figure 5.8. The initial CST of the mesophilic digestate before treatment was > 84000 s for all OLR tested. After F/T treatment, there was no improvement in CST for mesophilic digestate. In contrast, the thermophilic digestate showed a small improvement from an average of ~3060 s and ~3435 s to ~2872 s and ~3048 s at OLR 4 and 5 g VS l^{-1} day^{-1}, respectively.

Table 5.5. Results of CST and FIC tests on mesophilic and thermophilic digestate before and after F/T treatment

Parameters	initial				After F/T treatment			
	M-OLR4	M-OLR5	T-OLR4	T-OLR5	M-OLR4	M-OLR5	T-OLR4	T-OLR5
CST (s)	> 84000	> 84000	3060	3435	>84000	> 84000	2871	3048
Interface height/sample height (%)	100	100	25.7	26.8	100	100	42.9	42.2
Solid-liquid separation (%)	0	0	74.3	73.2	0	0	57.1	57.8
Solid cake (% WW)	100	100	19.4	17.9	100	100	25.8	24.8
Initial TS (% WW)	8.6	8.7	4.2	4.5	8.6	8.7	4.5	4.7
Final TS (% WW)	8.6	8.7	12.0	13.4	8.6	8.7	10.9	11.9
Initial VS (% WW)	6.5	6.9	3.1	3.2	6.6	6.8	3.4	3.5
Final VS (% WW)	6.5	6.9	8.0	8.0	6.6	6.8	8.0	7.9



(a) (b)

Figure 5.8. Results of dewaterability testing on digestates from mesophilic and thermophilic digesters before and after freeze/thawing treatment: (a) CST; (b) Final supernatant interface height as a % of original height of SBP digestate in FIC test

From Figure 5.8b, it can be seen that F/T treatment had no effect on the FIC results for mesophilic digestates, with 0% separation in all cases. For thermophilic digestates, after F/T treatment the supernatant separation at OLR 4 and 5 g VS l^{-1} day^{-1} decreased from ~74 % and ~73 % to ~57 % and ~58 %, respectively; however, the separated supernatant was clearer than in the untreated sample. The volume of cake solid in thermophilic digestate also increased from < ~20% to ~25%, indicating that F/T treatment was not a good option for enhancing dewaterability.

SEM. Figure 5.9 shows SEM images of the mesophilic and thermophilic digestate at OLR 4 g VS l^{-1} day^{-1} before and after F/T treatment. Freezing was able to promote particle aggregation, especially for the mesophilic digestate (Figure 5.9a and b). Yet there was no change in CST and FIC probably due to the duration of freezing process (24 hours) was not enough to break the colloids and/or to release the water in the digestate. Several authors have stated that a longer period (> 36 hours) of freezing gave improved dewaterability due to its effect on ice crystal formation, disruption of colloids, movement and aggregation of the solids, and/or the released of bound water (Vesilind and Martel, 1990; Örmeci and Vesilind, 2001; Wang et al., 2001; Chen and He, 2003). In the case of the thermophilic digestate, although there was a slight improvement in CST results, freezing for 24 hours apparently did not enhance the formation of larger particles (Figure 5.9c and d), possibly as the time spent frozen was insufficient.

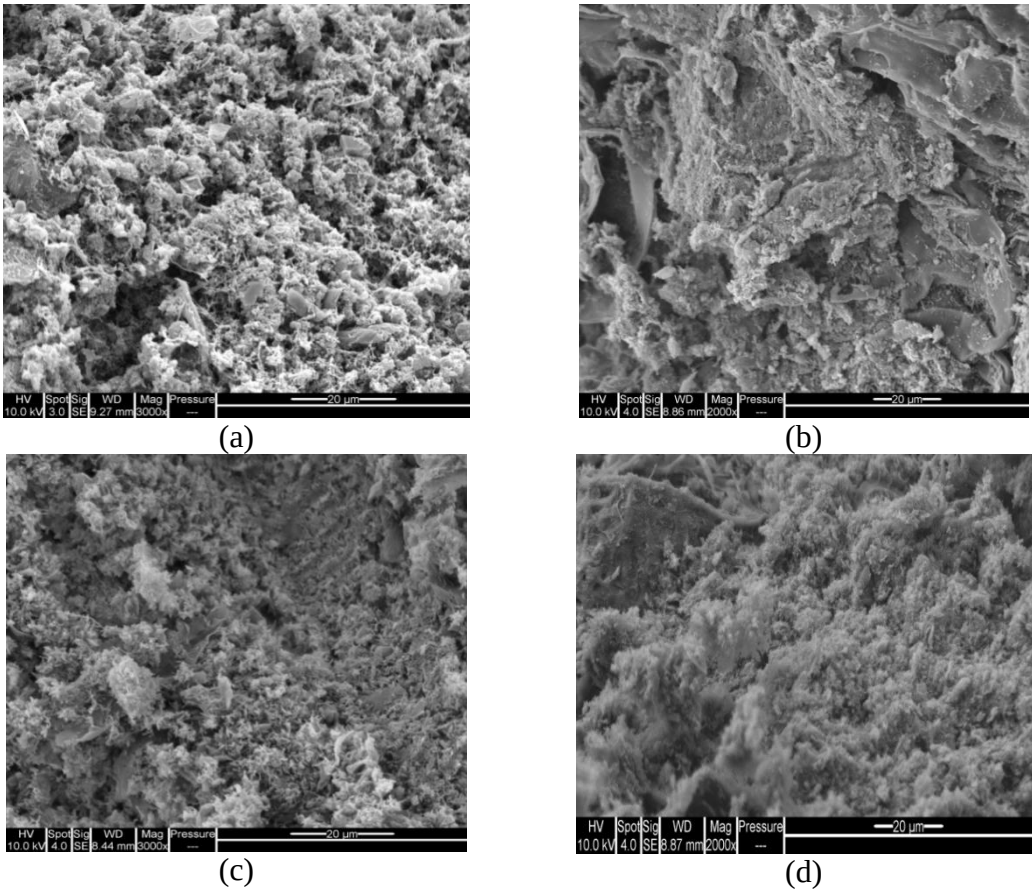


Figure 5.9. SEM images of digestate from AD of SBP fed at OLR 4 g VS l^{-1} day^{-1}: Mesophilic digestate before (a) and after F/T (b); thermophilic digestate before (c) and after F/T (d). (Magnification of 2000x treated and 3000x untreated, 10 kV, 20 µm)

Conclusions. Dewaterability of digestates from mesophilic and thermophilic digesters did not significantly improve after F/T treatment. F/T treatment for a duration of 24 hours slightly changed the floc structure of mesophilic digestate but not enough to release the water fraction in the digestate.

5.2.3. Effect of chemical pre-treatment on digestate dewaterability

These experiments investigated chemical coagulants as a means of improving dewaterability.

5.2.3.1. One-stage chemical treatment

Objective. To compare the effect of 7 commonly-used chemical coagulants on fresh digestate from mesophilic and thermophilic digesters.

Method. Seven commonly-used chemical coagulants (Table 5.6) were tested on fresh digestate from mesophilic (M1 and M4) and thermophilic (T1 and T3) digesters in each case after 3 HRT of operation (days 242 to 257 - see section 4.6). The jar test method (APHA, 2005) was used for making the assessment. The concentration of chemical added was 1.25 and 2.5% (w/w) as bulk chemical. In the first set of tests the digestate was not diluted whereas in the second set a 40% dilution was used, based on the results of section 5.2.2.1. The samples were measured for CST and FIC according to the methods in section 3.2.5. The basic procedure for the jar test was as follows:

1). Digestate (200 g) was placed in a beaker and stirred. When used at a 40% dilution 120 g digestate and 80 g water were used.
2). pH was adjusted to the desired value, depending on the chemical coagulant used.
3). Chemical dosage was immediately followed by rapid mixing at ~100 rpm for 2 min.
4). The sample was then gently agitated at ~20 rpm for 20 or more minutes to promote flocculation.
5). CST and FIC were measured on the treated digestate.

Table 5.6. Chemical coagulant used and adjusted pH

Coagulants	pH
Alum ($Al_2(SO_4)_3$)	4.5-7.0
Lime ($Ca(OH)_2$)	9.0-11.0
Ferrous Sulphate ($FeSO_4.7H_2O$)	4.0-7.0
Ferric Chloride ($FeCl_3$)	4.5-7.0
Calcium Carbonate ($CaCO_3$)	-
Sodium Silicate (Na_2SiO_3)	-
Cationic Polymer (FM 305 cationic)	-

Results

CST. CSTs before dilution were >84000 s (M-OLR4 and M-OLR5), ~5000 s (T-OLR4) and ~6000 (T-OLR5). After dilution the CST reduced significantly (see Table 5.7 and Figure 5.10). The results from the first set of tests showed that the addition of chemical coagulant on digestates without dilution had no positive effect on dewaterability characteristic, and this part of the experiment was therefore stopped. The diluted values were then taken as the untreated CST for comparison with the CST of the digestate samples after treatment with chemical coagulants.

From Figure 5.10, it can be seen that, in general, the thermophilic digestate samples performed better than the mesophilic samples after one-stage chemical treatment with all types and doses of chemical coagulant tested. Alum and ferric chloride were the most effective coagulants in terms of their impact on CST. Dosing with Alum at 2.5% reduced CST more than 100-fold, but the final values were still considerably above the guideline of < 10 s for effective dewatering (Table 5.7). Ferric chloride was the most effective coagulant tested, with a 2.5% dose reducing CST to the range 11-37 s. For other chemical coagulants such as lime, ferrous sulphate, calcium carbonate, Na_2SiO_3 and polymer FM 305 the CST for all samples tested was still above 800 s, with the exception of thermophilic digestate and polymer FM305 (cationic polymer) where a dose of 2.5% decreased the CST to 37 s at OLR 4 g VS l^{-1} day^{-1} and 150 s at OLR 5 g VS l^{-1} day^{-1}.

Table 5.7. CST of mesophilic and thermophilic digestate before and after one-stage chemical treatment

	CST (seconds)			
	Mesophilic		Thermophilic	
OLR (g VS l^{-1} day^{-1})	4	5	4	5
initial (no dilution)	> 84000	> 84000	4592	5255
initial (40% dilution)	13227	20516	1455	1587
1.25% Alum	1488	2811	86	150
2.5% Alum	160	1590	55	96
1.25% Lime	4523	16404	896	949
2.5% Lime	3501	14268	495	560
1.25% $FeSO_4.7H_2O$	9037	20231	887	1061
2.5% $FeSO_4.7H_2O$	8694	17885	828	986
1.25% $FeCl_3$	45	243	51	53
2.5% $FeCl_3$	11	37	36	31
1.25% $CaCO_3$	4018	16689	1370	1392
2.5% $CaCO_3$	3180	6883	1120	1237
1.25% Na_2SiO_3	8703	11387	1924	2239
2.5% Na_2SiO_3	6801	6164	1642	2170
1.25% Polymer FM305	3266	4880	1840	2180
2.5% Polymer FM305	836	2192	37	150
Standard CST for dewatering process*	< 10			

Note: * USEPA (1987)

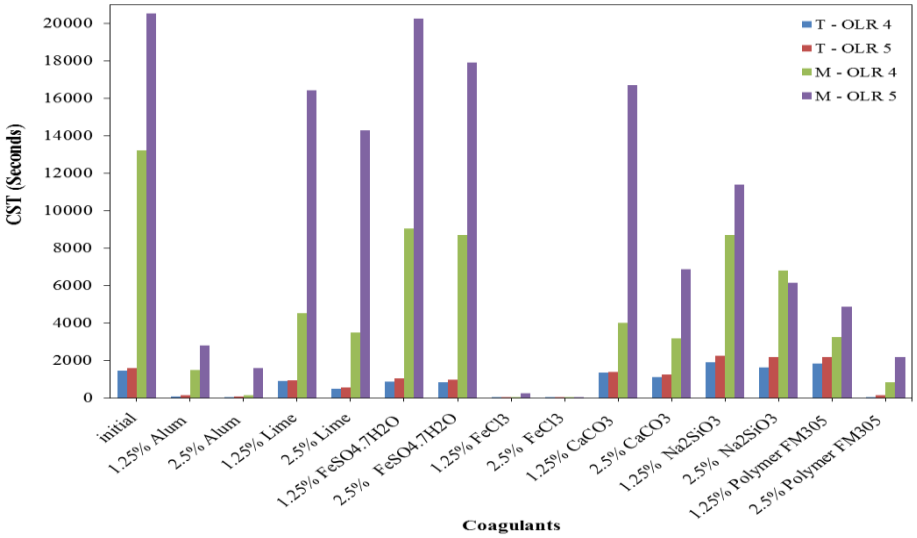


Figure 5.10. CST for SBP digestate after treatment with different coagulants

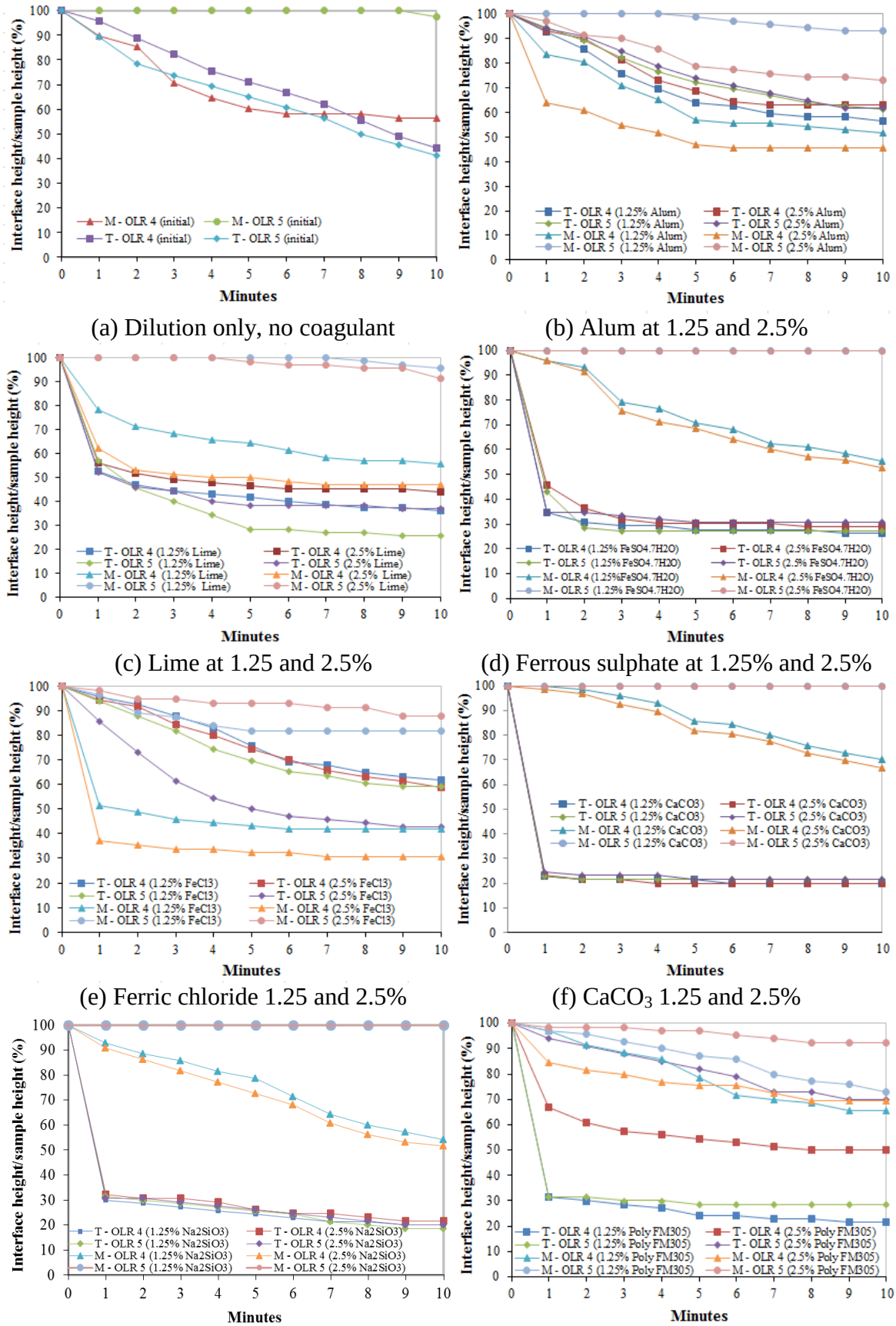
FIC. Table 5.8 and Figure 5.11 show the effect of type and dose of coagulant on the FIC results. In mesophilic conditions with dilution but no coagulant addition the digestate at OLR 4 g VS l^{-1} day^{-1} showed 41.7% separation after 6 minutes centrifugation, then little or no further change, while the digestate at OLR 5 g VS l^{-1} day^{-1} showed almost no separation even after 9 minutes (Figure 5.11a). In contrast the thermophilic digestates achieved 56% and 59% separation, with a rate of separation that appeared to be similar and approximately linear throughout the test period.

Alum dosing at 2.5% gave a larger increase in separation of mesophilic digestate at OLR 4 and a small improvement at OLR 5 g VS l^{-1} day^{-1}, although this again plateaued after ~6 minutes (Figure 5.11b). For the thermophilic digestates, both dosages of alum reduced the separation but there was an improvement in supernatant clarity and colour.

For the mesophilic digestate at OLR 4 addition of 1.25% lime had no effect, but increasing the dose to 2.5% improved the separation slightly (Figure 5.11c). At OLR 5 g VS l^{-1} day^{-1} lime had very little effect, with a maximum separation of only 8.6% at a 2.5% dose. In the thermophilic digestates addition of 1.25% lime increased both the total separation and the initial separation rate, which reached a plateau after 3-5 minutes. Increasing the lime dose to 2.5% led to a reduction in supernatant separation at both OLR. The maximum separation achieved was 74% at OLR 5 g VS l^{-1} day^{-1} and 1.25% lime.

Table 5.8. FIC results for mesophilic and thermophilic digestate before and after one-stage chemical treatment

		Interface height/sample height (%)				Solid-liquid separation (%)				Solid cake (%WW)				Maximum rate of separation (%WW)				Time at which stable interface is achieved (minutes)			
		Mesophilic		Thermophilic		Mesophilic		Thermophilic		Mesophilic		Thermophilic		Mesophilic		Thermophilic		Mesophilic		Thermophilic	
OLR (g VS l^{-1} day^{-1})		4	5	4	5	4	5	4	5	4	5	4	5	4	5	4	5	4	5	4	5
Initial (40% dilution)		56.3	97.5	44.4	41.3	43.8	2.5	55.6	58.7	30.5	99.5	39.3	38.6	16.4	0.3	7.0	12.7	6	>10	>10	>10
Alum	1.25%	51.4	92.9	56.5	61.1	48.6	7.1	43.5	38.9	55.5	99.3	57.4	60.2	15.1	0.7	8.6	6.1	5-6	9	5-7	9
	2.5%	45.4	72.9	62.7	61.5	54.6	27.1	37.1	38.5	48.5	80.5	62.1	63.4	36.5	3.8	7.6	5.3	5-6	8	6	9
Lime	1.25%	55.7	95.8	36.1	25.7	44.3	4.2	63.9	74.3	58.3	97.9	33.8	23.2	22.8	0.5	42.7	47.1	8	>10	3-5	5-7
	2.5%	47.0	91.4	43.8	36.9	53.0	8.6	56.2	63.1	62.1	87.7	42.3	36.6	38.0	0.9	48.6	22.8	7	6	6	5
$FeSO_4.7H_2O$	1.25%	55.6	100	26.4	27.1	44.4	0	73.6	72.9	57.5	100	24.4	27.6	7.6	0	71.4	61.8	>10	>10	3	3
	2.5%	52.9	100	28.8	30.6	47.1	0	71.2	69.4	56.4	100	27.1	29.0	8.6	0	54.7	71.4	>10	>10	4	5
$FeCl_3$	1.25%	41.7	81.8	61.5	59.1	58.3	18.2	38.5	40.9	53.2	85.8	63.3	59.8	53.2	5.7	5.1	6.5	6	5	>10	9
	2.5%	30.8	87.7	58.6	42.9	69.2	12.3	41.4	57.1	39.5	87.5	61.6	42.4	62.3	2.8	6.1	15.2	7	2	>10	9
$CaCO_3$	1.25%	70.0	100	20.0	21.4	30	0	80.0	78.6	65.6	100	16.2	17.7	3.2	0	67.8	82.1	>10	>10	2	2
	2.5%	66.7	100	20.0	21.5	33.3	0	80.0	78.5	69.8	100	18.7	19.9	3.4	0	76.0	74.5	>10	>10	4	5
Na_2SiO_3	1.25%	54.3	100	20.0	18.6	45.7	0	80.0	81.4	51.8	100	16.0	15.1	7.6	0	61.5	73.0	>10	>10	5-7	5-7
	2.5%	51.5	100	21.5	20.0	48.5	0	78.5	80.0	50.4	100	18.1	16.9	9.1	0	66.9	68.4	>10	>10	5-7	5-7
Polymer FM305	1.25%	65.7	72.9	21.4	28.6	34.3	27.4	78.6	71.4	68.1	85.8	19.4	24.6	5.1	3.8	60.3	73.0	4	>10	5	6
	2.5%	69.2	92.3	50.0	69.7	30.8	7.7	50	30.3	77.0	100	51.6	59.4	15.2	1.9	33.4	6.1	6	8	8	7



(a) Dilution only, no coagulant

(b) Alum at 1.25 and 2.5%

(c) Lime at 1.25 and 2.5%

(d) Ferrous sulphate at 1.25% and 2.5%

(e) Ferric chloride 1.25 and 2.5%

(f) $CaCO_3$ 1.25 and 2.5%

(g) Na_2SiO_3 1.25 and 2.5%

(h) Poly FM305 at 1.25 and 2.5%

Figure 5.11. FIC test results for digestates from mesophilic and thermophilic digesters at OLR 4 and 5 g VS l^{-1} day^{-1} using different coagulants

Addition of ferrous sulphate ($FeSO_4.7H_2O$) to mesophilic digestate at 1.25% and 2.5% gave a marginal increase in separation at OLR 4 g VS l^{-1} day^{-1}, and no separation at OLR 5 g VS l^{-1} day^{-1}. In the thermophilic digestates, however, ferrous sulphate addition at 1.25% led to an increase in separation to around 73% at both OLR, and a very rapid onset of separation which had stabilised after ~3 minutes (Figure 5.11d). Increasing the dose to 2.5% had no further effect.

Addition of 1.25% ferric chloride to mesophilic digestate at OLR 4 g VS l^{-1} day^{-1} caused a rapid increase in the initial rate of separation, and improved the final separation to 58%. Increasing the dose to 2.5% gave a further improvement in both rate of separation and final value, to 69%. At OLR 5 g VS l^{-1} day^{-1} separation improved to around 18% with 1.25% ferric chloride addition, but this reduced to around 12% separation when the dose was increased to 2.5%. Addition of 2.5% ferric chloride to the thermophilic digestate at OLR 5 g VS l^{-1} day^{-1} gave a slight increase in the initial rate of separation but had little or no effect on total separation: while for the other thermophilic digestates and doses tested the total separation was reduced (Figure 5.11e).

Calcium carbonate addition reduced the supernatant separation in mesophilic digestate at both of the OLRs and doses tested; the supernatant, however, was clearer and lighter in colour. In the thermophilic digestates there was a very rapid onset of separation with the interface stabilising within ~1 minute (Figure 5.11f), and an improvement in overall separation to ~80% at OLR 4 g VS l^{-1} day^{-1} and ~78.5% at OLR 5 g VS l^{-1} day^{-1} at both dosages.

The effects of sodium silicate (Na_2SiO_3) addition were similar in kind to those produced by calcium carbonate. There was little or no effect on separation in mesophilic digestate at the OLRs and doses tested. In the thermophilic digestates, however, the majority of separation occurred within ~1 minute (Figure 5.11g), with overall separation around ~80% at both OLR and dosages tested.

Mesophilic digestate at OLR 4 g VS l^{-1} day^{-1} showed a decrease in overall separation on addition of Poly FM305 at either dosage. At OLR 5 g VS l^{-1} day^{-1} there was an increase in separation at 1.25% polymer addition, but this reduced when the dose was increased (Figure 5.11h). The thermophilic digestates also showed an increase in separation with a

more rapid onset at 1.25% addition, which decreased sharply at the higher polymer dose.

Typical dosages for chemical coagulants used in dewatering of digested sludge, such as ferric chloride, lime and dry polymer, are in the range of 40 - 50 kg tonne^{-1} DM, 80 – 335 kg tonnes^{-1} DM, and 10 – 20 kg tonne^{-1} DM, respectively (USEPA, 1979). Hwa and Jeyaseelan (1997) found that the dewaterability of digested sludge improved after the addition of Alum alone at optimum dose of 4% (w/w), while adding at dose above 4% only resulted in a decrease in the solid volume and solid content of the sludge cake. Lo et al. (2001) found that addition of Alum at doses of 162.5 mg l^{-1} and 325 mg l^{-1} combined with the addition of salt to increase the salinity of digested sludge in the range of 5000 to 20000 mg l^{-1} enhanced dewaterability, in particular for the high salinity digested sludge, reducing SRF from ~24 x 10^{12} m kg^{-1} to ~17 x 10^{12} m kg^{-1} and ~15 x 10^{12} m kg^{-1}, respectively. Ferrous sulphate, also known as copperas, is also commonly used in dewatering (Bratby, 1980; IWPC, 1981) with a typical concentration for digested sludge of 40% on dry solids basis (IWPC, 1981). Several studies found that the addition of sodium silicate improved the flocculation and flotation of treated sludge (White et al., 1998; Tripathy and De, 2006; Rao et al., 2010), and at a dosage of 3 – 50 kg tonne^{-1} combined with other dewatering aids (e.g. reagent U developed by Virginia Tech) further improved dewaterability by reducing the cake moisture content from ~18% to ~13% on wet weight basis (Eraydin, 2004). The use of calcium carbonate is often preferred to other coagulants such as alum and iron salts as it is more economical and safe (Lee et al., 2007). Using calcium carbonate can improve settling performance and removal of dissolved organic matter in a slaughterhouse effluent, and when combined with other coagulants, such as Alum and poly-aluminum chloride at doses of 2.5 and 1 g l^{-1}, improved phosphorus removal by ~100% and solid cake volume by up to ~42% (Aguilar et al., 2002)

In this study the use of alum alone gave little improvement in terms of CST and liquid/solid separation efficiency, thus it is not recommended for full-scale application. Several studies of wastewater biosolids have reported that use of alum alone was not feasible in term of dewaterability performance and cost; however when combined with other coagulant aids (e.g. chitosan, cationic and anionic polymers), dewaterability was much improved, for instance a reduction in SRF from 0.512 x 10^{-12} mg kg^{-1} to 0.121 x

10^{-12} mg kg^{-1} (Lee et al., 2001) or a reduction in sludge volume by 60-70% (Haydar and Aziz, 2009).

Qi et al. (2011) described the main mechanisms of coagulation as changing or neutralising the surface charge of particles or bridging between small particles in the digestate, thus promoting agglomeration into larger flocs. In general it was clear that the addition of coagulants of this type resulted in an improvement in dewaterability and solid cake properties. In this study, the effect of chemical coagulants/flocculants was indicated by much easier solid–liquid separation through mechanical dewatering (centrifugation) and also by an improvement in CST from > 84000 s to ~11 s - ~21000 s; however the degree was dependent on the physical properties of the digestate and the type of coagulants/flocculants. Thermophilic digestate had better dewaterability either before or after chemical conditioning compared to that of mesophilic digestate.

Conclusions. The addition of chemical coagulant/flocculants alone improved CST and solid/liquid separation in SBP digestate. With respect to the low dose required, in the range of 12.5 - 25 kg $tonnes^{-1}$ WW (or ~1 – 2 kg $tonne^{-1}$ TS), and the great reduction of CST to ~11 - 250 s, the use of ferric chloride is recommended; however further consideration is needed of the costs, including those for disposal of additional solids, and of any effects on the environment.

5.2.3.2. Two-stage chemical treatment

Experiment 1 – Two-stage chemical treatment for digestates with and without TE addition

Objective. To evaluate the effect of two-stage chemical conditioning on dewaterability of digestates from the mesophilic digesters with and without TE addition.

Method. Digestate was taken from mesophilic digesters with (N9) and without (N3) TE addition, in each case after 3 HRT (see section 3.5.1). The test was performed in a single run for each sample. The chemical conditioners used were aluminium sulphate and cationic polymer (Polymer FM 305). The jar test method (APHA, 2005) was used for making the assessment with the procedure was as follows:

1). A representative 240 g of the digestate was placed in a 1-litre glass beaker. 160 g of tap water was added and the contents of the beaker were stirred for 5 minutes.

2). 5 ml of aluminium sulphate solution (~24-27% w/v) was added to the beaker and stirred for 10 minutes at ~100 rpm.

3). Mixing was reduced to a low speed and polymer solution (1-2 ml of a 1% w/w aqueous solution) was slowly added until flocculation occurred.

4). The resulting conditioned sludge was immediately tested using CST, FIC and filtration tests according to the methods in section 3.2.5 to determine its dewatering characteristics. SEM was also carried out for digestate samples before and after treatment according to the methods in section 3.2.6.

Results

CST. Figure 5.12 shows the results of tests carried out using the two-stage chemical approach. This strategy was successful in reducing CST values, but the same dosages were more effective on samples from the digester with TE addition. Table 5.9 shows that to reduce CST to < 20 s the amount of polymer solution needed for digestate with TE addition was 197 ml kg^{-1} digestate, while digestate without TE addition required 287 ml kg^{-1} digestate.

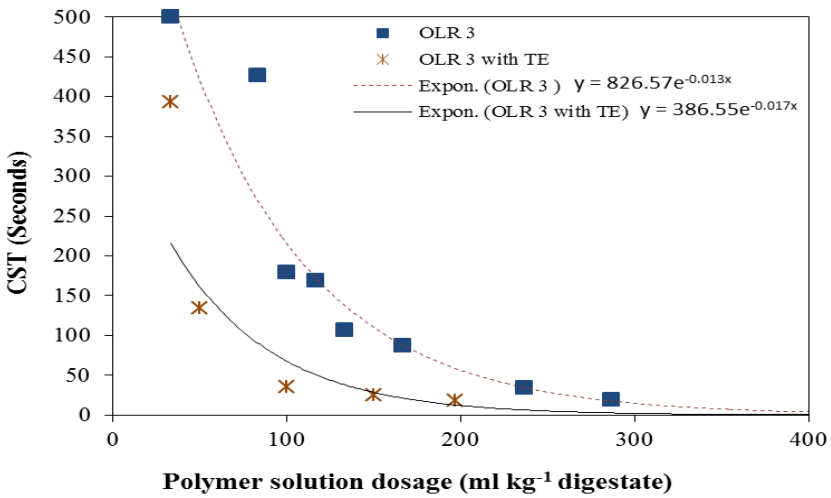


Figure 5.12. CST at aluminium sulphate dosing of 5 ml and different polymer doses for digestates with and without TE addition, with trend line fitted using exponential function

Table 5.9. Results of CST test on digestates after two-stage chemical treatment

OLR ($g\ VS\ l^{-1}\ day^{-1}$)	Polymer added ($ml\ kg^{-1}$ digestate)	CST (seconds)
2	167	15.7
3	287	19.7
4	390	19.1
5	557	12.3
3 (+ TE)	197	18.5

Filtration test. Two-stage chemical treatment was successful in allowing filter cake formation and dewatering for both digestates, but the results were inconclusive with respect to any difference between them as the method used did not record the rate of filtration but only the percentage of the original material present as filtrate after set intervals at different pressures (Table 5.10).

Table 5.10. Results of filtration test from two-stage chemical treatment with and without TE supplementation

Performance indicators	OLR ($g\ VS\ l^{-1}\ day^{-1}$)	
	3	3 (+ TE)
Digestate weight (g WW)	75.0	76.0
Solid weight (g WW)	15.0	19.6
Liquid weight (g WW)	60.0	56.4
Efficiency of filtration (%)	80.0	74.1
TS ($g\ kg^{-1}$ WW)	111.7	90.0
VS ($g\ kg^{-1}$ WW)	103.6	86.1

Note: all in average values of duplicate samples

FIC. The results of the FIC test are shown in Table 5.11 and Figure 5.13. With two-stage chemical treatment the time for dewatering in the FIC test was greatly reduced for both digestates tested in comparison with the untreated values, making it difficult to distinguish any difference using FIC. Approximately 50% of supernatant liquid could be separated from the digestate in less than 50 s (Figure 5.13).

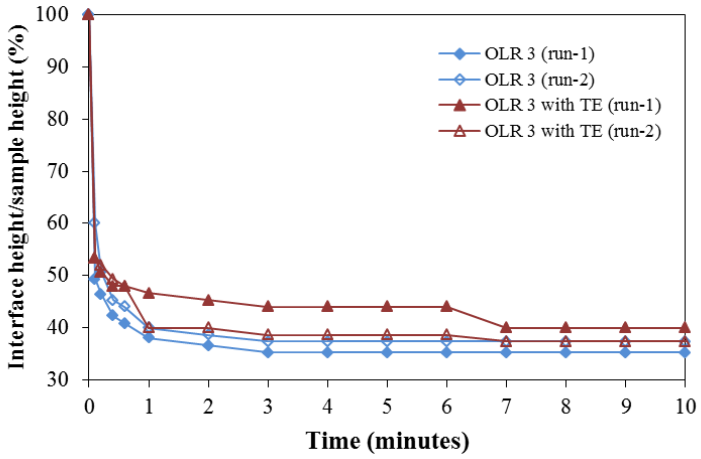


Figure 5.13. Supernatant interface height as a % of original height during FIC run on digestates with and without TE addition (treated digestates)

Table 5.11. Results of FIC test from two-stage chemical treatment for digestates with and without TE supplementation

Performance indicators	OLR (g VS l^{-1} day^{-1})			
	3		3 (+ TE)	
	run-1	run-2	run-1	run-2
Supernatant interface height (% of original height) after 10 minutes	35.2	37.3	40.0	37.3
TS (g kg^{-1} WW)	66.6	64.7	62.0	61.3
VS (g kg^{-1} WW)	45.8	46.1	46.6	46.3

SEM. Figure 5.14 shows SEM images of treated and untreated digestate from digesters with and without TE supplementation. In Figure 5.14a and c (untreated digestates) the digestate appears to form an open porous matrix in both cases, suggesting that water could be held within this sponge-like structure. In contrast, after chemical destabilisation and addition of the cationic polymer, the SEM images show a change in the structure of the digestates, with a dense smooth matrix in which small-scale pores are no longer evident (Figure 5.14b and d). Similar changes in digestate structure due to the addition of polymer have been found by Ayol (2005) for water treatment residuals (biosolids).

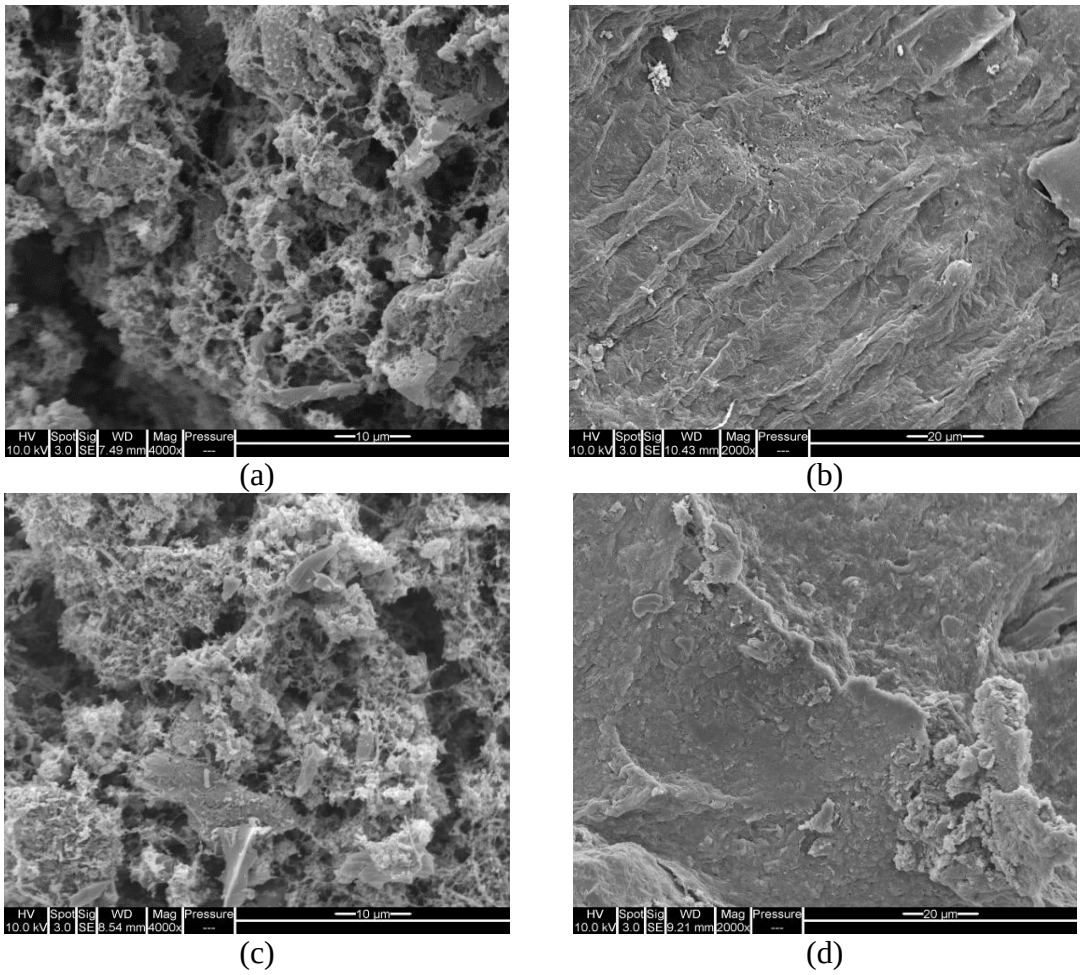


Figure 5.14. SEM images of the digestaste from AD of SBP fed at: OLR 3 g VS l^{-1} day^{-1} no TE (a) fresh/untreated and (b) treated; OLR 3 g VS l^{-1} day^{-1} TE added (c) fresh/untreated and (d) treated. (Magnification 2000x treated and 4000x untreated, 10 kV, 10-20 µm)

Conclusions. A two-stage chemical approach to dewatering using aluminium sulphate followed by polymer addition was successful in improving digestate dewaterability, indicated by a reduction in CST values to < 20 s and an increase in separation percentage to 50% for both digestates. Digestates from TE-supplemented digesters required a smaller polymer dose for dewatering, as determined by CST testing, compared to that for unsupplemented digestate. SEM did not show any strong difference between digestate samples with and without TE.

Experiment 2 – Two-stage chemical treatment for digestates at different OLR

Objective. To evaluate the effect of two-stage chemical conditioning on dewaterability of digestates from mesophilic digesters fed at different OLR.

Method. The methods used were the same as in experiment 1. The digestate was taken from mesophilic digesters fed at OLR 2 to 5 g VS l^{-1} day^{-1} (N1, N5 and N8) in each case after 3 HRT, as described in section 3.5.1.

Results

CST. The required polymer dose for the mesophilic digestate samples tested increased with OLR. For instance, the digestate fed at OLR 2 g VS l^{-1} day^{-1} required a dose of 167 ml kg^{-1} digestate to reduce the CST from ~12000 s to 15.7 s, while at OLR 5 g VS l^{-1} day^{-1} a decrease in CST from > 84000 s to 12.3 s was achieved at a dose of 557 ml kg^{-1} digestate. A typical FM305 polymer dosage curve for the two-stage chemical treatment of mesophilic digestates fed at different OLR is shown in Figure 5.15 and Table 5.9.

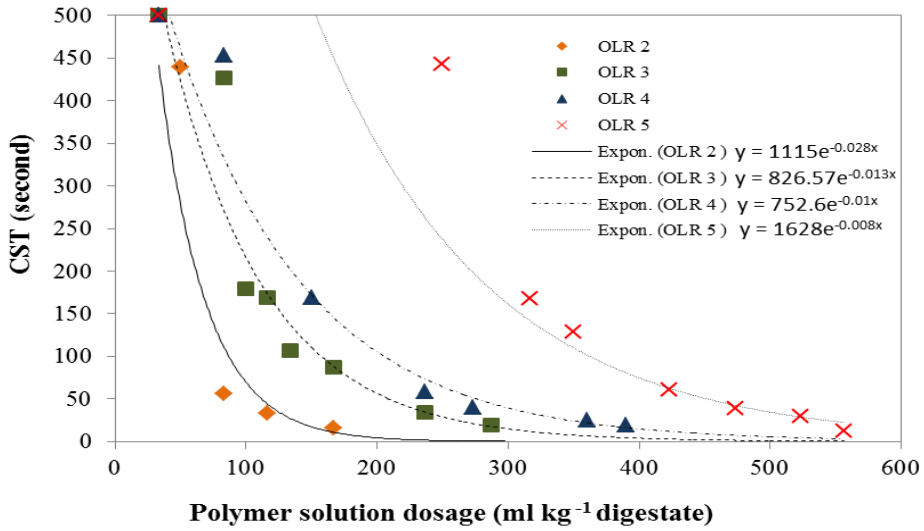


Figure 5.15. CST at aluminium sulphate dosing of 5 ml and different polymer doses for digestates from mesophilic digesters fed at different OLR, with trend line fitted using exponential function

Filtration test. As in experiment 1, the filtration test results showed that a two-stage chemical treatment was effective in allowing filter cake formation and dewatering for all digestate samples. The efficiency of filtration reduced as the OLR increased (Table 5.12).

Table 5.12. Results of filtration test from two-stage chemical treatment for digestates at different OLR

Performance indicators	OLR (g VS l^{-1} day^{-1})			
	2	3	4	5
Digestate weight (g WW)	80.0	75.0	78.5	79.5
Solid weight (g WW)	16.0	15.0	19.5	23.6
Liquid weight (g WW)	64.0	60.0	59.0	56.0
Efficiency of filtration (%)	80.0	80.0	75.2	70.4
TS (g kg^{-1} WW)	110.4	111.7	103.7	111.7
VS (g kg^{-1} WW)	106.1	103.6	97.6	103.6

Note: all in average values of duplicate samples

FIC. Results for the FIC test are given in Table 5.13 and Figure 5.16, and show that the two-stage chemical treatment greatly improved digestate separation by centrifugation. Approximately 50% of supernatant could be separated within 50 seconds at OLRs 2 and 4 g VS l^{-1} day^{-1}, making it difficult to differentiate between the digestates. After ten minutes of centrifugation, these values had risen to ~71% and ~68%. At OLR 5 g VS l^{-1} day^{-1} about 40% of supernatant liquid could be separated in less than 50 s, and after 10 minutes centrifugation this increased to 63%. This result further confirmed the negative effect of the higher OLR in liquid-solid separation of mesophilic digestates.

Table 5.13. Results of FIC tests from two-stage chemical treatment for digestates at different OLR

Performance indicators	OLR (g VS l^{-1} day^{-1})			
	2	3	4	5
Supernatant interface height (% of original height) after 10 minutes	29.9	36.3	31.9	37.2
TS (g kg^{-1} WW)	71.3	65.6	65.6	92.4
VS (g kg^{-1} WW)	50.7	46.0	49.2	64.4

Note: all in average values

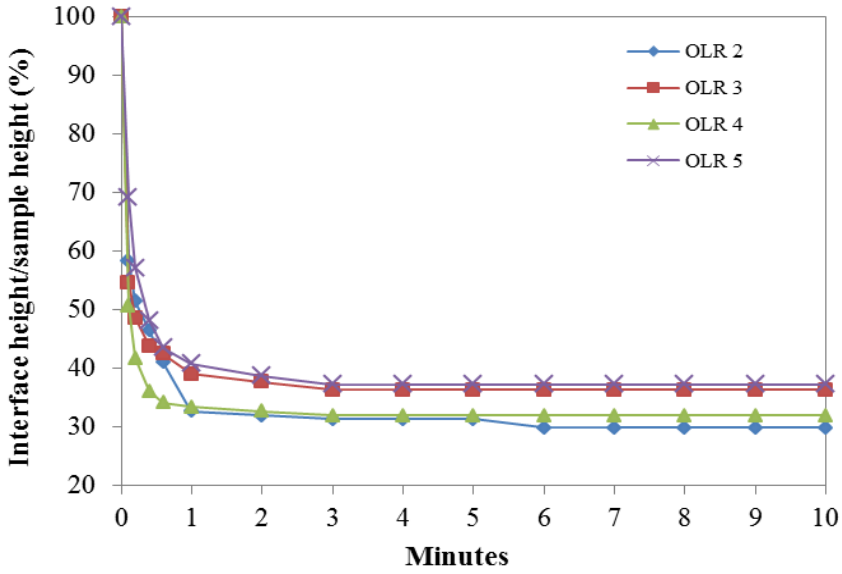


Figure 5.16. Supernatant interface height as a % of original height during FIC run on digestates with and without TE addition (treated digestates)

The two-stage chemical treatment was effective at improving the dewaterability in all cases at the polymer dosage range of 170 – 600 ml kg^{-1} digestate WW (or 6.8 – 48 kg $tonne^{-1}$ TS). Since a typical polymer dosage for dewatering of anaerobically digested sludge is in the range of 20 - 40 kg polymer $tonne^{-1}$ DM according to USEPA (1979) and USEPA (1987), this indicates some potential for full-scale application.

SEM. Figure 5.17 shows SEM images of treated and untreated digestate from the mesophilic digesters fed at OLR 2, 4 and 5 g VS l^{-1} day^{-1}. As in the previous images (Figure 5.9 and 5.14), the untreated digestate in Figures 5.17a, c and e appears to have an open porous matrix in all cases. At OLR 2 g VS l^{-1} day^{-1}, however, the structure is noticeably less open, possibly indicating less capacity to retain water. After chemical destabilisation and addition of the cationic polymer, the SEM image again showed a change in the structure of the digestate at all OLRs tested, creating a more compact and dense matrix (Figure 5.17b, d and f).

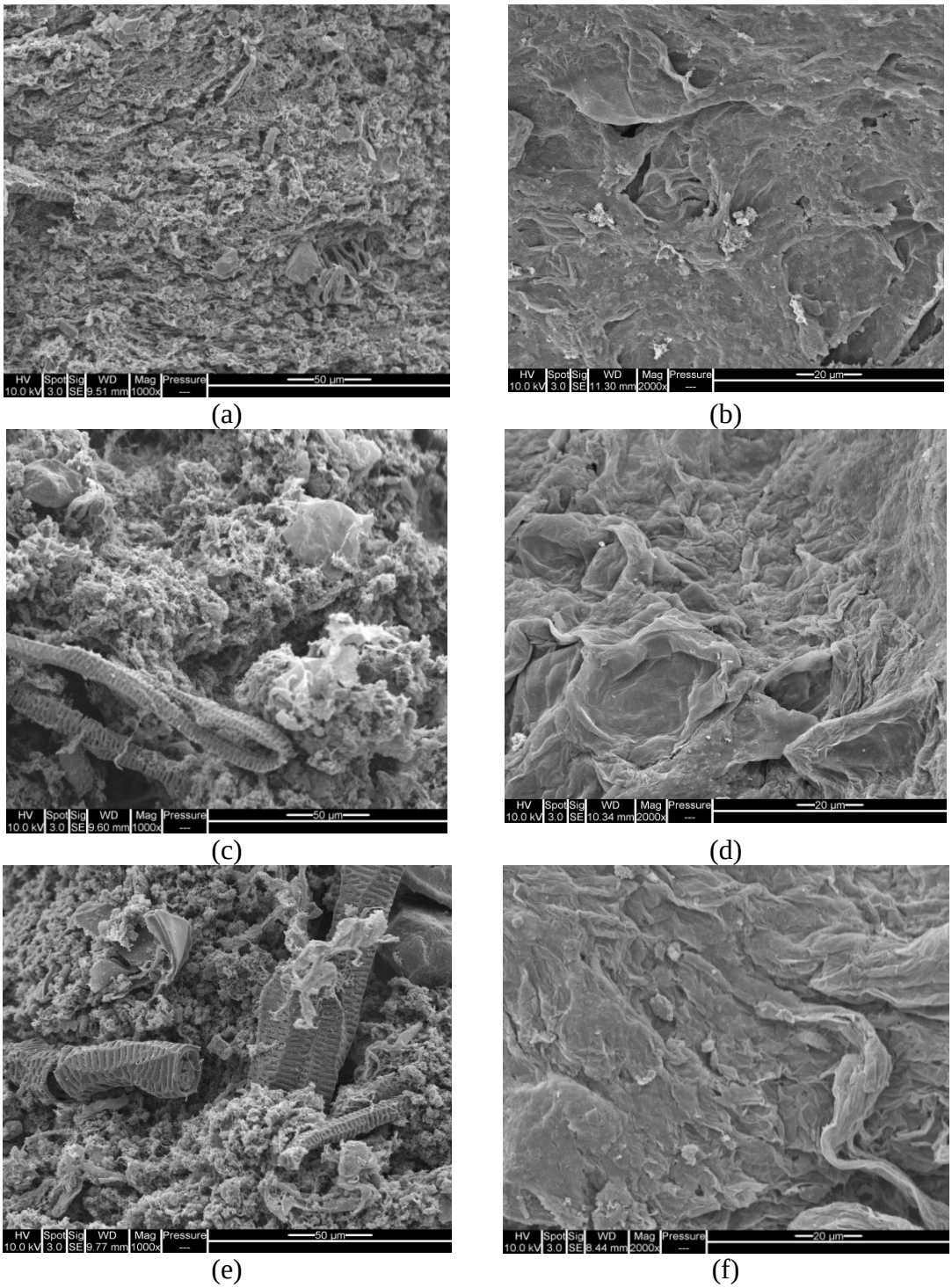


Figure 5.17. SEM images of the digestate from AD of SBP fed at: OLR 2 g VS l^{-1} day^{-1} (a) fresh/untreated and (b) treated; OLR 4 g VS l^{-1} day^{-1} (c) fresh/untreated and (d) treated; and OLR 5 g VS l^{-1} day^{-1} (e) fresh/untreated and (f) treated. (Magnification 1000x untreated and 2000x treated, 10 kV, 20 - 50 µm)

Conclusions. The above results confirmed that while increasing OLR changed the properties of the digestate. The results from two-stage chemical treatment indicate some potential for full-scale application in terms of dosages required.

Experiment 3 – two-stage chemical treatment for digestates from mesophilic and thermophilic digesters

Objective. To evaluate the effect of two-stage chemical conditioning on dewaterability of digestates from mesophilic and thermophilic digesters fed at different OLR.

Method. The digestates used were taken from mesophilic and thermophilic anaerobic digesters operating at OLR 4 and 5 g VS l^{-1} day^{-1} (see section 4.6). The samples were collected on day 137 from digesters M1, M4, T1 and T3. The experimental procedure was the same as in the previous experiments i.e. as in experiments 1 and 2.

Results

CST. Figure 5.18 shows polymer dosage curves for the digestate samples tested. In general, the thermophilic digestate needed less polymer than the mesophilic to achieve a given CST value. The higher OLR required more polymer, especially for mesophilic digestate (Table 5.14). For instance, at OLR 4 g VS l^{-1} day^{-1} the mesophilic digestate required 300 ml kg^{-1} digestate of polymer solution to reduce the CST from ~13300 s to 16.3 s; at OLR 5 g VS l^{-1} day^{-1} this almost doubled (Figure 5.18). In contrast, the thermophilic digestate samples required < 190 ml kg^{-1} digestate of polymer solution to reduce the CST to < 20 s and the increase in OLR had very little effect.

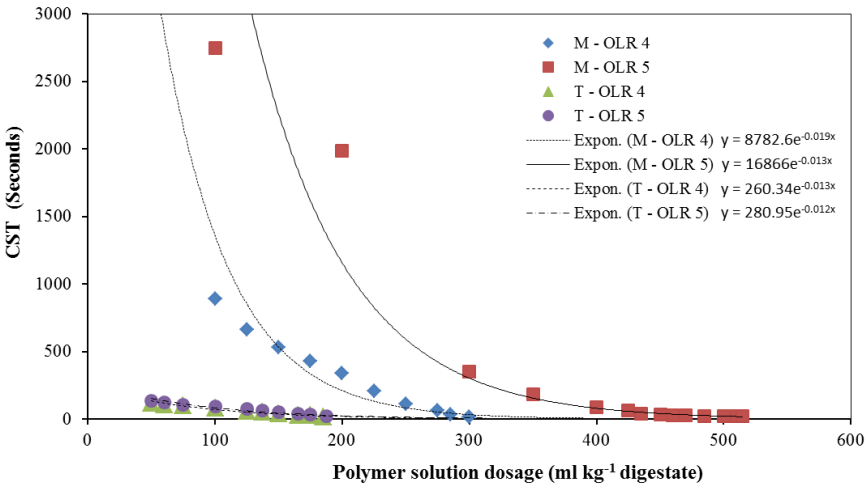


Figure 5.18. CST at aluminium sulphate dosing of 5 ml and different polymer doses for digestates from mesophilic and thermophilic digesters at all OLRs, with trend line fitted using exponential function

Table 5.14. Results of CST test from two-stage chemical treatment for digestates from mesophilic and thermophilic digester

Sample ID	Total polymer added (ml kg^{-1} digestate)	CST (seconds)
M - OLR 4	300.0	16.3
M - OLR 5	515.0	19.9
T - OLR 4	185.0	17.3
T - OLR 5	187.5	18.9

Filtration. Table 5.15 shows the filtration test results after two-stage chemical treatment. Again, the thermophilic digestate had higher filtration efficiencies of ~74% and ~60% at OLR 4 and 5 g VS l^{-1} day^{-1}, respectively. The efficiency for the mesophilic digestate was much lower at ~56% and ~54% for OLR 4 and 5 g VS l^{-1} day^{-1}, respectively. In this case, the effect of the higher OLR was more evident in the thermophilic digestates.

Table 5.15. Results of filtration tests from two-stage chemical treatment for digestates from mesophilic and thermophilic digestion

Performance indicators	M - OLR 4	M - OLR 5	T - OLR 4	T - OLR 5
Digestate weight (g WW)	65.0	67.0	65.0	64.0
Solid weight (g WW)	28.8	31.3	16.7	25.6
Liquid weight (g WW)	36.2	35.9	48.2	38.4
Efficiency of filtration (%)	55.6	53.5	74.2	60.1
TS (g kg^{-1} WW)	112.0	98.1	125.5	119.5
VS (g kg^{-1} WW)	104.2	81.2	101.8	92.5

Note: all in average values of duplicate samples

FIC. Figure 5.19 presents the FIC test results after two-stage chemical treatment. The time for dewatering in the FIC test was greatly reduced for all the digestates tested. The thermophilic digestate had much better separation than the mesophilic, and OLR made little difference in either case. Within less than 50 s, approximately 40% separation was achieved for the mesophilic digestates and about 55% for thermophilic. By the end of the test, separation increased to ~48-49% (mesophilic) and ~64-65% (thermophilic). The other performance parameters from the FIC test are shown in Table 5.16.

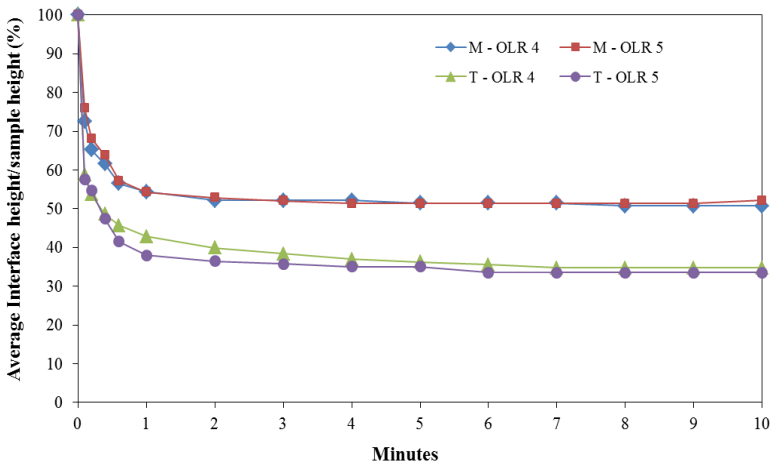


Figure 5.19. Supernatant interface height as a % of original height during FIC run on digestates from mesophilic and thermophilic digesters at all OLRs (treated digestates)

Table 5.16. Results of FIC test from two-stage chemical treatment for digestates from mesophilic and thermophilic digestion

Performance indicators	M - OLR 4	M - OLR 5	T - OLR 4	T - OLR 5
Supernatant interface height (% of original height) after 10 minutes	50.7	52.2	34.8	33.6
Solid volume (%)	51.7	53.9	30.9	31.8
TS (g kg^{-1} WW)	73.1	62.7	74.2	73.8
VS (g kg^{-1} WW)	56.6	49.2	57.1	54.5

Note: all values are averages of duplicate samples

SEM. Figure 5.20 shows SEM images of treated and untreated digestate from the mesophilic and thermophilic digesters fed at OLR 5 g VS l^{-1} day^{-1}. It can be seen that digestate from mesophilic digester tested has a more open porous matrix, which suggests that water could be held within this sponge-like structure (Figure 5.20a). In contrast the digestate from thermophilic digesters was visually different and had a finer and denser appearance without pores (Figure 5.20c), to some extent intermediate between the untreated mesophilic digestate and the two treated samples. This difference in structure may be responsible for the difference in dewatering characteristics. After chemical destabilisation and addition of the cationic polymer, the SEM image again showed the characteristic change in the structure of both digestates to a smooth matrix (Figure 5.20b and d).

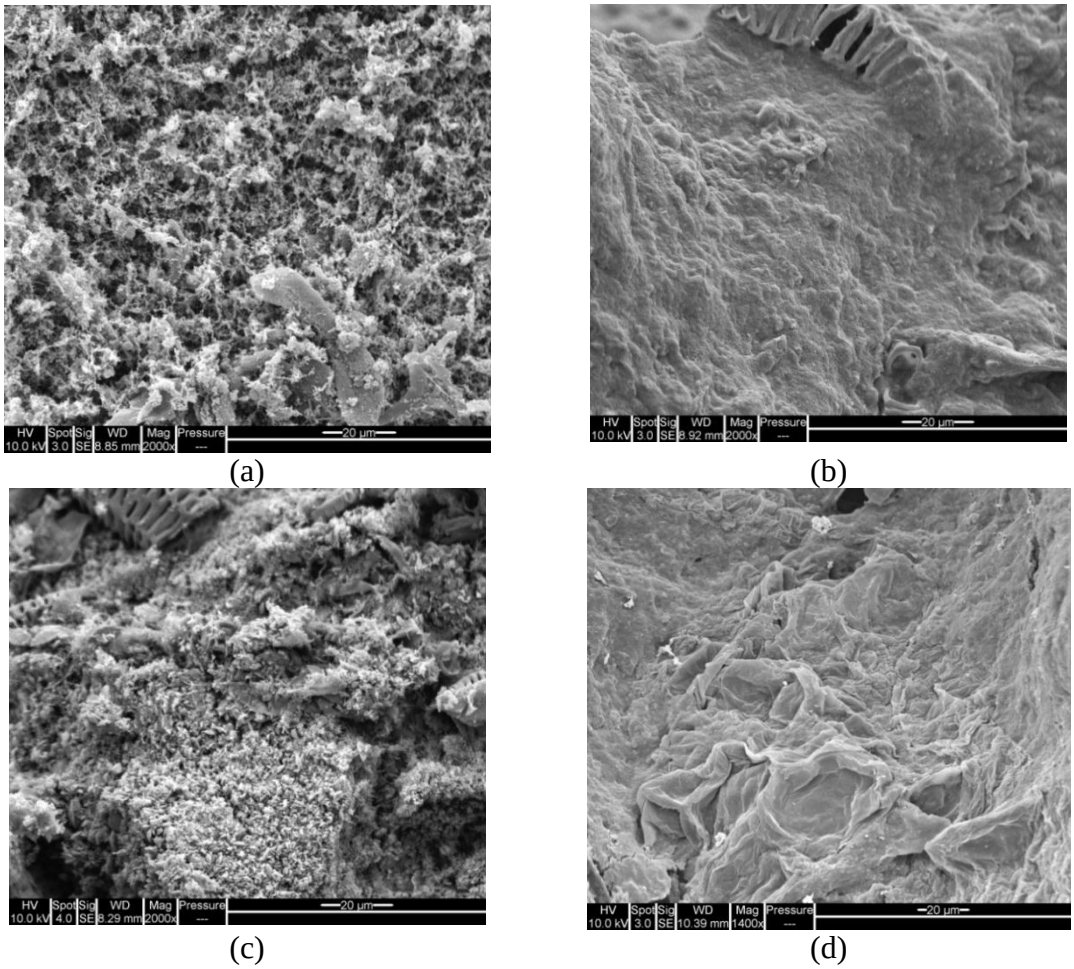


Figure 5.20. SEM images of the digestaste from AD of SBP fed at OLR 5 g VS l^{-1} day^{-1}: Mesophilic digestate before (a) and after two-stage chemical treatment (b); thermophilic digestate before (c) and after two-stage chemical treatment (d). (Magnification 1400x- 2000x treated and 2000x untreated, 10 kV, 20 µm)

From the above results it is clear that in addition to OLR, the operating temperature of the AD process may influence the dewaterability of SBP digestate and this is further investigated in section 5.2.5.

Conclusions. The above results again demonstrated that two-stage chemical treatment enhanced the digestate dewaterability. The findings also indicated that the dewaterability of thermophilic digestate was superior to that of mesophilic digestate.

5.2.4. Enzymatic treatment

Objective. To assess the effect of treatment by cellulolytic enzymes on digestate dewaterability

Method. The digestate used was from the first semi-continuous AD trial as described in section 3.5.1. A sample was taken on day 280 from digester N4 operating at OLR 3 g VS l^{-1} day^{-1}. Two enzymes were used obtained from Novozymes (Novozymes A/S, Denmark): NS 22083 (*xylanase endo-1,4*) and NS 22086 (*cellulase* and *xylanase endo-1,4*-). A 150 g sample of digestate was added to a 250 ml Erlenmeyer flask and the pH adjusted to 5 using phosphoric acid. A dose of 0.1, 0.5, 1.0 or 1.5 g kg^{-1} WW of enzyme solution was then added and the test flask was incubated in a Hybaid Maxi 14 incubator (Thermo Scientific, UK) at 55 ^{o}C. Every 24 hours, a 5 ml sample was taken for CST testing according to the method in section 3.2.5. The experiment ran for 42 days.

Results

The initial CST of the fresh digestate sample was ≥ 84000 s and there was no significant improvement in this after enzyme addition and incubation over 30 days, in any of the cases tested. Enzyme doses of 0.1 and 0.5 g kg^{-1} WW resulted in a small decrease in CST to 79171 s and 55594 s for NS 22083 and 81922 s and 75373 s for NS 22086, respectively. With higher enzyme doses the CST remained ≥ 84000 s (Table 5.17). It was clear that the xylanase and cellulase enzymes used in this experiment were not effective in breaking down the components responsible for the poor dewaterability of the digestate.

Table 5.17. Results of CST tests after enzyme treatment

Enzyme type	Concentration (ml l^{-1})	CST (seconds)
NS 22083	0.1	79172
	0.5	55594
	1.0	> 84000
	1.5	> 84000
NS 22086	0.1	81923
	0.5	75374
	1.0	> 84000
	1.5	> 84000

Conclusion. The results of treatment with cellulolytic enzymes confirmed that this method was not effective in treating digestate from AD of SBP.

5.2.5. Evolution of dewaterability characteristics in mesophilic and thermophilic digestion

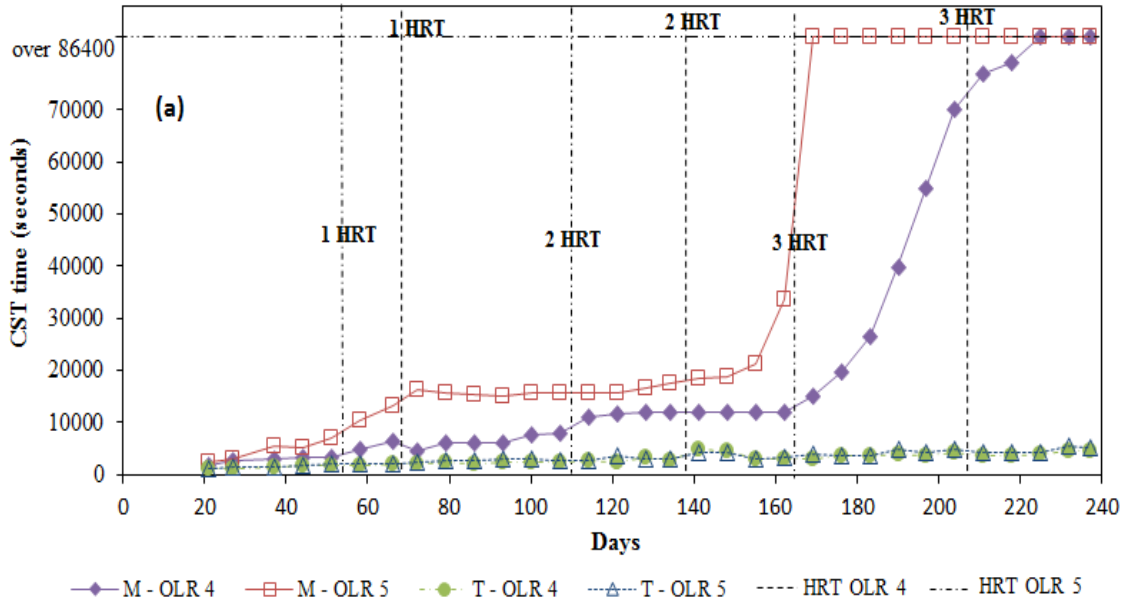
Objective. To evaluate the effects of temperature, loading rate and digester operating period on digestate dewaterability characteristics.

Method. Digestate samples were obtained from mesophilic and thermophilic digesters M1, M4, T1 and T4 as described in section 4.6, at weekly intervals throughout the experimental period. The dewaterability characteristics were measured using CST and FIC tests according to the methods in section 3.2.5.

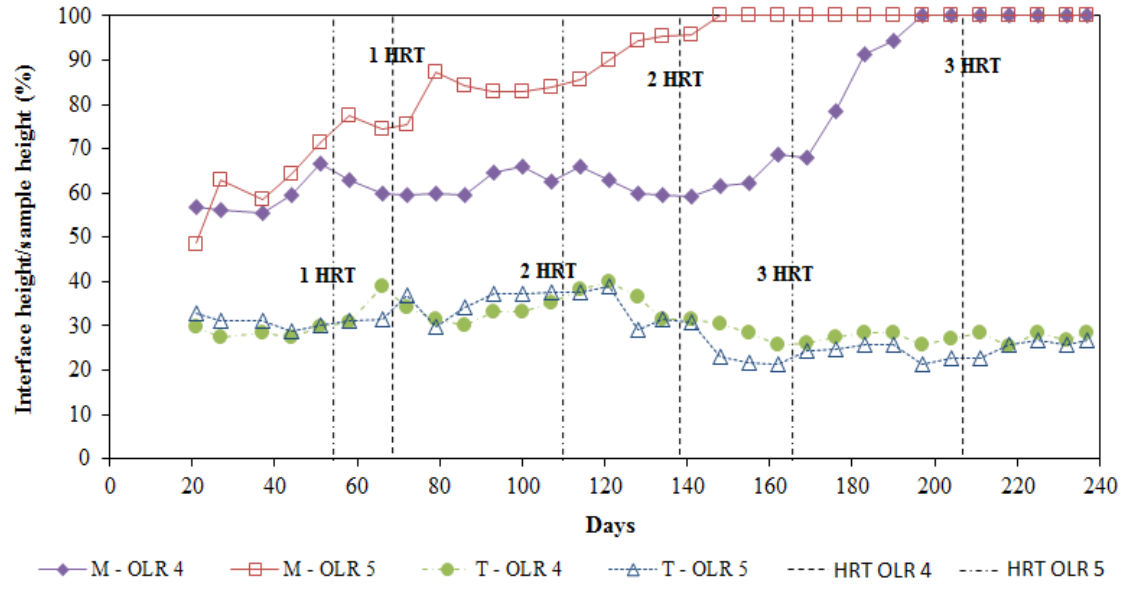
Results

The dewaterability characteristics of the digestates from mesophilic and thermophilic AD are shown in Figure 5.21. Weekly measurements of the capillary suction time (CST) showed that filterability of the mesophilic digestate deteriorated over the period of 3 HRT (Figure 5.21a). More specifically, at an OLR of 4 g VS l^{-1} day^{-1} the CST gradually increased from 369 s to 6000 s by the end of 1 HRT (day 69) and reached a value of ~11000 s after 147 days. This was followed by a further sharp increase to more than ~70000 s by day 207 (3 HRT), reaching the maximum value of over 84000 s (> 24 hours) by day 225. A similar but slightly more severe result was seen at the higher loading rate with an earlier onset of the sharp changes, presumably as a result of the shorter HRT. After 3 HRT the CST value at the higher loading was again > 24 hours. These extremely high CST values are usually interpreted to mean that the water is strongly bound to the digestate, and other observations supported this as the digestate itself became thicker. As neither filtration nor CST tests showed any appreciable dewatering, it was thought this could be due to either colloidal solids or high molecular weight extracellular polymers blinding the filter pores. In either case the digestate could be considered as very hydrophilic with the water apparently strongly bound between discrete particles (Colin and Gazbar, 1995).

Digestate from the thermophilic digesters showed better dewaterability characteristics: at both of the OLR tested there was no obvious difference in CST, which remained in the range of 5000 - 6000 s over the entire period of 3 HRT (165 and 207 days for each OLR respectively).



(a)



(b)

Figure 5.21. Results of dewaterability testing on digestates from mesophilic and thermophilic AD of SBP at different OLR. (a) CST; (b) Final supernatant interface height as a % of original height of SBP digestate in FIC test. Vertical lines indicate completion of one or more HRT at a given loading

The FIC test was also able to show a clear difference between the dewaterability of the mesophilic and thermophilic digestates (Figure 5.21b). Initially (day 15 - 155) for digestate from mesophilic AD operated at OLR 4 g VS l^{-1} day^{-1} approximately 40% of supernatant could be separated after one hour of centrifugation at 1100 rpm (100 g). After this time there was a rapid deterioration with 0% separation by day 225. At OLR 5 g VS l^{-1} day^{-1} the deterioration started earlier and was more gradual, so that the ~50% of

supernatant that could be separated initially had decreased to 20% at the end of 1 HRT (day 55) and to 0% shortly after 2 HRT (by day 148).

In contrast dewaterability of thermophilic digestate showed a small improvement with time as measured by the FIC test, although there was a slight fluctuation towards the end of 3 HRT. All the samples had an average of 80% supernatant separation after one hour of centrifugation at 100 *g*. The above result suggested that, despite the high CST values, dewatering may be possible without chemical destabilisation of the digestate matrix. The results from this work are therefore similar to those from previous studies on digestion of activated sludge and wastewater biosolids, which showed that thermophilic AD improved the digestate dewaterability characteristics (Buhr and Andrews, 1977; Chi et al., 2010; Amani et al., 2011; Zhou et al., 2002).

Conclusion. Digestion of SBP under thermophilic conditions gave better digestate dewaterability, and this was maintained over prolonged digestion periods.

5.3. Foaming

5.3.1. Antifoam serum bottle test

Objective. To evaluate the effect of antifoam on biogas production, and to allow estimation of any threshold concentration for antifoam inhibition of the digestion process.

Method: Seven antifoaming agents were tested, obtained from 4 different commercial chemical companies (Table 5.18). Triplicate 20 ml aliquots of sieved digestate were dispensed into a crimp top serum bottles with a capacity of 119 ml. To each of the triplicates antifoam was added at doses of 0.05, 0.1, 0.2, 0.5 or 1 ml l^{-1} digestate. A test with no antifoam added (blank) was also prepared. The headspace of each serum bottle was then flushed with N_2/CO_2 (80:20) (BOC, UK) to provide anaerobic conditions and the bottle sealed using a crimp cap PTFE coated silicone septa, with an initial pressure measurement taken to ensure there was no leakage. The serum bottles were placed into an incubator (Hybaid Maxi 14, Thermo Scientific, UK) at 37 ^{o}C without shaking and the pressure was measured every 2 hours up to 72 hours and then less frequently over a 21-day test period. Biogas production was calculated by converting pressure readings to gas volume in the headspace at STP conditions using the ideal gas law as described in

section 3.2.4.2. The pressure was measured using a Digitron 2025P absolute pressure meter (Electron Technology plc., UK) with care taken to avoid any pressure release during reading.

Table 5.18. Type of antifoams

Antifoam	Type	Appearance	Active ingredient (%)	Company
AF 530	Silicone emulsion	Milky off white liquid	-	Goldencrest-chemicals
BC 86/103	Silicone emulsion	Translucent yellowish grey viscous liquid	100	Basildon chemical
Bevaloid 6016I	Silicone emulsion	White, milky liquid	23	Kemira
Bevaloid 1725	Water based fatty alcohol emulsion	White milky emulsion	24	Kemira
Bevaloid 5000	Oil free, ester (surfactant)	Pale yellow liquid	100	Kemira
Bevaloid 2541	Oil based emulsifiable	Yellow opaque liquid	100	Kemira
J-Quell 19	Mineral oil defoamer	White, milky liquid	-	J1 Technologies

Results

Biogas production from the controls and average net biogas production from the digestate samples is shown in Figure 5.22. The controls showed very good agreement until the end of the experiment (about 500 hours) (Figure 5.22a).

In general, it can be seen that all the sample sets containing digestate with the addition of antifoams in the range of 0.05 – 0.2 ml l^{-1} digestate produced more biogas than the control samples. Of the three silicone emulsions tested (AF 530, BC 86/103 and Bevaloid 6016E), AF 530 showed a small net increase in biogas yield of about 0.01-0.07 ml g^{-1} digestate WW with increasing dosage from 0.05 – 0.2 ml l^{-1} digestate, all of which was produced within the first 75 hours of the test (Figure 5.22b). At a dosage of 1.0 ml l^{-1}, however, there was an initial peak in cumulative biogas production, but the cumulative net total started to fall after ~116 hours, leading to a negative value at the end of the experiment. Antifoam BC 86/103 at a dose of 0.5 ml l^{-1} and 1.0 ml l^{-1} caused severe inhibition. This reduced to mild inhibition at 0.2 ml l^{-1}, no inhibition at 0.1 ml l^{-1} and a small increase in net biogas production at 0.05 ml l^{-1} (Figure 5.22c). The addition

of Bevaloid 6016E at doses above 0.5 ml l^{-1} resulted in negative net biogas production at the beginning of the test then a small increase some days later (Figure 5.22d).

The addition of Bevaloid 1725 (water based fatty alcohol) at doses above 0.5 ml l^{-1} resulted in negative net biogas production. At doses of 0.05 to 0.2 ml l^{-1} net production was negative at the beginning of the test but gave a small positive value later (Figure 5.22e). For Bevaloid 5000 (oil-free ester) and Bevaloid 2541 (oil-based) similar trends was observed (Figure 5.22f and g), although for Bevaloid 5000 the net gas production was positive in all cases and at doses of less than 0.5 ml l^{-1} was around 0.03-0.04 ml biogas g^{-1} digestate WW. In contrast J-QUELL 19 showed little or no effect on gas production throughout the test at all concentrations tested and no sign of inhibition was observed. The serum bottle test therefore was able to provide substantial results for recommending the most effective antifoams to be added in digestion process in regard to eliminating foaming without any negative effect to digestion performance. However, further evaluation on the long-term effect of the addition of antifoam in digestion performance need to be performed in semi-continuous trials.

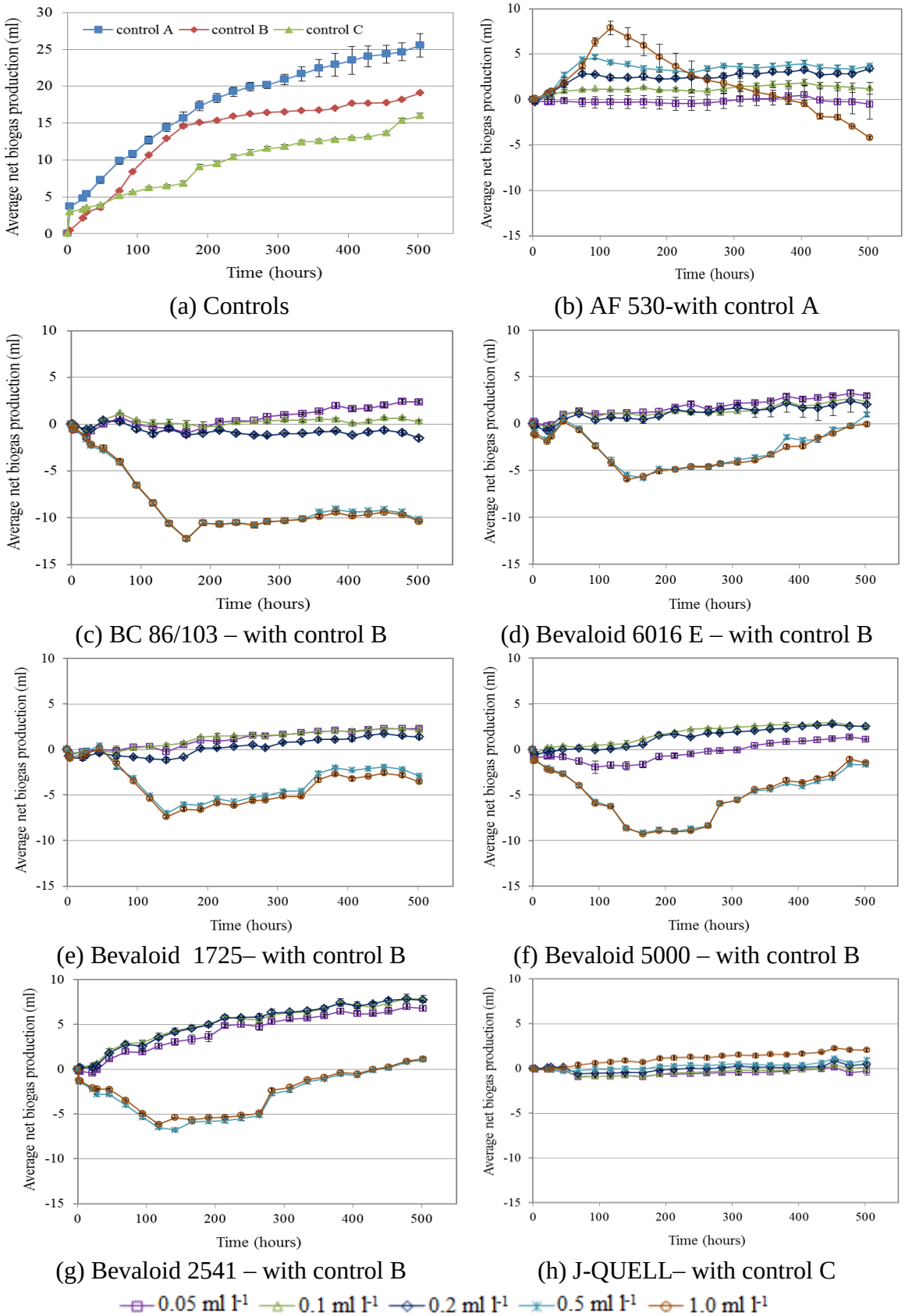


Figure 5.22. Average control biogas and net biogas production in serum bottle tests with different types and doses of antifoams. (If error bars are not seen on control samples, error is less than size of symbol, all the average values are based on triplicates samples)

The results confirmed that the choice of antifoam is important, due to its impact on both the physico-chemical properties and the microbial activity of the medium (or inoculum) (Koch et al., 1995). For instance, Routledge et al. (2011) compared the performance of five different type of antifoams, including Struktol J673A (oil based), antifoam A and C (silicone emulsion), Schell and Schelinger's Struktol SB2121 (polyalkylene glycol) and Fluka P2000 (a polypropylene glycol) at dosages of 0.1 - 1.0% (v/v), in the production of green fluorescent protein (GFP) from recombinant *Pichia pastoris*. The addition of oil based antifoam (Struktol J673A) had a positive effect in enhancing the amount of GFP secreted into the medium from 246 μg GFP (control) to 394 μg GFP. Calik et al. (2005), who studied two different antifoams containing surfactant and silicone oil in a model fermentation process for enzyme production by recombinant *Escherichia coli*, found that the addition of surfactant-based antifoams did not affect the metabolism of *E. coli*, in contrast to silicone oil based antifoams which had negative effects on enzyme production and cell concentration.

Conclusion. The above results indicated that the anaerobic digestate was active and still contained readily biodegradable material, but the addition of antifoams particularly at dosages of 0.5 m l^{-1} or more could inhibit gas production. The use of J-QUELL 19 antifoam had least effect on the serum bottle test results of any of the antifoams tested. Although J-QUELL 19 did not enhance biogas production, indicating it was not readily degradable in the system, there was nothing in the screening test to indicate any adverse effect.

5.3.2. Defoaming test

Objective. To compare the effectiveness of antifoams used in this study.

Methods. A Bartsch method was used to measure the activity of the antifoams, adapted from that outlined by Denkov et al. (2002), in which 100 ml of surfactant solution (Sodium lauryl sulphate/ Sodium dodecyl sulfate, 1% w/v) was placed in a 300 mL graduated glass cylinder (5 cm in diameter). Antifoam was then added at dose of 0.5 ml l^{-1}. The cylinder was tightly plugged by a bung, and the foam generated by shaking about 10 times in vertical position. Initial foam height was recorded and then foam height was recorded at 1 minute intervals for 6 minutes or until no foam remained (liquid surface). Foam volume was calculated by subtracting the volume of medium

from the total volume (foam plus medium) in the cylinder. The tests were performed in duplicate for each antifoam.

Results

As can be seen from Figure 5.23, the foam volume generated after the addition of antifoams was several times smaller than in the control sample. After 6 minutes observation the volume of the control sample remained the same, whereas with 0.5 ml l^{-1} of antifoam the foam volume reduced within 60 seconds in all cases. In most cases the foam reduction efficiency was 70% or more. Bevaloid 1725 which is a water based fatty alcohol emulsion (no silicone) gave a slightly lower percentage reduction at ~63% (Table 5.19), while J-QUELL 19 (mineral oil) was marginally the most effective (Figure 5.23). According to Routledge (2012), the addition of antifoams enhances oxygen transfer in the medium, leading to a decrease in bubble size followed by release into the aqueous phase. Denkov and Marinova (2006) also found that antifoam containing silicone oil was able to greatly reduce foaming occurrence, and the addition of silica improved this reduction further. The minor differences noted in foam reduction efficiency were probably due to the chemical characteristics and physico-chemical properties of the antifoams, as stated by Denkov and Marinova (2006) and Kougias et al. (2013a). Kougias et al. (2013a) found that antifoams containing natural oil (i.e. rapseed oil, sunflower oil), Long Chain Fatty Acids (LCFA) (i.e. oleic acid and octanoic acid), esters (tributylphosphate), and the commercial antifoams (natural fatty acids - Struktol SB 2080) were the most effective compounds to suppress foam in raw and digested cattle manure samples, with foam reduction efficiencies of 89–100%; while other antifoams containing silicone emulsion (Struktol SB 2113 Dimethylpolysiloxane), alchohol (ethanol), and salts (i.e. polyaluminium chloride, calcium chloride dihydrate and magnesium chloride hexahydrate) had foam reduction efficiencies of less than 30%.

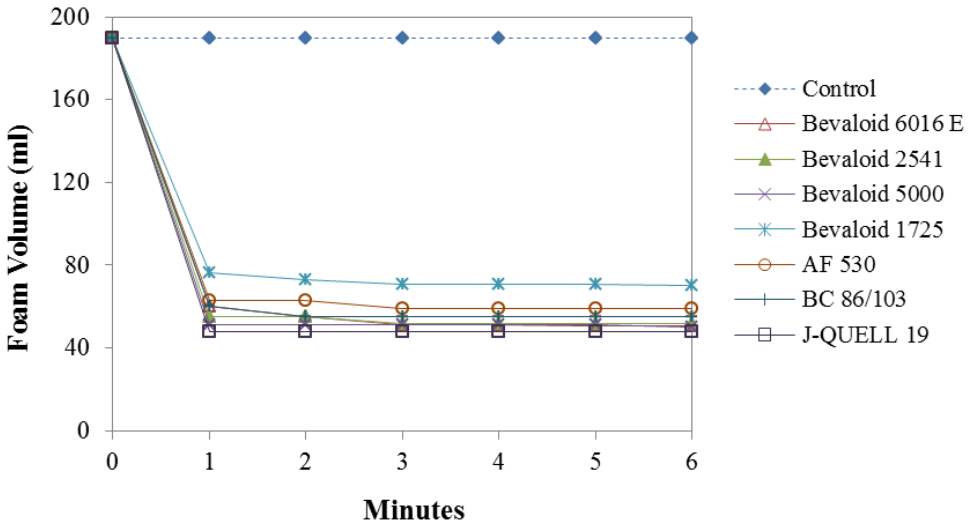


Figure 5.23. Foam volume versus time for seven antifoam and control, at antifoam dose of 0.5 ml l^{-1}

Table 5.19. Foam reduction efficiency of different type of antifoam

Antifoams	Reduction efficiency (%)
Bevaloid 6016 E	73.5
Bevaloid 2541	72.8
Bevaloid 5000	73.5
Bevaloid 1725	63.3
AF 530	69.2
BC 86/103	71.3
J-QUELL 19	74.8

Conclusion. While the current tests did not show major differences between the antifoams, J-QUELL 19 was marginally the most effective for foam destruction. Thus, based on the results of the tests in section 5.3.1 and 5.3.2 J-QUELL 19 was selected for further testing in a semi-continuous trial to determine its effect on long-term operation when used to minimise foaming (see section 4.5.2).

5.3.3. Effect of digestion temperature on foaming occurrence

Objective. To determine the effect of digestion temperature in terms of potential for foaming occurrence in AD of SBP.

Methods. Digestate samples were obtained from mesophilic and thermophilic digesters M1, M4, T1 and T4 on days 94, 166 and 266 (see section 4.6). The fresh digestate was

measured for potential to foam using the aeration foaming potential test according to the methods in section 3.2.5.4.

Results

Figure 5.24 shows the results of the foaming potential test over time for the digesters operated under mesophilic and thermophilic conditions (section 4.6). The samples from mesophilic AD at OLR 4 and 5 g VS l^{-1} day^{-1} both showed foaming potential, with a final value of 0.14 at the higher OLR compared to 0.10 for the lower OLR (Figure 5.24a). The foaming tendency showed a gradual increase over the experimental period, from ~0.01 to 0.04 ml ml^{-1} min^{-1} at OLR 4 g VS l^{-1} day^{-1}, and from ~0.02 to 0.06 ml ml^{-1} min^{-1} at OLR 5 g VS l^{-1} day^{-1} (Figure 5.24b). A similar trend was seen in foam stability which increased to 0.03 ml ml^{-1} min^{-1} and 0.04 ml ml^{-1} min^{-1} at OLR of 4 and 5 g VS l^{-1} day^{-1} respectively by the end of 3 HRTs (Figure 5.24c). There was a slight decrease in foaming propensity over the period of 3 HRT, with final values of 0.95 mm g^{-1} TS and 1.42 mm g^{-1} TS for OLR 4 and 5 g VS l^{-1} day^{-1} respectively (Figure 5.24d).

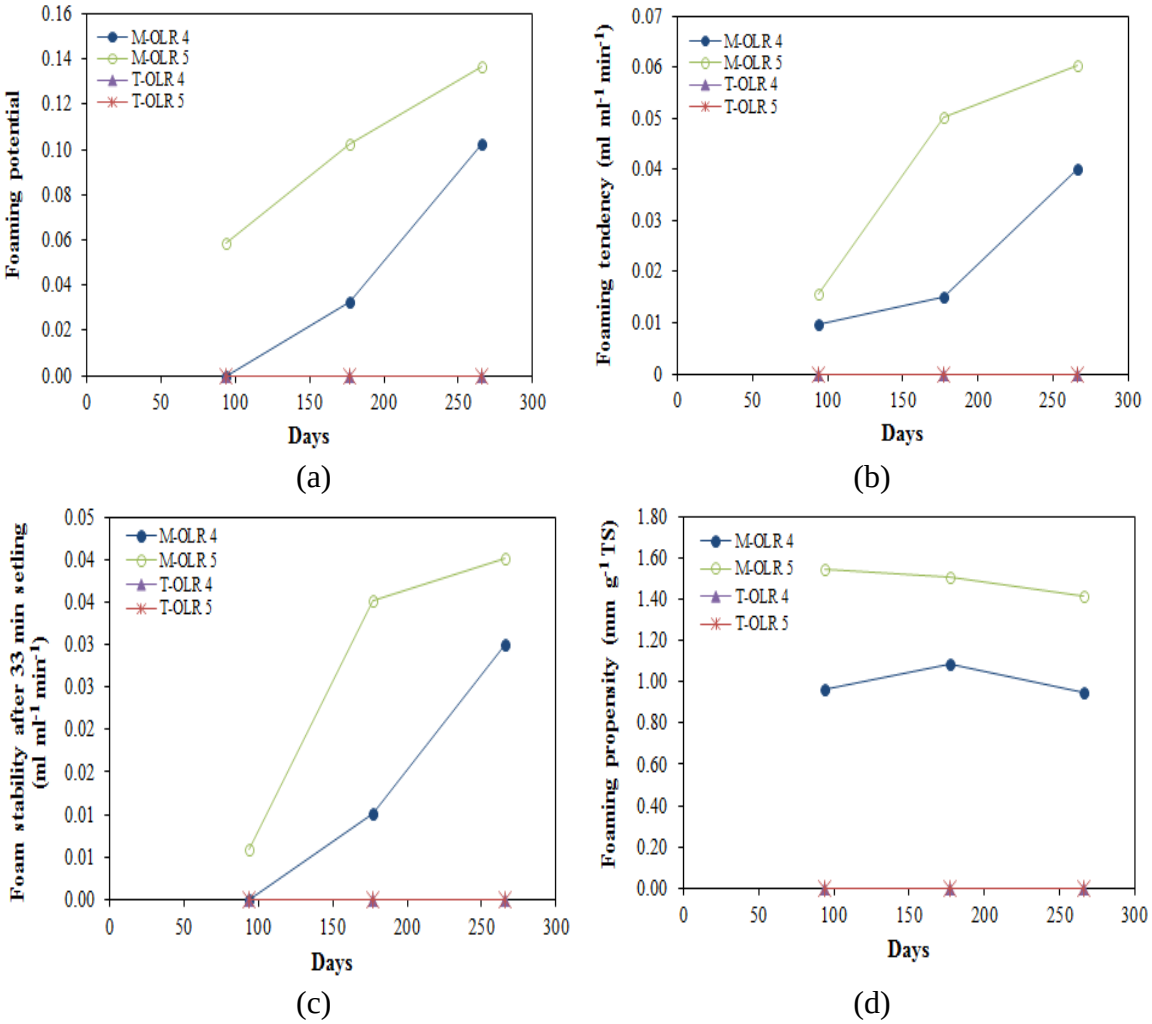


Figure 5.24. Foaming test results for mesophilic and thermophilic SBP digestates: (a) foaming potential; (b) foam stability; (c) foaming tendency; and (d) foaming propensity

Digestate for the thermophilic digesters gave values of zero in the foaming potential test, and as any foam that did form during the 5-minute aeration had disappeared within one minute after aeration stopped, all of the other foaming tests were also negative. This result agreed with the visual observations of the digester contents described above.

Conclusion. Thermophilic AD largely eliminates the foaming problems that occur in mesophilic AD of SBP.

5.4. Digestate structure and components

Throughout the experiments centrifugation had always proved better at digestate separation and dewatering than methods based on pressure-induced filtration. One

possible reason for this was the presence of a light fraction in the digestate that could not be separated by the gravitational forces seen in the FIC apparatus but that could cause 'blinding' of filter paper pores in both the CST and Buchner funnel tests. The overall aim of these experiments was therefore to determine whether the use of differential *g* forces could elucidate the composition of the digestates with respect to their separation characteristics.

5.4.1. Effect of centrifugation on separation of mesophilic digestate

Objective. To identify the centrifugation force(s) required for separating the liquid–solid fractions in SBP digestate.

Method. Mesophilic digestate samples were taken on days 146 and 480 from digester N1 fed at OLR 2 g VS l^{-1} day^{-1} (see section 3.5.1) and from digester N5 operated at OLR 4 g VS l^{-1} day^{-1} with feedstock dilution (section 3.5.2). 30 ml aliquots of each sample were placed in 50 ml centrifuge tubes and centrifuged at 613 - 24,175 *g* for 10 mins in a Sorvall Legend XTR Centrifuge (Fisher Scientific Ltd, UK) equipped with a Fiberlite F15-6x100 rotor (Fisher Scientific Ltd., UK). The centrifuged samples were examined to determine the supernatant interface height and identify any layered structure in the digestate pellet. FIC tests were also carried out according to the methods in section 3.2.5.

Results

The digestate fed at OLR 2 g VS l^{-1} day^{-1} showed a clear increase in separation as the relative *g* force increased from 613 to 21475 *g* (Figure 5.25). There was also a progressive change in supernatant colour and, after centrifugation at a *g*-force of 9803 or more, distinct layers were apparent consisting of four parts: a residue, biomass fraction, non-cellular light fraction, and supernatant liquid (Figure 5.26).

The same trend was noted in the samples from the digester fed at OLR 4 g VS l^{-1} day^{-1} with feedstock dilution; however, these samples showed greater separation at the lowest speed used (Figure 5.25), perhaps due to the effect of water on the digestate structure.

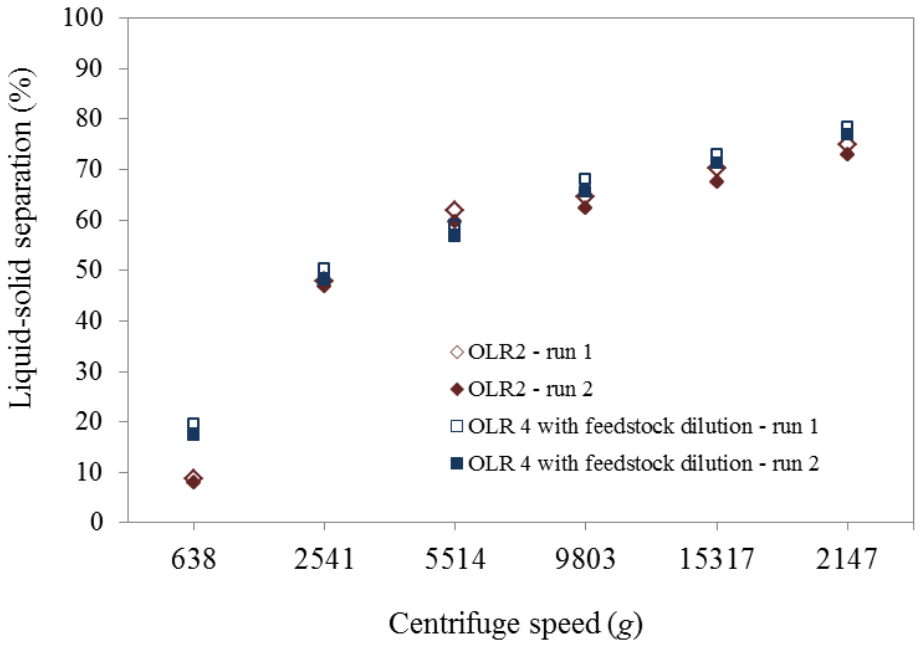


Figure 5.25. The percentage of separation for digesters without and with feedstock dilution at different centrifuge speed (1st trials)

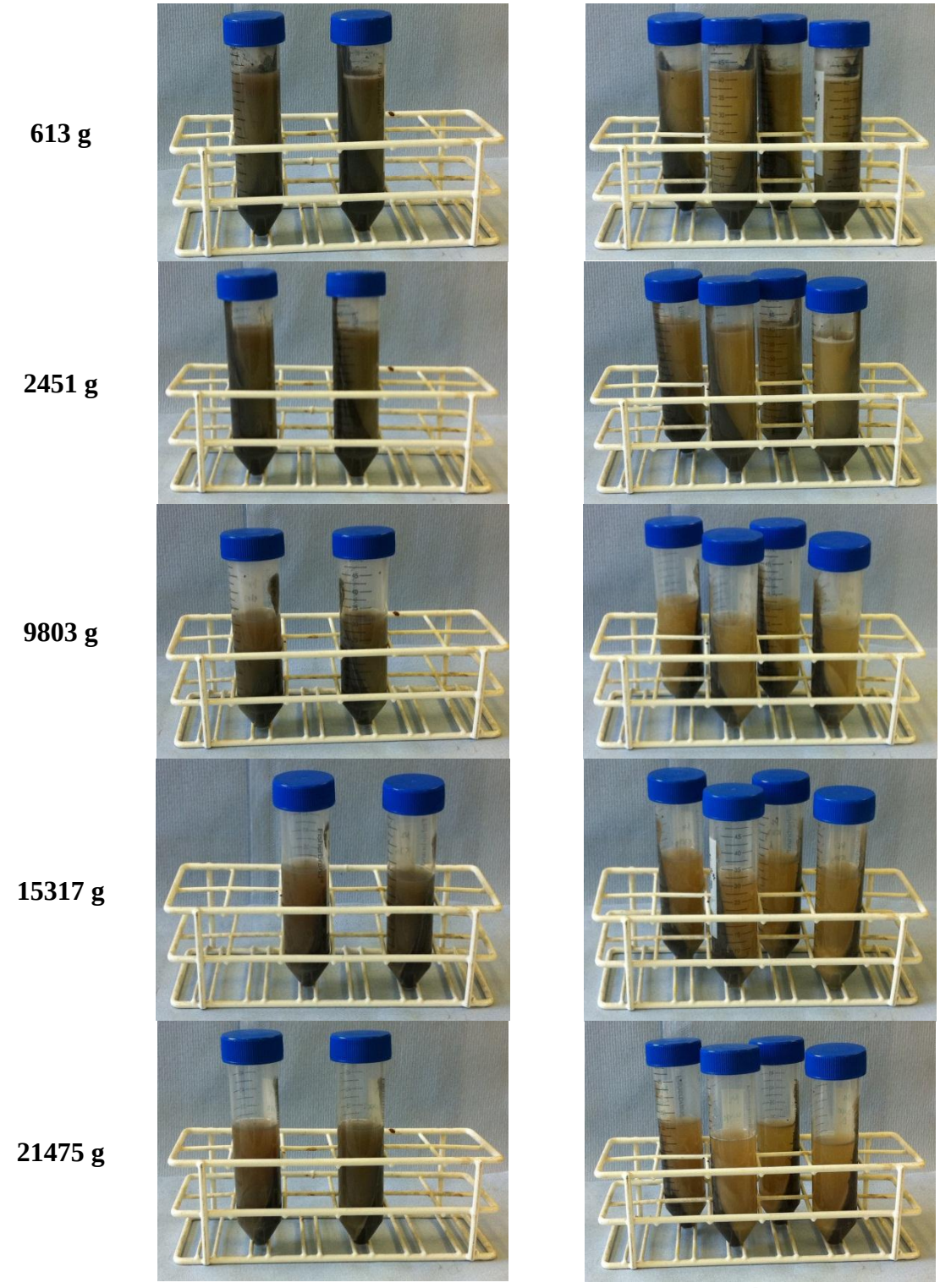


Figure 5.26. Image of digestate (solid-liquid fraction) after subjected to centrifugation at different speed: (a) OLR 2 g VS l^{-1} day^{-1} and (b) OLR 4 g VS l^{-1} day^{-1} (with feedstock dilution)

Conclusion. Centrifugation at 2451 *g* or above was able rapidly to separate the liquid fraction from both digestate samples, and to create a 4-layer structure in the centrifuged digestates.

5.4.2. Effect of centrifugation on separation of mesophilic and thermophilic digestates

Objective. To compare layer formation in mesophilic and thermophilic digestates at different OLR, and to isolate and identify a non-cellular light fraction in the supernatant, with a view to suggesting causes for differences in dewaterability characteristics.

Method. Digestate samples were taken from digesters M1, M4, T1 and T4 on day 120 of the comparative trial of AD of SBP at mesophilic and thermophilic conditions (see section 4.6). The methods used were as described in section 5.4.1.

Results

Centrifugation tests showed that the digestate separated into four parts: a solid residue, a biomass layer, a non-cellular light fraction, and a supernatant liquid (Figure 5.27). On centrifugation of mesophilic digestate at 438 *g* to 7012 *g* layers of solid residue, biomass and supernatant liquid could be seen, but no non-cellular light fraction could be detected and it appeared this material was still mixed with the supernatant. On centrifugation at 8875 *g* to 21475 *g* there was a clear difference between the supernatant and the non-cellular light fraction. For the thermophilic digestate on centrifugation at 438 *g* to 8875 *g* a thin layer of non-cellular light fraction could be seen. When the centrifugation speed was increased to 10956 - 21475 *g*, a very thin layer of the non-cellular light fraction was separated, but the quantity was much smaller than in the mesophilic digestate. The thickness of the non-cellular light fraction layer in the thermophilic digestate samples was less than the line thickness used in Figure 5.27, and thus it cannot be seen in the images.

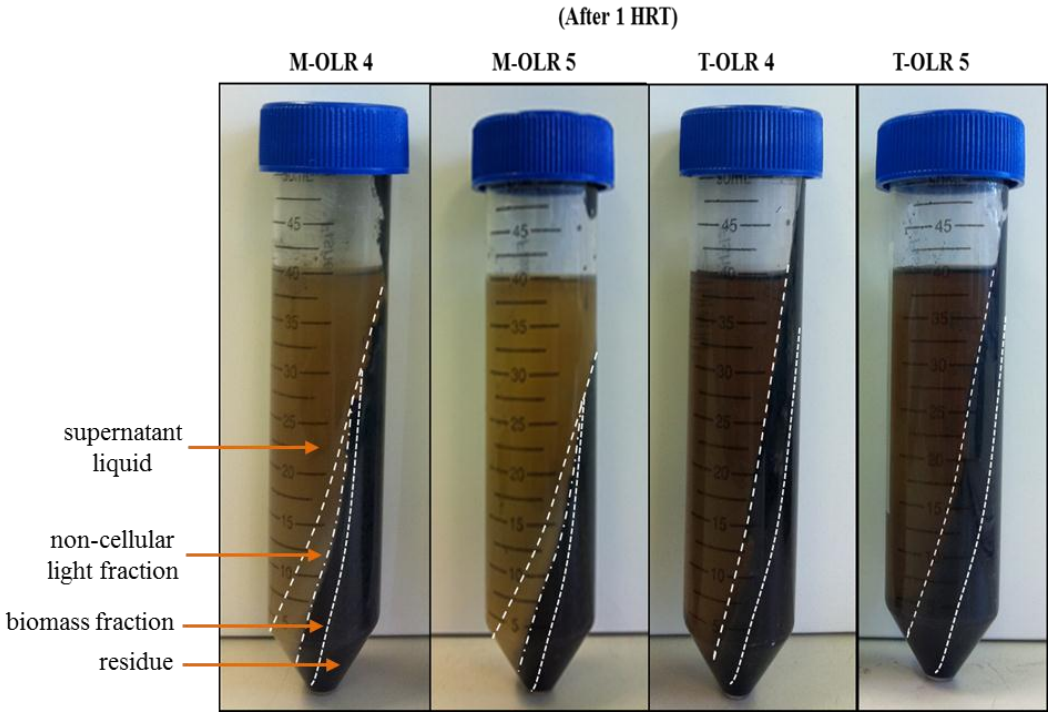


Figure 5.27. Separation of samples of mesophilic (M) and thermophilic (T) digestates at OLR 4 and 5 g VS l^{-1} day^{-1} by centrifugation at 21475 *g* (dotted lines indicate interface between solid residue, biomass layer, non-cellular light fraction, and supernatant liquid)

The non-cellular light fraction could be made up of extracellular polymer substances (EPS) or soluble microbial products (SMP): if so this may be responsible for the differences in dewaterability characteristics of the digestates tested and in particular the poor dewaterability and foaming in mesophilic digesters. EPS has been reported in wastewater sludges on a number of occasions and has been variously described as being composed of proteins, carbohydrate (polysaccharide), lipids, and humic acid (Mikkelsen and Keiding, 2002; Ramesh et al., 2006). SMP on the other hand are secreted by cells as soluble components: they contain less polysaccharide and no proteins, but have roughly the same amount of lipid and more humic substances compared with soluble EPS (Ramesh et al., 2006) . To allow determination of the nature of the non-cellular light fraction a larger sample was therefore obtained by centrifuging the entire contents of digester M4 at the end of the experimental run described in section 4.6. This was analysed for the parameters shown in Table 5.20 by the methods given in Chapter 3.

From Table 5.20, it can be seen that major component of the non-cellular light fraction was likely to be a protein (43.4%) and carbohydrates (polysaccharide) (7.4%). The protein content was calculated based on multiplying the TKN concentration by the standard conversion factor of 6.25, which is commonly used to convert Kjeldahl N into crude protein value for lignocellulosic materials such as sugar beet pulp (Hatakka and Pirhonen, 1985; Grajek and Gervais, 1987; Di Lena and Quaglia, 1992; Hartnell et al., 2005). Carbohydrate content of the non-cellular light fraction was composed of hemicellulose and cellulose, extracted using the Fibercap method described in section 3.2.3.7. The results suggested that the non-cellular light fraction was best classified as EPS since it mainly composed of carbohydrates and protein (Liu and Fang, 2002; Comte et al., 2006a, 2006b; Adav and Lee, 2008), which bonds together with bacteria causing difficulty in separating the liquid-solid fraction of sludge (Ayol et al., 2008). Ayol et al. (2008) noted that complex organic structures in particulate organic matter such as EPS are principally hydrolysed by enzymes such as glucosidases, lipases, and proteases. In the study of enzyme hydrolysis (section 5.2.4) using two commercial formulations of cellulolytic enzymes were not effective in improving dewaterability, further supporting the view that this material is EPS rather than SMP. Further study on enzyme treatment using different enzyme types, in particular proteases and lipases, is therefore recommended.

Table 5.20. Characteristics of non-cellular light fraction material

Parameter	Value
Elemental analysis (%TS)	
C	46.47
H	6.18
N	8.09
O	9.06
Total Kjeldahl Nitrogen (%TS)	6.95
Biochemical composition	
Crude Protein (%TKN * 6.25)	43.43
Hemicellulose (%TS)	4.91
Cellulose (%TS)	2.47
Lignin (%TS)	0.95
Monosaccharide sugars	n.d

Note: n.d = not detected

Figure 5.28 shows an SEM image of the extracted non-cellular light fraction which appears as a continuous folded sheet of material.

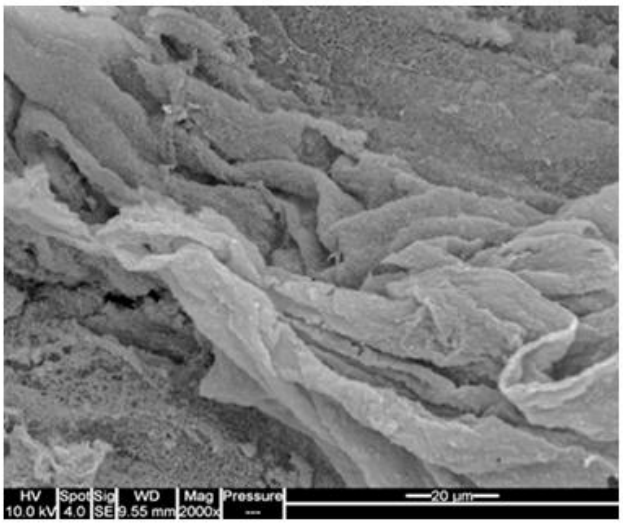


Figure 5.28. SEM image of separated non-cellular light fraction. (Magnification of 2000x, 10 kV and scale bar, 20 µm)

Conclusion. The results suggested that the light non-cellular fraction was EPS, and that the higher quantity of this in mesophilic digestate, especially at higher OLR, may account for its poor dewaterability and foaming behaviour.

5.4.3. Effect of heat treatment on mesophilic digestate

To test whether the EPS might be heat labile and therefore broken down in thermophilic digesters, as opposed to not being produced, a further experiment was carried out. This compared the CST and FIC values of mesophilic digestate before and after heating.

Objective. To assess whether heating mesophilic digestate to 55 ^{o}C, 60 ^{o}C and 65 ^{o}C improved dewaterability.

Method. 100 g of mesophilic digestate taken from digesters M1 and M4 on day 327 (section 4.6) was placed into a 250 ml Erlenmeyer flask and heated in a water bath at the selected temperature for 24 hours. Samples were taken before and after the treatment for CST and FIC analysis. Heat treated samples were also used to identify the presence or absence of the non-cellular light fraction (EPS) in the supernatant.

Results

The experimental results showed that increasing the temperature made no difference to the CST or FIC values or to the amount of non-cellular light fraction that could be recovered through centrifugation (Table 5.21). This indicated that the material was thermally stable at the temperatures tested, suggesting that the EPS was not produced in the thermophilic process, rather than being produced and then subsequently broken down. Barjenbruch and Kopplow (2003) showed that EPS in wastewater sludges could be broken down by autoclaving at 121 ^{o}C and that this led to a reduction in foaming during digestion, but heat treatments at lower temperatures were ineffective.

Table 5.21. Results of CST and FIC test on mesophilic digestates after thermal post-treatment

Parameters	OLR (g VS l^{-1} day^{-1})	Initial	After thermal post-treatment		
			55 ^{o}C	60 ^{o}C	65 ^{o}C
CST (seconds)	4	>84000	>84000	>84000	>84000
	5	>84000	>84000	>84000	>84000
Interface height/sample height (%) after 1-hr centrifugation	4	100	100	100	100
	5	100	100	100	100
Final solid cake volume (%)	4	0	0	0	0
	5	0	0	0	0
Changes in amount of layer non-cellular light fraction	4	no	no	no	no
	5	no	no	no	no

Conclusions. Heat treatment of mesophilic digestate at 55 ^{o}C, 60 ^{o}C and 65 ^{o}C does not improve dewaterability or lead to breakdown of EPS, indicating that EPS is not thermally labile and is not produced in thermophilic digestion of SBP.

5.4.4. Effect of removal of the non-cellular light fraction

Objective. To confirm that the non-cellular light fraction (EPS) affected digestate dewaterability, and to further investigate the effect of F/T treatment.

Method. A test was performed by removing the non-cellular light fraction (EPS) after centrifugation at different speeds. The digestate samples were obtained from mesophilic and thermophilic digesters (M1, M4 and T1) after 3 HRT, as described in section 4.6. The centrifugation method was the same as in section 5.4.1. The procedure used was as follows: 40 g digestate was weighed and centrifuged and two different techniques were

used to remove the non-cellular light fraction (EPS) in digestate: (1) EPS was carefully separated from the liquid supernatant soon after centrifugation using a disposable syringe; when the EPS was not clearly separated from the supernatant, particularly for mesophilic digestate at speed of < 7012 *g*, all of the supernatant fraction was removed and substituted it with tap water. (2) the centrifuged digestate was frozen for 24 hours and the non-cellular light fraction layer was separated by slicing manually during the thawing process. After removal of the non-cellular light fraction digestate samples were re-suspended and their weight made up to 40 g by water addition to compensate for the material removed. These reconstituted samples were then tested for their dewaterability using the CST test according to the methods in section 3.2.5. Prior to the CST measurements, the sample was thoroughly agitated to ensure that it was well mixed. The amount of EPS removed was taken as equal to the weight of water added to make up the sample to its initial weight (40 g).

Results

The results showed that in all treatments, removing EPS enhanced dewaterability for both mesophilic and thermophilic digestates (Figure 5.29). In thermophilic digestate, CST was reduced from ~5500 s (control) to ~900 - 1200 s with or without F/T treatment. F/T treatment made little or no difference in dewaterability of thermophilic digestate at all centrifuge speeds, reflecting the fact that only a small volume of EPS was found and possibly that this EPS was not disrupted by freezing. In contrast, for the mesophilic digestate removal of EPS generally resulted in a large fall in CST from > 84000 s to ~3000 s, while F/T treatment further decreased the CST to ~2000 s. This showed that removing EPS from mesophilic digestate improved the dewaterability characteristics, and also suggested that freezing slightly disrupted any residual EPS, similar to the previous F/T treatment in section 5.2.2.4. The CST was better at higher centrifugation speeds (> 13257 *g*) in mesophilic digestate, possibly due to more effective removal of the EPS layer as the interface between supernatant, EPS and biomass was clearly defined.

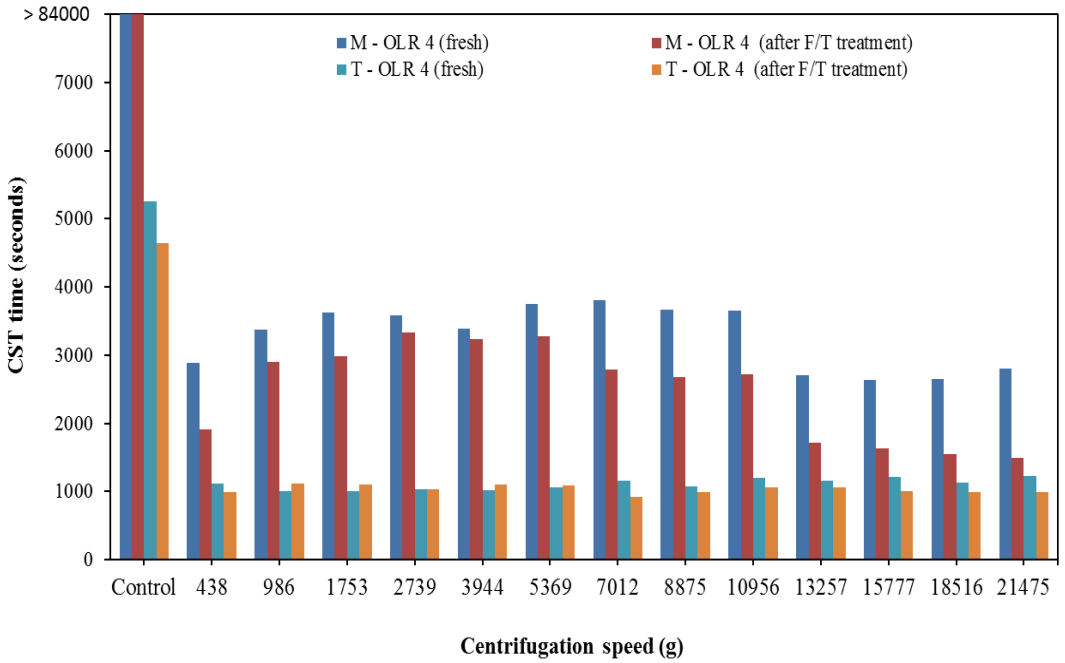


Figure 5.29. Results of CST testing from digestate treated at different centrifugation speed followed by removal of the non-cellular light fraction (fresh and after freeze/thaw)

Table 5.22 shows the amount of EPS removed from mesophilic and thermophilic digestate fresh and after F/T treatment. The results indicated that mesophilic digestate contains a larger amount of EPS compared to thermophilic digestate, which may be linked to the poorer dewaterability performance in mesophilic digestate. Further extraction methods such as EDTA, formaldehyde, sonification etc could be trialled to measure more accurately the quantity of EPS in the digestate.

Table 5.22. Amount of EPS layer removed from mesophilic and thermophilic digestate fresh and after F/T treatment

	EPS removed (g)			
	M-OLR4		T-OLR4	
Centrifugation speed (*g*)	fresh	after F/T	fresh	after F/T
Control	0	0	0	0
438	4.53*	4.95*	1.61	1.66
986	5.90*	6.11*	1.60	1.63
1753	6.03*	6.42*	1.59	1.62
2739	7.57*	7.97*	1.59	1.61
3944	8.77*	9.14*	1.56	1.58
5369	10.95*	11.16*	1.54	1.60
7012	2.74	3.20	1.45	1.54
8875	3.81	4.19	1.44	1.48
10956	5.10	5.52	1.43	1.45
13257	5.42	6.00	1.42	1.43
15777	6.06	6.62	1.42	1.43
18516	6.36	6.84	1.42	1.43
21475	7.00	7.08	1.41	1.43

Note: control = no EPS removed, *at speed of 438-5369 *g* the amount represents EPS and supernatant, as the interface layer between these two was not clear (EPS still mixed with supernatant)

These results confirmed that EPS is one of the main factors affecting dewaterability in SBP digestate: similar findings have been reported in studies of raw and digested wastewater biosolids suggesting that any control techniques that can be identified may be effective in both cases (Urbain et al., 1993; Poxon and Darby, 1997; Liao et al., 2001; Houghton et al., 2000a, 2000b, 2001; Houghton and Stephenson, 2002; Sponza, 2003; Jin et al., 2004; Li and Yang, 2007; Hosnani et al., 2010).

Conclusion. Removing the EPS fraction from mesophilic and thermophilic digestate enhanced the dewaterability characteristics. Combining with F/T treatment gave a further improvement in mesophilic digestate, but only a small change in thermophilic digestates.

5.5. Nutrients and PTE in digestate components

Digestate from AD system is not only a potentially valuable commodity as it has a high nutrient and organic material content, but also has high disposal costs. The current focus of digestate treatment is to recover the maximum amount of the nutrients (N, P, K) present for use as biofertiliser or soil conditioner, as well as to produce a dischargeable

or re-useable liquid fraction. Furthermore, knowing the nutrient and PTE content in digestate is critical with respect to the regulation of digestate application to land and to environmental and health effects.

Objective. To determine the nutrient content and other properties of whole digestate and liquid and solid digestate fractions from thermophilic and mesophilic digesters with respect to their potential for utilisation in agriculture or disposal.

Method. The digestate samples were obtained from mesophilic and thermophilic digester (M1, M4, T1 and T4) on day 230 of the experiment described in section 4.6. The whole digestate (WD) used was fresh digestate, which was subjected to centrifugation at 21475 *g* to separate the solid and liquid fractions. Certain analyses, such as alkalinity, TAN, COD, and turbidity, were performed directly after the centrifugation on the separated solid (SF) and liquid (SL) fractions, by the methods given in section 3.2.3. For TKN and elemental analyses, the samples were air dried to constant weight and then homogenised using a centrifugal grinder type ZM1 (Glen Creston, Stanmore, UK) and stored in sealed screw-top containers in a desiccator before analysis according to the methods in section 3.2.3.

Results

The characteristics of the whole digestate, solid and liquid fractions are presented in Table 5.23. The nutrient content (N, P, K) in whole digestate was quite high compared to that of from digested sewage sludge, card package etc., indicating its potential as biofertiliser.

Table 5.24 shows the actual digestate nutrient concentrations and estimated values based on specific biogas yield and % VS destruction. Both calculation methods gave approximately similar values for all nutrient parameters, supporting the accuracy of the experimental data. Measured N concentrations were in the range of ~92 - 100% of calculated values, indicating a good agreement. Measured K concentrations were in the range of ~70 - 90%, while the recovery of P was more than 100%, apart from in mesophilic digestate at OLR 5, possibly indicating some systematic error in analysis.

In term of nutrient N partitioning between liquid and solid fraction, it can be seen from Table 5.25 that mesophilic digestate from both OLR tested and thermophilic digestate at OLR 4 have moderate percentage recovery, with the value of 70%, 82% and 86%,. While for thermophilic at OLR 5, the N nutrient partitioning in liquid and solid fraction was overestimated by 30%.

The concentration of Fe was higher in thermophilic digestate than in the mesophilic digestates on both a fresh and dry weight basis, possibly due to higher VS destruction in the thermophilic system and/or to leaching from the stainless steel stirrer. For the potentially toxic elements (PTE) measured in this study, Ni was much lower (~0.23 – 0.75 mg kg^{-1} TS) than that the maximum concentration (50 mg kg^{-1} TS) of digested material for application to land (Table 2.5 in Chapter 2). This suggests that the digestate resulting from AD of SBP has the potential to be utilised on both agricultural and arable land.

One of the main purposes of separating the solid and liquid fractions of digestate is to reduce transportation costs; but if the liquid fraction is not to be used it may require treatment before disposal. It can be seen that COD of the liquid fraction of the mesophilic digestates was higher than of the thermophilic digestates at both OLRs. In mesophilic conditions the COD was much higher at the higher OLR, while no significant difference was observed between the thermophilic digestates. Before the supernatant could be discharged to a water course, further treatment would be required in order to meet the minimum UK standards of 125 mg COD l^{-1} (Legislation Government of UK, 1994). The turbidity values were also very high. In mesophilic WD samples the concentration of soluble salts, as estimated by electrical conductivity (EC), reduced from 9.7 mS cm^{-1} at M-OLR 4 to 1.3 mS cm^{-1} at M-OLR 5. These values were below those in the thermophilic digestate (14.1-14.6 mS cm^{-1}).

In addition, the concentrations of TKN and ammonia N in the supernatant were in the range of 1 - 2 g l^{-1} (assumed density of supernatant = 1 kg l^{-1}), very high for directly discharge to watercourses. Furthermore, the removal of ammonia N in wastewater treatment is energy intensive as it requires ~4.3 g O_2 per g N while typical transfer efficiencies in aeration plants range from 0.9 to 3.1 kg O_2 kWh^{-1} (Tchobanoglous et al., 2003). There is considerable interest in development of technologies to recover N from

liquid supernatants though struvite precipitation etc. (Liu et al., 2011b; Rahman et al., 2011), but these are not considered further here.

Table 5.23. Nutrient and trace elemental content of mesophilic and thermophilic digestates

Parameter	Mesophilic						Thermophilic					
	OLR 4			OLR 5			OLR 4			OLR 5		
	WD	SL	SF	WD	SL	SF	WD	SL	SF	WD	SL	SF
Macro nutrient (g kg^{-1}WW)												
TKN (N)	4.04	1.54	8.15	3.86	1.75	8.09	4.22	1.81	8.19	4.34	1.60	8.47
P	0.61	-	-	0.47	-	-	0.80	-	-	0.77	-	-
K	0.72	-	-	0.80	-	-	0.80	-	-	0.94	-	-
Elemental analysis (mg kg^{-1} WW)												
Co	0.27	-	-	0.23	-	-	0.34	-	-	0.30	-	-
Fe	66.16	-	-	39.73	-	-	115.32	-	-	112.96	-	-
Mo	0.14	-	-	0.19	-	-	0.12	-	-	0.11	-	-
Ni	0.74	-	-	0.94	-	-	0.68	-	-	0.57	-	-
Se	0.11	-	-	0.07	-	-	0.12	-	-	0.11	-	-
Other parameters												
Crude Protein (g kg^{-1} WW)*	25.23	18.58	9.63	24.12	20.81	10.92	26.35	23.94	11.29	27.09	37.94	9.98
Conductivity (mS cm^{-1})	9.7	-	-	1.3	-	-	14.1	-	-	14.6	-	-
Total dissolved salts (g l^{-1})**	7.76			1.04			11.28			11.68		
COD (g l^{-1})	-	96.48	-	-	197.93	-	-	66.09	-	-	68.67	-
Turbidity (NTU)	-	2435	-	-	8015	-	-	1255	-	-	1235	-
Alkalinity (g CaCO3 kg^{-1} WW)	16.1	8.53	39.41	11.6	5.61	16.99	16.9	8.65	34.99	16.7	9.08	39.39
TAN (g NH_3-N kg^{-1} WW)	1.40	1.25	1.85	0.80	0.55	0.85	2.09	1.56	1.90	1.98	1.63	2.04

Note: WD = whole digestate, SL = separated liquid, SF = separated fibre, * = TKN x 6.25, OLR is expressed in g VS l^{-1} day^{-1},
** TDS = k_eEC, where k_e is a conductivity factor ranging from 0.55 - 0.8 (0.55 for natural water, 0.65 for hard/alkaline water, and 0.8 for inorganic nutrients), while EC is the electrical conductivity in µS cm^{-1} at 25 ^oC (Lloyd and Heathcote, 1985). For the purposes of the calculation, a factor of 0.8 is selected.

Table 5.24. Estimation of nutrient balance in feedstock and digestate

	Unit	Input	Mesophilic		Thermophilic	
			OLR4	OLR5	OLR4	OLR5
SBP added	g WW	1000				
VS	% WW	22.6				
Mass SBP added	g VS	226				
TKN	g kg^{-1} WW	3.48				
P	g kg^{-1} WW	0.41				
K	g kg^{-1} WW	0.84				
Measured nutrient values in digestate						
N	g kg^{-1} WW		4.04	3.86	4.22	4.34
P	g kg^{-1} WW		0.61	0.47	0.80	0.77
K	g kg^{-1} WW		0.72	0.80	0.80	0.94
Estimation using specific biogas production						
CH_4	%		53	52	52	52
CO_2	%		47	48	48	48
Specific Biogas production	l g^{-1} VS		0.554	0.549	0.664	0.681
Biogas produced*	g		162.5	162.5	196.6	201.6
Mass of residual digestate	g WW		837.5	837.5	803.4	798.4
Digestate VS remaining	g VS		63.5	63.5	29.4	24.4
Estimated digestate VS conc.	g VS kg^{-1} WW		75.9	75.8	36.6	30.5
Actual digestate VS conc.**	g VS kg^{-1} WW		43.1	67.6	33.8	33.8
Predicted N in digestate	g kg^{-1} WW		4.15	4.16	4.33	4.36
Predicted P in digestate	g kg^{-1} WW		0.49	0.49	0.51	0.51
Predicted K in digestate	g kg^{-1} WW		1.00	1.00	1.04	1.05
Recovery of N	%		97.2	92.9	97.4	99.6
Recovery of P	%		124.6	96.0	156.8	149.9
Recovery of K	%		72.2	80.2	77.0	89.9
Estimation using VS destruction						
VS destruction**	%		85.2	76.6	88.3	88.2
Total weight of VS destroyed	g VS		193	173	200	199
Mass of residual digestate	g WW		807	827	800	801
Digestate VS remaining	g VS		33	53	26	27
Estimated digestate VS conc.	g VS kg^{-1} WW		41.4	64.0	33.0	33.3
Actual digestate VS conc.**	g VS kg^{-1} WW		43.1	67.6	33.8	33.8
Predicted N in digestate	g kg^{-1} WW		4.31	4.21	4.35	4.35
Predicted P in digestate	g kg^{-1} WW		0.51	0.50	0.51	0.51
Predicted K in digestate	g kg^{-1} WW		1.03	1.01	1.04	1.04
Recovery of N	%		93.7	91.7	97.1	99.9
Recovery of P	%		120.1	94.8	156.2	150.4
Recovery of K	%		69.6	79.2	76.7	90.1

Note: value for WD, OLR is expressed in g VS l^{-1} day^{-1}, assume that water vapour is negligible, *CH_4 density= 0.71 kg m^{-3} and CO_2 density= 1.96 kg m^{-3} (on basis = 1 kmol of a perfect gas occupies 22.4 m^3), ** data obtained from Table 4.8 in Chapter 4

Table 5.25. Nutrient N partitioning between liquid and solid fraction

Temperature	OLR	WD g kg^{-1} WW	SL g kg^{-1} WW	SF g kg^{-1} WW	SL % WW	SF % WW	Total g kg^{-1} WW	Recovery %
Mesophilic	4	4.04	1.54	8.15	70	30	3.52	87
Mesophilic	5	3.86	1.75	8.09	70	30	3.65	95
Thermophilic	4	4.22	1.81	8.19	70	30	3.72	88
Thermophilic	5	4.34	1.60	8.47	70	30	5.66	84

Note: OLR is expressed in g VS l^{-1} day^{-1}

5.6. General conclusions

Thermophilic digestion of SBP enhanced the digestate dewaterability characteristics and eliminated its propensity to foam. Two-stage chemical treatment facilitated dewatering, while sludge ageing, physical treatment, treatment with cellulolytic enzymes and thermal post-treatment either did not enhance digestate dewaterability or appeared unlikely to be feasible at a large scale in the UK. All antifoams tested worked very well in terms of foam removal. Some showed a slight enhancement of biogas production at low dosages and signs of inhibition at higher dosages; however the serum bottle test by itself is not necessarily sufficient to indicate toxicity. Mesophilic digestion, especially at higher OLR, produced considerable amounts of EPS which were associated with poor dewaterability and possibly with foaming; this EPS did not appear to be generated in thermophilic conditions, accounting for the better dewaterability of thermophilic digestates. Digestates from mesophilic and thermophilic AD of SBP contained useful quantities of N, P and K, with an acceptable Ni concentration with respect to limits for PTE. After separation, both liquid and solid fractions of digestates still have a high amount of nutrient N. These results confirm that the digestate has potential for application on agricultural land to substitute the use of mineral N, P and K fertiliser both as whole digestate, solid and/or liquid fraction. Carbon, energy and Nitrogen balances for some of these options are considered in the next chapter.

CHAPTER 6. CARBON, ENERGY AND NUTRIENT FOOTPRINT

6.1. Chapter Summary

The main goals of this part of the study were to evaluate quantitatively the carbon, energy and nutrient (CEN) footprints of the AD process for SBP. The results from this analysis can be used to suggest alternatives to the current practice or to identify its environmental impacts and opportunities for reducing them. The selected parameter data related to this study was collected both from the results of the experimental work conducted as part of this thesis, and from the literature or personal communication. Calculation of the CEN balance was carried out using a model developed at the University of Southampton (Salter and Banks, 2009). The difference between mesophilic and thermophilic AD of SBP with respect to the CEN footprint was also assessed.

6.2. System boundaries

The system boundary for AD of SBP, in both mesophilic and thermophilic conditions, includes the anaerobic digestion process, feedstock and digestate transportation, digestate dewatering and application, and biogas utilisation/upgrade as shown in Figure 6.1. In this analysis, feedstock pre-processing was not included in the system boundary as the particle size of SBP is small enough and the material homogeneous enough for it to be fed directly to digesters. Furthermore, the experimental results indicated that SBP should be fed directly to the digester without dilution since the dilution process significantly reduced specific biogas and methane production.

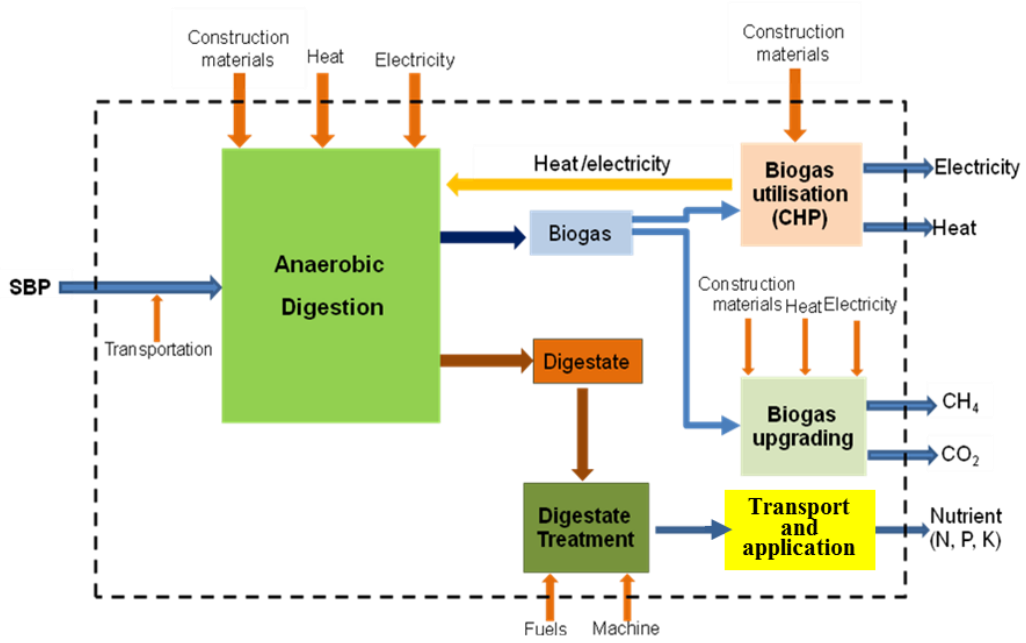


Figure 6.1. System boundary for the energy balance analysis on AD of SBP

6.3. Energy potential of mesophilic and thermophilic AD based on experimental results

6.3.1. 1st semi-continuous AD of SBP in mesophilic conditions with different OLR

Results of energy calculations using the Buswell and Dulong Equations for the first semi-continuous trial are presented in Table 6.1. The energy value of the methane recovered from mesophilic digestion at OLR 2 g VS l^{-1} day^{-1} was 12.6 MJ kg^{-1} VS based on a conversion factor of 39.84 MJ m^{-3} (British Standards, 2005), which is equivalent to 69.4% of the measured calorific value of the substrate. These values were higher than for the mesophilic digesters at OLR 3-5 g VS l^{-1} day^{-1}. The addition of TE at OLR 3 g VS l^{-1} day^{-1} improved the methane yield, thus increasing the energy value to 12.5 MJ kg^{-1} VS or ~69% of the calorific value as methane.

Table 6.1. Comparison of energy potential from mesophilic AD of SBP (1st experiment)

Parameter	Unit	2	3	3 (+TE)	4	5
OLR	kg VS m^{-3} day^{-1}					
Actual CH_4 yield	m^3 kg^{-1} VS	0.316	0.293	0.313	0.286	0.294
Recovery as CH_4	MJ kg^{-1} VS	12.6	11.7	12.5	11.4	11.7
% Recovery of measured CV	%	69.4%	64.4%	68.8%	62.8%	64.8%
Lower heat value	MJ kg^{-1} VS			2.1		

Note: the methane yields values were taken from Table 4.5 in Chapter 4

6.3.2. 2nd semi-continuous AD of SBP at mesophilic and thermophilic condition with different OLR

Table 6.2 shows the energy potential from the mesophilic and thermophilic digestion trial with SBP. The energy potential of thermophilic AD was found to be superior compared to that in mesophilic conditions. The improvement in the thermophilic energy yield is associated with the higher specific biogas production resulting from higher degradation: Zábranská et al. (2000) found similar results for the energy yield from AD of WAS. In the current study the energy value of the methane recovered in thermophilic AD was 13.7 and 14.1 MJ kg^{-1} VS at OLR of 4 and 5 kg VS m^{-3} day^{-1}, equivalent to 75.8% and 78% of the measured calorific value of the substrate. These values were much higher than those for the mesophilic digesters of 11.6 and 11.3 MJ kg^{-1} VS with 64.2% and 62.2% recovery of measured CV. Researchers working with other substrates (corn grain, fruit and vegetable waste) have also found that thermophilic digestion increased the net energy production due to higher biogas and methane production compared to that in mesophilic conditions (Bouallagui et al., 2004; Agler et al., 2008) .

Table 6.2. Comparison of energy potential from mesophilic and thermophilic AD of SBP

Parameter	Unit	Thermophilic		Mesophilic	
OLR	kg VS m^{-3} day^{-1}	4	5	4	5
Actual CH_4 yield	m^3 kg^{-1} VS	0.345	0.355	0.292	0.283
Recovery as CH_4	MJ kg^{-1} VS	13.7	14.1	11.6	11.3
% Recovery of measured CV	%	75.8	78.0%	64.2%	62.2%
Lower heat value	MJ kg^{-1} VS		2.1		

Note: the methane yields values were taken from Table 4.8 in Chapter 4

Operation in the thermophilic range in the full-scale digesters could result in improved operation of existing facilities and consequently avoid digester overloading. Furthermore, the relation between the temperature and the daily production of biogas showed that for an equal amount of biogas produced, the size or number of thermophilic digester units can be decreased compared to mesophilic digesters. van Lier et al. (2001) and Bouallagui et al. (2004) stated that temperature strongly affects the activity of microorganisms and the bioconversion rate of anaerobic organisms, thus a smaller reactor volume will be sufficient at thermophilic temperatures compared to mesophilic conditions.

6.4. Anaerobic digestion scenario modelling (CEN footprint from AD of SBP)

6.4.1. Basic parameters and input variables

The basic model parameters and input variables used are presented in Table 6.3 and 6.4. Values obtained from the laboratory studies and required for full-scale digester design and energy balance assessment are summarised in Table 6.4. Although OLR 2 kg VS m^{-3} day^{-1} gave the highest specific biogas and methane production, similar to the values obtained from BMP test, as well as a stable process with no indication of any inhibition during the laboratory-scale trial, scenarios with different OLR (3, 4 and 5 kg VS m^{-3} day^{-1}) have also been evaluated due to their high volumetric biogas methane yield. The results of the laboratory study confirm that semi-continuous testing is important to estimate the digester design parameters and energy yields in full-scale operation

Table 6.3. Basic model parameters used in energy balance model

Parameters	unit	Value
Input OLR	kg VS m^{-3} day^{-1}	3 - 8
Simulation time digester[a]	days	165-411
Digester temperature	^{o}C	35-55
Volume of gas head space	%	10
Biogas losses	%	1
Parasitic	kWh $tonne^{-1}$ WW	20
CHP electrical efficiency	%	35
CHP heat efficiency	%	50
Heat utilization	%	40
Heat energy source		natural gas
Construction materials		steel

[a] length of run

Table 6.4. Input variables used in energy balance model

Inputs	unit	Value
Mass of feedstock	tonnes WW $year^{-1}$	100000
TS	% WW	22
VS	% TS	94
CH_4 composition	%	50
Proportion of fixed carbon		0.45
Proportion converted		0.94
residual TS	% WW	3.07
N	g kg^{-1} WW	3.48
P	g kg^{-1} WW	0.41
K	g kg^{-1} WW	0.84

6.4.2. Main Scenarios

Two sets of scenarios were developed based on the production of electricity and heat in a combined heat and power (CHP), and of methane through biogas upgrading. In each scenario, the assumptions made were as follows:

1. Feedstock mass of 100000 tonnes WW $year^{-1}$. This was based on the production of SBP from the British Sugar Factory in Wissington at 273750 tonnes $year^{-1}$: the choice of 100,000 tonnes $year^{-1}$ was based on the assumption that a proportion of the material would be used as cattle feedstock.
2. Operation of the AD plant at mesophilic (35 ^{o}C) or thermophilic (55 ^{o}C) temperatures
3. OLR of 3 kg VS m^{-3} day^{-1} at mesophilic temperature, and 3 - 8 kg VS m^{-3} day^{-1} at thermophilic. This was because the experimental results showed that operating at an OLR of 4-5 kg VS m^{-3} day^{-1} in mesophilic conditions was difficult due to severe foaming problems, which also caused a decline in biogas and methane production. At thermophilic temperatures such problems were not observed.
4. The AD plant was operated as a 'complex' and ‘simple’ system. The 'complex' process consisted of a digester followed by a pasteuriser, with biogas stored in a separate gas-holder and then burnt in a CHP unit, and with digestate storage, including digestate separation and composting. The ‘simple’ process is a system without digestate treatment.
5. No pre-treatment of SBP prior to digestion was required as based on the experimental result, the use of non-treated SBP as a feedstock was satisfactory.
6. In all cases, it was assumed that the digesters were constructed of steel with a separate gas holder (capacity for 2 hours production of biogas).
7. Pasteurisation occurred after digestion.
8. It was assumed that there was 1% process loss of biogas.
9. Biogas generated is used in a CHP unit to produce electricity at 35% conversion efficiency and heat. The CHP unit has a load factor of 8300 hours.
10. Biogas volumes are based on the amount of methane and carbon dioxide only (i.e. volume of dry biogas without water vapour, and not taking any other gases into consideration), assuming densities of 0.71 kg CH_4 m^{-3} and 1.96 kg CO_2 m^{-3} and that 1 kmol of an ideal gas occupies 22.4 m^3 at STP.
11. Digestate was stored in a steel tank with volume capacity for up to 6 months.

12. Digestate separation was by belt press with separation efficiency for dry matter of 56%, and for N, P and K of 32%, 29% and 27%, respectively. Using this equipment the volume reduction is assumed to be 29% with the energy required for the process 0.7 kWh m^{-3} (Burton and Turner, 2003).
13. Ambient temperatures were based on Wissington (UK).

The scenarios are identified using the following codes:

M= mesophilic

T= thermophilic

S= simple system

C= complex system

E= biogas used in CHP unit for electricity and heat generation

B= biogas upgrading to biomethane

Unless specified, all loading rates are 3 kg VS m^{-3} day^{-1}

6.4.3. Energy balance for electricity and heat production (AD + CHP)

Table 6.5 shows a summary energy balance for production of electricity and heat in a CHP unit, and detailed energy inputs and outputs are presented in Table 6.6.

Table 6.5. Summary energy balances for electricity and heat production

	Units	MSE3	MCE3	TSE3	TCE3
Energy balance total	GJ $year^{-1}$	171751	163401	164208	155858
	GJ $tonne^{-1}$ waste	1.72	1.63	1.64	1.56
Energy balance electrical	GJ $year^{-1}$	61214	52864	57442	49092
	GJ $tonne^{-1}$ waste	0.61	0.53	0.57	0.49

As expected the quantity of raw biogas and methane was the same for all scenarios. The complex system required more energy for processing than the simple system, and increasing temperature from mesophilic to thermophilic condition caused an increase in heat requirement. For all the cases considered, the energy available from AD of SBP is sufficient to provide both electricity and heat demand for the operation of the plant. The model from these scenarios also shows that AD of SBP has potential as a renewable energy source. This is because the electricity and heat produced were not only able to fulfill the energy required (electricity and heat) for operating the plant, but also to provide electricity and heat for export to other users. For example, since 2000 the

British Sugar plc factory in Wissington has been able to provide heat from the CHP to maintain the temperature of the 5 ha Cornerways Nursery which was built next to the factory (British Sugar, 2012). The energy available for export from thermophilic digestion was lower than from mesophilic digestion. The difference was mainly due to the potential for exported heat in thermophilic digestion which was 1047 MWh $year^{-1}$ less than in mesophilic conditions.

Table 6.6. Energy inputs and outputs for electricity and heat production

Details	Units	MSE3	MCE3	TSE3	TCE3
digester input	tonnes	100000	100000	100000	100000
digester loading rate	kg m^{-3} day^{-1}	3	3	3	3
total digester capacity required	m^3	4155	4155	4155	4155
retention time	days	69	69	69	69
methane produced	m^3	7651600	7651600	7651600	7651600
methane available	m^3	7575084	7575084	7575084	7575084
biogas	m^3	13912000	13912000	13912000	13912000
=	tonnes	17788	17788	17788	17788
digestate	tonnes	82212	82212	82212	82212
Energy balance (year^{-1})					
pre-processing electricity	GJ	0	0	0	0
digester electricity requirement	GJ	3600	3600	3600	3600
electricity for upgrading	GJ	0	0	0	0
electricity for composting	GJ	0	883	0	883
heat for digester	GJ	13017	13017	23695	23695
heat for pasteuriser	GJ	12115	12115	5209	5209
diesel for composting	GJ	0	6574	0	6574
Total	GJ	28732	36189	32504	39961
Embodied energy					
digester embodied	GJ	1200	1200	1200	1200
pasteuriser embodied	GJ	7	7	7	7
CHP embodied	GJ	51	51	51	51
upgrading embodied	GJ	0	0	0	0
gas holder embodied	GJ	14	14	14	14
ABPR building embodied	GJ	18	18	18	18
digestate storage	GJ	125	125	125	125
separator embodied	GJ	0	10	0	10
feedtank embodied	GJ	8	8	8	8
Total	GJ	1423	1433	1423	1433
on-site boiler/CHP		CHP	CHP	CHP	CHP
CHP electrical capacity	kW	3179	3179	3179	3179
energy in methane produced	GJ	274080	274080	274080	274080
generated electricity	GJ	94969	94969	94969	94969
generated heat	GJ	135670	135670	135670	135670
imported electricity	GJ	0	0	0	0
imported heat	GJ	0	0	0	0
exported electricity	GJ	91369	90486	91369	90486
	MWh	25382	25137	25382	25137
exported heat	GJ	110538	110538	106766	106766
	MWh	30707	30707	29660	29660

6.4.4. Energy balance for biogas upgrading to biomethane (AD + biogas upgrading)

Table 6.7 shows a summary energy balance for the scenarios based on production of biomethane with a biogas upgrading unit (e.g. wet scrubbing unit), and detailed energy inputs and outputs are presented in Table 6.8.

Table 6.7. Summary energy balance for biomethane production

	Units	MSB3	MCB3	TSB3	TCB3
Energy balance total[a]	GJ year^{-1}	171669	163319	167442	158029
	GJ tonne^{-1} waste	1.72	1.63	1.67	1.58
Energy balance biomethane[b]	GJ year^{-1}	171214	161801	167442	158029
	GJ tonne^{-1} waste	1.71	1.62	1.67	1.58

Note: [a] including upgraded biomethane and exported heat, [b] including upgraded biomethane but not exported heat

The trends in these scenarios are similar to those for electricity and heat production: as expected, the energy requirement for the complex system was higher than for the simple system. The energy input required for operating thermophilic digestion in all scenarios was higher than that obtained in mesophilic digestion, due to heating the feedstock and digester to a higher temperature (55 ^{o}C). Operation at thermophilic temperature reduced the amount of heat available for export by up to 77%.

The amount of biomethane produced in these scenarios also confirmed the potential of SBP as feedstock for AD systems as an alternative renewable energy sources, replacing the use of fossil fuels or other non-renewable energy.

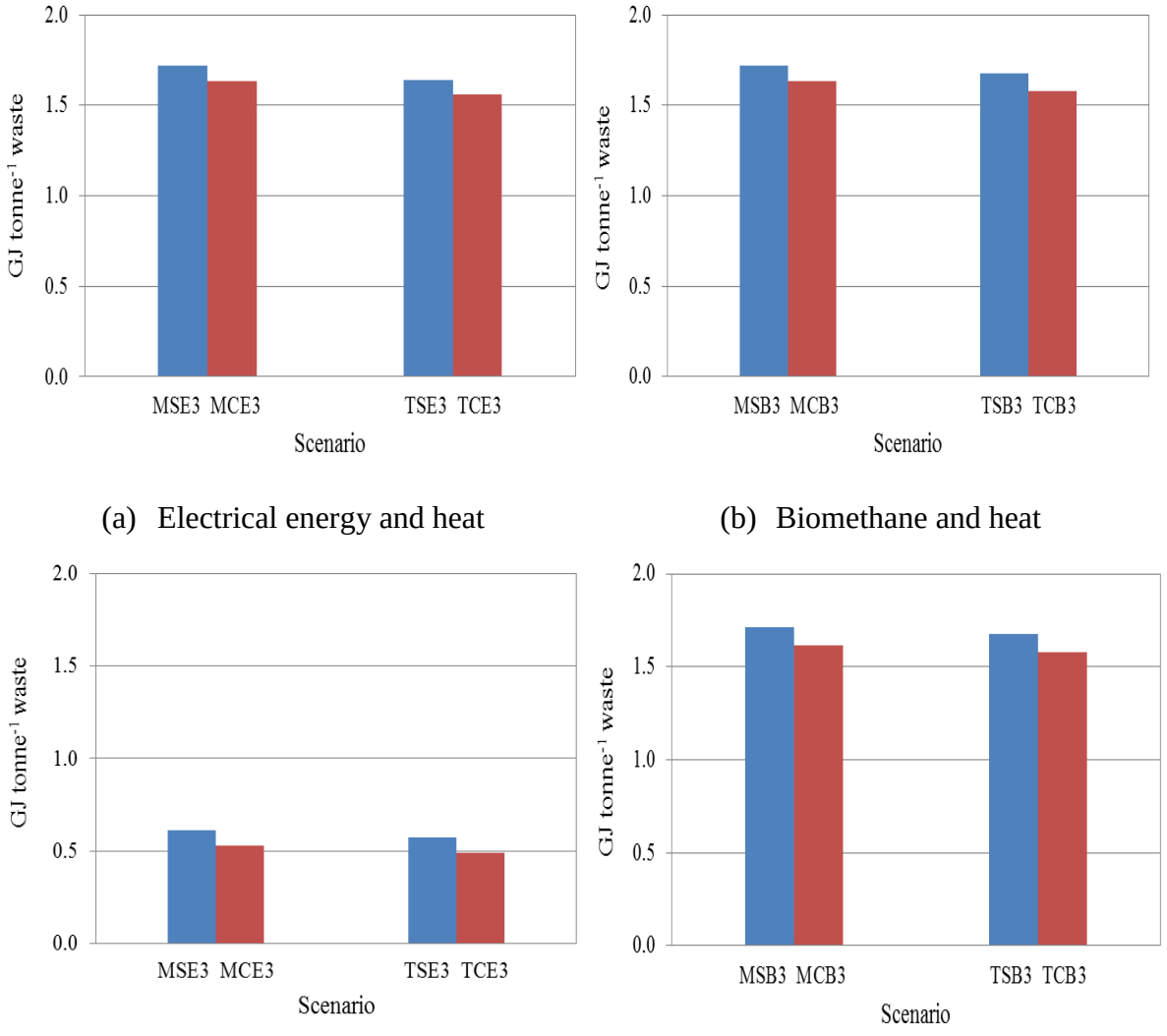
Table 6.8. Energy inputs and outputs for biomethane production

Details	Units	MSB3	MCB3	TSB3	TCB3
digester input	tonnes	100000	100000	100000	100000
digester loading rate	kg m^{-3} day^{-1}	3	3	3	3
total digester capacity required	m^3	4155	4155	4155	4155
retention time	days	69	69	69	69
methane produced	m^3	7651600	7651600	7651600	7651600
methane available	m^3	7575084	7575084	7575084	7575084
biogas	m^3	13912000	13912000	13912000	13912000
=	tonnes	17788	17788	17788	17788
digestate	tonnes	82212	82212	82212	82212
Energy balance (year^{-1})					
pre-processing electricity	GJ	0	0	0	0
digester electricity requirement	GJ	3600	3600	3600	3600
electricity for upgrading	GJ	14311	13734	14311	13734
electricity for composting	GJ	0	883	0	883
heat for digester	GJ	13017	13017	23695	23695
heat for pasteuriser	GJ	12115	12115	5209	5209
diesel for composting	GJ	0	6574	0	6574
Total	GJ	43043.06	50362	46814.46	54133
Embodied energy					
digester embodied	GJ	1200	1200	1200	1200
pasteuriser embodied	GJ	7	7	7	7
CHP embodied	GJ	15	16	15	16
upgrading embodied	GJ	118	114	118	114
gas holder embodied	GJ	14	14	14	14
ABPR building embodied	GJ	18	18	18	18
digestate storage	GJ	125	125	125	125
separator embodied	GJ	0	10	0	10
feedtank embodied	GJ	8	8	8	8
Total	GJ	1505	1514	1505	1514
CHP electrical capacity	kW	599	624	599	624
energy in methane produced	GJ	274080	274080	274080	274080
generated electricty	GJ	17911	18656	17911	18656
generated heat	GJ	25587	26651	25587	26651
imported heat	GJ	0	0	3317	2253
exported heat	GJ	455	1519	0	0
	MWh	126	422	0	0
upgraded biogas	m^3	6023516	5965293	6023516	5965293
energy in upgraded CH4	GJ	215762	213677	215762	213677

6.4.5. Comparison of energy balance for electricity and biomethane production

Figure 6.2 shows the energy balance for electricity and biomethane production from AD of SBP. The total exportable energy resulting from the scenarios with electricity and heat production was similar to that for biomethane production, in the range of ~1.6 - 1.7

GJ tonnes^{-1} waste (mesophilic conditions) and ~1.5-1.6 GJ tonnes^{-1} waste (thermophilic conditions) (Figure 6.2a and b). Exportable energy in terms of electricity and biomethane only, not taking into account the potential energy from exported heat (Figure 6.2c and d) gave a much lower value for the electricity option value ~0.5 ~0.6 GJ tonnes^{-1} waste. As expected, the complex systems gave lower net energy output compared to simple systems due to the higher energy requirement for digesting and treating digestate.



(a) Electrical energy and heat

(b) Biomethane and heat

(c) Electrical energy only

(d) Biomethane only

Figure 6.2. Comparison of energy balances for electricity and biomethane production (AD + CHP)

6.4.6. GHG emissions

Tables 6.9 and 6.10 show the emission balances for all scenarios. The results showed that the potential emission savings generated from AD of SBP were derived from two major sources: replacement of electricity and heat generated from fossil fuels. The AD + CHP scenario has ~9-10% higher potential emission savings compared to those of from AD + biogas upgrading. Complex systems have lower potential emission savings compared to the equivalent simple system, due to the higher energy requirement for treating the digestate. Increasing temperature has no effect on the potential emissions saving from N fertilizer, but reduced the potential emissions saving from the heat exported. Biogas losses during operation are another source of emissions in AD plant, which need to be minimised as these account for ~8-10% of the emission savings.

Table 6.9. Emission inputs and output for electricity and heat production (AD + CHP)

tonne CO_{2eq}	MSE3	MCE3	TSE3	TCE3
diesel for composting	0.00	491.56	0.00	491.56
embodied carbon ($year^{-1}$)				
digester embodied	87.50	87.50	87.50	87.50
pasteuriser embodied	0.51	0.51	0.51	0.51
CHP embodied	2.39	2.39	2.39	2.39
upgrading embodied	0.00	0.00	0.00	0.00
gas holder embodied	1.21	1.21	1.21	1.21
ABPR building embodied	2.05	2.05	2.05	2.05
digestate storage	12.34	12.34	12.34	12.34
separator embodied	0.00	0.42	0.00	0.42
feedtank embodied	0.95	0.95	0.95	0.95
Total	106.95	107.37	106.95	107.37
process loss	1494.6	1494.6	1494.6	1494.6
electricity generation replaced	11471.9	11361.0	11471.9	11361.0
export heat source replaced	6312.6	6312.6	6097.2	6097.2
potential emission savings from N	2328.3	2328.3	2328.3	2328.3
total emissions	1601.5	2093.5	1601.5	2093.5
emission saving (total)	18511.3	17908.4	18295.9	17693.0
emissions balance (electricity)	9870.3	9267.5	9870.3	9267.5
emissions balance (electricity + heat)	16182.9	15580.1	15967.5	15364.7

Note: emission from CHP is not included since biogas is produced from waste rather than fossil fuels

Table 6.10. Emission inputs and output for biomethane and heat production (AD + biogas upgrading)

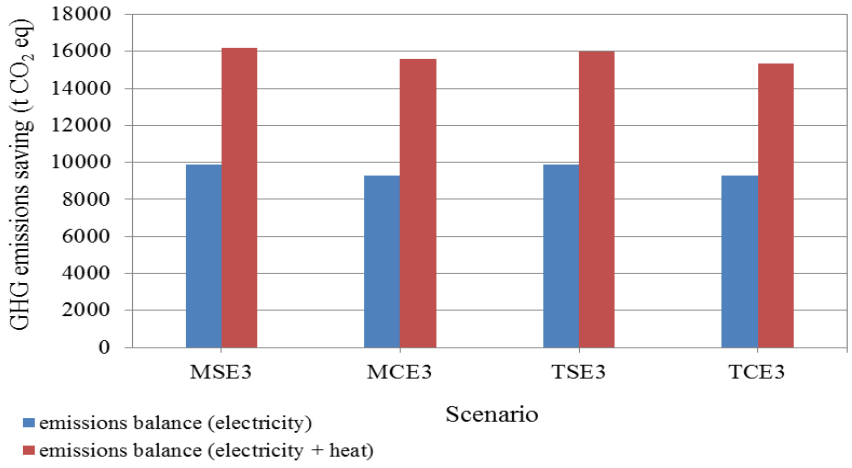
tonne CO_{2eq}	MSB3	MCB3	TSB3	TCB3
diesel for composting	0.00	491.56	0.00	491.56
embodied carbon ($year^{-1}$)				
digester embodied	87.50	87.50	87.50	87.50
pasteuriser embodied	0.51	0.51	0.51	0.51
CHP embodied	0.82	0.88	0.82	0.88
upgrading embodied	11.74	11.34	11.74	11.34
gas holder embodied	1.21	1.21	1.21	1.21
ABPR building embodied	2.05	2.05	2.05	2.05
digestate storage	12.34	12.34	12.34	12.34
separator embodied	0.00	0.42	0.00	0.42
feedtank embodied	0.95	0.95	0.95	0.95
Total	117.12	117.19	117.12	117.19
process loss	1494.6	1494.6	1494.6	1494.6
imported heat	0.0	0.0	189.4	0.0
export heat source replaced	26.0	279.5	0.0	64.1
potential emission savings from N	2328.3	2328.3	2328.3	2328.3
energy source replaced (biomethane)	16133.5	15977.6	16133.5	15977.6
total emissions	1611.7	2103.3	1801.1	2232.2
emission saving (total)	16876.2	16289.1	16660.8	16073.7
emissions balance (biomethane)	14521.9	13874.0	14332.4	13745.4
emissions balance (biomethane + heat)	14547.8	13960.8	14332.4	13745.4

Note: emission from CHP is not included since biogas is produced from waste rather than fossil fuels

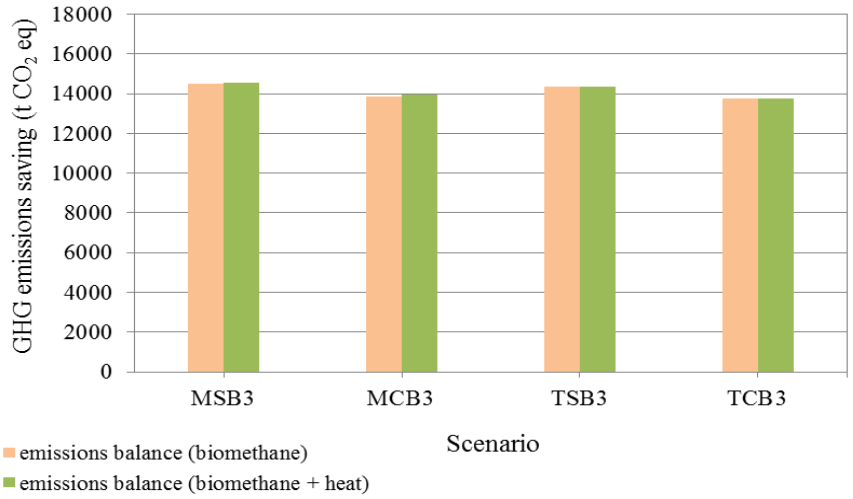
Tables 6.11 and Figure 6.3 show the relative emissions saving from all scenarios. It can be seen that there was no difference in the potential emissions on a per tonne waste basis from AD + CHP and AD + biogas upgrading both in mesophilic and thermophilic conditions. The major difference is between simple and complex system scenarios, due to the higher energy requirement in the complex system caused a reduction in biogas available for upgrading to biomethane. In general the model showed that if all heat exported can be used to replace fossil fuels, scenarios with AD + CHP provided better results as indicated by higher total emission savings and energy balance (electricity + heat) compared to AD + biogas upgrading, by up to 2000 tonne CO_{2eq} $tonne^{-1}$.

Table 6.11. Emission balances for electricity and biomethane production

tonne CO_{2eq} tonne^{-1} waste	MSE3	MCE3	TSE3	TCE3
AD + CHP				
total emissions	0.016	0.021	0.016	0.021
emission saving (total)	0.185	0.179	0.183	0.177
emission balance (electricity)	0.099	0.093	0.099	0.093
emission balance (electricity + heat)	0.162	0.156	0.160	0.154
AD + Biogas upgrading	MSB3	MCB3	TSB3	TCB3
total emissions	0.016	0.021	0.018	0.022
emission saving (total)	0.169	0.163	0.167	0.161
emissions balance (biomethane)	0.145	0.139	0.143	0.137
emissions balance (biomethane + heat)	0.145	0.140	0.143	0.137



(a)



(b)

Figure 6.3. Potential emission savings from electricity (a) and biomethane (b) production with and without use of heat

6.4.7. Effect of OLR and temperature on energy balance and GHG emissions

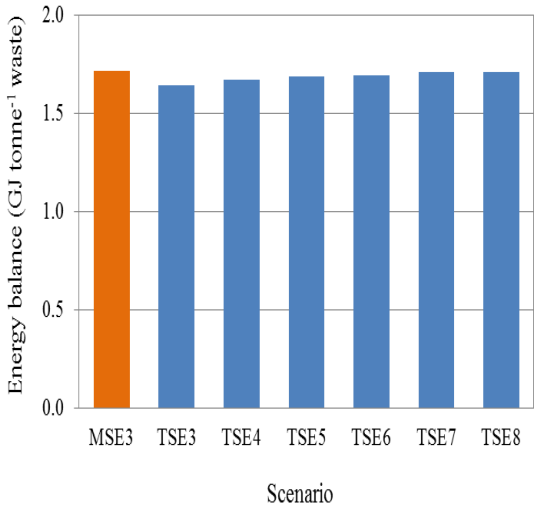
6.4.7.1. Energy balances

The model was used to assess the effect of changing OLR and digester temperature from mesophilic to thermophilic to determine its potential application. For modelling purposes it was assumed than the specific methane production for thermophilic digestion at OLR 6-8 kg VS m^{-3} day^{-1} was similar to that at OLR 5 kg VS m^{-3} day^{-1}.

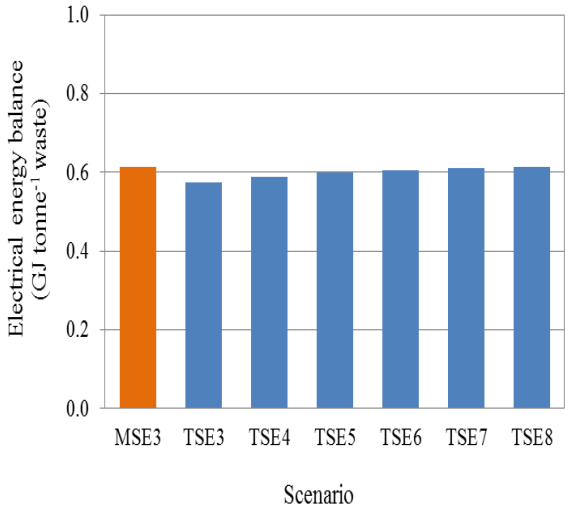
The total energy balance was higher in the mesophilic than in the thermophilic system for both electricity and methane production in all cases considered. For example at 1.72 GJ $tonne^{-1}$ waste compared to 1.64 GJ $tonne^{-1}$ waste for the AD combined with CHP in simple configuration (Table 6.5). This is a result of an increase in heat requirement from the feedstock and digester, as the amount of SBP is the same. Increasing the OLR reduced the size of digesters required, thus decreasing the parasitic and embodied energy demand per tonne on waste input. The effect of increasing the OLR from 3 to 8 kg VS m^{-3} day^{-1} on the energy balance per tonne of waste in thermophilic condition is shown in Figure 6.4 and 6.5.

For AD + CHP in both simple and complex configuration, it can be seen that increasing OLR increased the total and electrical energy balance per tonne of waste until these were equal or more than that for the mesophilic system (Figure 6.4). However, there was no effect on the heat required to raise the temperature of the feedstock as it remained the same depending on the ambient temperature and the amount of waste.

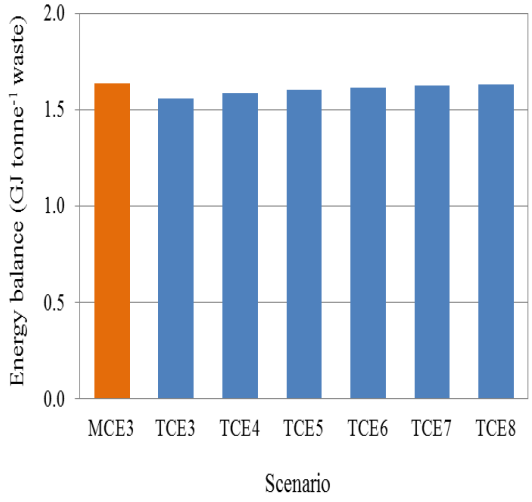
The same trends were also identified for AD + biogas upgrading, as OLR increases the overall and biomethane energy balance per tonne of waste approaches that of the mesophilic system in both simple and complex configuration (Figure 6.5). The difference in the energy balance between mesophilic and thermophilic was due to the changes in the imported heat.



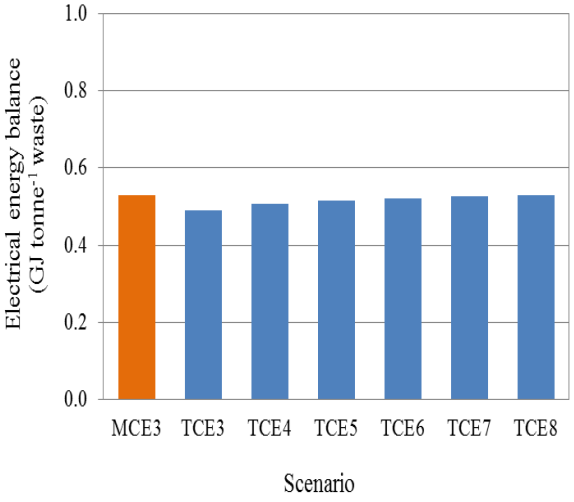
(a) overall energy balance-simple system



(b) electrical energy balance-simple system

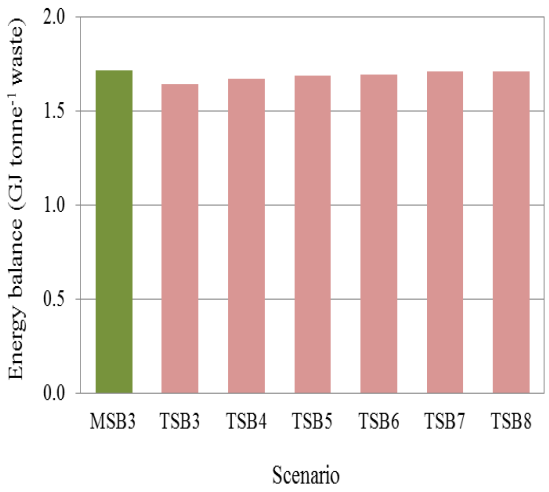


(c) overall energy balance-complex system

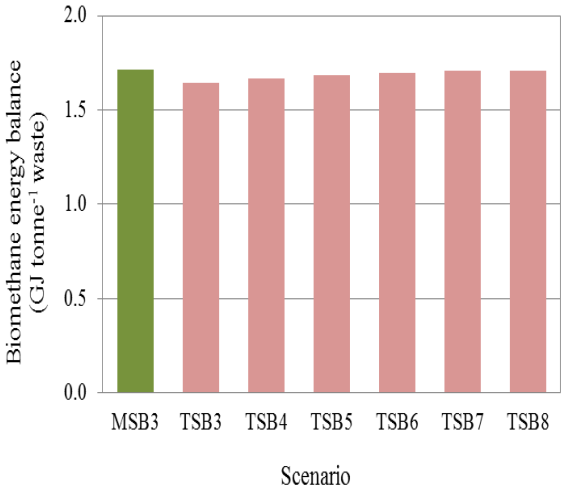


(d) electrical energy balance-complex system

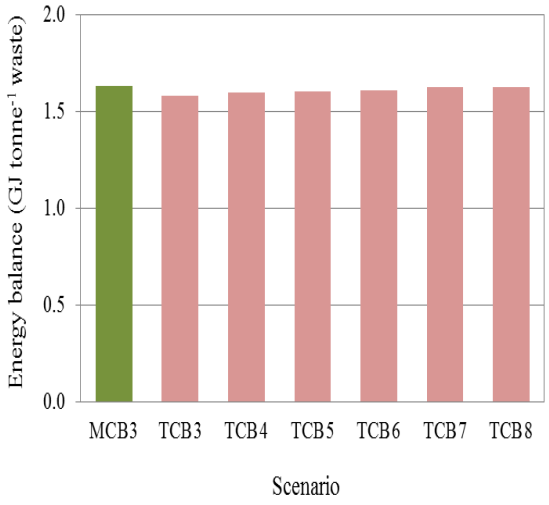
Figure 6.4. Effect of OLR on overall and electrical energy balances in simple and complex system of AD combined with CHP unit



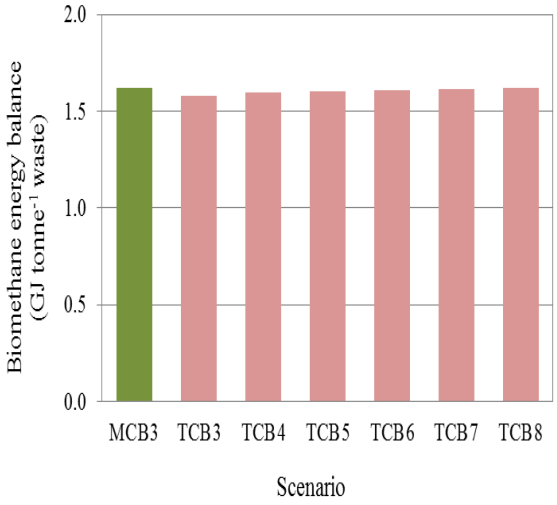
(a) overall energy balance-simple system



(b) biomethane energy balance-simple system



(c) overall energy balance-complex system



(d) biomethane energy balance-complex system

Figure 6.5. Effect of OLR on overall and electrical energy balances in simple and complex system of AD combined with CHP unit

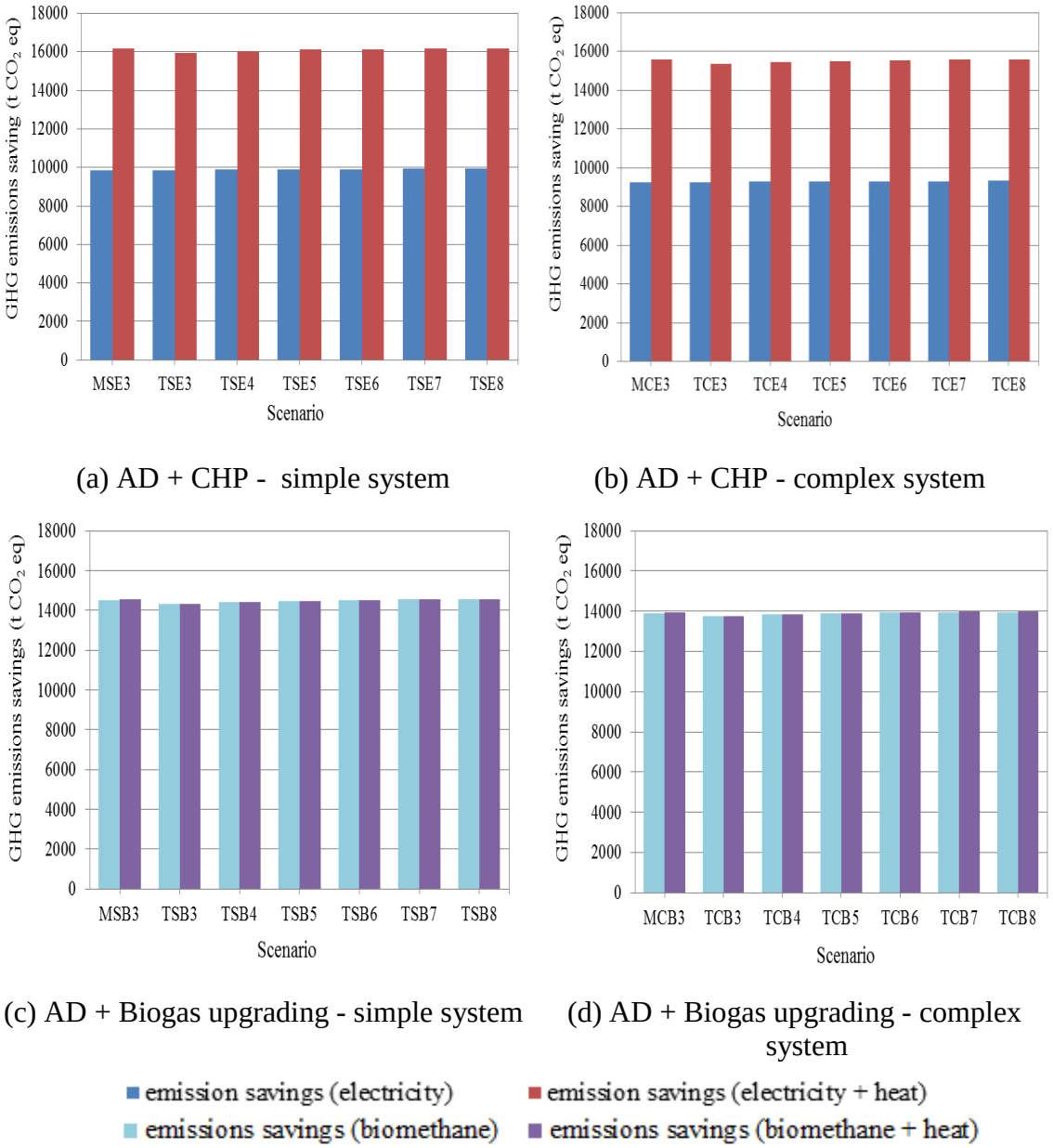
From above findings, it was clear that the increase in OLR, in both AD + CHP and AD + biogas upgrade, was the main cause of a decrease in the energy required (imported electricity and heat) used in operating the digesters due to a decrease in the size and number of digester units and retention time. By increasing the OLR up to 8 kg VS m^{-3} day^{-1} it is possible for the thermophilic process to achieve similar energy balance per tonne of waste to the mesophilic.

6.4.7.2. GHG emissions

In terms of potential emissions, the increase of OLR had little effect, as the amount of energy and fertiliser produced is related to feedstock volume (Table 6.12). For AD + CHP, increasing OLR lead to a slight increase in the potential emission savings from either electrical and heat or electrical only, which mainly derived from the potential heat exported (Figure 6.6a and b). An increase in the size of CHP unit resulted in greater potential of exported heat for replacing the use of fossil fuels, thus reducing the amount of potential emissions from the AD operation. For AD + Biogas upgrading, the increase in the potential emission savings due to an increase in OLR resulted from a reduction in imported heat (Figure 6.6c and d).

Table 6.12. Summary of emission balances from electricity and biomethane production scenario

tonnes CO_{2eq}							
AD + CHP							
Simple system	MSE3	TSE3	TSE4	TSE5	TSE6	TSE7	TSE8
emission savings (electricity)	9870	9870	9891	9905	9911	9921	9924
emission savings (electricity + heat)	16183	15968	16055	16118	16144	16186	16200
Complex system	MCE3	TCE3	TCE4	TCE5	TCE6	TCE7	TCE8
emission savings (electricity)	9267	9267	9288	9302	9308	9318	9321
emission savings (electricity + heat)	15580	15365	15453	15515	15541	15583	15597
AD + Biogas upgrading							
Simple system	MSB3	TSB3	TSB4	TSB5	TSB6	TSB7	TSB8
emission savings (biomethane)	14522	14332	14420	14483	14509	14551	14564
emission savings (biomethane + heat)	14548	14332	14420	14483	14509	14551	14564
Complex system	MCB3	TCB3	TCB4	TCB5	TCB6	TCB7	TCB8
emission savings (biomethane)	13874	13745	13833	13896	13915	13925	13928
emission savings (biomethane + heat)	13961	13745	13833	13896	13922	13964	13977



(a) AD + CHP - simple system

(b) AD + CHP - complex system

(c) AD + Biogas upgrading - simple system

(d) AD + Biogas upgrading - complex system

Figure 6.6. Effect of varying OLR on emissions balances from scenario AD + CHP and AD + Biogas upgrading

Conclusions from scenario modelling

The model showed that all the scenarios have strong positive energy balances. The AD of SBP in thermophilic condition and complex configuration had a slightly lower net energy yield in all cases. If the potential heat exported can be fully used, the AD + CHP scenario is preferable as the electricity production shows a marginally higher net energy output. However, if the heat cannot be utilised effectively, the AD + biogas upgrading option is more effective in terms of maximising the utilisation of the available energy. The potential emission savings are better for biomethane than CHP electricity

production alone, but lower if the heat generated from CHP unit can be exported as a replacement for fossil fuels. Increasing the OLR reduced the parasitic and embodied energy demand, due to the shorter HRT and the smaller number of digesters required, thus increasing the potential energy available.

6.5. Overall energy and GHG emissions balances from waste to field

In this study, the AD model was also used to estimate energy and emissions for digestate utilisation, to establish overall energy and emission balances for the complete system selected (see Figure 6.1).

6.5.1. Energy and emissions in digestate transport and application

Table 6.13 shows the energy and emission factors used for digestate and mineral fertiliser application. For this study, the biogas plant was assumed to be built inside the beet sugar factory, thus the distance in transportation consisted of 0.2 km for loading SBP from the waste area to the biogas plant and 50 km for digestate transport to the sugar beet agricultural land. This distance was selected based on the fact that British Sugar plc in Wissington used three million tonnes of sugar beet produced by 1,500 UK growers, at an average distance of 50 km from the factory (British Sugar, 2012). The transportation was assumed to be HGV (heavy goods vehicle) artic >33 tonnes and artic < 33 tonnes as these have a high capacity and low fuel needs.

Table 6.13. Energy and emission in digestate transport and application

	diesel use	emissions	embodied energy
mineral fertiliser application	2.9 l ha^{-1} [(a)]	7.78 kg ha^{-1} [(b)]	8.5 MJ ha^{-1} [(c)]
digestate transport	2.07 MJ $tonne^{-1}$ km^{-1} [(d)]	0.155 kg $tonne^{-1}$ km^{-1} [(b)]	36.27 GJ $year^{-1}$ [(d)]
whole digestate/liquor application	3.8 l ha^{-1} [(a)]	10.2 kg ha^{-1} [(b)]	42.8 MJ ha^{-1} [(c)]
fabric fraction application	9.5 l ha^{-1} [(a)]	25.5 kg ha^{-1} [(b)]	47 MJ ha^{-1} [(c)]

Source: [(a)] VALORGAS (2013a); [(b)] 0.075 kg CO_{2eq} MJ^{-1} diesel (AEA, 2010); [(c)] Salter, et al. (2011); [(d)] VALORGAS (2013b)

The maximum application rate for N is 170 kg N ha^{-1} based on limit for Nitrate Vulnerable Zones (NVZ) in the EU Nitrates directive (91/676/EEC) (European Commission, online), and this was used to determine the area required for digestate application. For this modelling purpose the nutrients (N, P, K) of SBP obtained from the

experimental work at values of 3.48 gN kg^{-1} WW, 0.41 g P kg^{-1} WW and 0.84 g K kg^{-1} WW were used, with the nutrient values of digestate chosen were from mesophilic digesters fed at OLR 4 g VS l^{-1} day^{-1}: 4.04 g N kg^{-1} WW, 0.61 g P kg^{-1} WW, and 0.72 g K kg^{-1} WW (see Table 5.23 in Chapter 5).

The digestate application was assumed to replace fossil fuel based mineral nitrogen fertiliser requiring 42.9 MJ kg^{-1} to produce and deliver to site with an emissions value of 6.81 kg CO_{2eq} kg^{-1} N (Mortimer et al., 2010). In this study, the energy balances and emission savings were based on N fertiliser substitution only as this was used as the limiting factor for land application and was the most significant component in replacing the fossil fuels.

6.5.2. Overall energy and emissions balances

The system employed in this study is shown in Table 6.14. The energy and emission balance in this section is combined with the results of scenarios fed at OLR 3 kg VS m^{-3} day^{-1} with an input of 100000 tonnes of wastes from section 6.4.

Table 6.14. Whole system scenarios

	MSE/MSB	MCE/MCB	TSE/TSB	TCE/TCB
collection	yes			
pre-treatment	no	no	no	no
digestion	mesophilic	mesophilic	thermophilic	thermophilic
digestate treatment	simple (no-separation)	complex (separation, composting)	simple (no-separation)	complex (separation, composting)
digestate application	single (whole digestate)	separate fibre and liquor application	single (whole digestate)	separate fibre and liquor application

Note: M= mesophilic, T= thermophilic, S= simple, C= complex, E= electricity production, and B= biomethane production

In all cases, the total digestate produced was 82212 tonnes which is enough to meet the nitrogen requirement for 1954 ha of crop. In the AD system with simple configuration, digestate is applied as whole digestate thus the energy required derived from transportation and application. In the AD system with complex configuration, the digestate is applied as separated fibre and liquid, with the fibre fraction further treated by composting, thus reducing the mass of digestate. For modelling purposes it was assumed that composting reduced the moisture of the separated fibre by 50% (Salter,

2013). Both fractions are applied separately to land. The energy for transport and application in this scenario is shown in Table 6.15. It was assumed that the digestate replaced 332136 kg of fossil fuel based on nitrogen which required 14249 GJ to produce and deliver with emission value of 2261.8 tonnes CO_{2eq} kg^{-1}.

Table 6.15. Energy and emission from digestate transport and application

	amount (tonnes)	transport (GJ)	transport (t CO_{2eq})	application (GJ)	application (t CO_{2eq})	embodied energy (GJ)
simple	82212	8509.0	637.14	265.3	19.93	119.9
complex-fibre	11921	1233.8	92.39	663.2	49.8	128.1
complex-liquor	58371	6041.4	452.38	265.3	19.93	119.9

6.5.2.1. Energy balances

Table 6.16 shows the results for energy inputs and outputs from all cases, while Figure 6.7 shows the energy balances resulting from energy production from electricity/biomethane only, electricity/biomethane and heat, electricity/biomethane and fertiliser replacement, and electricity/biomethane, heat and fertiliser. The energy required for transportation in collection was the same in all cases. It can be seen the energy balance was positive in all cases, indicating that the energy produced from AD of SBP in both simple and complex configuration was able to fulfil the energy required for processing the plant. As previously stated in section 6.4, increasing complexity and digestion temperature reduces the overall energy balances and the AD + CHP option gives a slightly higher energy balances than the AD + biogas upgrading if all of the heat can be effectively used to replace fossil fuels.

In complex system, although the reduction in mass of the solid fraction (fibre) gave a decrease in energy required for transport and application, this was outweighed by the additional energy required for separation of the digestate into solid and liquid fractions. Separation and composting treatment may be not effective as there is still an energy requirement for disposing of the liquor fraction of digestate, therefore if it is desired to reduce the volume for transport other disposal routes must be considered for the liquid fraction.

Table 6.16. Whole system energy inputs and outputs

GJ	MSE3	MCE3	TSE3	TCE3	MSB3	MCB3	TSB3	TCB3
collection inc. embodied	24	24	24	24	24	24	24	24
digestion inc. embodied	30155	37622	33927	41393	44548	51876	51637	57900
digestate transport & application inc. embodied	8894	8452	8894	8452	8894	8452	8894	8452
exported electricity/biomethane	91369	90486	91369	90486	215762	213677	215762	213677
exported heat	110538	110538	106766	106766	455	1519	0	0
mineral N fertiliser replaced	14249	14249	14249	14249	14249	14249	14249	14249
total energy balance	177082	169175	169539	161632	177000	169093	169457	161550
electricity/biomethane	52296	44389	48525	40617	162296	153326	155208	147301
electricity/biomethane + heat	162833	154926	155291	147383	162751	154844	155208	147301
electricity/biomethane + fertiliser	66545	58637	62773	54866	176545	167574	169457	161550
electricity/biomethane + heat + fertiliser	177082	169175	169539	161632	177000	169093	169457	161550

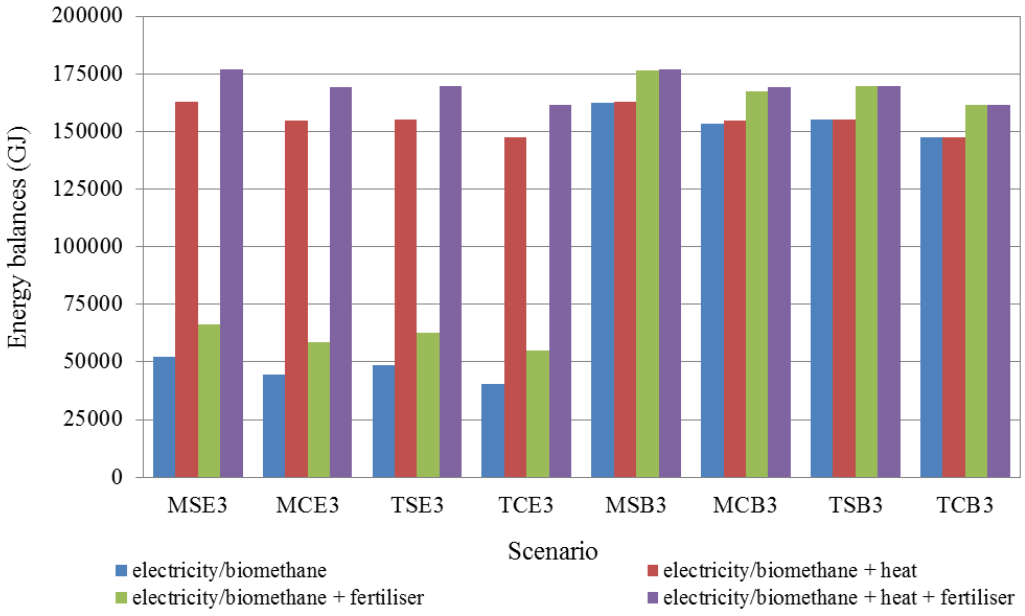


Figure 6.7. Whole system energy balances

6.5.2.2. GHG emissions

Table 6.17 shows the results from GHG emission from the whole system and Figure 6.8 shows the relative emission savings resulting from the replacement of electricity/biomethane, heat and fertiliser produced from fossil fuels. The potential GHG emissions resulting from complex system were higher than those of from simple system in all cases due to more energy requirement for the extra processing of digestate output for production of the same amount of fertiliser, lead to a decrease in potential emissions savings. Potential emissions from process losses of biogas accounted for up to 11% of the net emissions saving, thus their reduction would provide a greater improvement of emissions savings in both scenarios.

There was no difference between mesophilic and thermophilic digestion or between simple and complex configuration in both scenarios. In general, there was a little difference in potential emission savings between thermophilic and mesophilic operation in this case.

Table 6.17. Whole system energy emissions

tonnes CO_{2eq}	MSE3	MCE3	TSE3	TCE3	MSB3	MCB3	TSB3	TCB3
collection inc. embodied	1.7	1.7	1.7	1.7	1.7	1.7	1.7	1.7
digestion inc. embodied	1601.5	2093.5	1601.5	2093.5	1611.7	2103.3	1801.1	2232.2
digestate transport & application inc. embodied	657.1	614.5	657.1	614.5	657.1	614.5	657.1	614.5
process losses	1494.6	1494.6	1494.6	1494.6	1494.6	1494.6	1494.6	1494.6
replaced grid-produced electricity/diesel fuels	11471.9	11361.0	11471.9	11361.0	16133.5	15977.6	16133.5	15977.6
replaced fossil fuel based heat	6312.6	6312.6	6097.2	6097.2	26.0	86.7	0.0	0.0
replaced mineral N fertiliser	2328.3	2328.3	2328.3	2328.3	2328.3	2328.3	2328.3	2328.3
total emissions savings	16357.9	15797.6	16142.5	15582.2	14722.8	14178.3	14507.4	13962.9
electricity/biomethane	9211.5	8651.2	9211.5	8651.2	13863.0	13257.8	13673.6	13129.1
electricity/biomethane + heat	15524.1	14963.8	15308.7	14748.4	13889.0	13344.5	13673.6	13129.1
electricity/biomethane + fertiliser	11539.9	10979.6	11539.9	10979.6	16191.4	15586.1	16002.0	15457.5
electricity/biomethane + heat + fertiliser	17852.4	17292.1	17637.1	17076.8	16217.3	15672.8	16002.0	15457.5

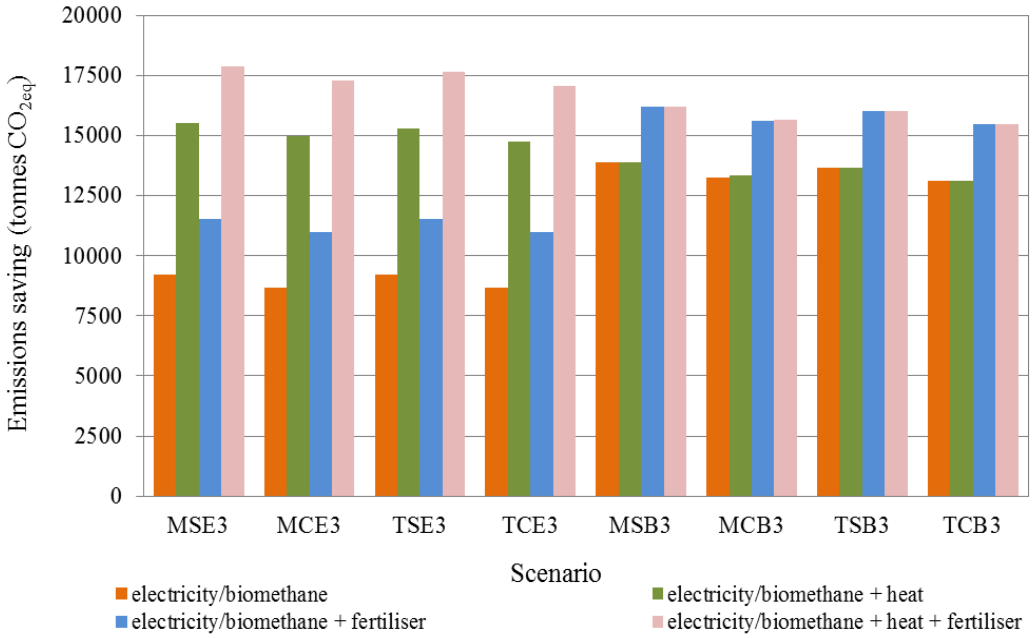


Figure 6.8. Whole system emissions savings

The potential mineral N fertilizer replacement was in the range of 14-18% of the net savings in GHG emissions (Figure 6.9). This indicates that the use of digestate as a biofertiliser has a useful role to play in terms of carbon balance or GHG emissions or environmental contribution as it makes up about 1/3 of the emissions saving in these cases

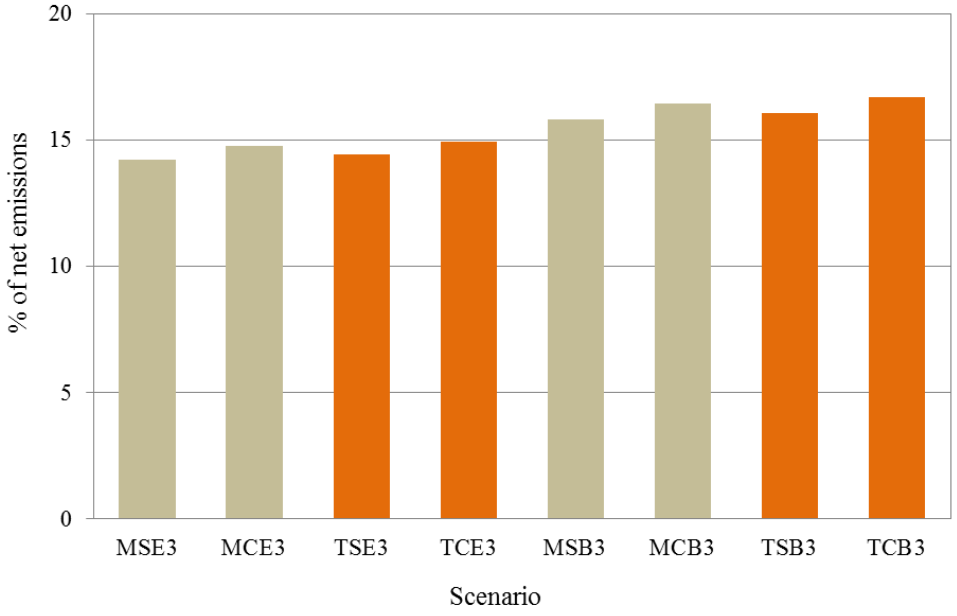


Figure 6.9. Mineral N fertilizer replacement as % of net GHG emissions savings

6.5.3. Operation without digestate utilisation

In certain situations it is possible that digestate from an AD system cannot be applied to land for reasons such as local farming practice; soil or hydrological condition; regulatory requirements; higher COD concentration, or unacceptable level of contamination (e.g. high concentrations of PTE or plastics, etc.). In this case, the digestate cannot be considered as mineral fertiliser replacement in the energy and emission balances. Therefore in such cases it is assumed that the digestate is separated into solid and liquid fraction (complex system) and the liquid fraction is treated to an acceptable standard for recycling or discharge to sewer at an assumed energy cost of 48 MJ tonne^{-1} liquor (VALORGAS, 2012). The separation process is assumed to use the same equipment as in section 6.4. The fibre fraction of the digestate, equal to 23842 tonnes, is assumed to be transported 50 km to a landfill site for disposal without composting treatment using HGV artic < 33 tonnes.

Table 6.18 shows the energy inputs and outputs for these scenarios, while the emission balances are shown in Table 6.19. In this case, the complex digestion scenarios are selected as in the simple scenarios the digestate receives no post-treatment. It can be seen that the total energy balance, while lower than that for digestate application, is still positive. Figure 6.10 shows the comparative energy and emissions balances for the whole systems with either application of fibre and liquor fraction of the digestate to the field or without application (separation and treatment and disposal of the two fractions).

Table 6.18. Input and output energy for separated digestate without application to land

GJ	MCE3	TCE3	MCB3	TCB3
collection inc. embodied	24	24	24	24
digestion inc. embodied	33172	36943	46488	50809
digestate transport and application inc. embodied	2558	2558	2558	2558
exported electricity/biomethane	84523	84523	199596	199596
exported heat	105056	101285	3221	0
mineral N fertiliser replaced	0	0	0	0
total energy balance	153825	146282	153747	146204

Table 6.19. Emission balances for separated digestate without application to land

tonnes CO_{2eq}	MCE3	TCE3	MCB3	TCB3
collection inc. embodied	1.7	1.7	1.7	1.7
digestion inc. embodied	107.3	107.3	116.8	148.2
digestate transport and application inc. embodied	182.0	182.0	182.0	182.0
process losses	7472.8	7472.8	7472.8	7472.8
replaced grid-produced electricity/diesel fuels	10612.3	10612.3	14924.7	14924.7
replaced fossil fuel based heat	5999.5	5784.2	184.0	0.0
replaced mineral N fertiliser	0.0	0.0	0.0	0.0
total emissions savings	8848.0	8632.6	7335.3	7119.9

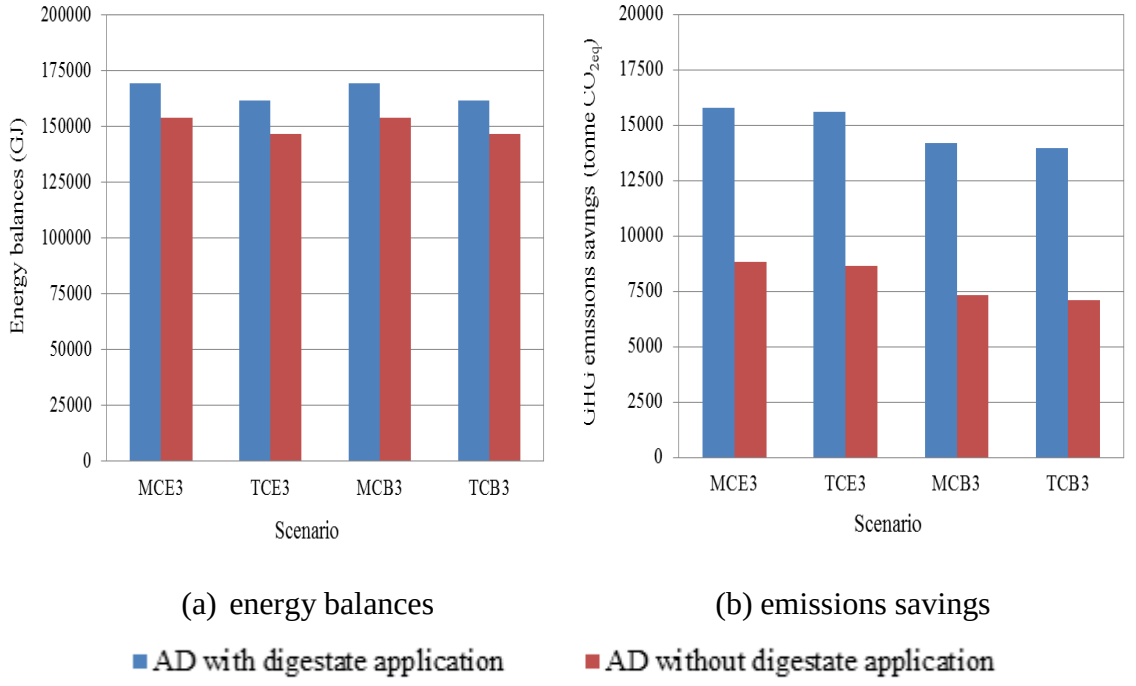


Figure 6.10. Energy and emissions balances

The results showed that treating liquor rather than using it as a nutrient source for crop production reduced the energy balance by 9-10% due to an increase in energy requirement for processing in the plant which reduces the exportable electricity fraction. The emission savings decreased by 44-49% due to the reduction in electricity available for export and for non-substitution of fossil fuel based fertiliser.

Conclusions for whole system assessment

The positive net energy productions found for all scenarios confirm that AD of SBP is an effective option, as expected from its high energy potential and status as a waste product. AD operation in thermophilic condition and complex configuration with and without digestate application reduced the energy for export and potential emissions savings. The application of digestate to land provide benefits not only increasing the exportable energy fraction and potential emission savings, but also providing biofertiliser to replace the use of mineral fertilizer. In general, the AD scenarios with digestate application gave a better performance in terms of the potential exported energy and emissions savings.

6.6. Conclusions

The AD model in this study was used to evaluate several scenarios for the same amount of SBP feedstock to determine the most efficient system in terms of energy or GHG emissions. All cases from the selected scenarios gave a positive energy and GHG emissions balances. Both mesophilic and thermophilic operations have a strongly positive energy and emissions balance and the differences between them are relatively small. Thermophilic operation has a higher energy demand and consequently less energy available for export in comparison with mesophilic operation, but increasing the OLR increases the energy and emissions savings, so at higher OLR the performance of thermophilic systems in terms of energy production per tonne and GHG emissions is nearly as good as mesophilic. The model only considers energy and GHG balances, but in practice decisions are made based on many other factors: for example in this case thermophilic operation may be chosen because it is easier to operate due to absence of foaming, requires a smaller area etc. This work confirmed that the thermophilic AD of SBP can be a feasible option to valorize SBP as renewable energy. As supported by the findings from the experimental results that operating thermophilic digestion of SBP was possible without any sign of disturbance in digestion process. The option of AD combined with CHP is preferable if the potential exported heat can be effectively utilized as replacement of fossil fuels. Combining an AD system with digestate application appears likely to be favourable in term of environmental aspects, as the utilization of digestate to land as replacement of mineral fertilizer reduced the potential emissions. This option is better suited for AD of SBP as the experimental results showed that digestate from this operation complies with the regulation for digestate

application (PAS110). In general, the modelling work further confirmed that the use of SBP as feedstock for biogas production is feasible and effective in terms of energy production and environmental benefits. The modelling output also provides the basic data (e.g. digester size, transport and energy costs, etc.) on which an economic analysis could be carried out in future work.

CHAPTER 7. CONCLUSIONS AND RECOMMENDATIONS

The following main conclusions can be drawn from the results obtained:

7.1. Characterisation and Anaerobic Digestion Trials

- SBP has a high organic content consisting of approximately 50% fibre, comprised mainly of hemicelluloses (29.0%) with cellulose (13.3%) and lignin (8.2%) also present.
- BMP test results suggested that SBP had a specific methane yield of 0.321 1 CH_4 g^{-1} VS, indicating a significant potential for use as feedstock into an AD system to produce renewable energy.
- In BMP tests started sequentially over a period of 7 days after obtaining the fresh inoculum, the age of the inoculum appeared to have no significant effect on specific biogas or methane yields, or on the final values of physico-chemical parameters such as pH and VFA.
- Semi-continuous mesophilic digestion at OLR of 2 to 5 g VS l^{-1} day^{-1} resulted in VS degradation of ~84-91% VS destruction with methane yields of 0.293 - 0.316 m^3 CH_4 kg^{-1} VS, and volumetric methane production of ~0.64-1.44 m^3 CH_4 m^{-3} digester day^{-1}.
- Increasing the OLR in mesophilic digestion led to a deterioration in digestion process stability parameters, indicated by an increase in IA/PA ratio from 0.28 to 0.57 and a reduction in: pH from 7.56 to 7.12, total alkalinity from ~19 to ~14 g $CaCO_3$ kg^{-1} WW, VS destruction from ~91% to ~84%, biogas and methane production from 0.621 to 0.579 m^3 biogas kg^{-1} VS and from 0.316 to 0.294 m^3 CH_4 kg^{-1} VS. Although total VFA increased from 82 to 376 mg l^{-1}, these values were acceptable for digester operation. Foaming was also observed in digesters fed at OLR 4 and 5 g VS l^{-1} day^{-1}.
- At OLR 3 g VS l^{-1} day^{-1}, TE supplementation of mesophilic digestion appeared to offer advantages in terms of a lower IA/PA ratio and an increase in specific methane production from 0.293 to 0.313 m^3 CH_4 kg^{-1} VS, and no occurrence of foaming.
- In mesophilic conditions, dilution of feedstock with water on a 1:1 (w/w) basis did not prevent foaming, and had an adverse effect on performance including a reduction in specific methane yield from 0.286 to 0.234 m^3 CH_4 kg^{-1} VS and an

increase in IA/PA ratio from 0.42 to ~1.07, in comparison with operation at the same OLR without dilution.

- Thermophilic digestion of SBP showed performance advantages over mesophilic, including higher specific methane production (0.345-0.355 m^3 CH_4 kg^{-1} VS), better solids destruction (~88%) and enhanced buffering capacity with lower IA/PA ratios (~0.32). Mesophilic digestion showed clear signs of instability at an OLR of 4 g VS l^{-1} day^{-1} while the thermophilic process could operate stably at OLR 5 g VS l^{-1} day^{-1}, with no foaming.
- Residual biogas and methane production from digesters fed on SBP at an OLR of 5 were 37.31 l biogas and 23.55 l CH_4 over a period of 222 days with specific residual values of 0.015 l biogas g^{-1} VS and 0.010 l CH_4 g^{-1} VS.

7.2. Dewaterability Trials

- SBP digestate from digesters at all OLR tested had poor dewaterability characteristics, as shown by high CST values, poor filtration capacity and little or no liquid separation in FIC tests.
- Dewaterability of thermophilic digestate was superior to that of mesophilic digestate. After 3 HRT at OLR 4 and 5 g VS l^{-1} day^{-1}, the CST for mesophilic digestate was >84000 s with 0% supernatant liquid separation in FIC test, while for thermophilic digestate the CST was between ~5000-6000 s with an average of 80% supernatant separation.
- SEM images showed that mesophilic digestate had a more open porous matrix, whereas thermophilic digestates had a finer and denser appearance without pores. This difference in structure may be responsible for the difference in dewatering characteristics, with water potentially being held within the sponge-like mesophilic matrix.
- In mesophilic conditions, increasing OLR resulted in a reduction in digestate dewaterability, with a fall in supernatant separation from 40% to 0%,
- TE supplementation of a mesophilic digester at OLR 3 g VS l^{-1} day^{-1} gave a slight improvement in digestate dewaterability, at indicated by an increase in supernatant separation to 40% in the FIC test compared to 30% for the same OLR without TE addition.

- Dilution in the range 180:20 – 80:120 of digestate:water generally had a positive impact on dewaterability: CST reduced from > 84000 s to ~14000-21000 s for mesophilic digestate and from ~5000-6000 s to ~1500 s for thermophilic, with an increase in liquid-solid separation from 0% to ~1.54-30% (mesophilic digestate) and from ~75-79% to ~79-87% (thermophilic digestate).
- Dilution of feedstock with water on a 1:1 (w/w) did not improve dewaterability in terms of CST or FIC results.
- An ageing study on mesophilic digestates fed at OLR 3 g VS l^{-1} day^{-1} and maintained at 4 ^{o}C, room temperature and 35 ^{o}C showed only limited improvement in dewaterability after storage for up to 9 months: as CST values were still well above the target value of < 10 s, this option is unlikely to be favourable.
- F/T treatment had no significant effect on the dewaterability of fresh mesophilic and thermophilic SBP digestate; however, SEM images revealed a slight change in the floc structure of mesophilic digestate. No equivalent change was observed in thermophilic digestate.
- One-stage chemical treatment using 7 common chemical coagulants (e.g. Alum, $FeCl_3$, lime, Polymer solution) improved SBP digestate dewaterability, as indicated by a reduction in CST from > 84000 s to ~11 - 21000 s in mesophilic digestate and from ~5000 - 6000 s to ~31 - 2300 s for thermophilic; and an increase in solid/liquid separation from ~2.5 - 43.8% to ~7.1 - 69.2% for mesophilic digestate.
- The use of two-stage chemical treatment using aluminium sulphate and polymer solution (1% w/w, Polymer FM 305) further reduced CST values to less than 20 s, allowed higher cake formation in filtration tests to ~71-80%, and reduced the time for separation in FIC tests from > 10 minutes to < 50 s.
- Cellulolytic enzymatic treatment was ineffective in reducing the CST

7.3. Foaming Trials

- In mesophilic digestion, digesters fed at OLR 4 and 5 g VS l^{-1} day^{-1} showed operational problems due to foaming. In the case of one digester fed at OLR 5 g VS l^{-1} day^{-1} this led to explosive loss of digestate, and subsequent digestion failure. The cause of this failure is unknown, but may be related to processes taking place during foaming and/or pressurisation.
- All antifoams tested were able to reduce foam by around 70%.

- In batch tests some types of antifoams, such as water-based fatty alcohol and silicone emulsion, showed a slight enhancement of biogas production up to 0.04 ml biogas g^{-1} digestate WW at low doses (< 0.2 ml l^{-1}) and inhibition of biogas production at higher doses (> 0.5 ml l^{-1}).
- Mineral oil antifoam was tested in a semi-continuous digestion trial, and repeated dosing at more than 1 ml l^{-1} at fortnightly intervals was found necessary to control foaming: this dose is considerably higher than values in typical industrial use. The repeated addition of mineral oil antifoam caused failure of the digestion process, as indicated by low biogas and methane production, a rise in VFA to over 12 g l^{-1} and IA/PA ratio to 5.56, and a fall in pH to 6.2.
- No foaming was observed during thermophilic digestion trials at OLR 4 and 5 g VS l^{-1} day^{-1}.

7.4. Digestate structure and components

- Effective separation of SBP digestate was not possible at speed of less than 438 g
- At 2451 *g* and above digestate could be separated into four fractions: a solid residue, biomass layer, non-cellular light fraction, and supernatant liquid. The non-cellular light fraction was identified as EPS.
- In mesophilic conditions the amount of EPS increased with an increase in OLR from 4 to 5 g VS l^{-1} day^{-1}. Only a small amount of EPS was observed in thermophilic conditions.
- Thermal post-treatment of mesophilic digestates at 55 ^{o}C to 65 ^{o}C had no effect on reducing and/or eliminating EPS or improving the dewaterability characteristics. This indicates that the EPS is not heat labile and is in fact not produced in thermophilic conditions, rather than being produced and then broken down.
- Removing EPS and replacing it with water improved the CST time in fresh mesophilic digestates from > 84000 s to ~3000-~4000 s and in fresh thermophilic digestates from ~5500 to ~1000-~1200 s.
- Removal of EPS combined with freezing and thawing of the sample gave a further improvement in CST, in the range of~2000 s to ~3000s for mesophilic and ~900 to ~1100 for thermophilic digestates, suggesting that freezing slightly disrupts any residual EPS.

- Digestates from mesophilic and thermophilic AD of SBP contained nutrients N, P and K, of 0.48, 0.41 and 0.84 g kg^{-1} WW respectively, while the concentration of Ni (a PTE) was ~66 - 210 times lower than the regulatory limit, confirming the potential for application of digestate to agricultural land as mineral fertiliser replacement.

7.5. CEN Footprint Study

- In thermophilic digestion 76% and 78% of the calorific value of SBP was recovered as methane at OLR 4 and 5 respectively, while in mesophilic conditions the respective recoveries were 64% and 62%.
- The results of a CEN footprint study with system boundaries including anaerobic digestion, biogas utilisation, digestate treatment, and digestate application demonstrated that AD is an effective technology for valorising SBP to provide renewable energy as it limits the GHG emissions and offers alternative organic nutrients for enriching the soil as a substitute for inorganic fertiliser.
- Modelling identified that both mesophilic and thermophilic operations have a strongly positive energy and emissions balance, with relatively small differences between them.
- Mesophilic AD has a lower energy demand and higher energy available for export compared to thermophilic digestion. In AD + CHP scenario, mesophilic operation required energy of 30155 - 37622 GJ $year^{-1}$ to produce available energy (heat and electricity) of 201024 - 201907GJ $year^{-1}$. Operating in thermophilic condition needed 33927 - 41394 GJ $year^{-1}$ resulting in 197252 - 198135 GJ $year^{-1}$ of available energy. Similarly, in the AD + biogas upgrading option, mesophilic operation required less energy at 44548 - 51876 GJ $year^{-1}$ with available energy of 215196 - 216217 GJ $year^{-1}$ (heat and biomethane). The energy demand for themophilic operation was higher at 51637 - 57900 GJ $year^{-1}$ with energy availbale less than that of in mesophilic counted for 213677 - 215762 GJ $year^{-1}$. However, increasing the OLR in thermophilic conditions could increase the energy and emissions savings until they are close to those in mesophilic operation.
- With the assumptions used, modelling indicated that AD of SBP combined with CHP, in mesophilic and thermophilic conditions, was more favourable than AD +

biogas upgrading if the potential exported heat can be effectively utilized to replace the use of fossil fuels.

7.6. Recommendations

- Single-stage digestion of SBP at OLR higher than 3 g VS l^{-1} day^{-1} showed operational problems. Digestion at thermophilic temperatures is therefore recommended to ensure stable performance at higher OLR.
- Batch testing of antifoams can provide an indication of their degradability and potential to contribute to or to inhibit gas production but semi-continuous trials may be necessary to identify longer-term effects.

7.7. Future Work

- Further work on enzymatic treatments with the use of protease or lipase enzymes is needed to evaluate its potential for improving digestate dewaterability.
- Better screening protocols for cumulative inhibition by antifoam need to be developed, with more work to clarify what types of antifoam are effective.
- In-depth study of the nature of EPS is needed to see if any highly targeted chemical techniques are able to degrade it. Further work on identifying other possible causes of poor dewaterability such as particle size distributions, viscosity, bound water etc is also recommended.
- Future work is needed to evaluate and expand the CEN footprint from AD of SBP in combination with economic analysis to identify the most efficient options.

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APPENDICES

Appendix 1. Two-stage chemical treatment (dosage of polymer on CST test)

a. Mesophilic digestate (First semi-continous trial)

Dosage of polymer required on CST test of mesophilic digestate

Polymer dosage (ml kg^{-1} digestate)	CST (seconds)				
	OLR 2	**OLR 3**	**OLR 4**	**OLR 5**	**OLR 3 +TE**
33	> 900	> 900	> 900	> 900	393.3
50	439.4	> 900	> 900	> 900	134.4
67	188.8	502.2	552.1	> 900	82.8
83	56.1	426.4	453	> 900	52.8
100	43.8	179.6	-	> 900	46.7
117	33.5	169.1	-	> 900	35
133	27.9	107	216	> 900	29.4
150	22.6	-	169.1	> 900	25.2
167	**15.7**	87.2	-	> 900	21.6
197		76.0	-	> 900	**18.5**
203		56.4	-	> 900	
217		40.3	-	603.7	
237		34.1	57.9	-	
250		25.9	-	442.9	
273		20.6	39.7	-	
287		**19.1**	31.7	262.8	
307			-	-	
317			29.1	167.4	
350			26.5	128.1	
360			24.5	78.7	
390			**19.1**	74.2	
423				61.2	
473				38.4	
523				29.3	
557				**12.3**	

Note: OLR= organic loading rate (kg VS l^{-1} day^{-1})

b. Mesophilic and thermophilic digestate (Second semi-continous trial)

Dosage of polymer required on CST test of mesophilic and thermophilic digestates

Polymer dosage (ml kg^{-1} digestate)	CST (seconds)			
	Mesophilic		Thermophilic	
	OLR 4	OLR 5	OLR 4	OLR 5
50.0	>6000	> 6000	115	132
60.0	>3000	> 4000	107.1	121.9
75.0	>1000	> 3000	97.7	106.6
100.0	889.4	2745.3	80.4	92.4
125.0	661.3	-	58.8	73.2
137.5	-	-	49.3	61.2
150.0	531.5	-	40.1	52.9
165.0	-	-	28.7	41.8
175.0	428.9	-	25.2	31.2
185.0	-	-	**17.3**	-
187.5	-	-		**18.9**
200.0	337.7	1984		
225.0	208.4	-		
250.0	113.2	-		
275.0	61.5	-		
285.0	31.2	-		
300.0	**16.3**	349.7		
350.0		184.3		
400.0		86.9		
425.0		63.5		
435.0		41.6		
450.0		30.5		
460.0		28		
470.0		26.5		
485.0		23.2		
500.0		21.9		
510.0		20.8		
515.0		**19.9**		

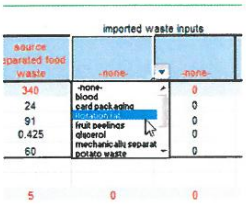
Note: OLR= organic loading rate (kg VS l^{-1} day^{-1})

Appendix 2. Manual ADtool

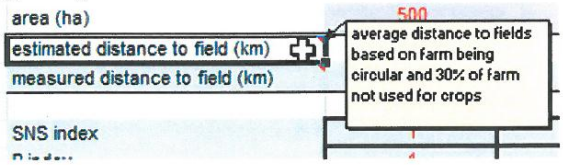
Introduction

The various aspects of the energy tool have been combined into a spreadsheet based tool in order to allow for the calculation of potential energy balances and emissions using a waste based AD system. The tool enables the user to get a 'snap shot' view based on a single year but with the flexibility to easily change feedstock materials.
User inputs are indicated in the tool by red text

or drop down lists (red text, blue background and thick border).

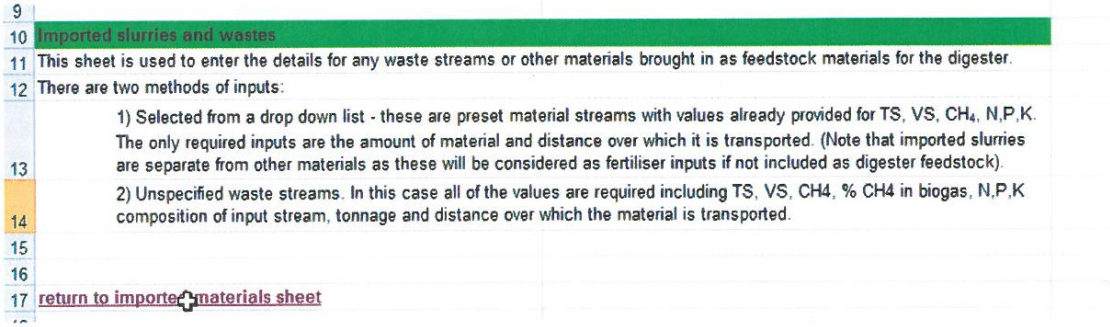


Some cells have a small red triangle in the top right corner. Placing the mouse pointer over the cell will cause a comment box to appear proving some help regarding the information in that row.



There are also various 'help' links which when selected lead to a help page.

Clicking on the relevant 'return' link from this page returns the user to the selected input sheet.



Further detail regarding the theoretical basis of the tool and associated data sources is available in VALORGAS Deliverable D6.3.

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Imported materials

A number of specified import streams including wastes can be entered (Figure 1). Selection can be made from a range of animal slurries including cattle pigs and poultry. Once selected the only other inputs required are the amount and the distance. A range of preselected crop and other waste streams are also available. These also require tonnage and distance.
Finally the user is able to enter up to 5 waste streams of their own specification in which case the user is required to specify the amount, total solids, volatile solids (as proportion of total solids), methane yield and %methane in biogas. Anticipated nutrient values (N, P and K) for these streams are also required in order to provide information for the digestate analysis.

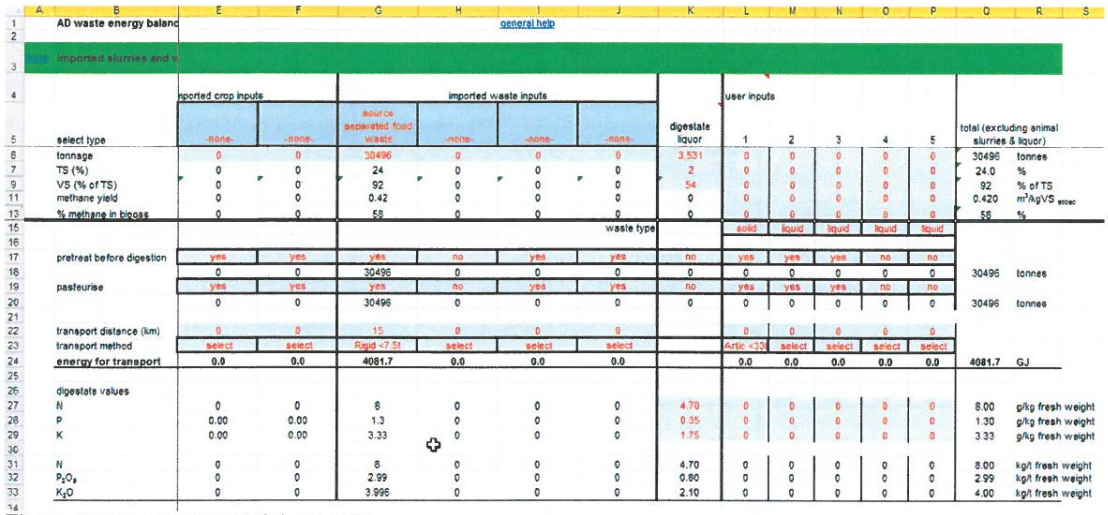

Figure 1: Imported material streams

If the user input waste stream is used, the type of waste (liquid or solid) should be selected (Figure 2). This is used in defining the parasitic electrical energy requirements.

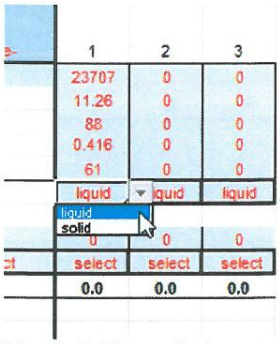

Figure 2: Manually inputted waste stream type

Options are available for pasteurisation and pre-treatment for each waste stream, these will have effect on the digester sheet. Select pre-treatment if the waste requires pre-sorting before entering the digestion system, This gives an energy value separate from the parasitic energy requirement for pre-treatment.

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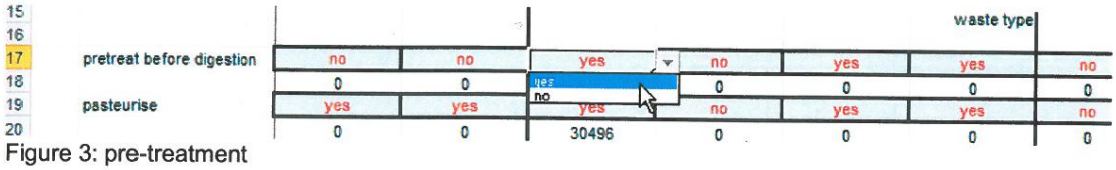

Figure 3: pre-treatment

Different waste streams may or may not require pasteurisation. Select if it is required for that stream Figure 3.

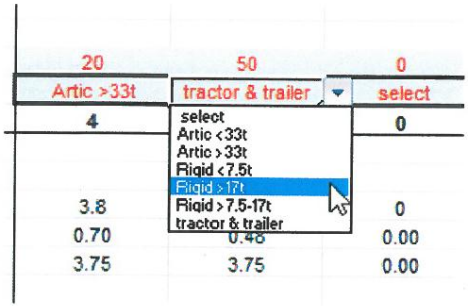

Figure 4: Selecting transport type

If transport energy is to be considered then distance over which the waste is transported to the digester can be specified. The amount of energy required will vary according to the type of transport used. It is possible to select from a range of lorry types based on the DEFRA/DECC guidelines for GHG factors for company reporting (DEFRA, 2009, AEA, 2010). Energy requirements for tractor transport are based on values from KTBL (2009). The type of transport is selected using the relevant drop down list as shown in Figure 4.

The digesters

From the amount of feedstock materials specified, the tool calculates the required digester size and energy requirements as shown in Figure 5.

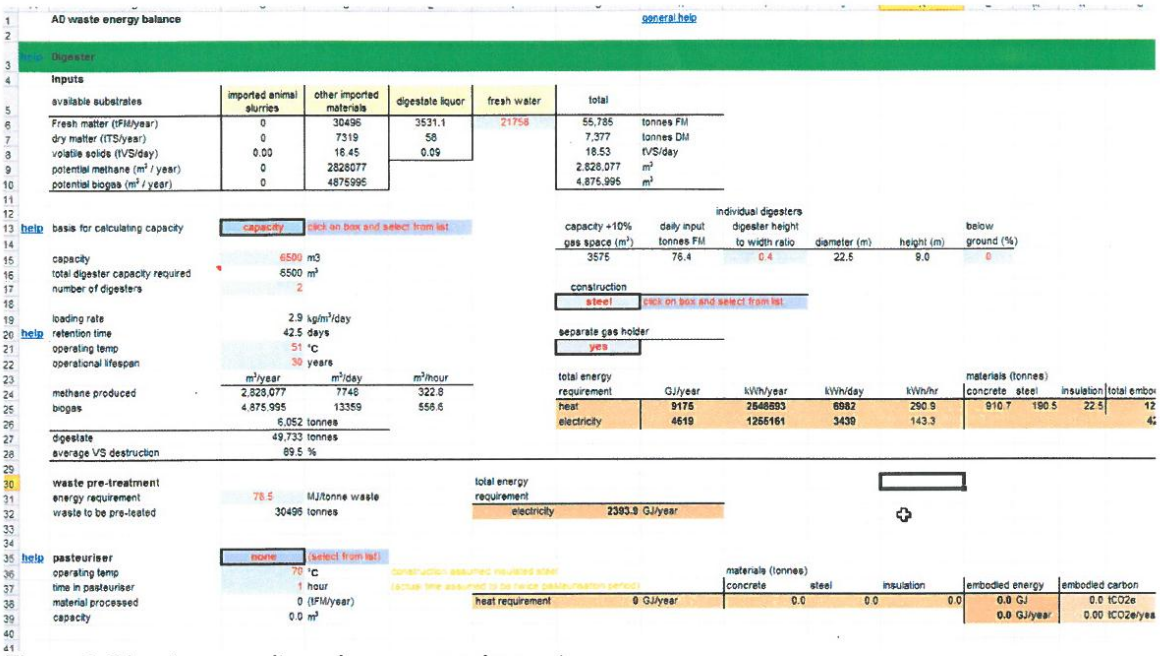

Figure 5: Digester capacity and energy requirements

Overall digester capacity here is calculated based on three options; capacity, loading rate or retention time. Research has shown that a loading rate in the region of 3kg VS/m^3/day is good for CSTR digesters using these types of feedstock materials. Overloading the digester can lead to a reduction in efficiency, methane output and stability. Retention time is also important because it determines the average length of time over which the material is held in the digester. If the retention time is too short then not all of the potential biogas will be released, leading to biogas being produced in the following stages of digestion, storage or after the digestate has been applied to fields. The capacity, loading rate or retention time can be selected as shown in Figure 6. If the selection criteria leads to unreasonable values these are indicated by warning messages. The values should then be reconsidered.

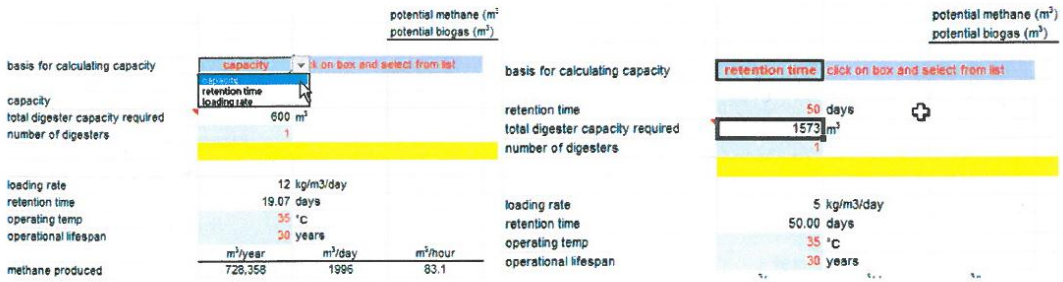

Figure 6: Selection of capacity criteria

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The number of digesters over which this capacity is spread can also be specified, typically a single digester will not be larger than 3500 m^3. The user can specify if the digester is of steel or concrete construction and whether a pasteuriser is included. Energy requirement will be affected by the temperature the digester is operated at (which can be specified), embodied energy is calculated per year based on expected lifespan (which can also be specified).

A number of design options are available using the various input boxes.

The construction materials for the digester can be selected as either concrete based or steel based. A concrete digester is modelled as having a reinforced concrete wall and floor surrounded by an insulation layer and protective sheet metal skin. A flexible gas dome is modelled as the roof for the digester. A steel digester is modelled as a cylinder constructed of two layers of steel separated by a layer of insulation. The floor of the digester is constructed from reinforced concrete. In both designs 10% of the volume is added to the working volume for gas storage.

The height to width ratio and amount of digester buried below ground level can be input.

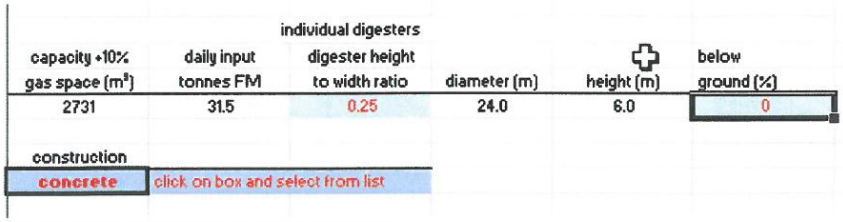


Figure 7: inputs for digester dimensions

Pasteurisation is an option either before digestion for materials selected as requiring pasteurisation in the imported materials sheet or for after digestion in which case all of the digestate is pasteurised. The heat requirements are calculated based on the different options, in the case of pre-pasteurisation it is assumed that the material requires no further heat before being added to the digester.

Figure 8: Pasteurisation

Biogas storage can be done either in the digester, in which case 30% is added to the digester volume to allow for this or in a separate gas storage unit, in which case 10% is added to the working volume of the digester as freeboard. If a separate gas storage unit is specified then it is assumed to be spherical, constructed of two layers of PVC and situated on a reinforced concrete base. The size of the unit is determined by the maximum storage period required.

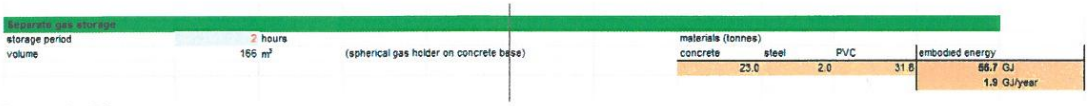


Figure 9: Biogas storage

VALORGAS

Digestate storage facilities can also be specified. The storage period determines the volume of storage required and it is possible to specify the construction materials and whether a roof is included and, if so, its construction. The digestate storage is taken to be a cylindrical tank on a reinforced concrete base without insulation or heating.

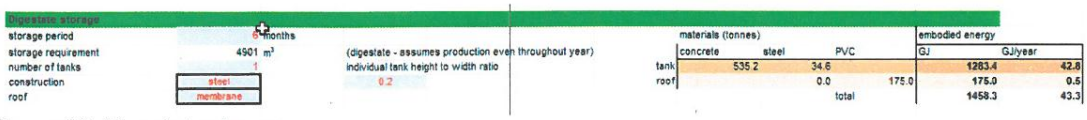


Figure 10: Digestate storage

If pre-treatment of wastes has been selected (on the input materials sheet) then the total energy required for treatment is calculated based on a user input value (given initially as 78.5 MJ tonne^{-1} waste).

Figure 11: pre-treatment

Many plants processing meat or animal based waste products will require compliance with animal by-product regulations including the provision of a building which separates input waste materials from digestates produced. The embodied energy of the building required is calculated based on user specified dimensions and assuming construction is a steel frame covered with corrugated steel cladding.

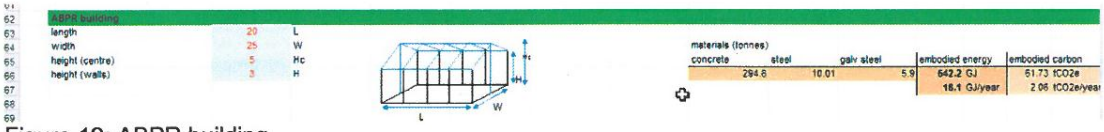


Figure 12: ABPR building

Digestate

The fertiliser value of the digestate is calculated on the basis of nutrients contained in the materials used for digestion with no losses.

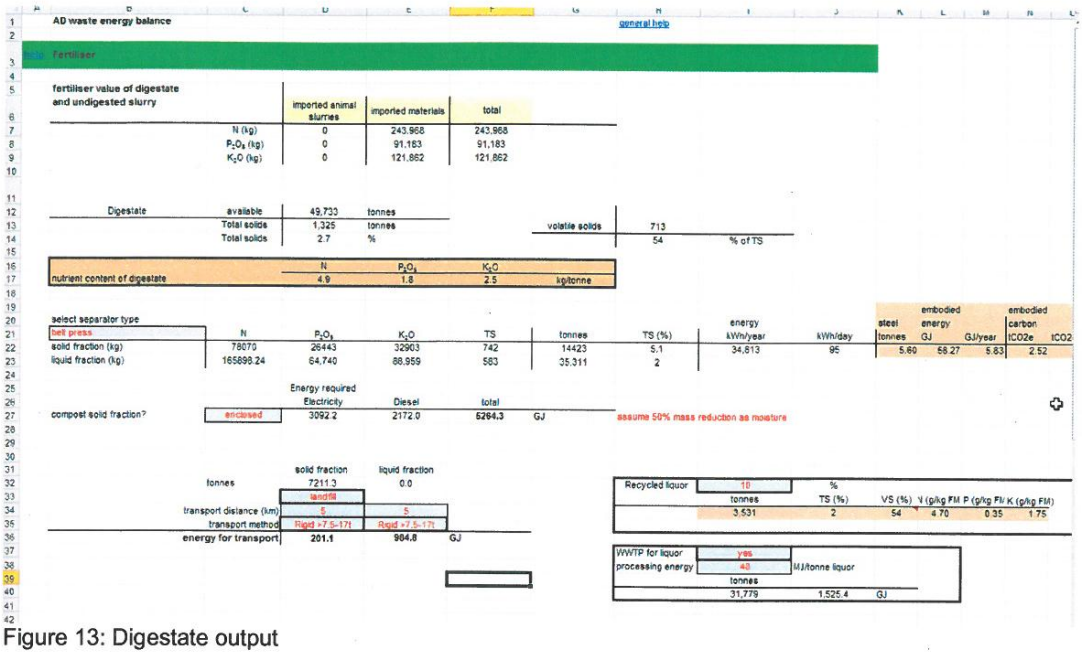


Figure 13: Digestate output

If on site separation is available then the potential separation of solids and nutrients can be determined using different types of separators. This also includes the energy requirement for the separator and embodied energy.
If separation is selected then this can be followed by composting for the fibre fraction. The composting can be either in open windrows or enclosed, each having different requirements for diesel and electricity as shown in

Table 1.

Table 1. Composting energy requirement

	electricity (MJ/t)	diesel (MJ/t)
enclosed	214.4	150.6
none	0	0
open	28.4	275.7

The separated liquor has three paths of use.
a) transported to fields for application
b) recycled to the digester to assist in the dilution of input feedstock, for this use the % of liquor recycled must be specified.
c) any liquor not recycled can be sent to a waste water treatment plant (WWTP) for treatment where it is assumed 48.3 MJ tonne^{-1} liquor is required for the treatment process.

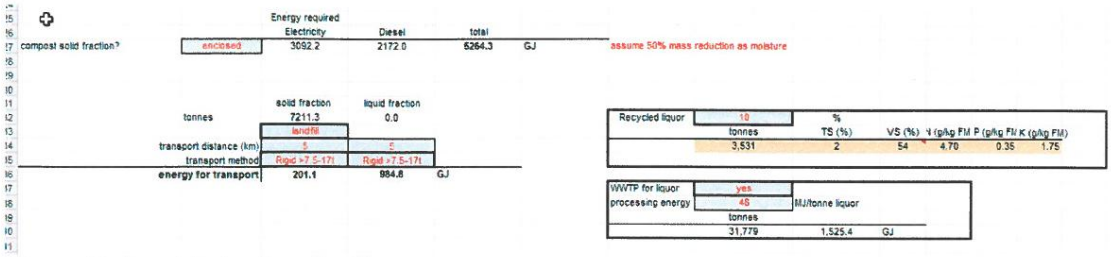


Figure 14. digestate treatment options

Energy requirement for the transport of each fraction of the digestate is calculated based on the type of transport and distance to be travelled.

Biogas use

Energy production is determined from the production and use of the biogas. Electrical energy requirement on-site can be supplied from the grid or through the use of on-site combined heat and power (CHP). The user can specify if the biogas is upgraded or upgraded and compressed. If on-site CHP is selected and no upgrading then it is assumed that all of the biogas is used for CHP (Figure 15).

Heat energy required can be supplied via an on-site boiler. If CHP is selected then there is the potential for heat generated to be used. The tool assumes that heat will initially be used for maintaining digester temperature and heating feedstock materials – any remaining heat is available for export. In this case it is possible to specify the expected heat utilisation (as a percentage of the heat available for export). If no on-site biogas use is selected then heat must be generated from other, imported fuel sources which can be selected.

Process losses (biogas lost before use in the CHP/upgrading) can be entered and are deducted from the potential total available.

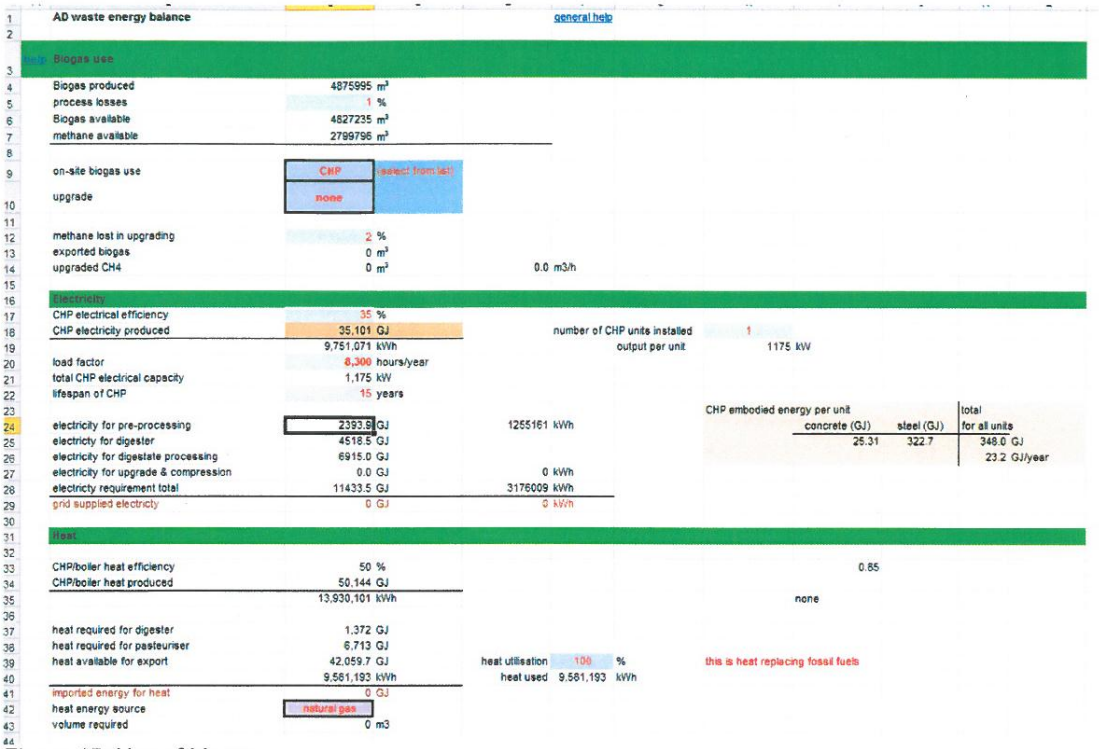


Figure 15: Use of biogas

Where CHP is not included it is assumed that heat and electricity are imported. In the case of electricity this is assumed to be from the national grid, in the case of heat the source can be selected from the drop down list (Figure 16).

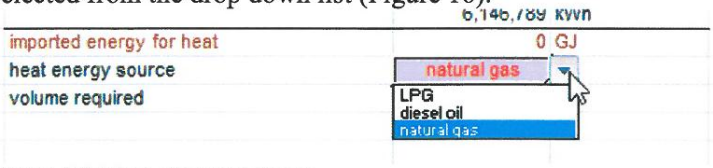


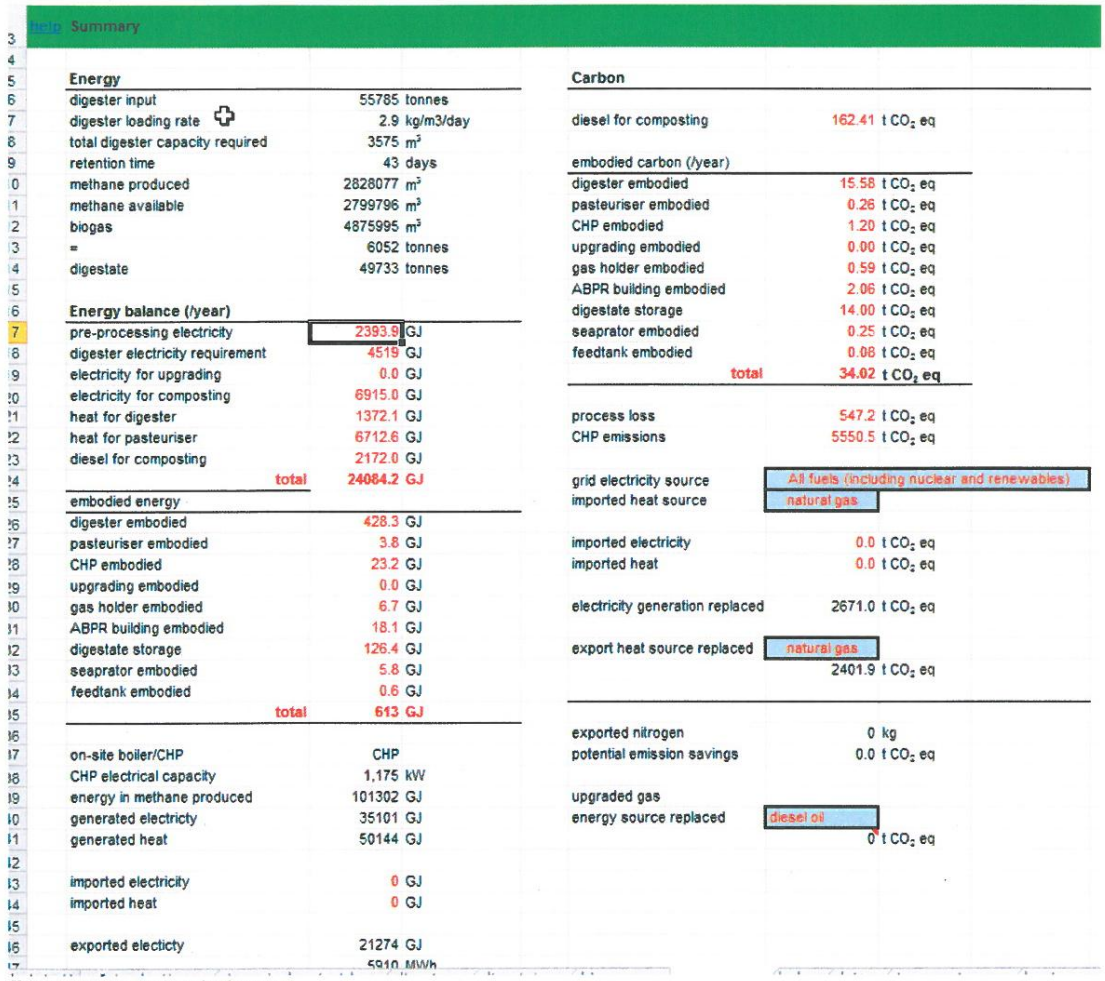
Figure 16: Heat energy sources

If upgrading is selected then the energy required for upgrading and for compression of the upgraded gas can be selected. These are user input with an initial value of 1.08 MJ m^{-3} gas.

Figure 17: Upgrading and compression

Summary sheet

Finally, a summary is given of the energy requirements and balances and emissions produced and potentially saved (Figure 18).



help Summary

Energy

digester input	55785	tonnes
digester loading rate	2.9	kg/m3/day
total digester capacity required	3575	m^3
retention time	43	days
methane produced	2828077	m^3
methane available	2799796	m^3
biogas	4875995	m^3
=	6052	tonnes
digestate	49733	tonnes

Energy balance (/year)

pre-processing electricity	2393.9	GJ
digester electricity requirement	4519	GJ
electricity for upgrading	0.0	GJ
electricity for composting	6915.0	GJ
heat for digester	1372.1	GJ
heat for pasteuriser	6712.6	GJ
diesel for composting	2172.0	GJ
total	24084.2	GJ

embodied energy

digester embodied	428.3	GJ
pasteuriser embodied	3.8	GJ
CHP embodied	23.2	GJ
upgrading embodied	0.0	GJ
gas holder embodied	6.7	GJ
ABPR building embodied	18.1	GJ
digestate storage	126.4	GJ
seaprator embodied	5.8	GJ
feedtank embodied	0.6	GJ
total	613	GJ

on-site boiler/CHP	CHP	
CHP electrical capacity	1,175	kW
energy in methane produced	101302	GJ
generated electricty	35101	GJ
generated heat	50144	GJ
imported electricity	0	GJ
imported heat	0	GJ
exported electicty	21274	GJ
	5910	MWh

Carbon

diesel for composting	162.41	t CO_2 eq

embodied carbon (/year)

digester embodied	15.58	t CO_2 eq
pasteuriser embodied	0.26	t CO_2 eq
CHP embodied	1.20	t CO_2 eq
upgrading embodied	0.00	t CO_2 eq
gas holder embodied	0.59	t CO_2 eq
ABPR building embodied	2.06	t CO_2 eq
digestate storage	14.00	t CO_2 eq
seaprator embodied	0.25	t CO_2 eq
feedtank embodied	0.08	t CO_2 eq
total	34.02	t CO_2 eq

process loss	547.2	t CO_2 eq
CHP emissions	5550.5	t CO_2 eq
grid electricity source	All fuels (including nuclear and renewables)	
imported heat source	natural gas	
imported electricity	0.0	t CO_2 eq
imported heat	0.0	t CO_2 eq
electricity generation replaced	2671.0	t CO_2 eq
export heat source replaced	natural gas	
	2401.9	t CO_2 eq
exported nitrogen	0	kg
potential emission savings	0.0	t CO_2 eq
upgraded gas energy source replaced	diesel oil	
	0	t CO_2 eq

Figure 18: Energy balances

If no CHP is provided it is assumed that all heat and electricity for the AD plant is imported from the national grid for electricity and selectable source for the heat (i.e natural gas, LPG, or diesel oil). When calculating the emissions resulting from the generation of electricity for the national grid, various options can be selected including generation from coal to all sources including renewable (Figure 19). Values used in the tool are based on UK electricity production and will vary for other countries according to the fuel sources used.

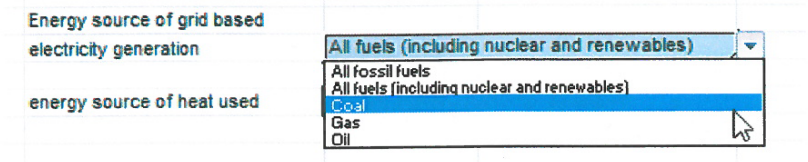


Figure 19: Sources for electricity generation

The emissions saved from exported energy are based on the same selected fuel sources. Emissions saved from the use of heat captured from the CHP are based on the amount of heat utilised as determined on the biogas use sheet. In both the case of electricity and heat the amount available for export is assumed to be that generated les the amount required for use at the AD plant including any biogas upgrading specified.

Temperatures

The temperatures sheet contains information relating to average monthly temperatures for the chosen location. The values used are those contained in column B. These values can be altered to match the users location. If the soil temperatures are unknown then a close estimate can be made by using the average air temperatures.

References for manual

AEA (2010) 2010 Guidelines to Defra / DECC's GHG Conversion Factors for Company Reporting. London, DEFRA.

DEFRA (2009) Guidance on How to Measure and Report Your Greenhouse Gas Emissions. London, DEFRA.

KTBL (2009) *Betriebsplannung Landwirtschaft 2008/09,* Darmstadt, KTBL.

Appendix 3. Modelling: AD + CHP

a. Energy balance

Energy input and outputs for electricity and heat production at different OLR (complex system)

Details	Units	MCE3	TCE3	TCE4	TCE5	TCE6	TCE7	TCE8
digester input	tonnes	100000	100000	100000	100000	100000	100000	100000
digester loading rate	kg m^{-3} day^{-1}	3	3	4	5	6	7	8
total digester capacity required	m^3	4155	4155	3895	4155	3462	4452	3895
retention time	days	69	69	52	41	34	30	26
methane produced	m^3	7651600	7651600	7651600	7651600	7651600	7651600	7651600
methane available	m^3	7575084	7575084	7575084	7575084	7575084	7575084	7575084
biogas	m^3	13912000	13912000	13912000	13912000	13912000	13912000	13912000
=	tonnes	17788	17788	17788	17788	17788	17788	17788
digestate	tonnes	82212	82212	82212	82212	82212	82212	82212
Energy balance ($year^{-1}$)								
pre-processing electricity	GJ	0	0	0	0	0	0	0
digester electricity requirement	GJ	3600	3600	3600	3600	3600	3600	3600
electricity for upgrading	GJ	0	0	0	0	0	0	0
electricity for composting	GJ	883	883	883	883	883	883	883
heat for digester	GJ	13017	23695	22514	21673	21326	20758	20577
heat for pasteuriser	GJ	12115	5209	5209	5209	5209	5209	5209
diesel for composting	GJ	6574	6574	6574	6574	6574	6574	6574
total	GJ	36189	39961	38780	37939	37592	37024	36843
embodied energy								
digester embodied	GJ	1200	1200	919	720	638	502	460
pasteuriser embodied	GJ	7	7	7	7	7	7	7
CHP embodied	GJ	51	51	51	51	51	51	51
upgrading embodied	GJ	0	0	0	0	0	0	0
gas holder embodied	GJ	14	14	14	14	14	14	14
ABPR building embodied	GJ	18	18	18	18	18	18	18
digestate storage	GJ	125	125	125	125	125	125	125
separator embodied	GJ	10	10	10	10	10	10	10
feedtank embodied	GJ	8	8	8	8	8	8	8
total	GJ	1433	1433	1152	953	871	735	693
on-site boiler/CHP		CHP	CHP	CHP	CHP	CHP	CHP	CHP
CHP electrical capacity	kW	3179	3179	3179	3179	3179	3179	3179
energy in methane produced	GJ	274080	274080	274080	274080	274080	274080	274080
generated electricty	GJ	94969	94969	94969	94969	94969	94969	94969
generated heat	GJ	135670	135670	135670	135670	135670	135670	135670
imported electricity	GJ	0	0	0	0	0	0	0
imported heat	GJ	0	0	0	0	0	0	0
exported electicty	GJ	90486	90486	90486	90486	90486	90486	90486
	MWh	25137	25137	25137	25137	25137	25137	25137
exported heat	GJ	110538	106766	107947	108788	109135	109703	109883
	MWh	30707	29660	29988	30221	30318	30476	30526
Energy balance total	GJ $year^{-1}$	163401	155858	158501	160381	161157	162430	162833
	GJ $tonne^{-1}$ waste	1.63	1.56	1.59	1.60	1.61	1.62	1.63
Energy balance electrical	GJ $year^{-1}$	52864	49092	50554	51594	52023	52727	52950
	GJ $tonne^{-1}$ waste	0.53	0.49	0.51	0.52	0.52	0.53	0.53

Energy input and outputs for electricity and heat production at different OLR (simple system)

Details	Units	MSE3	TSE3	TSE4	TSE5	TSE6	TSE7	TSE8
digester input	tonnes	100000	100000	100000	100000	100000	100000	100000
digester loading rate	kg m^{-3} day^{-1}	3	3	4	5	6	7	8
total digester capacity required	m^3	4155	4155	3895	4155	3462	4452	3895
retention time	days	69	69	52	41	34	30	26
methane produced	m^3	7651600	7651600	7651600	7651600	7651600	7651600	7651600
methane available	m^3	7575084	7575084	7575084	7575084	7575084	7575084	7575084
biogas	m^3	13912000	13912000	13912000	13912000	13912000	13912000	13912000
=	tonnes	17788	17788	17788	17788	17788	17788	17788
digestate	tonnes	82212	82212	82212	82212	82212	82212	82212
Energy balance ($year^{-1}$)								
pre-processingelectricity	GJ	0	0	0	0	0	0	0
digester electricity requirement	GJ	3600	3600	3600	3600	3600	3600	3600
electricity for upgrading	GJ	0	0	0	0	0	0	0
electricity for composting	GJ	0	0	0	0	0	0	0
heat for digester	GJ	13017	23695	22514	21673	21326	20758	20577
heat for pasteuriser	GJ	12115	5209	5209	5209	5209	5209	5209
diesel for composting	GJ	0	0	0	0	0	0	0
total	GJ	28732	32504	31323	30482	30135	29567	29386
embodied energy						0		
digester embodied	GJ	1200	1200	919	720	638	502	460
pasteuriser embodied	GJ	7	7	7	7	7	7	7
CHP embodied	GJ	51	51	51	51	51	51	51
upgrading embodied	GJ	0	0	0	0	0	0	0
gas holder embodied	GJ	14	14	14	14	14	14	14
ABPR building embodied	GJ	18	18	18	18	18	18	18
digestate storage	GJ	125	125	125	125	125	125	125
separator embodied	GJ	0	0	0	0	0	0	0
feedtank embodied	GJ	8	8	8	8	8	8	8
total	GJ	1423	1423	1143	943	861	726	683
on-site boiler/CHP		CHP	CHP	CHP	CHP	CHP	CHP	CHP
CHP electrical capacity	kW	3179	3179	3179	3179	3179	3179	3179
energy in methane produced	GJ	274080	274080	274080	274080	274080	274080	274080
generated electricty	GJ	94969	94969	94969	94969	94969	94969	94969
generated heat	GJ	135670	135670	135670	135670	135670	135670	135670
imported electricity	GJ	0	0	0	0	0	0	0
imported heat	GJ	0	0	0	0	0	0	0
exported electicty	GJ	91369	91369	91369	91369	91369	91369	91369
	MWh	25382	25382	25382	25382	25382	25382	25382
exported heat	GJ	110538	106766	107947	108788	109135	109703	109883
	MWh	30707	29660	29988	30221	30318	30476	30526
Energy balance total	GJ $year^{-1}$	171751	164208	166851	168731	169507	170779	171183
	GJ $tonne^{-1}$ waste	1.72	1.64	1.67	1.69	1.70	1.71	1.71
Energy balance electrical	GJ $year^{-1}$	61214	57442	58903	59943	60373	61076	61299
	GJ $tonne^{-1}$ waste	0.61	0.57	0.59	0.60	0.60	0.61	0.61

b. GHG emissions

Emissions inputs and outputs for electricity production at different OLR (complex system)

tonne CO_{2eq}	MCE3	TCE3	TCE4	TCE5	TCE6	TCE7	TCE8
diesel for composting	491.56	491.56	491.56	491.56	491.56	491.56	491.56
embodied carbon (year^{-1})							
digester embodied	87.50	87.50	67.06	52.50	46.50	36.65	33.53
pasteuriser embodied	0.51	0.51	0.51	0.51	0.51	0.51	0.51
CHP embodied	2.39	2.39	2.39	2.39	2.39	2.39	2.39
upgrading embodied	0.00	0.00	0.00	0.00	0.00	0.00	0.00
gas holder embodied	1.21	1.21	1.21	1.21	1.21	1.21	1.21
ABPR building embodied	2.05	2.05	2.05	2.05	2.05	2.05	2.05
digestate storage	12.34	12.34	12.34	12.34	12.34	12.34	12.34
separator embodied	0.42	0.42	0.42	0.42	0.42	0.42	0.42
feedtank embodied	0.95	0.95	0.95	0.95	0.95	0.95	0.95
total	107.37	107.37	86.92	72.37	66.36	56.51	53.39
process loss	1494.57	1494.57	1494.57	1494.57	1494.57	1494.57	1494.57
electricity generation replaced	11360.98	11360.98	11360.98	11360.98	11360.98	11360.98	11360.98
export heat source replaced	6312.57	6097.20	6164.64	6212.64	6232.45	6264.92	6275.22
potential emission savings from N	2328.33	2328.33	2328.33	2328.33	2328.33	2328.33	2328.33
total emissions	2093.49	2093.49	2073.05	2058.49	2052.49	2042.64	2039.52
emission saving (total)	20001.88	19786.51	19853.95	19901.95	19921.76	19954.23	19964.53
emissions balance (electricity)	9267.48	9267.48	9287.93	9302.48	9308.49	9318.34	9321.46
emissions balance (electricity + heat)	15580.06	15364.68	15452.57	15515.12	15540.94	15583.26	15596.67

Emissions inputs and outputs for electricity production at different OLR (simple system)

tonne CO_{2eq}	MSE3	TSE3	TSE4	TSE5	TSE6	TSE7	TSE8
diesel for composting	0.00	0.00	0.00	0.00	0.00	0.00	0.00
embodied carbon (year^{-1})							
digester embodied	87.50	87.50	67.06	52.50	46.50	36.65	33.53
pasteuriser embodied	0.51	0.51	0.51	0.51	0.51	0.51	0.51
CHP embodied	2.39	2.39	2.39	2.39	2.39	2.39	2.39
upgrading embodied	0.00	0.00	0.00	0.00	0.00	0.00	0.00
gas holder embodied	1.21	1.21	1.21	1.21	1.21	1.21	1.21
ABPR building embodied	2.05	2.05	2.05	2.05	2.05	2.05	2.05
digestate storage	12.34	12.34	12.34	12.34	12.34	12.34	12.34
separator embodied	0.00	0.00	0.00	0.00	0.00	0.00	0.00
feedtank embodied	0.95	0.95	0.95	0.95	0.95	0.95	0.95
total	106.95	106.95	86.51	71.95	65.95	56.10	52.98
process loss	1494.57	1494.57	1494.57	1494.57	1494.57	1494.57	1494.57
electricity generation replaced	11471.86	11471.86	11471.86	11471.86	11471.86	11471.86	11471.86
export heat source replaced	6312.57	6097.20	6164.64	6212.64	6232.45	6264.92	6275.22
potential emission savings from N	2328.33	2328.33	2328.33	2328.33	2328.33	2328.33	2328.33
total emissions	1601.52	1601.52	1581.07	1566.52	1560.51	1550.66	1547.54
emission saving (total)	20112.77	19897.39	19964.83	20012.83	20032.65	20065.12	20075.41
emissions balance (electricity)	9870.35	9870.35	9890.79	9905.35	9911.35	9921.20	9924.32
emissions balance (electricity + heat)	16182.92	15967.54	16055.43	16117.98	16143.80	16186.12	16199.54

Appendix 4. Modelling: AD + Biogas upgrading

a. Energy balance

Energy input and outputs for biomethane and heat production at different OLR (complex system)

Details	Units	MCB3	TCB3	TCB4	TCB5	TCB6	TCB7	TCB8
digester input	tonnes	100000	100000	100000	100000	100000	100000	100000
digester loading rate	kg m^{-3} day^{-1}	3	3	4	5	6	7	8
total digester capacity required	m^3	4155	4155	3895	4155	3462	4452	3895
retention time	days	69	69	52	41	34	30	26
methane produced	m^3	7651600	7651600	7651600	7651600	7651600	7651600	7651600
methane available	m^3	7575084	7575084	7575084	7575084	7575084	7575084	7575084
biogas	m^3	13912000	13912000	13912000	13912000	13912000	13912000	13912000
=	tonnes	17788	17788	17788	17788	17788	17788	17788
digestate	tonnes	82212	82212	82212	82212	82212	82212	82212
Energy balance ($year^{-1}$)								
pre-processing electricity	GJ	0	0	0	0	0	0	0
digester electricity requirement	GJ	3600	3600	3600	3600	3600	3600	3600
electricity for upgrading	GJ	14173	14173	14173	14173	14173	14173	14173
electricity for composting	GJ	883	883	883	883	883	883	883
heat for digester	GJ	13017	23695	22514	21673	21326	20758	20577
heat for pasteuriser	GJ	12115	5209	5209	5209	5209	5209	5209
diesel for composting	GJ	6574	6574	6574	6574	6574	6574	6574
total	GJ	50362	54133	52952	52112	51765	51196	51016
embodied energy								
digester embodied	GJ	1200	1200	919	720	638	502	460
pasteuriser embodied	GJ	7	7	7	7	7	7	7
CHP embodied	GJ	15	15	15	15	15	15	15
upgrading embodied	GJ	117	117	117	117	117	117	117
gas holder embodied	GJ	14	14	14	14	14	14	14
ABPR building embodied	GJ	18	18	18	18	18	18	18
digestate storage	GJ	125	125	125	125	125	125	125
separator embodied	GJ	10	10	10	10	10	10	10
feedtank embodied	GJ	8	8	8	8	8	8	8
total	GJ	1514	1514	1234	1035	952	817	774
CHP electrical capacity	kW	624	624	624	624	624	624	624
energy in methane produced	GJ	274080	274080	274080	274080	274080	274080	274080
generated electricty	GJ	18656	18656	18656	18656	18656	18656	18656
generated heat	GJ	26651	26651	26651	26651	26651	26651	26651
imported heat	GJ	0	2253	1072	231	0	0	0
exported heat	GJ	1519	0	0	0	116	684	865
	MWh	422	0	0	0	32	190	240
upgraded biogas	m^3	5965293	5965293	5965293	5965293	5965293	5965293	5965293
energy in upgraded CH_4	GJ	213677	213677	213677	213677	213677	213677	213677
diesel equivalent	liters	5979556	5979556	5979556	5979556	5979556	5979556	5979556
Energy balance total	GJ $year^{-1}$	163319	158029	159490	160530	161076	162348	162751
	GJ $tonne^{-1}$ waste	1.63	1.58	1.59	1.61	1.61	1.62	1.63
Energy balance biomethane	GJ $year^{-1}$	161801	158029	159490	160530	160960	161663	161886
	GJ $tonne^{-1}$ waste	1.62	1.58	1.59	1.61	1.61	1.62	1.62

Energy input and outputs for biomethane and heat production at different OLR (simple system)

Details	Units	MSB3	TSB3	TSB4	TSB5	TSB6	TSB7	TSB8
digester input	tonnes	100000	100000	100000	100000	100000	100000	100000
digester loading rate	kg m^{-3} day^{-1}	3	3	4	5	6	7	8
total digester capacity required	m^3	4155	4155	3895	4155	3462	4452	3895
retention time	days	69	69	52	41	34	30	26
methane produced	m^3	7651600	7651600	7651600	7651600	7651600	7651600	7651600
methane available	m^3	7575084	7575084	7575084	7575084	7575084	7575084	7575084
biogas	m^3	13912000	13912000	13912000	13912000	13912000	13912000	13912000
=	tonnes	17788	17788	17788	17788	17788	17788	17788
digestate	tonnes	82212	82212	82212	82212	82212	82212	82212
Energy balance ($year^{-1}$)								
pre-processing electricity	GJ	0	0	0	0	0	0	0
digester electricity requirement	GJ	3600	3600	3600	3600	3600	3600	3600
electricity for upgrading	GJ	14311	14311	14311	14311	14311	14311	14311
electricity for composting	GJ	0	0	0	0	0	0	0
heat for digester	GJ	13017	23695	22514	21673	21326	20758	20577
heat for pasteuriser	GJ	12115	5209	5209	5209	5209	5209	5209
diesel for composting	GJ	0	0	0	0	0	0	0
total	GJ	43043	46814	45633	44793	44446	43877	43697
embodied energy						0	0	0
digester embodied	GJ	1200	1200	919	720	638	502	460
pasteuriser embodied	GJ	7	7	7	7	7	7	7
CHP embodied	GJ	15	15	15	15	15	15	15
upgrading embodied	GJ	118	118	118	118	118	118	118
gas holder embodied	GJ	14	14	14	14	14	14	14
ABPR building embodied	GJ	18	18	18	18	18	18	18
digestate storage	GJ	125	125	125	125	125	125	125
separator embodied	GJ	0	0	0	0	0	0	0
feedtank embodied	GJ	8	8	8	8	8	8	8
total	GJ	1505	1505	1225	1026	943	808	765
CHP electrical capacity	kW	599	599	599	599	599	599	599
energy in methane produced	GJ	274080	274080	274080	274080	274080	274080	274080
generated electricty	GJ	17911	17911	17911	17911	17911	17911	17911
generated heat	GJ	25587	25587	25587	25587	25587	25587	25587
imported heat	GJ	0	3317	2136	1295	948	380	199
exported heat	GJ	455	0	0	0	0	0	0
	MWh	126	0	0	0	0	0	0
upgraded biogas	m^3	6023516	6023516	6023516	6023516	6023516	6023516	6023516
energy in upgraded CH_4	GJ	215762	215762	215762	215762	215762	215762	215762
diesel equivalent	liters	6037918	6037918	6037918	6037918	6037918	6037918	6037918
Energy balance total	GJ $year^{-1}$	171669	164126	166768	168648	169425	170697	171100
	GJ $tonne^{-1}$ waste	1.72	1.64	1.67	1.69	1.69	1.71	1.71
Energy balance biomethane	GJ $year^{-1}$	171214	164126	166768	168648	169425	170697	171100
	GJ $tonne^{-1}$ waste	1.71	1.64	1.67	1.69	1.69	1.71	1.71

b. GHG emissions

Emissions inputs and outputs for biomethane production at different OLR (complex system)

tonne CO_{2eq}	MCB3	TCB3	TCB4	TCB5	TCB6	TCB7	TCB8
diesel for composting	491.56	491.56	491.56	491.56	491.56	491.56	491.56
embodied carbon (year^{-1})							
digester embodied	87.50	87.50	67.06	52.50	46.50	36.65	33.53
pasteuriser embodied	0.51	0.51	0.51	0.51	0.51	0.51	0.51
CHP embodied	0.83	0.83	0.83	0.83	0.83	0.83	0.83
upgrading embodied	11.64	11.64	11.64	11.64	11.64	11.64	11.64
gas holder embodied	1.21	1.21	1.21	1.21	1.21	1.21	1.21
ABPR building embodied	2.05	2.05	2.05	2.05	2.05	2.05	2.05
digestate storage	12.34	12.34	12.34	12.34	12.34	12.34	12.34
separator embodied	0.42	0.42	0.42	0.42	0.42	0.42	0.42
feedtank embodied	0.95	0.95	0.95	0.95	0.95	0.95	0.95
total	117.45	117.45	97.01	82.45	76.45	66.60	63.48
process loss	1495	1495	1495	1495	1495	1495	1495
imported heat	0	129	61	13	0	0	0
export heat source replaced	87	0	0	0	7	39	49
potential emission savings from N	2440	2440	2440	2440	2440	2440	2440
energy source replaced (biomethane)	15978	15978	15978	15978	15978	15978	15978
total emissions	2104	2232	2144	2082	2063	2053	2050
emission saving (total)	18393	18306	18306	18306	18313	18345	18355
emissions balance (biomethane)	13874	13745	13833	13896	13915	13925	13928
emissions balance (biomethane + heat)	13961	13745	13833	13896	13922	13964	13977

Emissions inputs and outputs for biomethane production at different OLR (simple system)

tonne CO_{2eq}	MSB 3	TSB3	TSB4	TSB5	TSB6	TSB7	TSB8
diesel for composting	0.00	0.00	0.00	0.00	0.00	0.00	0.00
embodied carbon (year^{-1})							
digester embodied	87.50	87.50	67.06	52.50	46.50	36.65	33.53
pasteuriser embodied	0.51	0.51	0.51	0.51	0.51	0.51	0.51
CHP embodied	0.82	0.82	0.82	0.82	0.82	0.82	0.82
upgrading embodied	11.74	11.74	11.74	11.74	11.74	11.74	11.74
gas holder embodied	1.21	1.21	1.21	1.21	1.21	1.21	1.21
ABPR building embodied	2.05	2.05	2.05	2.05	2.05	2.05	2.05
digestate storage	12.34	12.34	12.34	12.34	12.34	12.34	12.34
separator embodied	0.00	0.00	0.00	0.00	0.00	0.00	0.00
feedtank embodied	0.95	0.95	0.95	0.95	0.95	0.95	0.95
total	117.12	117.12	96.67	82.12	76.11	66.26	63.14
process loss	1495	1495	1495	1495	1495	1495	1495
imported heat	0	189	122	74	54	22	11
export heat source replaced	26	0	0	0	0	0	0
potential emission savings from N	2440	2440	2440	2440	2440	2440	2440
energy source replaced (biomethane)	16134	16134	16134	16134	16134	16134	16134
total emissions	1612	1801	1713	1651	1625	1583	1569
emission saving (total)	18488	18462	18462	18462	18462	18462	18462
emissions balance (biomethane)	14522	14332	14420	14483	14509	14551	14564
emissions balance (biomethane + heat)	14548	14332	14420	14483	14509	14551	14564

Appendix 5. Modelling: AD without digestate application

a. Energy balance

Energy input and outputs for AD without digestate application (complex system)

Energy	Units	MCE3	TCE3	MCB3	TCB3
digester input	tonnes	100000	100000	100000	100000
digester loading rate	kg m^{-3} day^{-1}	3	3	3	3
total digester capacity required	m^3	4155	4155	4155	4155
retention time	days	69	69	69	69
methane produced	m^3	7651600	7651600	7651600	7651600
methane available	m^3	7269020	7269020	7269020	7269020
biogas	m^3	13912000	13912000	13912000	13912000
=	tonnes	17788	17788	17788	17788
digestate	tonnes	82212	82212	82212	82212
Energy balance ($year^{-1}$)					
pre-processing electricity	GJ	0	0	0	0
digester electricity requirement	GJ	3600	3600	3600	3600
electricity for upgrading	GJ	0	0	13239	13239
electricity for composting	GJ	3009	3009	3009	3009
heat for digester	GJ	13017	23695	13017	23695
heat for pasteuriser	GJ	12115	5209	12115	5209
diesel for composting	GJ	0	0	0	0
total	GJ	31741	35513	44980	48751
embodied energy					
digester embodied	GJ	1200	1200	1200	1200
pasteuriser embodied	GJ	7	7	7	7
CHP embodied	GJ	49	49	16	16
upgrading embodied	GJ	0	0	110	110
gas holder embodied	GJ	14	14	14	14
ABPR building embodied	GJ	18	18	18	18
digestate storage	GJ	125	125	125	125
separator embodied	GJ	10	10	10	10
feedtank embodied	GJ	8	8	8	8
total	GJ	1431	1431	1508	1508
on-site boiler/CHP		CHP	CHP	CHP	CHP
CHP electrical capacity	kW	3050	3050	664	664
energy in methane produced	GJ	274080	274080	274080	274080
generated electricty	GJ	91132	91132	19848	19848
generated heat	GJ	130188	130188	28354	28354
imported electricity	GJ	0	0	0	0
imported heat	GJ	0	0	0	550
exported electicty/biomethane	GJ	84523	84523	0	0
	MWh	23480	23480	0	0
exported heat	GJ	105056	101285	3221	0
	MWh	29185	28137	895	0
upgraded biogas	m^3	0	0	5572185	5572185
energy in upgraded CH_4	GJ	0	0	199596	199596

b. GHG emissions

Emissions inputs and outputs for AD without digestate application (complex system)

tonne CO_{2eq}	MCE3	TCE3	MCB3	TCB3
diesel for composting	0.00	0.00	0.00	0.00
embodied carbon (year^{-1})				
digester embodied	87.50	87.50	87.50	87.50
pasteuriser embodied	0.51	0.51	0.51	0.51
CHP embodied	2.32	2.32	0.85	0.85
upgrading embodied	0.00	0.00	10.99	10.99
gas holder embodied	1.21	1.21	1.21	1.21
ABPR building embodied	2.05	2.05	2.05	2.05
digestate storage	12.34	12.34	12.34	12.34
separator embodied	0.42	0.42	0.42	0.42
feedtank embodied	0.95	0.95	0.95	0.95
total	107.29	107.29	116.82	116.82
process loss	7472.8	7472.8	7472.8	7472.8
CHP emissions	14410.5	14410.5	3138.5	3138.5
imported electricity	0.0	0.0	0.0	0.0
imported heat	0.0	0.0	0.0	31.4
electricity generation/biomethane replaced	10612.3	10612.3	14924.7	14924.7
export heat source replaced	5999.5	5784.2	184.0	0.0
total emissions savings	8848.0	8632.6	7335.3	7119.9
total emissions	7580.12	7580.12	7589.65	7621.06
emission saving (total)	9031.71	8816.33	7519.00	7303.62
emissions balance (biomethane)	3032.18	3032.18	7335.03	7303.62
emissions balance (biomethane + heat)	9031.71	8816.33	7519.00	7303.62