



Critical Assessment and Optimisation of Sewage Sludge Mesophilic Anaerobic Digestion Processes at Operational Wastewater Treatment Plant

Jin LIU

Supervisor: Professor Stephen R. Smith

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Department of Civil and Environmental Engineering, Imperial College London

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Abstract

Anaerobic digestion (AD) is a long-established method for treating wastewater sludge and has been extensively researched, but there is still a lack of generic or practical modelling tools available to guide operators to optimise the process at full industrial scale. Detailed kinetic models, incorporating fundamental AD mechanisms, such as the Anaerobic Digestion Model No.1 (ADM1), are available, but are considered to be too complex as a practical tool for full-scale process management. The Water Industry collects extensive data records on operational mesophilic anaerobic digestion (MAD) process variables, but there has been no attempt to integrate this information into a comprehensive and systematic process optimisation strategy. A novel, multiple regression analysis approach was applied to operational data from 84 full-scale MAD sites (including 66 conventional, 8 thermal hydrolysis process (THP) and 10 enzymic) in five major UK Water Utility companies, demonstrating a wide range of AD performance, to construct a universal, operationally based MAD model. The model incorporated a categorical site or process type factor, and specific calibration methods were used, to account for inter-site variation. This approach to modelling the full-scale AD process explained the variation caused by local data measurement and other unrecorded factors at specific sites, and the major operational factors influencing the process were quantified based on relative changes in digester performance, using a centred predictors approach. Digestion process performance was modelled using biogas yield (BY) or electricity yield (EY) as the dependent variable, and two sub-models were also developed on volatile solids reduction (VSR) and digested sludge dewaterability. The BY of the conventional and THP MAD processes was positively correlated to the main effects of temperature and HRT, and negatively correlated to the dry solids (DS) content in feed sludge. The effect of HRT on BY also depended on the concentration of the DS feed and increasing HRT, and had a stronger effect on BY at higher DS concentrations. The EY of the enzymic MAD process also decreased with increasing DS, and increased in relation to both temperature and volatile fatty acid (VFA) feed. The magnitude of the response in EY to temperature increased at higher VFA feed concentrations. Eight MAD sites were selected for sludge sample collection on 6 sampling events, over a period of approximately one year, to examine the effects of detailed fat, protein, carbohydrate and fibre (cellulose, hemicellulose and lignin) composition on BY. However, no significant relation was detected between BY and the major organic energy constituents (such as protein and fat) in feed sludge, but the compositional derived BY_c , which is based on the proportions of substrates destroyed during AD, was significantly correlated to the observed BY. The models can be applied universally to any full-scale, stable, MAD process, including THP and enzymic pretreatment, by adopting one or more calibration options, to account for site specific conditions, by

using local site: (a) biogas data, (b) electricity generation, or (c) sludge composition results. The results showed that full-scale digester performance was controlled by the combined and interactive effects of multiple process parameters; therefore, it is necessary to balance all the principal operational parameters to optimise the AD performance. Furthermore, the highest BY or EY does not necessarily correspond to the optimum AD performance in terms of the maximum biogas volume or net energy output. Thus, to ensure a positive energy balance, a conventional process should only be operated in the high mesophilic range $>39^{\circ}\text{C}$, when the DS of the feed sludge is $\geq 3.0\%$. A greater energy surplus is obtained with increasing feed DS content, which is a major advantage of THP pretreatment. Conventional MAD processes have an average DS feed of 4.5% and there is considerable potential to modify current operational practices to improve the overall energy balance of the system. The model provides AD operators with a practical management tool to aid decision support to balance the principal operational parameters and improve the efficiency and net energy output of industrial scale sewage sludge AD.

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Acronyms

AER	Advanced energy recovery
AD	Anaerobic digestion
ADM1	Anaerobic Digestion Model No.1
AA	Amino acids
ANOVA	Analysis of variance
BY	Biogas yield
BY _e	Electricity derived biogas yield
BY _c	Sludge composition derived biogas yield
BOD	Biochemical oxygen demand
BMP	Biomethane potential
BNR	Biological nutrient removal
BSA	Bovine serum albumin
COD	Chemical oxygen demand
CPR	Chemical phosphorus removal
CSTR	Continuous stirred-tank reactor
DS	Dry solids
EH	Enzymic hydrolysis
EEH	Enhanced enzymic hydrolysis
<i>E. coli</i>	<i>Escherichia coli</i>
ECP	Extracellular polymer
FOG	Fat oil and grease
FVSW	Fruit and vegetable solid waste
FAS	Ferrous (iron II) ammonium sulphate
GHG	Greenhouse gas
HRT	Hydraulic retention time

HpH	Heating, pasteurisation and hydrolysis
HDPE	High-density polyethylene
IC ₅₀	Half maximal inhibitory concentration
LCFA	Long chain fatty acids
MAD	Mesophilic anaerobic digestion
MS	Monosaccharides
MLSS	Mixed liquor suspended solids
OLR	Organic loading rate
PS	Primary sludge
PHA	Polyhydroxyalkanoates
R ²	Coefficient of determination
SD	Standard deviation
SS	Sums of squares
SRB	Sulphate reducing bacteria
SAS	Surplus activated sludge
SBY	Specific biogas yield
SEY	Specific electricity yield
TOC	Total organic carbon
TS	Total solids
THP	Thermal hydrolysis process
VS	Volatile solids
VSS	Volatile suspended solids
VSR	Volatile solids reduction
VSR _{MB}	Mass balance volatile solids reduction
VSR _{VK}	Van Kleeck volatile solids reduction
VFA	Volatile fatty acids
WWT	Wastewater treatment

Chemical Formulae and Abbreviations

CO ₂	Carbon dioxide
CH ₄	Methane
Ca	Calcium
Cu	Copper
Cr	Chromium
Co	Cobalt
Cd	Cadmium
Fe	Iron
FeCl ₃	Ferric chloride
Fe ₂ (SO ₄) ₃	Ferric sulphate
H ₂ S	Hydrogen sulphide
H ⁺	hydrogen cation
K	Potassium
Mg	Magnesium
Mn	Manganese
Mo	Molybdenum
Na	Sodium
Ni	Nickel
NH ₃	Ammonia
NH ₄ ⁺	Ammonium
OH ⁻	Hydroxide anion
Pb	Lead
S	Sulphur
Se	Selenium

P

Phosphorus

Z

Zinc

Chapter 1.Introduction

1.1. Background

The Water Industry consumes up to 3% of the total energy used (Howe, 2009), is the fourth most energy intensive sector (POST, 2007) and contributes approximately 1% of national greenhouse gas (GHG) emissions in the UK (Water UK, 2009); specifically, wastewater treatment (WWT) contributes almost 60% of overall GHGs emitted by the industry (Ainger *et al.*, 2009). However, the Water Industry is also a significant producer of renewable energy, for example, 20% of the energy consumed in Thames Water is from renewable sources supplied through anaerobic digestion (AD) of sewage sludge (Thames Water, 2019).

Anaerobic digestion is well established as a process for the stabilisation and treatment of residual sewage sludge from WWT. Considerable attention is being paid to the optimisation of the mesophilic anaerobic digestion (MAD) process to increase revenues by maximising potential renewable energy generation. A particular advantage of this process above other sludge treatment options, therefore, is that it improves the energy balance of WWT and contributes to the renewable energy obligations of the Industry.

Scientific models of the AD process have been developed for almost 40 years, motivated by the need for efficient operation of anaerobic systems (Donoso-Bravo *et al.*, 2011). The complexity of the AD process requires a modelling approach to balance the various influencing operational parameters, and specific models have been developed for different purposes (Kythreotou *et al.*, 2014). The simple stoichiometric equation first proposed by Buswell and Muller (1952) calculated the maximum biogas potential of the substrates, however, this approach does not account for the biodegradable fraction in sludge. The next generation of models focussed on the rate limiting step of the AD process and were based, for example, on the rates of conversion of fatty acids, methanogenesis or the hydrolysis of suspended solids (Eastman and Ferguson, 1981). These models were simple and easy to use, but were unable to adequately describe the overall process performance (Donoso-Bravo *et al.*, 2011). More complex models incorporated additional processes, microbial species and detailed kinetics, including inhibitory mechanisms, based on improved microbiological understanding of the process. For example, Hill (1982) used the volatile fatty acid (VFA) concentration as a key parameter and separated the kinetics of acidogenesis and acetogenesis into individual stages. The most advanced model yet developed, by Batstone *et al.* (2002), the Anaerobic Digestion Model No.1 (ADM1) described the dynamics of 24 bacteria species and included 19 bioconversion processes, with the aim of providing a comprehensive, generic model of the AD system. Whilst valuable in research, the complexity and the large number of input parameters restricts the application of dynamic models at a practical level for optimisation of full-scale industrial AD systems. Moreover, a

constant-volume and completely-mixed system is assumed by ADM1 and this rarely achieved at full-scale (Kythreotou *et al.*, 2014).

The biogas yield (BY) of sewage sludge under MAD is influenced by both operational conditions and raw sludge chemical characteristics. The effect of individual operational parameter, such as digestion temperature and hydraulic retention time (HTR) have been well studied by several authors at the lab scale. For example, digester temperature has a major impact on the physico-chemical and biological conditions within the digester environment, and controls the chemical reaction rates, solubility of the organic compounds, and metabolism and growth rate of microorganisms (Smith *et al.*, 2005; Appels *et al.*, 2008; Lang and Smith, 2008). A positive effect of temperature on BY has been confirmed by several lab-scale studies (Boušková *et al.*, 2005; Kim and Lee, 2016; Nielsen *et al.*, 2017). Hydraulic retention time is also an important factor, which has a positive effect on BY (Alepu *et al.*, 2016). It is essential to ensure an adequate retention time to maintain a sufficient quantity and concentration of bacteria in the digester for the efficient conversion of complex organic matter to methane (CH₄) and carbon dioxide (CO₂) (Parkin and Owen, 1986).

However, few studies have focussed on the characterisation of the organic matter in input sludge and the relation between the organic matter properties and the anaerobic conversion performance. The intrinsic chemical quality of the organic matter input in sludge is not currently included in operational process performance monitoring criteria, but it could have a major influence in explaining the variation in performance at and between different AD sites. The current, simplistic approach to monitoring sludge characteristics only routinely records the dry solids (DS) and volatile solids (VS) and, for some advanced MAD treatment sites, also the VFA concentration in sludge. The VS represents the amount of organic matter in sewage sludge. Thus, increasing VS in sludge, combined with increasing DS, raises the organic loading rate (OLR), which is positively related to absolute gas production as well as the BY (Rus *et al.*, 2013). However, understanding the detailed intrinsic chemical quality of the organic matter input in sludge, including, protein, fat, carbohydrate and fibre contents (lignin and cellulose, hemicellulose), which is not currently included in operational process performance monitoring criteria, would give greater insight into the potential AD bioenergy value of sludge. For example, differences in the relative composition of these substrates can impact on BY due to the different CH₄ yields from the different substrate sources under AD; thus fat has the highest yield of 1014 m³ CH₄/t DS compared to lignin which is virtually undegradable in AD (Weiland, 2010; Li *et al.*, 2018). The substrate composition of sludge also plays an important role in digester stability, for example, Cook *et al.* (2017) reported that the sludge AD process was more stable when the protein proportion was between 5 and 25 %DS across a range of carbohydrate and fat contents. Therefore, significant progress in process understanding, control and optimisation could be provided from the current simplistic approach of monitoring only the DS and VS content by collecting more comprehensive data on sludge composition. For instance, this could be an important factor explaining the variation in performance at and between different AD sites.

Sludge pre-treatment method, upstream WWT processes and asset condition are also potential factors influencing full-scale AD performance. Conventional MAD plants are fully mixed, semi-continuous reactors operated in the mesophilic temperature range from 35 to 40 °C (Zupančič and Grilc, 2012). At advanced MAD site, different types of sludge pre-treatment method are employed before conventional MAD, which are designed to increase biodegradability and promote substrate hydrolysis in the following MAD process, and also to increase digester solids inputs (Werker *et al.*, 2007; Abu-Orf and Goss, 2012; Kubeshnee, 2014). The upstream WWT process variables, including surplus activated sludge (SAS) age and primary:SAS ratio can also affect BY of sludge AD. For example, a short sludge age coupled with a high primary:SAS ratio is considered favourable for raising the overall BY in AD due to the increased sludge digestibility (Gossett and Belser, 1982; WEF, 1987).

The complexity of full-scale AD plants means that it is not practicable to assess the effects of single operational parameters directly on digester performance or to apply simple modelling strategies to manage and optimise the process. Consequently, the extension of fundamental and AD modelling research to full-scale processes has not yet been achieved due to a lack of adequate modelling tools that can be practically applied by operators managing full-scale plant. Therefore, understanding the influence and relative significance of major operational variables affecting full-scale process control could be beneficial for wide-scale industrial optimisation of AD performance, specifically to increase BY.

Past research has quantified the effects of AD conditions on process performance in controlled laboratory environments. However, it is less clear how digestion conditions affect AD performance in full-scale, industrial AD plants. Specific AD plants develop unique process conditions, consequently, stable and optimal operation of full-scale AD processes depends heavily on local experience. The Water Industry has collected extensive data archives on operational MAD process variables, but little use is made of this information for process optimisation. Statistical analysis of this data using different approaches could increase understanding of the influence and relative significance of major operational factors affecting process control and optimisation of full-scale industrial MAD systems.

The aim of this research, therefore, was to understand the influence and significance of major operational variables affecting process control and optimisation of full-scale AD systems, and to develop a statistically based, decision support tool suitable for process operators to optimise AD conditions.

Chapter 2.Literature Review

2.1. Introduction to Sewage Sludge AD

Sewage sludge is an inevitable by-product of wastewater treatment and contains organic matter and essential plant nutrient resources which can be recycled for crop production and to improve soil structural properties. In the UK, more than 60% of sewage sludge was treated using AD in 2010 - 2011, and this increased to 80% in 2014 – 2015 (Ofwat, 2016). Figure 2-1 presents a typical flow diagram of full-scale wastewater treatment process with MAD. Primary sludge is produced through primary sedimentation by a combination of physical (sedimentation) or chemical (coagulation and flocculation) processes to remove suspended solids. Surplus activated sludge is produced from biological WWT process to remove soluble and colloid chemical oxygen demand (COD) after primary treatment (Grady *et al.*, 2011). Surplus activated sludge is generated from the metabolic transformation of biodegradable substrates in wastewater and typically comprises approximately 50% of overall sludge production at a wastewater treatment plant (WWTP) (Smith, 2014). Primary sludge and SAS are typically thickened separately, by polymer addition and a mechanical thickening process, such as a thickening belt or drum thickener and are then mixed together, with imported sludges received at the WWTP, before entering AD.

Sludge treatment is necessary to stabilise the organic fraction, to reduce nuisance and odour, and to reduce pathogens and vector attraction, and to convert the material into a product suitable and acceptable for beneficial recycling to land, usually for agricultural application. Anaerobic digestion is one of the most successful, practical and cost-effective methods for achieving these treatment objective, and the importance of the process has increased significantly due to its ability to also produce a renewable energy source in the form of biogas. Advanced AD processes are designed to increase the treatment capacity of the conventional system, and also provide other possible benefits. For example, the thermal hydrolysis process (THP) is one of the most widely deployed advanced AD processes involving a pretreatment stage to improve sludge digestibility, dewaterability and pathogen removal (Barber 2016). Mesophilic anaerobic digestion is controlled principally by modifying the digestion temperature, hydraulic retention time (HRT) and feed sludge dry solids (DS) concentration, to maintain a stable operation at full-scale site. The biogas produced from digesters is used on-site by combined heat and power plant (CHP) to generate heat and electricity, which can be used locally for digester heating and on-site electricity consumptions, surplus electricity may also be exported to the grid. The digested sludge is dewatered and recycled to land for beneficial use, or it may be supplied to a sludge incineration plant.

The wastewater sector has a long history of recycling sludge to agricultural land, successfully increasing proportion recycled to land from 44% in 1992 to 80% in 2010 in the UK (Ofwat, 2016). Recycling treated sewage sludge to agricultural land is recognized as the Best Practicable

Environmental Option for sludge management (Kroiss and Zessner, 2007). In Europe, recycling sludge to agriculture is a highly regulated activity by the EU Sewage Sludge Directive 86/278/EEC (1986) and, in the UK, the Directive was implemented in 1989 through The Sludge (Use in Agriculture) Regulations (SI, 1989 amended in 1990). Sludge management is further supported by the Code of Practice for the Agricultural Use of Sewage Sludge (DoE, 2018), which provides a further control measures and details of all aspects of sludge recycling to land. Furthermore, the Safe Sludge Matrix (ADAS, 2001) prescribes the operational and numerical microbiological parameters for sludge treatment processes as well as land-use restrictions of biosolids in relation to their classification. Two generic types of treated sewage sludge are defined by the Safe Sludge Matrix, and they are conventionally treated sludge and enhanced treated sludge. The conventionally treated sludge should ensure at least 2 log pathogen destruction, determined by the removal of the indicator bacteria, *Escherichia coli*, while the enhanced treated sludge should be free from Salmonella and be treated to ensure 6 log reduction of *Escherichia coli* (*E. coli*). The enhanced treated sludge can be achieved by advanced AD using pretreatment methods including: pasteurisation, THP, heating, pasteurisation and hydrolysis (HpH) process, or by the enhanced enzymic hydrolysis (EEH) process.

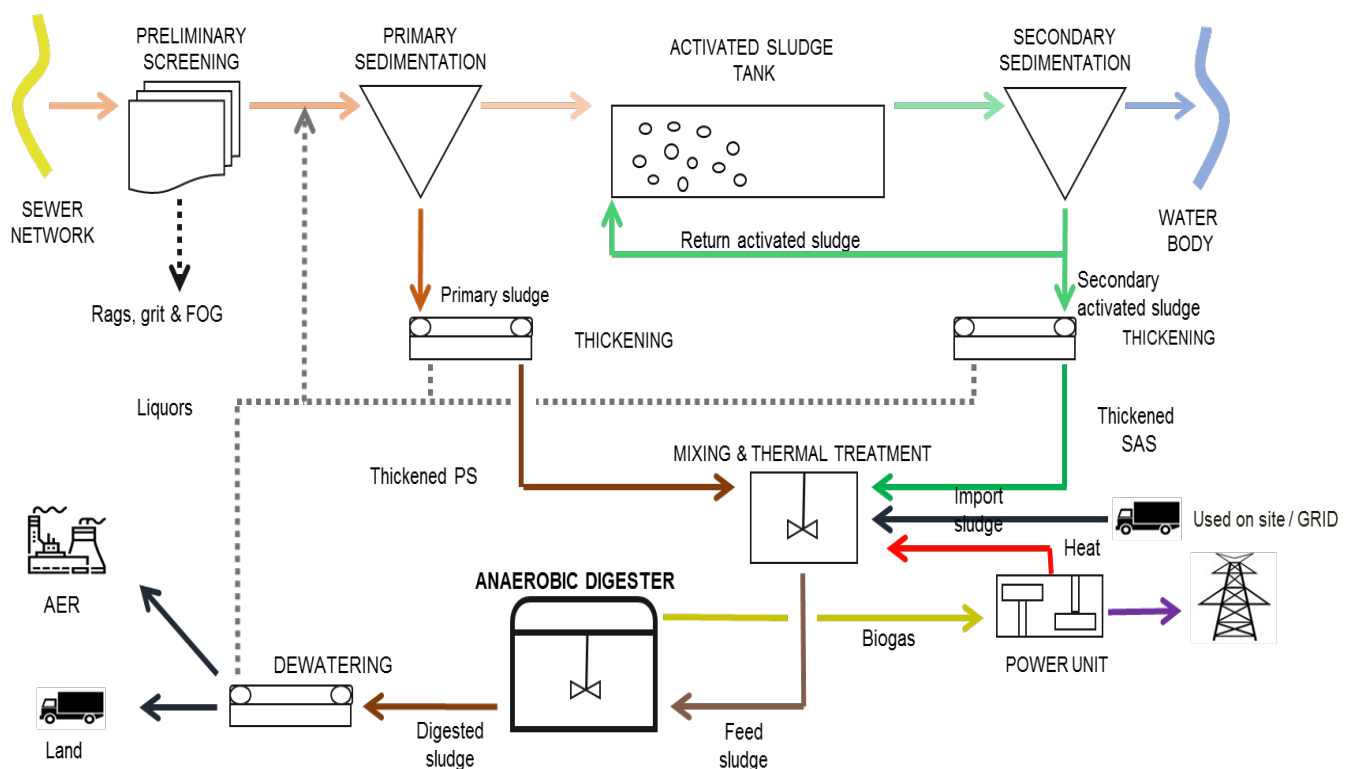


Figure 2-1 Typical process diagram of wastewater treatment with anaerobic digestion

2.2. Fundamentals Aspects of the AD Process

2.2.1. Microbiological and biochemical stages in AD

Anaerobic digestion is the process of decomposition of organic materials by a complex mixture of symbiotic microorganisms under an oxygen-free environment (Ward *et al.*, 2008). It is a naturally occurring process, mainly found in watercourses, sediments, or waterlogged soils, where oxygen is absent.

The AD of sewage sludge takes place in an enclosed reactor, where the temperature can be maintained within an optimum range. A methane (CH₄) rich biogas is produced, typically containing 60% CH₄, 40% carbon dioxide (CO₂), and trace amounts of water vapour and hydrogen sulphide (H₂S) (Chynoweth *et al.*, 2001).

The AD process has four key microbiological and biochemical stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Figure 2-2). Catalysed by a mixed group of bacteria, this series of reactions gradually converts biodegradable organic carbon present in biopolymer and other biodegradable materials into its most-oxidised form, carbon dioxide (CO₂), and its most-reduced form, CH₄, in the absence of oxygen (Madsen *et al.*, 2011). There is also a residual organic fraction, which is stable and not biodegradable, which leaves the process as a component of the residual, treated sewage sludge. The hydrolysis step consists of chemical reactions involving the splitting of covalent bonds with the addition of the hydrogen (H⁺) cation and the hydroxide (OH⁻) anion from water. This is a critical step to convert complex and insoluble organic polymer molecules into their component monomer molecules, which are a size and form that can pass through bacterial cell walls for use as energy or nutrient sources (Parkin and Owen, 1986). This process also involves the catalysis by extracellular enzymes such as lipases, proteases, and hydrolases. During acidogenesis, the monomer products from the hydrolysis step, including fatty acids, amino acids, and sugars, are metabolised by fermentative bacteria producing volatile fatty acids (VFAs) such as acetate, butyrate, and propionate, with simpler organic compounds such as alcohols, as well as CO₂ and hydrogen (H₂). During the acetogenesis step, acetate and hydrogen are produced by obligatory hydrogen producing acetogenic (OHPA) bacteria. Finally, CH₄ is produced from acetate or H₂ and CO₂ by specialised methanogenic bacteria.

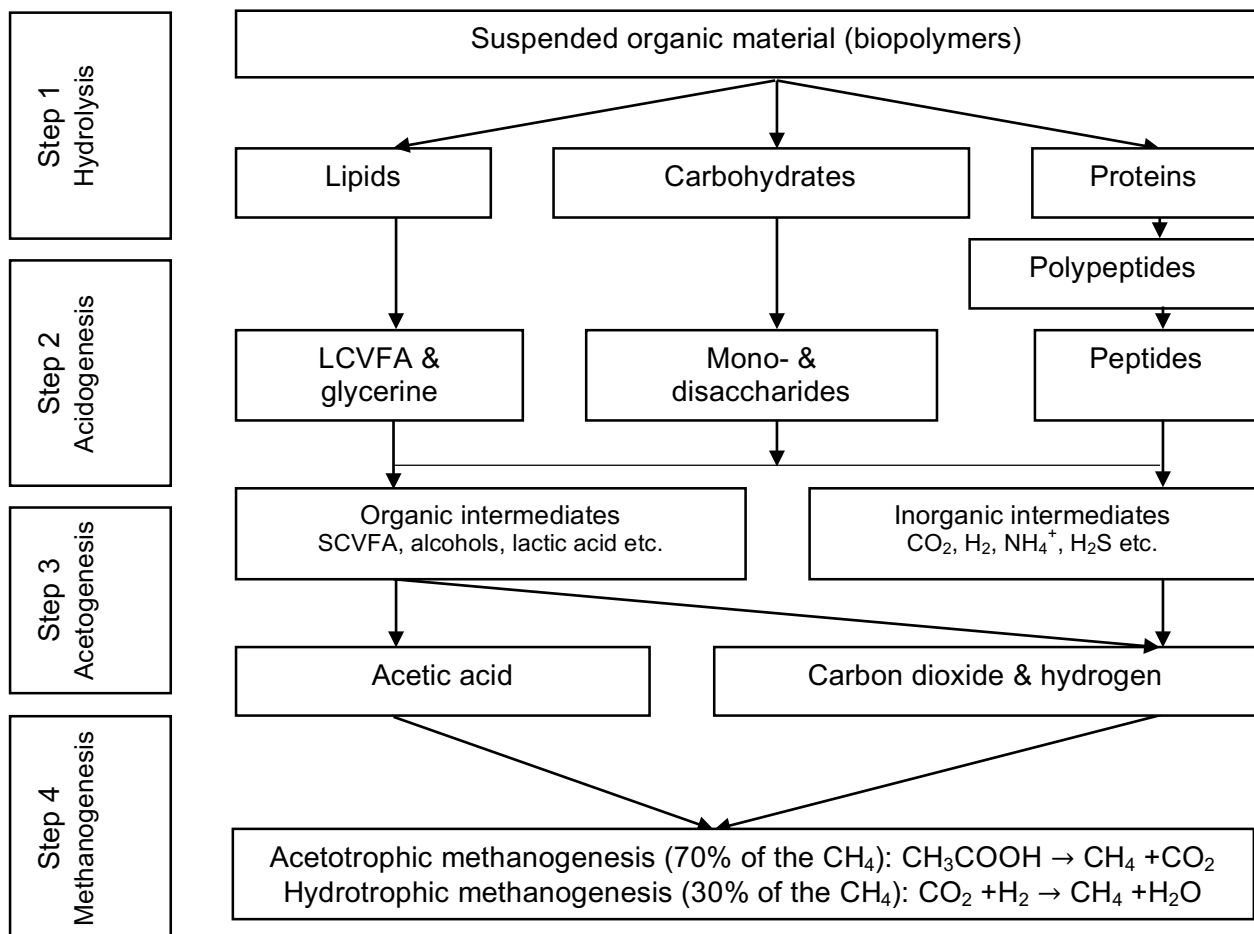


Figure 2-2 Four principle reaction steps involved in the decomposition of organic matter by anaerobic digestion (Madsen *et al.*, 2011)

The reaction times required for the individual steps of the AD process differ significantly. Hydrolysis reactions are usually considered as the main rate limiting step of AD processes, and their duration depends on the chemical environment and substrate complexity; in a well-operated AD process, they usually require a period of hours to days to complete. In contrast, methanogenesis proceeds in seconds to minutes, hence there is an instantaneous conversion of substrate entering methanogenesis. Thus, the main rate limiting step to the overall process is the hydrolysis stage of insoluble organic substrates (Eastman and Ferguson, 1981), and well-managed AD systems usually maintain low levels of VFAs (Pind *et al.*, 2003). In the digestion of soluble organic matter, however, the rate limiting step is methanogenesis (Ma *et al.*, 2013), as methanogenic bacteria are extremely fastidious and sensitive to changes in pH, alkalinity, temperature and concentrations of potentially inhibitors, such as ammonia (NH₃) (Gerardi, 2003). Therefore, it is essential to monitor and maintain the operational conditions within optimum ranges to maintain a stable operation. However, defining the optimum process control conditions for AD is challenging, because the operational conditions and parameters are strongly interrelated and changes in one may directly or indirectly affect the others. Furthermore, due to the presence of multiple, and dynamic bacterial groups within AD, the

optimum operational conditions can vary between these different groups of bacteria (Gerardi, 2003). The effect of operational condition on AD will be discussed in details in the following sections.

2.3. Critical Factors Influencing AD

2.3.1. Temperature

Digestion temperature has a significant influence on the physico-chemical and biological conditions within the digester environment. For instance, it has a major influence on the microbial population dynamics, controlling the metabolism and growth rate of microorganisms; methanogens are particularly sensitive to digestion temperature changes due to its influence on growth and metabolic activity (Appels *et al.*, 2008; Wang *et al.*, 2019). For example, Wang *et al.* (2019) gradually reduced the temperature of a semi-continuous methanogenic digester from 35 to 20 °C and found that both total biogas volume and CH₄ content dropped sharply below 25 °C (Figure 2-3), indicating the significant inhibition of methanogenic bacteria metabolic activity at low digestion temperatures. The major benefits of increasing temperature are enhancing biological and chemical reaction rates, increasing solubility of the organic compounds and increasing the pathogen reduction rate (Smith *et al.*, 2005; Appels *et al.*, 2008; Lang and Smith, 2008). However, increasing digestion temperature can also raise the concentration of free ammonia (NH₃), which is potentially inhibitory to the AD process (Chen *et al.*, 2008). At constant total ammonium (NH₄⁺) concentration of 4200 mg N/L and a pH of 7.65, Marcelo *et al.* (2009) found that the free NH₃ concentration was almost doubled at 35 °C, and equivalent to 200.4 NH₃-N/L compared to 102mg NH₃-N/L at 25 °C for the AD of swine manure. The general opinion (Braun *et al.*, 1981; Parkin and Miller, 1983) is that thermophilic anaerobic digestion (50-60°C) is more vulnerable to the inhibitory effects of high NH₃ concentrations than operating at the mesophilic temperature range (30-40 °C) for AD. This is because the ratio of free NH₃ to the total NH₄⁺ will be much higher at higher temperatures, and hence higher temperatures can inhibit methanogenesis for CH₄ production in anaerobic digesters. Thus, NH₄⁺, urea⁻, and protein-rich wastewaters are difficult to treat under thermophilic conditions. Bocher *et al.* (2008) showed that the average free NH₃ of protein-rich brewery wastewater under mesophilic and thermophilic AD were 74.1 and 321 mg NH₃-N/L, respectively, and that thermophilic digesters also showed more periods of unstable conditions as free NH₃ exceeded 400 mg NH₃-N/L and caused inhibition to methanogens.

Turovskiy and Mathai (2006) recommended that a stable and uniform temperature is necessary to avoid temperature effects on AD bacteria as methanogens are sensitive to frequent fluctuations in temperature and process failure is possible when temperature changes >1 °C/day are experienced. Chae *et al.* (2008) examined the effect of fluctuating temperature conditions on the operational stability and biogas production rate of swine manure digestion in a batch process for 500 h. In a digester normally operated at 35 °C, the temperature was suddenly lowered to 30 °C, maintained for

170 h, and then raised again to 32 °C till the end of the test (Figure 2-4). As would be expected in a batch process, a consistent reduction in biogas production rate was observed for the control group, due to the consumption of available substrates. The temperature shock, from 35 to 30 °C, led to a further decrease in the biogas production rate compared to the control, which was operated at a constant 35 °C. The biogas production rate resumed to the value of the control reactor in about 40 h. The second temperature shock, from 30 to 32 °C, again showed a decrease in the biogas production rate, but returned to its former level more rapidly. However, no lasting damage was seen in the digestion performance, even with fairly large changes in temperature. The temperature shock reactor showed only 7.2% less ultimate gas production compared to the control under the excessive HRT conditions (500 h). Therefore, although methanogens are relatively sensitive to temperature, they have considerable ability to adapt to temperature changes (Chae *et al.*, 2008). This result suggests that an effective adaptation of the mesophilic bacteria to different temperatures is possible as long as the change occurs smoothly.

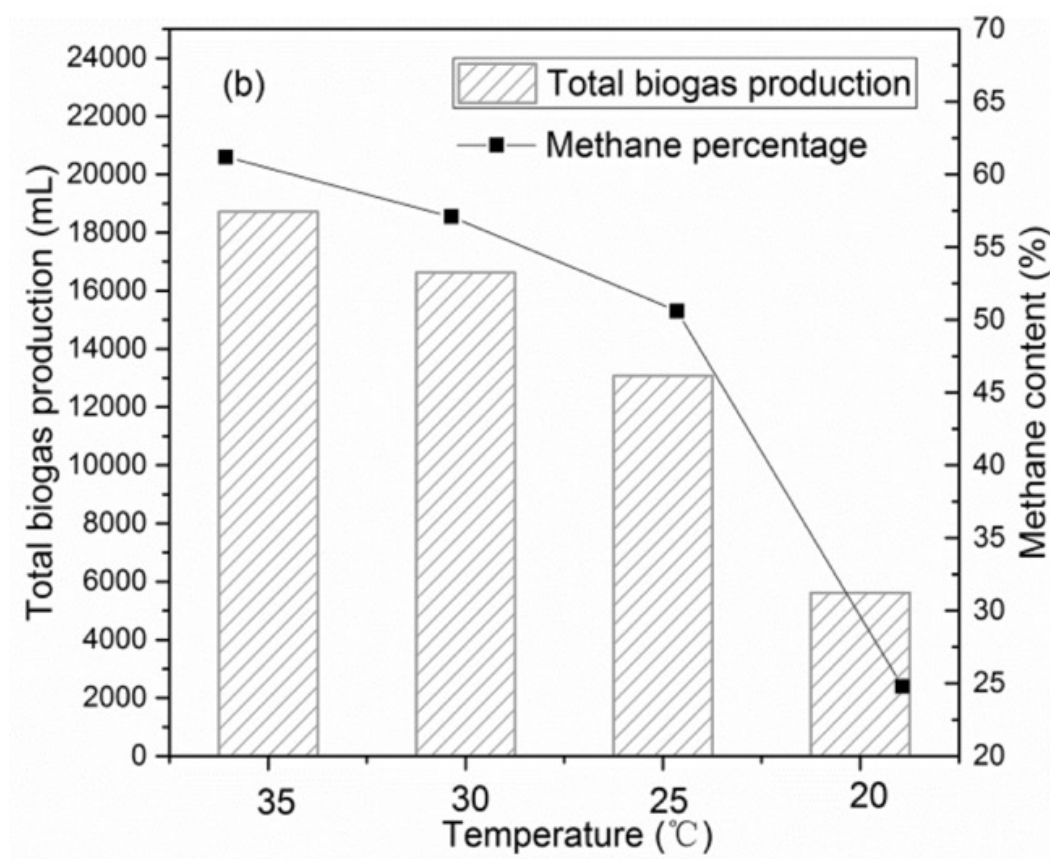


Figure 2-3 The effect of temperature on the total biogas volume and CH₄ content produced from the methanogenic phase of anaerobic digestion, using the acidizing mixture of cow manure and corn straw from an acidogenic reactor (Wang *et al.*, 2019)

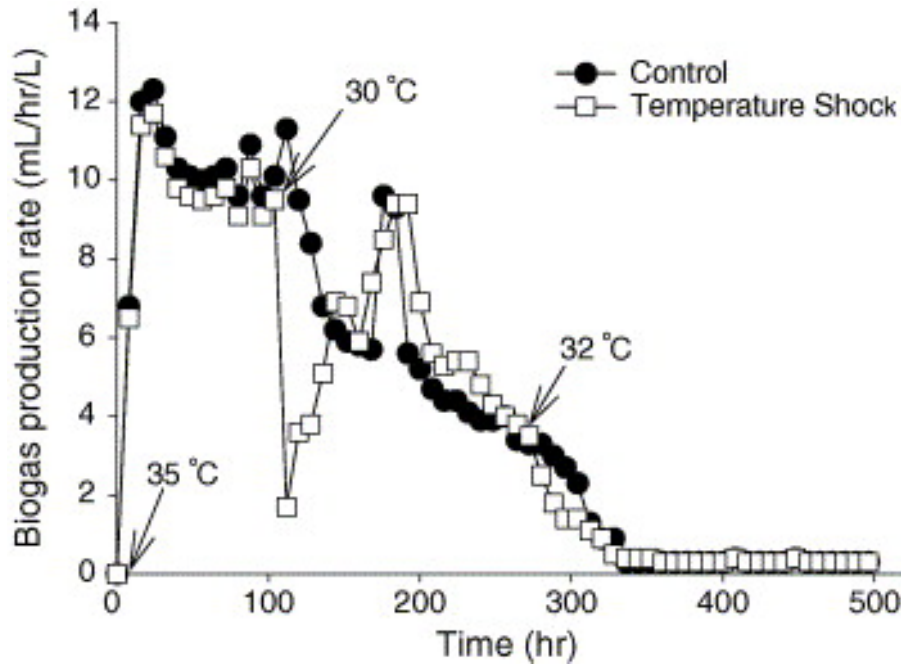


Figure 2-4 Influence of temperature shock on the digestion of swine manure at a 20% (v/v) feed load in batch digesters operated for 500 h (Chae *et al.*, 2008)

2.3.2. Dry solids, volatile solids, hydraulic retention time and organic loading rate

Volatile solids (VS), DS, HRT and organic loading rate (OLR) are four routinely measured parameters at wastewater treatment plants (WWTPs) which are used to control digester performance. Dry solids is a measurement of dry matter in sludge of both organic and inorganic matters and is determined by drying the wet sludge at 103 - 105 °C in a drying oven until it reaches a constant mass. Volatile solids is the amount of organic matter in sewage sludge and represents around 75% of the total DS in feed sludge that are lost on ignition at 550°C; the remaining solids are called fixed solids which are mostly non-biodegradable (Mattocks, 1984). Hydraulic retention time is the average time that liquid sludge is retained within the digester, and can be expressed as (Equation 2.1)

$$\text{HRT} = \frac{m_{\text{reactor}}^3}{\text{daily feed volume} \left(\frac{m^3}{\text{day}} \right)} \quad (2.1)$$

The organic loading rate refers to the amount of organic matter fed to a digester per unit of time, which can be expressed as a function of feed volume, DS, VS, digester volume and HRT (Equation 2.2).

$$\begin{aligned} \text{OLR} \left(\frac{\text{kgVS}}{m^3 * \text{day}} \right) &= \frac{\text{daily feed volume} \left(\frac{m^3}{\text{day}} \right) * \text{DS}(\%) * \text{VS}(\%) * 10}{m_{\text{reactor}}^3} \\ &= \frac{\text{DS}(\%) * \text{VS}(\%) * 10}{\text{HRT}} \end{aligned} \quad (2.2)$$

The OLR is a useful parameter to represent the total throughput of organic matter to a digester, which combines the effects of DS, VS and HRT. A critical objective of WWT operation is to increase the OLR and the total throughput of sludge to maximise site intensification. This is achieved by increasing the DS of the feed sludge, and minimising the HRT, which are under the control of the operator, or by increasing the VS concentration in the feed sludge, which is not usually possible to control. However, increasing OLR can overload the digester, which inhibit the methanogens by accumulation of VFA and process acidification, and hence disrupt the AD process (Meegoda, 2018). Although HRT, DS and VS contribute to the OLR, the impact of these parameters on process performance can vary between different digesters (Parkin and Owen, 1986; Rus, *et al.*, 2013; Alepu *et al.*, 2016).

The HRT is one of the key factors for bacteria growth and is strongly linked to digestion performance (Alepu *et al.*, 2016). Adequate retention time is necessary to allow substrate metabolism and to prevent washout of AD bacteria, and hence to maintain a sufficient quantity and concentration of bacteria in the digester for the efficient conversion of complex organic matter to CH₄ and CO₂ (Parkin and Owen, 1986). The relationship between digester performance and digester retention time is presented in Figure 2-5, based on a large number of digestion systems operating in the US (Parkin and Owen, 1986). Volatile solids reduction (VSR) in AD is positively related to both retention time and feed sludge VS concentration, and a rapid drop of VSR rate at 10 days in Figure 2-5 indicates the depletion of easily digestible substrates. However, other factors, such as digestion temperature, primary:SAS ratio, and different pretreatment methods can also affect digestion performance, and the relationship between HRT and VSR. Thus, effective thickening of digester feed sludge to raise the DS content, and thus the VS concentration on a wet sludge with an adequate HRT, to allow complete metabolism of degradable substrates, increases the overall VSR (Figure 2-5).

Dry solids content of feed sludge is another key factor influencing the performance of AD, and conventional MAD systems typically receive sludge with DS contents in the range of 2 - 6% (Appels, *et al.*, 2008). Rus *et al.* (2013) showed a 50% improvement in maximum specific CH₄ yield from 150 to 225 m³ CH₄/t VS, with increasing OLR from 1.0 to 4.0 kg VS/m³/d by adjusting DS feed and HRT in a series of batch digestion tests of mixed primary and SAS (3:2) under 37°C. No further improvement was observed as OLR increased above 4.0 kg VS/m³/d, and this may be explained because microbial access to substrates and mixing can be impaired with increasing solids loading (Cheng and Li, 2015; Parkin and Owen, 1986), and higher DS concentrations in feed sludge can negatively influence CH₄ production and raise the cost and operational challenges of conventional AD systems associated with heat transfer, and sludge mixing and pumping (Jolis, 2008). For example, Zhang *et al.* (2015) studied the influence of DS on CH₄ production through a series of static AD experiments at DS contents of 3% - 15%. They suggested that 6% of DS was the performance boundary between low and high solids AD, as the accumulative CH₄ yield declined exponentially for DS values larger than 6%. Increasing the DS can cause an exponential rise in sludge viscosity but

also an exponential decrease in the diffusive coefficient, and hence reduce microbial access to substrates within the AD system (Liao, *et al.*, 2014; Zhang *et al.*, 2015). Consequently, NH_3 and VFAs may accumulate within the system and result in a reduced CH_4 yield in high-solids AD (Zhang *et al.*, 2015). The increased DS was also reported to affect methanogenic activity. Lay *et al.* (1997) showed the relative activities of methanogens decreased by about 53% and the lag phase during the start-up period was prolonged when the DS concentration of feed sludge increased from 4% to 10%. A similar result was reported by Fernández, *et al.* (2010); for batch operated processes, the highest biogas production was detected in the period 15-40 days with 20% DS concentration, whereas the maximum production was observed during the period 35-75 days for 30% DS concentration.

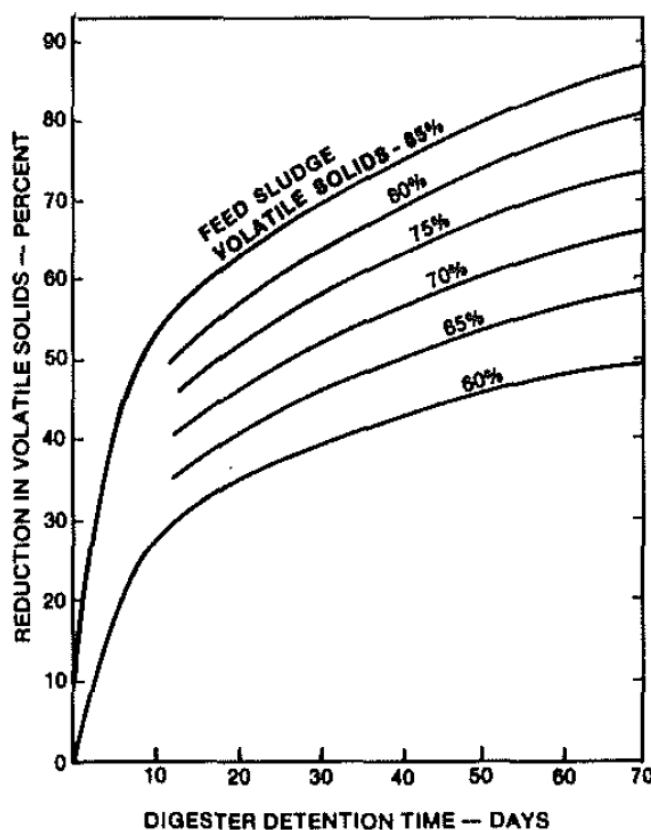


Figure 2-5 Relationship between volatile solids reduction, feed sludge volatile solids concentration and solids retention time (Parkin and Owen, 1986)

2.3.3. Digester mixing

Effective mixing of sludge digesters is essential to provide a uniform temperature and substrate concentration, and to maintain intimate contact between the feed sludge, bacterial population and enzymes. Furthermore, mixing also ensures the efficient utilisation of the whole digester volume, preventing the formation of surface scum layers and also the deposition of sludge at the base of the digester. Therefore, providing a uniform environment by effective mixing is critical for digestion process optimisation. Inefficient mixing reduces the effective digester volume and therefore the HRT.

Insufficient retention may lead to the wash out of bacteria and eventually to system failure. Monteith and Stephenson (1981) studied the performance of full-scale AD and showed that inefficient mixing could reduce the effective digester volume by 50%. Parkin and Owen (1986) examined the effects of different temperature conditions and mixing efficiencies on the stability of digester output residues, based on earlier data from O'Rourke (1968) (Figure 2-6). This showed that, at 20 days HRT, inefficient mixing reduced the effective digester volume by 50% and biochemical oxygen demand (BOD) removal by 5 %, compared to an effectively mixed system at an equivalent digestion temperature of 35°C. Reducing the digestion temperature by 5°C reduced the BOD removal by 16% when the digester was inefficiently mixed. A large reduction in BOD removal efficiency, equivalent to 54%, was observed when the HRT was reduced to 10 days for inefficiently mixed digesters. A further reduction in digestion temperature to 30 °C caused the process to fail.

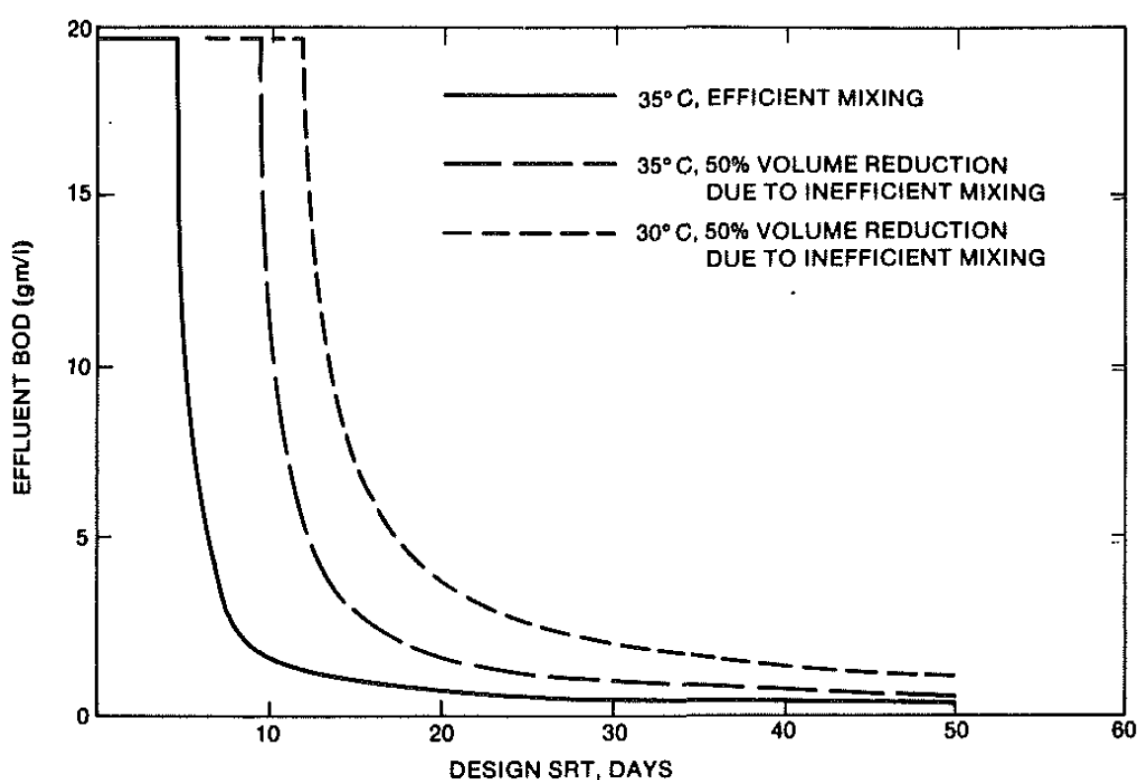


Figure 2-6 Effect of mixing on digester performance (Parkin and Owen, 1986) (SRT is equivalent to HRT in these semi-continuous flow through process, without a sludge biomass return flow)

Digester mixing efficiency can be effected by sludge viscosity. Viscosity is defined as the ratio of shear stress to shear rate, and can be calculated from the flow curves measured by rheometers, such as rotational and capillary rheometers (Eshtiaghi *et al.*, 2013). Sludge behaves as a non-Newtonian fluid, and its apparent viscosity is highly affected by temperature and DS concentration (Cao *et al.*, 2016). The effect of DS concentration on the viscosity of sludge has been examined in a great number of studies. It was found that the sludge viscosity increases with DS concentration. At higher DS contents, stronger inter-particle interactions are observed and, hence, a higher sludge

viscosity. This behaviour can be described by an exponential function or power model (Lotito *et al.*, 1997; Basim *et al.*, 2010). Moreover, decreasing particle size has a similar effect on sludge viscosity (Pevere *et al.*, 2006) to increasing the DS concentration, as both increase inter-particle interactions (Eshtiaghi *et al.*, 2013). Similarly, the temperature dependency of sludge viscosity has been well examined. In general, increasing temperature reduces the viscosity (Baudez *et al.*, 2013), but the response to temperature significantly reduced at room or lower temperature ranges (Moreau *et al.*, 2009). Thus, sludge viscosity increases with DS and decreases with temperature (Cao *et al.*, 2016), hence mixing energy requirements are raised at high sludge DS concentrations and low digestion temperature.

The mixing efficiency and effective digester volume can be evaluated by a tracer test by adding a known mass of tracer and studying the concentration change of the tracer in the effluent stream using an ion selective electrode, spectroscopy or other chemical analysis (Lindmark *et al.*, 2014). The commonly used tracer for anaerobic digesters is lithium chloride as it has no harmful effects on the AD process at low concentrations (Anderson *et al.*, 1991). Terashima *et al.* (2012) considered the mixing of non-Newtonian sludge in a full-scale digester (volume = 1100 m³) with mechanical draft mixing. The degree of mixing achieved in the digester was evaluated using a uniformity index, based on the ratio of the number cells containing tracer relative to the total cells in the mesh at any given time. As may be expected, the results demonstrated that increasing the rate of mixing produced a more uniform distribution of tracer throughout the digester. Furthermore, a comparison of the uniformity index across simulations with different sludge rheology showed that uniformity within the digester was achieved more quickly when mixing less viscous sludges compared to more viscous sludges.

The energy demand for mixing in a full-scale digester is substantial and can vary from 14% to 54% of the total energy demand of the AD plant (Rivard *et al.*, 1995; Kowalczyk *et al.*, 2013). The energy consumed by mixing is highly correlated to the mixing mode, for example, the energy demand may be significantly reduced by switching from continuous mixing to intermittent mixing. For example, Lindmark *et al.* (2014) observed a 29% energy reduction by mixing for 2 h and pausing mixing for 1 h compared to continuous mixing mode, and when mixing for 7 h and pausing for 1 h, the energy demand was reduced by 12%.

2.3.4. Ammonia

Ammonia is released from the biological degradation of nitrogenous organic matter, primarily from proteins and urea (Kayhanian, 1999) and is present as both free NH₃ and NH₄⁺ in aqueous solutions (Chen *et al.*, 2008). Ammonia can be beneficial for AD by supporting bacterial growth at low NH₃ concentrations (50-200 mg NH₄-N/L) (McCarty, 1964). However, high NH₃ concentration may cause severe inhibition of AD performance, resulting in reduced CH₄ production rates and the increase in concentrations of intermediate digestion products such as VFAs (Rajagopal, *et al.*, 2013). The upper

limit of NH_3 inhibition was approximately 3000 – 4000 mg $\text{NH}_4\text{-N/L}$ total ammoniacal-N concentration. McCarty (1964) reported that AD processes are inhibited at any pH when the ammoniacal-N concentration exceeds 3000 mg/L. Similarly, Hobson and Shaw (1967) reported that a total ammoniacal-N concentration equivalent to 3300 mg/L could inhibit methanogenesis completely. However, Sawayama *et al.* (2004), Procházka *et al.* (2012) and Lauterböck *et al.* (2012) consistently report a higher inhibition limit for methanogenesis, above 4000 – 6000 mg/L ammoniacal-N concentration. The wide range of inhibiting NH_3 concentrations reported in the literature may be explained because, inhibition is not only dependent on the total ammoniacal-N, but also to the free NH_3 .

Free NH_3 is generally considered as the most toxic form and the main cause of microbial inhibition due to its free permeability through cell membranes causing potassium (K) deficiency and proton imbalance (Sprott and Patel, 1986; Gallert *et al.*, 1998). The concentration of free NH_3 is mainly governed by total ammoniacal concentration, temperature and pH (Hansen *et al.*, 1998). Thus, as discussed earlier, increasing the digestion temperature may raise the free NH_3 concentration. Furthermore, the rise in alkalinity and pH associated with increasing $\text{NH}_3\text{-N}$ concentrations may also increase the ratio of free ionized NH_3 and the risk of microbial toxicity (Chen *et al.*, 2008). Kayhanian (1999) showed that the free NH_3 concentration increased six times at thermophilic temperatures (55 °C) compared to mesophilic temperature (35 °C) at the same pH. A similar study conducted by Fernandes *et al.* (2012) reported that the percentage of free NH_3 increased from less than 1% to 10% of total ammoniacal-N, by increasing the pH from 7 to 8 at 35 °C. Fernandes *et al.* (2012) also summarised the interactive relationship between free NH_3 , and total ammoniacal-N, temperature and pH as shown in Figure 2-7.

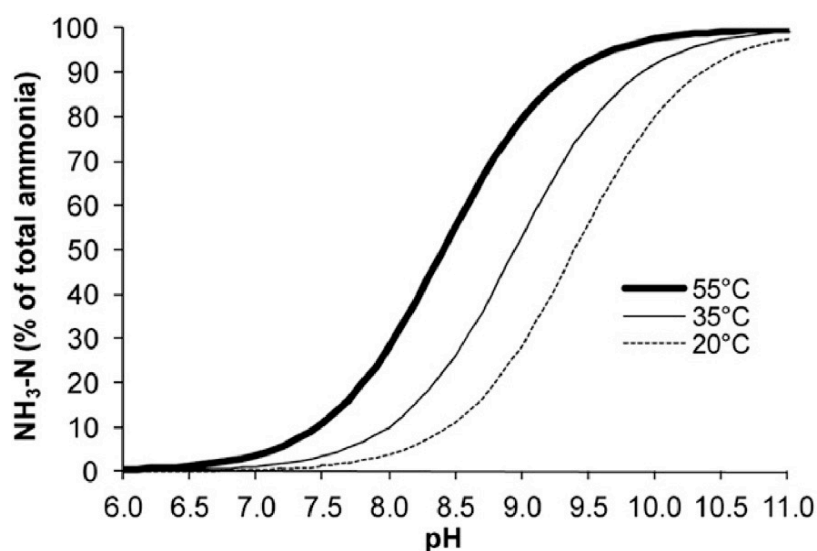


Figure 2-7 Proportion of free NH_3 and NH_4^+ present in solution at 20, 35 and 55 °C at varying pH (Fernandes *et al.*, 2012)

2.3.5. Sulphide

Many industrial effluents, such as those from the fermentation, paper pulp and food beverage industries contain high concentrations of sulphate (up to 40 g/L) (Colleran *et al.*, 1995). Sulphur is a required element for methanogenic archaea, and hence very low amounts of sulphate (1 to 25 mg S/L) are beneficial for methanogenesis (O'Flaherty *et al.*, 1999; Chen *et al.*, 2008; Siles *et al.*, 2010). Under anaerobic conditions, sulphate is reduced to sulphide by sulphate reducing bacteria (SRB), which are heterotrophic and utilise organic carbon sources for metabolism. Therefore, sulphate reduction during AD is generally undesirable because the competition with methanogens for carbon sources can reduce the CH₄ yield. Sulphate reducing bacteria can utilise a broad range of substrates and sulphate reduction has a thermodynamic advantage over methanogenesis for utilising hydrogen and acetate (Paulo *et al.*, 2015) as presented in the Equation 2.3 – 2.6 below.

Sulphate reduction:



$$\Delta G^{o'} = -151.9 \text{ kJ}$$



$$\Delta G^{o'} = -47.6 \text{ kJ}$$

Methanogenesis:



$$\Delta G^{o'} = -135.6 \text{ kJ}$$



$$\Delta G^{o'} = -31 \text{ kJ}$$

Where $\Delta G^{o'}$ is Gibbs free energies at 25 °C calculated at standard conditions (solute concentrations of 1 M and gas partial pressure of 10⁵ Pa).

Accumulated hydrogen sulphide (H₂S) is also toxic to various bacteria groups (Chen *et al.*, 2008) and particularly towards methanogens. Hydrogen sulphide diffuses across the cell membrane and is responsible for protein denaturation, enzyme inhibition, and interference with the sulphur uptake metabolism (Paulo *et al.*, 2015). The toxicity response is strongly dependent on pH, for example, at pH <6 it is mainly present in the toxic form as H₂S, whereas at higher pH (8 -12), deprotonation produces the less toxic HS⁻ form (Lens *et al.*, 1998). Thus, the reported half maximum inhibitory concentration (IC₅₀) for methane producing bacteria was 250 mg H₂S/L at pH 6.4 – 7.2 and 90 mg H₂S/L at pH 7.8 – 8.0, respectively (Chen *et al.*, 2008). The concentrations of H₂S found in influents to WWTPs can also be influenced by the length of the sewer network. Microbial sulphate reduction

occurs under the anaerobic conditions encountered in gravity sewers, and a higher concentration of H₂S reaching WWTPs can be expected with longer sewers (Carrera *et al.*, 2015).

2.3.6. pH

Different bacteria groups have varying pH optima and methanogenic bacteria are particularly sensitive to pH change, whereas acidogenic types tolerate a much wider range of pH conditions (Appels *et al.*, 2008). The optimal, tolerable and inhibitory ranges of pH for different groups of AD bacteria are presented in Table 2-1. Furthermore, the pH can also have different impacts on bacterial growth rate under different substrate concentrations (Figure 2-8) (Pavlostathis and Gossett, 1986).

Changing pH conditions can alter the concentration and balance of both free NH₃ and VFAs. Thus, the increased toxicity caused by high free NH₃ can affect process stability, and cause the accumulation of VFAs. For example, Angelidaki and Ahring (1994) observed rapid increase in VFA concentrations (5000 mg/L) due to the inhibition caused by high free NH₃ concentrations (700 mg/L) at thermophilic temperatures. Accumulated VFAs reduce the pH value, and therefore lower the concentration of free NH₃. This condition is described as an “inhibited steady state”, where the AD process is apparently stable, but with a reduced CH₄ production (Angelidaki and Ahring, 1993; Angelidaki *et al.*, 1993).

Table 2-1 pH tolerance range for different AD biological steps (Gerardi, 2003; Hwang, *et al.*, 2004; Nielsen, 2006 and Giacalone, 2018)

Biochemical group / pH	4	5	6	7	8	9
Hydrolysis	Red	Yellow	Green	Green	Green	Green
Acidogenesis	Yellow	Yellow	Green	Green	Yellow	Red
Aceticlastic / hydrogen methane	Red	Yellow	Yellow	Green	Green	Red
Aceticlastic methanogenesis	Red	Red	Yellow	Green	Green	Red

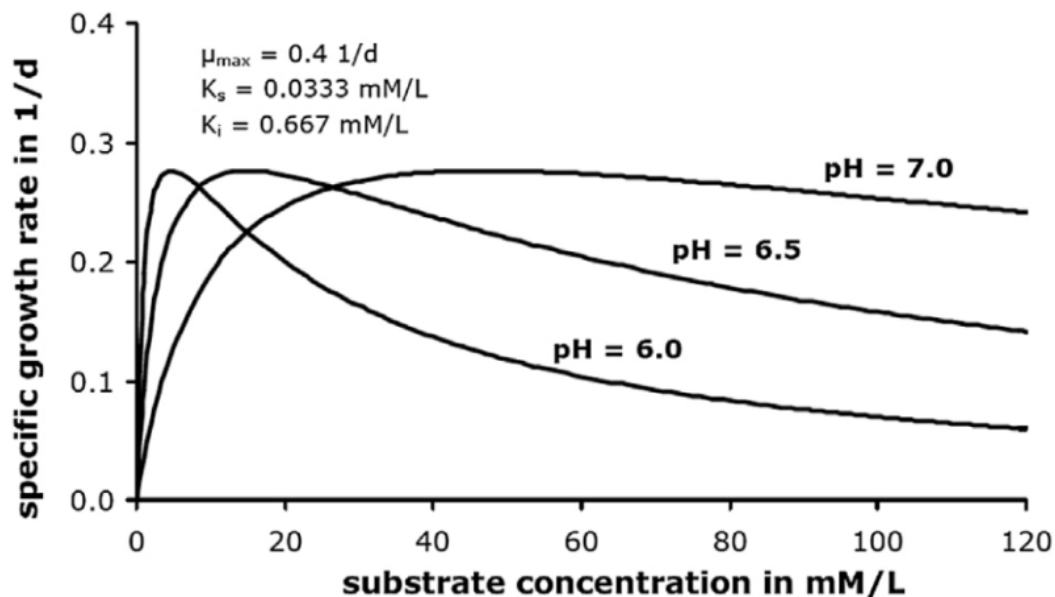


Figure 2-8 Maximum specific bacterial growth rate depending on substrate concentration for different pH values derived from Pavlostathis and Gossett (1986) kinetic model for AD of biological sludge

2.3.7. Iron dosing

Iron (Fe) salts can be used as a precipitant for chemical phosphorus (P) removal (CPR) to remove P from wastewater, generating sludge rich in P and Fe. The effect of CPR, especially when Fe is used as a precipitant, on AD performance has been widely discussed in the literature. Several authors (Grigoropoulos *et al.*, 1971; Ghyoot and Verstraete, 1997) found that CPR had no significant effect on AD performance, while others (Dentel and Gosset, 1982; Gosset *et al.*, 1978) showed that CPR may adversely affect the AD process and that iron-rich sludge generates less biogas than sludge without Fe dosing. Dentel and Gosset (1982) reported that using ferric chloride (FeCl_3) in WWT reduced the anaerobic digestibility and gas production of the resulting sludge. Gosset *et al.* (1978) similarly showed that FeCl_3 dosing for CPR reduced the performance of AD in several ways, including reduced: total gas production, CH_4 production, chemical oxygen demand (COD) and VSR. Both studies suggested the adverse effect of Fe dosing on AD was not attributed to Fe toxicity, but to the decreased biodegradability of substrates. These results contrast with the results of Lagrotta (2015), which showed increased digestibility and gas production with Fe dosing in primary WWT. Particle size analysis of sludge collected with Fe-dosing (ferric sulphate ($\text{Fe}_2(\text{SO}_4)_3$) and FeCl_3) into the primary WWT process showed that this response was attributed to greater capture of fine, highly digestible particulate material. Anecdotal evidence from operational experience within Thames Water (Paul Fountain, pers. comm., 2017) supported these findings and similarly suggested Fe dosing into primary WWT tanks not only increased primary sludge production, but proportionally raised the biogas yield (BY) from sludge AD. Thus, the impact of Fe-dosing on sludge characteristics and AD may depend on the location where Fe is introduced and it is apparently beneficial if supplied

in primary WWT. However, Lagrotta (2015) did not detect any negative effects of introducing Fe directly into sludge immediately predigestion.

2.3.8. Sludge type and age

Under optimum digestion condition, a typical CH₄ yield of 190 -240 Nm³/t VS can be expected for SAS, which is much lower compared to primary sludge, which has a typical CH₄ production rate of 315 - 400 Nm³/t VS under equivalent digestion conditions (Bachmann *et al.* 2015). The biodegradability of SAS is lower than primary sludge due to its biological origin and lower availability to the anaerobic biomass, because of the inherent resistance to lysis of living cells and the large dimension of the flocs (Bolzonella *et al.*, 2005; Batstone and Jensen, 2011; Mottet *et al.*, 2010).

Sludge age refers to the number of days of ambient temperature storage for primary sludge and the retention time for activated sludge (AS) in the secondary WWT process. The sludge age can affect the BY of both primary sludge and SAS. The decrease in the degradability of SAS may be explained by the changing physicochemical properties of SAS biomass with aging. The SAS composition is also highly related to biological nutrient removal (BNR) strategies, such as temperature, aerobic/anoxic management, and especially the sludge age. Conventional BNR has a typical sludge age of more than 10 days, and the degradability can be less than 35% for this long sludge-age SAS (>15 days) (Gossett and Belser, 1982). By contrast, SAS generated from high-rate aerobic treatment processes has a very short sludge age (normally <5 days), and has a significant potential for CH₄ production in AD. Compared to long sludge-age SAS, the degradability of short sludge-age SAS has much better degradability equivalent to 85, 73, and 63% at 2, 3 and 4 days, respectively (Ge *et al.*, 2013). Consequently, a 30% increase in CH₄ production to 115 Nm³/t VS from 90 Nm³/t VS was observed for SAS by reducing sludge age from 12 to 4 days, and a further reduction of sludge age to 2 days showed an additional increase of CH₄ production to 170 Nm³/t VS (Ometto *et al.*, 2017).

2.3.9. Pretreatment

Pretreatments for sludge are designed to increase the capacity of conventional MAD systems, and also provide other possible benefits, such as better sludge digestibility and higher pathogen reduction. Different pretreatment methods have been developed and can be divided into the three main categories of: mechanical, thermal, and biological processes.

Mechanical pretreatments include the methods: sonication, high-pressure homogeniser and lysis-centrifuge, and are applied specifically to reduce the substrate particle size by disintegrating solids particles to release cell compounds and increase the specific surface area (Ariunbaatar *et al.*, 2014). Increasing the surface area provides better contact between anaerobic bacteria and substrate, and hence enhances the AD process (Carrère *et al.*, 2010; Ariunbaatar *et al.*, 2014). Ultrasound pretreatment uses a vibrating probe that mechanically disrupts the cell structure and floc matrix, and the mechanisms for ultrasonic treatment are different under low and high frequencies. The formation

of radicals such as $\text{OH}^\cdot$, $\text{HO}_2^\cdot$, $\text{H}^\cdot$ at high frequency cause the oxidation of solid substrates (Bougrier *et al.*, 2006). At low frequency (20-40 kHz), cavitation reduces the particle size; low frequency ultrasound is the most efficient method for sludge pretreatment (Carrère *et al.*, 2010).

Thermal treatment is one of the most studied pretreatment methods with successful full-scale industrial application. Pasteurisation and THP are two of the most widely used thermal pretreatment processes at operational WWTP. Thermal treatment was first applied to improve sludge dewaterability (Haug *et al.*, 1978), and it also leads to better pathogen removal and reduced viscosity of the digestate in the following treatment process. Thermal pretreatment at 70 °C is highly effective at pathogen reduction (Skiadas *et al.*, 2005). At full-scale WWTP, pasteurisation pretreatment at 70 °C is usually applied for at least one hour. However, this has minimal effect of MAD of primary sludge with respect to both CH_4 yield and production rate, but a 20% increase in CH_4 yield was observed for SAS under MAD after thermal pretreatment at 70 °C for 1 day (Gavala *et al.*, 2003). For a shorter retention time of one hour, Appels *et al.* (2010) showed that the CH_4 yield of SAS was not affected by pretreatment at 70 °C, but a significant increase in CH_4 yield was observed for a pretreatment temperature of 80 and 90 °C, also for one-hour retention time.

High temperature pretreatment of sludge is typically in the temperature range of 160 – 180 °C (Carrère *et al.*, 2010). Several studies have demonstrated the significant increased gas production by MAD following a high temperature pretreatment. For example, Li and Noike (1992) observed more than 100% increase of BY from 108 to 216 $\text{ml g}^{-1} \text{COD}_{\text{in}}$ for SAS in a continuous stirred-tank reactor (CSTR) (5 days HRT and 35 °C) following sludge pretreatment at 175 °C for one-hour. Similarly, a 51% increase in CH_4 yield from 145 to 256 $\text{ml g}^{-1} \text{VS}_{\text{in}}$ for SAS in CSTR (20 days HRT and 35 °C) with 175 °C 30 min pretreatment was observed by Bougrier *et al.* (2006). The THP is one of the most widely deployed, high temperature pretreatment processes to improve sludge digestibility, dewaterability and pathogen removal (Barber 2016). The full-scale THP MAD process chain involves thermal hydrolysis pretreatment at 160 and 180 °C, for a time period between 20 and 40 min, followed by conventional MAD (Barber 2016). Thermal hydrolysis breaks down cellular material and long chain organic fatty acids to increase sludge digestibility (Guireff *et al.* 2011). In addition, it reduces the viscosity of sludge improving mixing and enabling significantly higher solids loading rates, in the range 8 - 12% DS, with typical digester mixing equipment, compared to standard processes, which have a typical upper loading rate limit of 2 - 6% DS (Appels *et al.*, 2008; Whitlock *et al.*, 2009; Zupančič and Grilc 2012). One of the benefits of THP is to change the sludge rheology which allows higher loading rates, and significantly improves sludge dewaterability, thus increasing digestion volume capacity and reducing downstream transport and processing requirements. These benefits provide capital savings for the construction of new digestion plant and by expanding the capacity of existing digestion infrastructure (Abu-Orf and Goss, 2012). The process also meets enhanced status for pathogen removal (ADAS, 2001), enabling greater flexibility in recycling routes for the treated product (Barber 2016).

Biological pretreatment processes consist of a broad range of methods of both aerobic and anaerobic configurations. The aim of anaerobic pretreatment is to separate the hydrolysis and acidification stages from the following reactions of acetogenesis and methanogenesis, to provide optimal conditions for each AD reaction (Ariunbaatar *et al.*, 2014). Optimising the first hydrolysis stage by physically separating acidogens from the methanogens stimulates the acidogenic microbes to produce more specific enzymes to increase the degradation of substrates (Parawira *et al.*, 2005), and hence enhance the CH₄ production and COD removal efficiency at a shorter HRT compared to single-stage digesters (Hartmann and Ahring, 2006). The simplest configuration for biological pretreatment is two-stage AD, which consists of a hydrolytic-acidogenic stage followed by the methanogenic stage. The advantages of two-stage AD include, increased stability with better pH control, higher loading rate, CH₄ yield and VS destruction (Blonskaja *et al.*, 2003; Riau *et al.*, 2012).

2.4. Sewage Sludge Characteristics Influencing AD

2.4.1. Summary of factors influence biogas production during AD

The biodegradability and CH₄ production of organic compounds in full-scale sewage sludge digesters is affected by several physical, chemical and physiological factors, including the operational conditions discussed above and also the characteristics of the sewage sludge feed. Several complex characteristics of the sewage sludge feed may influence biogas production including the total quantity of organic matter in the sludge feed, usually measured and reported as the VS content as well as its composition (e.g. content of carbohydrates, lipids, proteins, etc.) and quality in terms of degradation efficiency (Chandler *et al.*, 1980; Hashimoto, 1986; Robbins *et al.*, 1979) (Figure 2-9). This is because different substrate types (proteins, fats and carbohydrates) vary in their CH₄ yield and produce different qualities of biogas during AD as summarised in Table 2-2 (Weiland, 2010; Li *et al.*, 2018). Therefore, the sludge composition will influence the theoretical CH₄ yield.

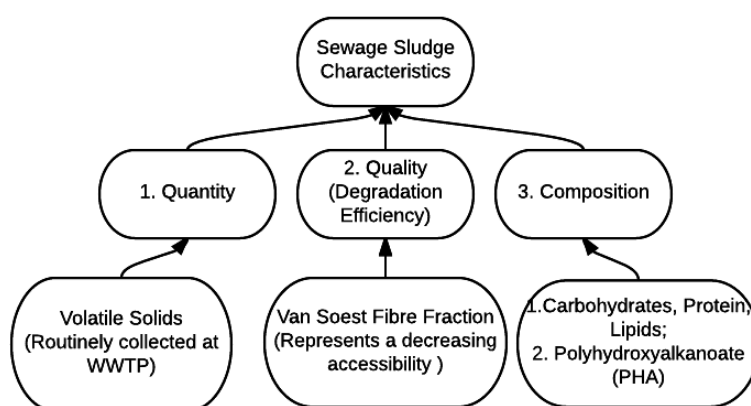


Figure 2-9 Sewage sludge feed characteristics influencing biogas production during AD (Chandler *et al.*, 1980; Hashimoto, 1986; Robbins *et al.*, 1979)

Table 2-2 Theoretical CH₄ yield and contents of biogas from different organic substrates (Weiland, 2010; Li *et al.*, 2018)

Substrate	CH ₄ yield (Nm ³ /t DS)	CH ₄ content of biogas (%)
Lipids	1014	67
Protein	496	70
Cellulose	414	50
Hemicellulose	409	50
Lignin	0	NA
Carbohydrates	400	50

2.4.2. Correlation between chemical composition and biochemical methane potential

Several studies have confirmed that the digestibility and CH₄ potential of different biomass types are highly correlated with their chemical composition. For example, batch trials carried out by Luna (2011) evaluated the biomethane potential (BMP) of 61 different agricultural/industrial substrates. The CH₄ yield of these agro-industrial substrates was markedly influenced by the lignin, hemicellulose, total protein and organic matter (VS) contents, while other variables such as lipids and cellulose did not have a significant influence (Table 2-3). As expected, VS content and protein were the main parameters, which positively affected CH₄ yield. Hemicellulose correlated positively with CH₄ yield, although the relationship was poor. No significant correlation was found between cellulose and CH₄ yield. Both cellulose and hemicellulose can be converted to CH₄ by AD. However, the conversion rates depend mainly on whether they are in a crystalline form or lignin-incrusted (Klimiuk *et al.*, 2010). Lignin content was the only variable that significantly reduced both the CH₄ yield and kinetic rate constant. These results agree with other reports (Weiland, 2010, Klimiuk *et al.*, 2010) showing that lignin is a complex plant constituent with very low CH₄ yield and production rate, which is very difficult to digest by anaerobic bacteria.

Table 2-3 Pearson's correlation of cumulative CH₄ yields and kinetic rate constants with the chemical composition of agro-industrial substrates (Luna, 2011)

Variable	n	Cumulative methane yield		Kinetic rate constant	
		r	p	r	p
TS	60	-0.168	0.221	-0.109	0.413
VS	60	0.785	<0.001*	0.033	0.8
TOC	7	0.36	0.427	0.425	0.401
Total protein	37	0.767	<0.001*	0.249	0.136
Fats	7	0.365	0.421	-0.139	0.765
Hemicellulose	45	0.343	0.029*	-0.514	<0.001*
Cellulose	20	-0.1	0.722	-0.505	0.023*
Lignin	18	-0.917	<0.001*	-0.789	0.008*
P	26	-0.473	0.016*	0.741	<0.001*
Ca	26	-0.563	0.002*	0.702	<0.001*
Mg	26	0.059	0.771	0.513	0.007*
K	26	-0.613	<0.001*	0.764	<0.001*

*: Statistically significant correlations ($p < 0.05$); r: Pearson's correlation coefficient; p: probability

For a mixture of energetic crops, pig manure slurry, agro-industrial waste, and the organic fraction of municipal solid waste (OFMSW) in a full-scale biogas plant, Schievano *et al.* (2008) found that the BMP was significant and positively correlated with VS, total organic carbon (C)/total Kjeldahl N ratio, oxygen demand in a 20-h respirometric test (OD20), total organic carbon (TOC), total solids (TS), and cell solubles (dry matter minus neutral detergent fibre), and significant negatively correlated with NH₃, total Kjeldahl N, NH₃/total Kjeldahl N ratio, total P, acid detergent fibres, and lignin.

By contrast, Gunaseelan (2007) found only a weak correlation between the lignin content and the BMP of fruit and vegetable solid waste (FVSW) ($R^2 = 0.49$), and sorghum and napier grass ($R^2 = 0.37$) when considering only this single compositional characteristic of these substrate types. This may be explained by the interactive effects of other biomass constituents which were not considered, or was due to the limited range of lignin contents in the biomass materials investigated in this study.

2.4.3. Correlation between the sewage sludge composition and biochemical methane potential

2.4.3.1. Sludge macromolecular composition

Only a limited number of published papers are available that consider the effect of sewage sludge composition on anaerobic biodegradability and CH₄ potential (Mottet *et al.*, 2010; Astals *et al.*, 2013). Astals *et al.* (2013) collected seven mixed sewage sludge samples from different WWTPs operating AD processes and examined the relationship between sewage sludge composition and biodegradability. The organic constituents and ultimate CH₄ production for the different sewage sludge samples are presented in Table 2-4, where the ultimate CH₄ represents the cumulative CH₄ production for 90 days of incubation. The BMP tests showed the ultimate CH₄ production varied and was in the range 324.5 - 379.7 mL CH₄/g VS_{fed}. However, no statistically significant relationship was found between the ultimate CH₄ production and the sewage sludge characteristics (protein, lipids (fats) and carbohydrates). These represent major constituents of the biodegradable fraction in sludge, but the lack of significance could be explained because CH₄ production was insensitive to the range of protein, carbohydrate and lipid contents found in this study (see Figure 2-10).

Table 2-4 Characterisation and ultimate CH₄ production of mixed primary sludge and SAS samples from seven WWTPs in Barcelona metropolitan area at 90 days of incubation (Astals *et al.*, 2013)

Protein (%DS)	Carbohydrates (%DS)	Lipids (%DS)	B ₀ VS (mL CH ₄ /g VS _{fed})	B ₀ COD (mL CH ₄ /g COD _{fed})
6.2 -13.52	4.5-17.2	2.23-7.96	324.5-379.7	188.3-214.2

B₀VS: Ultimate CH₄ production based on VS feed; B₀COD: Ultimate CH₄ production based on BOD feed

Mottet *et al.* (2010) investigated the relationship between the initial chemical characteristics of SAS and the thermophilic anaerobic biodegradability of the sludge. The anaerobic biodegradability was determined by the ratio between specific CH₄ production using a standard BMP test and the theoretical maximum CH₄ yield of 350mL CH₄ g COD⁻¹. The results indicated that carbohydrate, protein and lipid concentrations had a significant impact on SAS biodegradability under thermophilic anaerobic conditions, but there was no significant effect under MAD. In addition, the Van Soest fractionation of the fibre content (cellulose, hemicellulose, and lignin fractionation) also showed a significant influence on biodegradability of SAS, and the degree of reduction varied considerably during the AD of SAS. Cellulose was significantly reduced and to a greater extent ($51 \pm 10\%$) compared to hemicellulose and lignin during AD, which showed average reductions of $33 \pm 21\%$ and $33 \pm 15\%$, respectively. The Van Soest method is based on consecutive extraction of fibrous material by solvents with increasing extractive power, and hence representing fractions with decreasing microbial accessibility. Further studies are required to understand the effect of sewage sludge composition on CH₄ production.

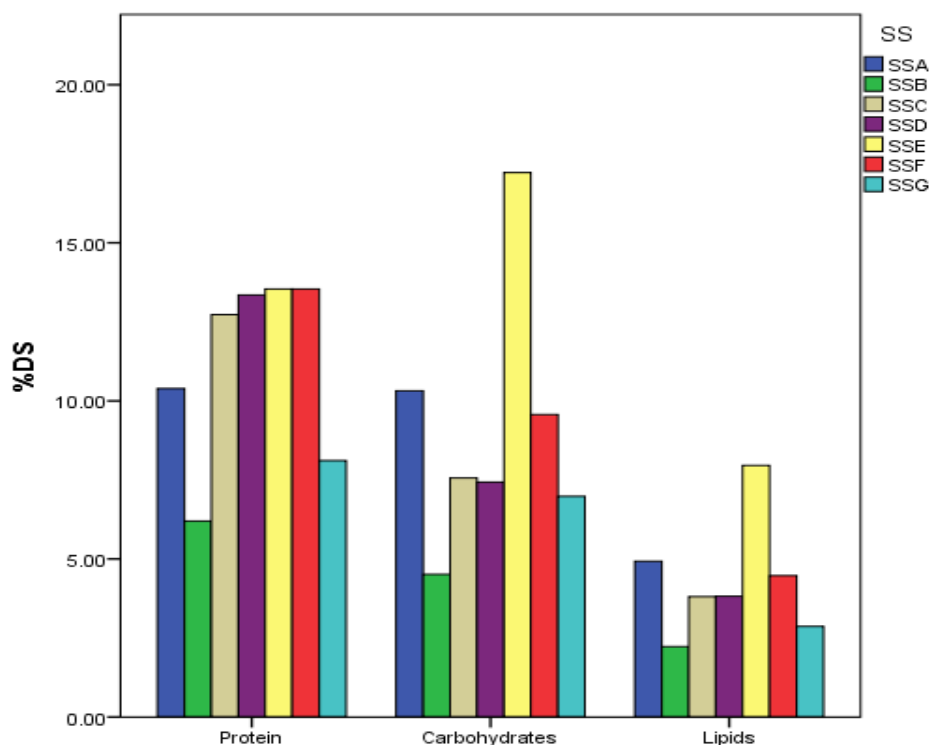


Figure 2-10 Concentrations of protein, carbohydrates and lipids in of raw mixed primary sludge and SAS samples collected from seven WWTPs (SSA to SSG) in Barcelona metropolitan area (Astals *et al.*, 2013)

2.4.3.2. Primary sludge and SAS

Primary sludge contains a greater quantity of lipids and fibre (7%-15% DS and 18%-25% DS), but less protein (16%-30% DS) compared to SAS (30%-60% DS). Surplus activated sludge consists of a large fraction of proteins arising from the assimilation of soluble N during the activated sludge

process, and a small amount of lipids and fibre (Table 2-5). Furthermore, SAS has a considerable amount of polyhydroxyalkanoate (PHA) polymers (20-60%DS), which are fully biodegradable; poly-3-hydroxybutyrate (PHB) is the most common type (Smith, 2014) (Table 2-5). PHA is an energy and carbon storage polymer accumulated under substrate feast-famine conditions by certain heterotrophic microorganisms during WWT (Wang *et al.*, 2015). Recent studies have demonstrated PHA could enhance CH₄ yield of SAS during AD. Polyhydroxyalkanoates are mostly located inside the cell and, according to Wang *et al.* (2015), increased PHA could enhance cell disruption of PHA cells to release intracellularly bound protein and carbohydrate. Moreover, sludge high in PHA showed greater sludge hydrolysis and acidification during AD than sludge samples with smaller PHA contents. For example, the fraction of small soluble substrates increased linearly with activated sludge PHA content after 2 days of digestion ($R^2 = 0.97$) (Figure 2-11).

In addition, PHA also exhibits a higher BMP than either proteins or carbohydrate. Wang *et al.* (2015) presented a standard BMP test result for dextran (a model polysaccharide compound), bovine serum albumin (BSA) (a model protein compound), and PHA, and showed that the CH₄ yield for PHA was 260.5 mL/g VSS, whereas the corresponding yield from dextran and BSA were 214.7 and 233.9 mL/g VSS, respectively.

Several studies have demonstrated the increased digestibility of primary sludge over SAS; typical ranges of VS destruction for primary and SAS are 55-60% and 30-45%, respectively (Gossett & Belser, 1982; WEF, 1987). Molecular formulae for primary sludge and SAS were determined by extensive data analysis by Barber (2014). The calorific value of primary sludge (C₂₃H₃₅O₈N) and SAS (C₇H₁₁O₃N) was calculated by using a sludge-specific version of the Dulong equation and sludge composition data. In Europe, primary sludge typically contains 30% inert material compared with 20% in SAS, and hence the calorific values of primary and SAS were estimated as 18000kJ kg⁻¹ VS and 17400kJ kg⁻¹ VS, respectively (Barber, 2014).

Toilet paper is a major constituent in wastewater, and consists primarily of cellulose fibre. Primary sludge, therefore contains a significant amount of fibre material mainly originating from toilet paper disposal that settles with the primary solids during primary WWT (Honda *et al.*, 2002). However, the behaviour of toilet paper in wastewater has not been examined to any extent and wastewater characterisation studies have neglected toilet paper in most cases (Ruiken *et al.*, 2013). The consumption of toilet paper per person varies between countries. In Europe, toilet derived cellulose accounts for 40% of the influent suspended solids and also a considerable fraction of the COD in influent wastewater (25-30%) (Ruiken *et al.*, 2013). Therefore, given the varying degradability's of fibre materials during AD it would appear to be essential to characterise the fibre fraction and assess its performance in sewage sludge AD.

Table 2-5 Reported values for organic constituents in primary sludge and SAS

Constituents	Sludge type	Average values (%DS) and source			
		Gonzalez, 2006	Barber, 2014	Smith, 2014	Range
Fibres	Primary	18-25%	22%	19%	18-25%
	SAS	0-1%	1%	0-10%	0-10%
Lipids	Primary	6-15%	13%	7-35%	7-15%
	SAS	1-2%	3%	5-12%	1-12%
Carbohydrates	Primary	18-27%	23%	N/A	18-27%
	SAS	25-28%	22%	N/A	22-28%
Proteins	Primary	16-23%	22%	20-30%	16-30%
	SAS	33-45%	42%	30-60%	30-60%
Polyhydroxyalkanoates (PHAs)	Primary	N/A	N/A	N/A	N/A
	SAS	N/A	N/A	20-60%	20-60%

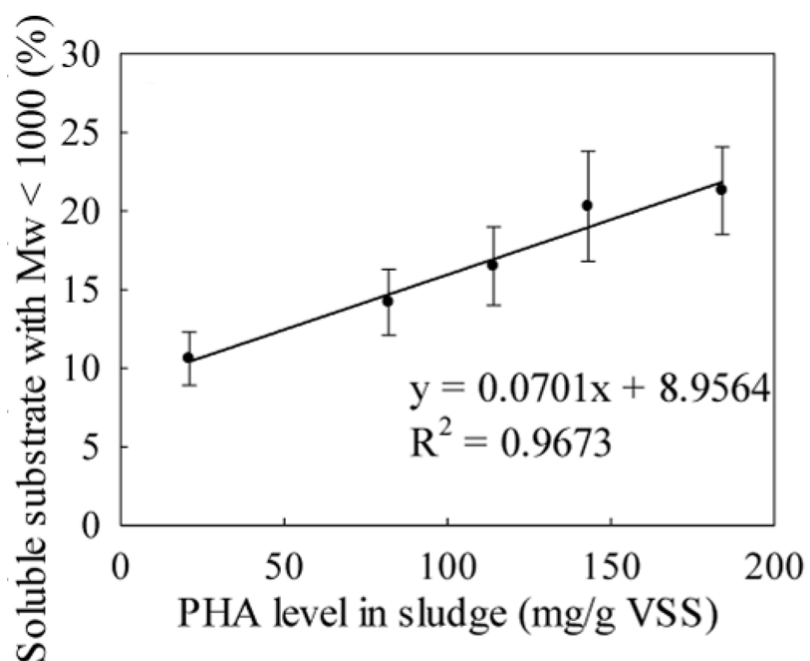


Figure 2-11 Correlation between soluble substrate with $M_w < 1000$ and activated sludge PHA concentration after 2 days of MAD (Wang *et al.*, 2015). M_w - molecular weight; VSS- volatile suspended solids

2.4.4. Biodegradability tests

The total quantity of organic matter in the sludge feed is routinely measured as VS at some of the full-scale WWTPs, based on the total mass lost following ignition of dry sludge. Similar parameters, such as COD and BOD, also assess the content of organic matter in sewage sludge. Chemical oxygen demand is an index that determines the amount of oxygen supplied by oxidants needed for the oxidation of organic compounds as well as some inorganic elements (such as sulphates), and the BOD is an index showing the amount of oxygen required for the oxidation of organic compounds by microorganisms (Suchowska-Kisielewicz and Jędrzak, 2019).

However, these tests do not provide information on how much of the organic substance present in the sewage sludge is biodegradable. The actual content of easily biodegradable matter in sewage sludge subjected to AD can be found through anaerobic and aerobic (respiratory) tests, which require time and specialised equipment. The BMP test is a relatively simple and inexpensive anaerobic measurement to estimate the digestibility of, and maximum CH₄ production from, various organic substrates in the AD process. It is achieved by adding a known quantity of organic substrate (for example, sewage sludge) to an active anaerobic inoculum (usually fresh sludge digestate) in an air-tight serum bottle where the biogas produced is measured, and its CH₄ content is determined (Esposito *et al.*, 2012). However, whilst simple, this technique is usually time consuming, and can require long incubation periods of 20 to 100 days (Da Silva *et al.*, 2018). Alternative aerobic tests include the static respiratory test (i.e. AT4) and dynamic respiratory test (i.e. DR4 and DR 100) (Suchowska-Kisielewicz and Jędrzak, 2019). Respirometry is generally defined as the measurement and interpretation of the rate of biological consumption of an inorganic electron acceptor under well-defined experimental conditions, and the respiration rate is usually measured with a respirometer (Van *et al.*, 2016). Among which, the AT4 index is being adopted increasingly for the analysis of substrates used in fermentation processes because AT4 takes a considerably shorter time (for example, 4 days) than the BMP test, but provides the required information on the biodegradability of substrates. For example, Suchowska-Kisielewicz and Jędrzak (2019) demonstrated a good agreement between the biodegradability of chicken manure with maize silage, grass and tomato stalks by the AT4 and BMP tests ($R^2 = 0.88 - 0.95$).

2.5. Factors Influencing Sludge Dewaterability

As discussed in Section 2.1, recycling treated sewage sludge to agricultural land is recognized as the Best Practicable Environmental Option for sludge management after AD (Kroiss and Zessner, 2007). Dewatering of the digested sludge prior to land application is essential to reduce the overall sludge volume and assist in the handling process (Houghton *et al.*, 2000). Effective dewatering relies heavily on the integration of dewatering equipment, sludge type and characteristics (including initial DS concentrations and pre-treatment), and polymer dosing (Minall *et al.*, 2014). Centrifuges,

dewatering belts and filter presses are three of the main types of dewatering equipment used at UK full-scale municipal WWTPs to dewater sludge to a DS concentration in the range 15 to 30 % DS concentrations (Minall, *et al.*, 2014). Different mechanical sludge dewatering devices have different operating principles and shear intensities, which greatly affects the efficiency of sludge dewatering (To *et al.*, 2016). For example, centrifuge dewatering applies a centrifugal force, between 500 and 3000 times that of gravity, to accelerate the separation of the solid fraction from the liquid. By comparison, a belt press operates at lower shear and pressure at approximately 1.2 to 1.5 bar. A filter press operates in a batch cycle and can dewater sludge to a higher degrees of dryness compared to either centrifuge and belt press (Minall *et al.*, 2014).

The level of natural extracellular polymer (ECP) present in the biological SAS fraction has a profound influence on the sludge dewaterability (Houghton *et al.*, 2000). ECP is an extremely hydrated polymer and prevents the desiccation of bacterial cells under natural environmental conditions. It is produced by bacteria particularly within the activated sludge process (Kang *et al.*, 1989) and sludge with high levels of ECP is particularly difficult to dewater (Houghton *et al.*, 2000). Anaerobic digestion of sewage sludge can change the ECP content in sludge and hence affects the sludge dewaterability (Nielsen *et al.*, 1996). Houghton *et al.* (2000) reported there was a 25% reduction in the ECP content of digested sludge on average compared to raw sludge, and suggested the optimum ECP concentration for maximum sludge dewaterability was 10 mg ECP g⁻¹ SS for digested sludge.

Polymer addition is necessary for mechanical dewatering of sewage sludge and is a high molecular weight, long chain, water soluble synthetic organic flocculant. The polymer dosage influences the DS quality achieved by a mechanical dewatering process and depend on the sludge type and compositions (Minall *et al.*, 2014). Typical polymer dosage values for different sludge types are summarised in Table 2-6.

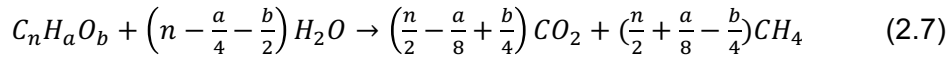
Table 2-6 Typical polymer dosage rate for mechanical dewatering different sludge types throughout UK (Minall *et al.*, 2014)

Sludge Type	Dose (kg/t DS)	Cake solids (% DS)
100% primary	2 - 4	30 - 40
67% primary : 33% SAS	3 - 5	25 - 35
67% SAS : 33% primary	6 - 8	20 - 25
100% SAS	7 - 12	15 - 20
Conventional digested	3 - 8	20 - 28
THP digested	8 - 15	26 - 32

2.6. AD Models

Scientific models of the AD process have been developed for almost 40 years motivated by the need for efficient operation of anaerobic systems (Donoso-Bravo *et al.*, 2011). The complexity of the AD process requires a modelling approach to balance the various influencing operational parameters, and specific models have been developed for different purposes (Kythreotou *et al.*, 2014).

The simple stoichiometric equation first proposed by Buswell and Muller (1952) calculated the maximum biogas potential of the digestible substrates in sludge (Equation 2.7).



Based on the elemental composition of different substrates, the theoretical maximum CH₄ yield can be calculated (Table 2-2). This approach usually overestimates the actual CH₄ yield as it does not take substrate biodegradability and digestion conditions into account, but it provides a simple method to calculate a benchmark of AD based on substrate composition.

The next generation of models focussed on the rate limiting step of the AD process, and were based on idea that the slowest step controls the global rate (Hill and Barth, 1977). The rate limiting steps can vary depending upon the operating conditions and substrate types fed into the digester (Speece, 1983). Eastman and Ferguson (1981) generated a rate-limiting model based on the hydrolysis of particulates to soluble substrates, for a CSTR treating primary sewage sludge (Figure 2-12). The hydrolysis kinetics were expressed by a first-order equation with respect to the particulate biodegradable COD.

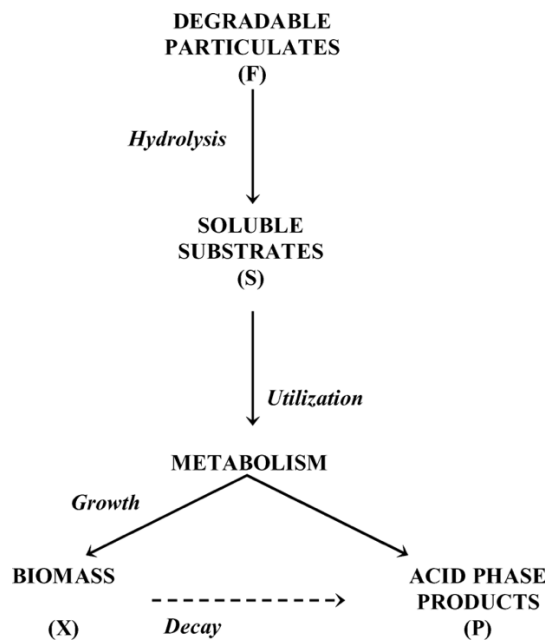


Figure 2-12 Schematic diagram of the Eastman and Ferguson (1981) model of the anaerobic digestion process

Similarly, Pavlostathis and Gossett (1986) developed a biological sludge model based on a more complex conversion process presented in Figure 2-13, where the digestion kinetics were determined by two possible limiting process: cell death/lysis and hydrolysis. Through a comparison of the performance between SAS and autoclaved sludge (bypassing the cell lysis step), the results showed that the degradable COD in the effluent of autoclaved sludge from the digester consisted of 80% non-hydrolysed protein. This result indicated that the hydrolysis of SAS is the critical step in the AD process. These rate-limiting models were simple and easy to use, but were unable to adequately describe the overall process performance (Donoso-Bravo *et al.*, 2011).

More complex models incorporated additional processes and microbial species and included more detailed kinetics, as well as inhibitory mechanisms, based on improved microbiological understanding of the process. For example, Hill (1982) used the volatile fatty acids concentration as a key parameter and separated the kinetics of acidogenesis and acetogenesis into individual stages. The basic microbiology of Hill's model is presented in Figure 2-14, which used soluble organics, acetic acid, propionic acid, butyric acid, H_2 , CO_2 , CH_4 and five bacterial cultures to describe the microbial pathways of the model.

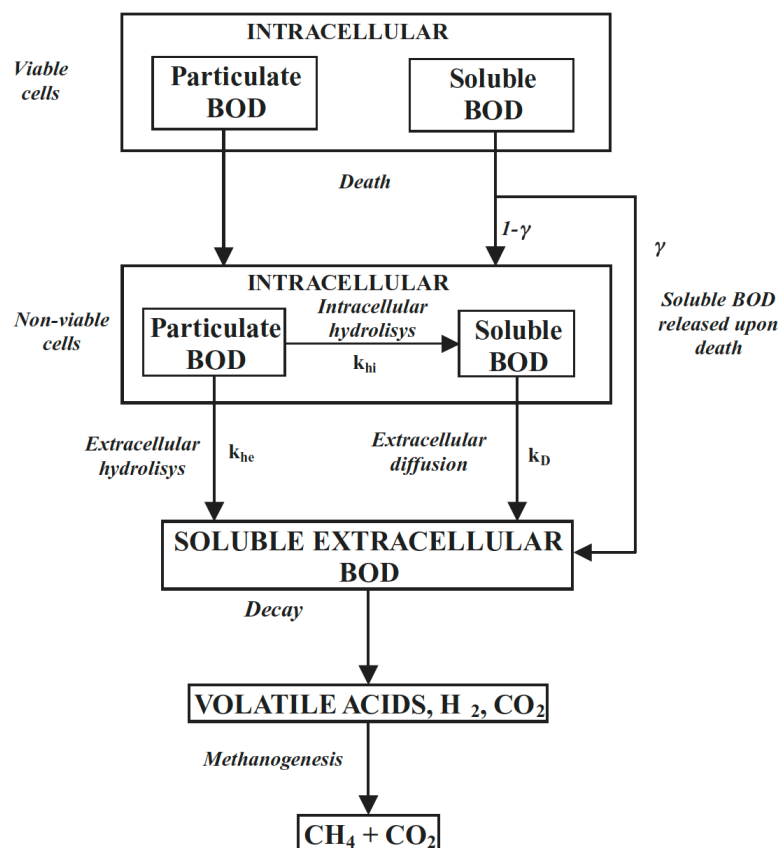


Figure 2-13 Conceptual model for anaerobic digestion of biological solids (k_d : death rate coefficient; k_{hi} : intracellular hydrolysis rate coefficient; k_{he} : extracellular hydrolysis rate coefficient; k_d : soluble BOD diffusion rate coefficient; γ : cell soluble BOD immediately released, fraction of total BOD) (Pavlostathis and Gossett, 1986)

More recently, the Anaerobic Digestion Model No.1 (ADM1), developed by Batstone *et al.* (2002), describes the dynamics of 24 species and includes 19 bioconversion processes, and aims to provide a generic model of fundamental AD mechanisms. The model process flow diagram is presented in Figure 2-15, and it describes both biochemical and physicochemical process of AD process. Biochemical processes include 4 complete AD steps from the first step of extracellular hydrolysis of carbohydrate, proteins and fats to the last step of converting acetate or H_2 and CO_2 into CH_4 by specialised methanogenic bacteria. Physicochemical processes are represented by equations describing ion association and dissociation and gas-liquid transfer. The ADM1 model has been widely applied and studied in simulating the AD of different organic wastes including sewage sludge from WWTPs and provided reasonable predictions of the dynamic behaviour of a pilot scale two-stage digestion process (Blumensaat and Keller, 2005). However, the complexity of such dynamic models and the large number of input parameters may limit their practical application to full-scale industrial AD systems. Moreover, a constant-volume and completely-mixed system is assumed by ADM1 and this is often difficult to achieve at full-scale (Kythreotou *et al.*, 2014).

Consequently, the extension of fundamental and AD modelling research to full-scale processes has not yet been achieved due to a lack of simple modelling tools that can be practically applied by operators managing full-scale plant.

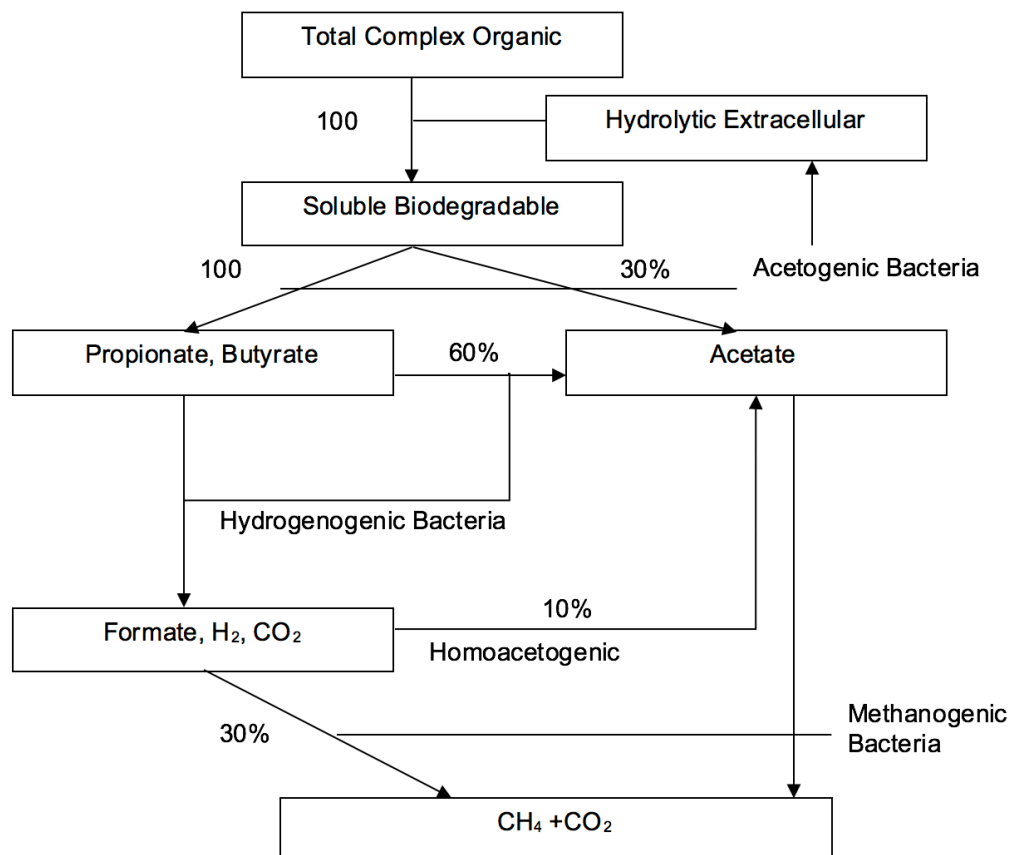


Figure 2-14 Basic microbiology and mass flows of the Hill model of anaerobic digestion (Hill 1982)

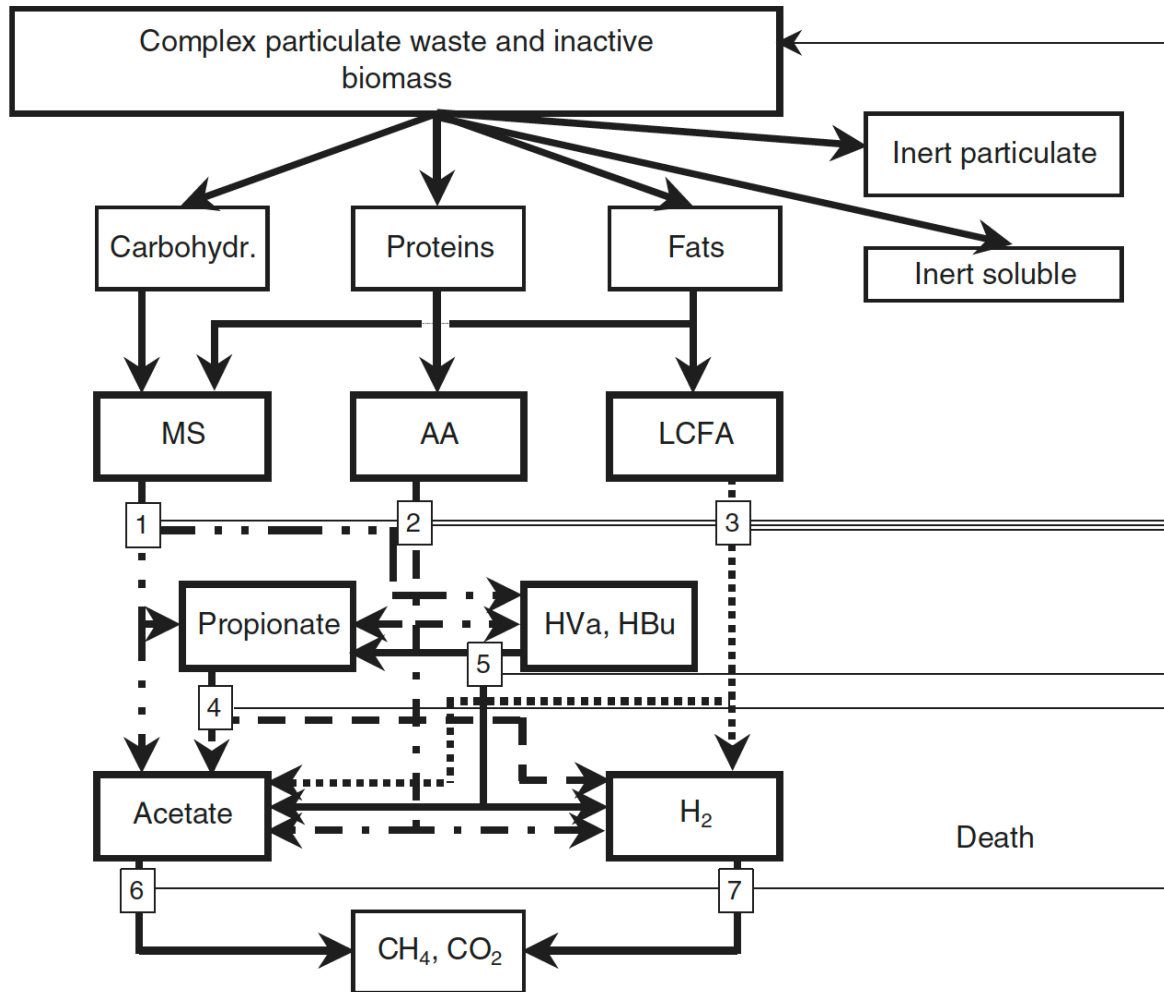


Figure 2-15 Process flow diagram of ADM1. (1) acidogenesis from sugars, (2) acidogenesis from amino acids, (3) acetogenesis from LCFA, (4) acetogenesis from propionate, (5) acetogenesis from butyrate and valerate, (6) aceticlastic methanogenesis, and (7) hydrogenotrophic methanogenesis. (MS: monosaccharides; AA: amino acids; LCFA: long chain fatty acids)

2.7. Objectives of the Study

Anaerobic digestion is a long-established method for treating wastewater sludge and has been extensively researched, but there is still a lack of generic or practical modelling tools available to guide operators and maximise the energy output. Detailed kinetic models have been developed, but they are too complex as a practical tool for full-scale application in the Water Industry. The parameters that are routinely recorded at WWTP for AD management are usually relatively restricted; the most widely recorded and controllable parameters available to AD operators include digestion temperature, HRT, and the DS content of the feed sludge to the digester. Sites with advanced MAD may record additional chemical parameters, such as VS, VFA, and pH. The effects of these principal operational parameters on the AD process have been extensively studied individually in controlled

laboratory experiments (Boušková *et al.*, 2005; Alepu *et al.*, 2016; Nielsen *et al.*, 2017). However, it is less clear how digestion conditions affect the performance of full-scale, industrial AD plants, when interactive effects of multiple process parameters exist. This thesis used up to 7 years of operational data from 97 full-scale MAD sites in the UK, demonstrating a wide range of AD performance, to construct several operationally-based, biogas and energy balance AD models. Modelling full-scale AD performance with limited operational parameters on multiple sites can be challenging, due to the complexity of the full-scale MAD process and relative differences in operational conditions, sludge composition and data recording between sites. Therefore, it is difficult to accurately predict an absolute BY value, based solely on the principal operational parameters (temperature, HRT, DS) currently available at full-scale sites. To overcome this challenge, a novel multiple regression modelling approach was applied to scrutinise AD data from multiple sites, with a hierarchical, clustered structure. A categorical factor was included to account for inter-site variations not explained by the continuous operational parameters in the model. Furthermore, the effects of the operational parameters on digester performance were represented by the relative changes in BY, based on a centred data approach (Aiken *et al.*, 1991). Local, absolute BY values were calculated by calibrating the model to the specific site conditions based on the local site BY, electricity generation or sludge chemical composition data. The aim of this research was to develop a statistically based, decision support tool, to predict and optimise AD performance using operational parameters available at full-scale sites, that can be applied by plant operators to optimise the BY and energy balance of full-scale sewage sludge digestion processes. The main objectives of the project were as follows:

1. Collect and collate AD operational data and related site information from full-scale conventional and advanced AD sites in five major Water Utility companies in the UK, perform quality assurance screening of the data and unify the data from multiple sources into a consistent format.
2. Perform a descriptive and preliminary statistical analysis, including linear correlation tests and cluster analysis, to visualise the performance data and check the data quality to ensure it provides meaningful interpretation in terms of AD performance response.
3. Perform multiple regression analysis to determine the statistically significant operational and environmental variables influencing the AD process and BY; the combined and interactive effects of multiple parameters influencing the AD performance will be assessed to provide quantitative coefficients for each predictor of the target dependent variables, including: BY, electricity yield (EY), volatile solids reduction (VSR) and digested sludge dewaterability.
4. Determine the content of major organic AD substrate components in feed sludge to the AD process by collecting and analysing sludge samples from eight selected AD sites over a period of one year on six occasions at intervals of 6-8 weeks. This will include the

determination of the total concentrations of proteins, fats and carbohydrates, as well as the amounts of different fibre components in sludge, including: cellulose, hemicellulose and lignin.

5. Develop empirical statistical models of BY incorporating principal operational parameters (temperature, HRT and DS feed), upstream WWT process variables (SAS age and primary:SAS ratio), and sludge chemical composition (VS, VFA and macromolecular composition) parameters. Separate models will be developed for conventional and for specific advanced AD technologies.
6. Validate the models against selected, specific WWTP data that are: (a) used in model development, and (b) independent, being collected subsequently, and not used to develop the model.
7. Develop model calibration methods, by using local site: (a) biogas data, (b) electricity generation, or (c) sludge composition results, to account for the site-specific conditions that are not captured by the routinely measured parameters in the models, to enable the models to be applied universally to any full-scale MAD process.
8. Develop optimisation strategies to achieve the maximum net energy output from full-scale AD under different MAD process conditions.
9. Develop a user interface to enable operators to use the model to evaluate local AD process performance and provide practical recommendations and measures for process improvement and optimisation.

Chapter 3. Materials and Methods

3.1. General Structure and Research Strategy

Anaerobic digestion is a complex process that is influenced by multiple factors (Chapter 2), and the Water Industry has collected extensive data archives on operational MAD process variables. The overall approach to data collection and statistical analysis is shown in Figure 3-1. The historical data (2009 – 2017) within five participating water companies (Anglian Water Services Ltd, Severn Trent Plc, Thames Water Utilities Limited, United Utilities Group PLC, Yorkshire Water Services Ltd) were collected and unified into a consistent format. Statistical screening methods were applied to the datasets to remove outlier values arising from various sources of error, such as misrecording and measurement errors. Descriptive and preliminary statistical analysis of biogas and electricity data in relation to other operational parameters, using simple linear correlation test and cluster analysis, were conducted to check the quality of data and that it provided meaningful interpretation in terms of AD performance response. Several statistical models were generated using multiple regression analysis techniques based on the full-scale operational data from conventional and advanced AD processes. A programme of sampling and analysis of sludge composition was completed for 8 selected sites representing different AD processes and performance levels, to determine the effects of detailed sludge composition on digestion performance. Additional operational data were also collected at these selected sites to validate the models generated based on the historical operational datasets. A detailed description of each stage of the research programme and the statistical, modelling and laboratory techniques applied is presented in the following sections.

3.2. Site Information

Mesophilic anaerobic digestion of sludge is typically operated as a fully mixed, semi-continuous reactor, operated in the mesophilic temperature range from 35 to 40 °C (Zupančič and Grilc 2012). In general, sludge is loaded into the digester once to several times a day, and the digester is usually a single stage equipped with a preparation tank, where various types of sludges can be mixed and prepared for loading, and which also serves as a buffer tank (Figure 3-2). An unmixed post-treatment tank is used to store the treated sludge at ambient temperature to complete the stabilisation process and prepare the sludge for further treatment (mechanical dewatering in most cases). Compared to the conventional MAD process, advanced MAD may utilise existing conventional MAD facilities with an additional pre-treatment step to increase the biodegradability and promote the hydrolysis of the substrate in the following MAD process, and/or also for pathogen destruction. There are several pre-treatment methods available and include: THP, EH (Enzymic hydrolysis), EEH and

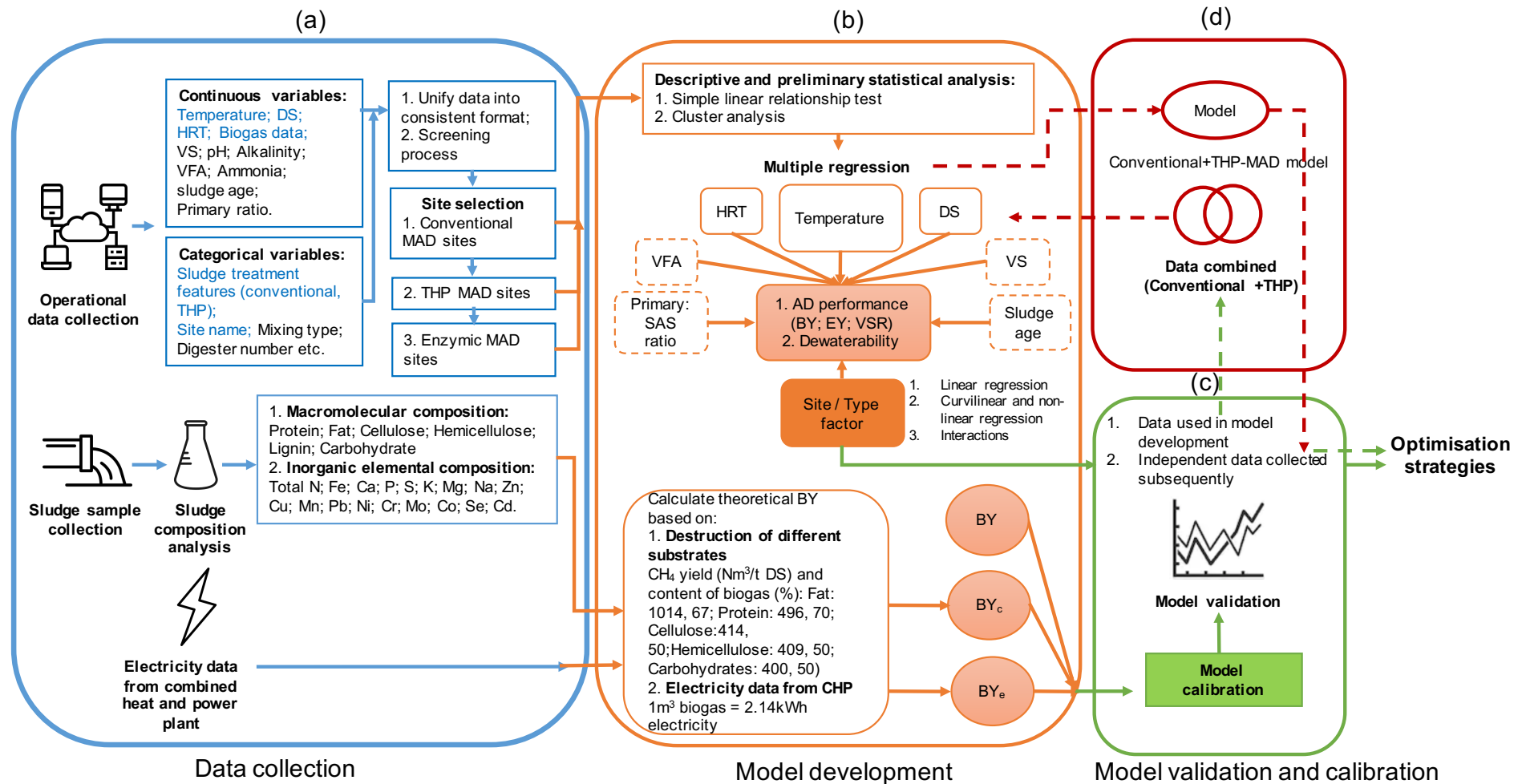


Figure 3-1 Overall approach to data collection, model development, validation and calibration. (a) Operational parameter data available in all companies are marked in blue. (b) Theoretical CH₄ yield and CH₄ contents of biogas produced from different specific organic substrates are from Weiland (2010) and Li *et al.* (2018). Electricity conversion factor from combined heat and power (CHP) is based on Banks (2009). (c) Model validation. (d) Combined conventional-THP MAD model development. Solid lines: general MAD model development pathway; dashed lines: combined conventional + THP-MAD model development pathway; dashed box: parameters used in sub-model development

pasteurisation processes. The effects of pretreatment is discussed in detail in Section 2.3.8 and detailed flow diagrams of each generic process is presented in Appendix 3-1.

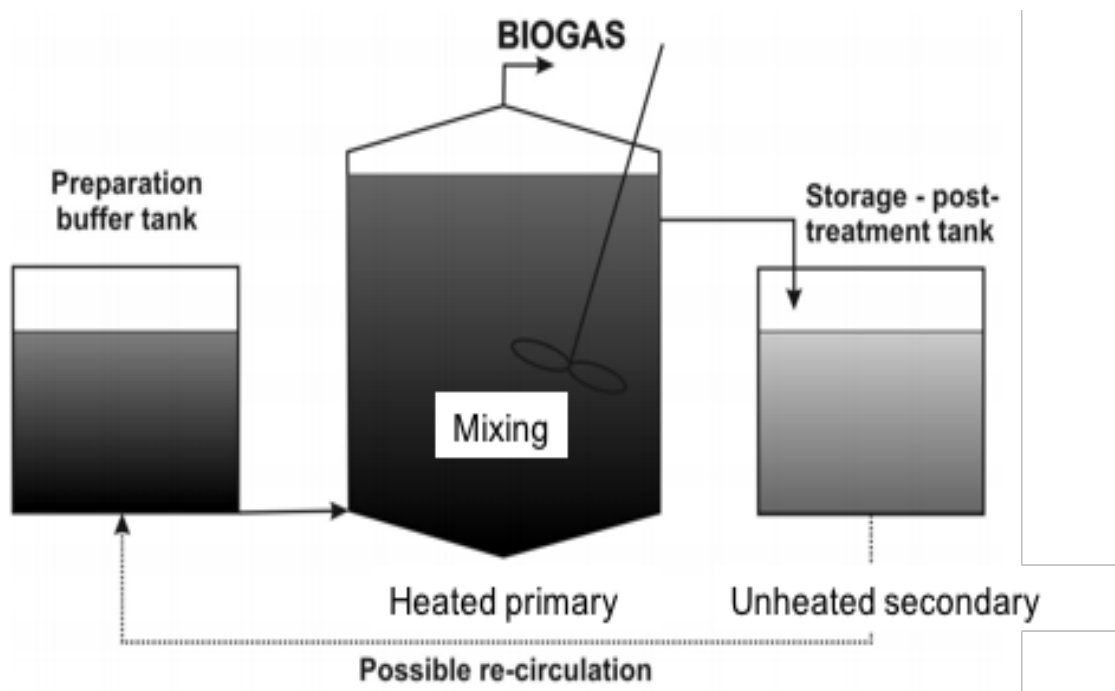


Figure 3-2 Conventional single stage anaerobic digestion process (Zupančič and Grilc 2012)

A grand total of 97 WWTPs operated MAD sludge treatment facilities within the five participating water companies. The MAD sites were divided into two main groups, and these included (1) conventional and (2) advanced MAD sites. The advanced MAD sites were further sub-divided into two sets according to the type of pre-treatment: (a) THP or (b) enzymic (including: EH, EEH, HpH) MAD. 66 conventional MAD (including three pasteurisation sites), eight THP MAD and 10 enzymic MAD sludge treatment facilities were selected for analysis based on data quality criteria (the average recorded BY, where available, was less than 700m³/t DS and more than 50% of the daily records of the AD operational parameters were complete). Operational data collected on a daily frequency were available for each of the selected sites for periods of 2 to 7 years, between 2009 and 2017. The distribution of conventional and advanced sites between the different companies is presented in Figure 3-3.

3.3. Data Collection

3.3.1. Operational Data Information

The Water Industry has collected an extensive data archive on operational MAD process variables, however, each company had a different system for data collection and reporting and it was therefore necessary to construct a unified, centralised dataset. Figure 3-4 shows a typical WWTP flow diagram and indicates the parameters recorded by the water companies and included in the database. The variables in red represent key parameters that were collected by all of the companies and the

variables in green were only available in specific companies. A detailed description of the collected variable information for each company is presented in Table 3-1. The critical data collected by all of the companies and used for modelling the MAD process included: digestion temperature (°C), HRT (d), and the DS content of the sludge feed (%).

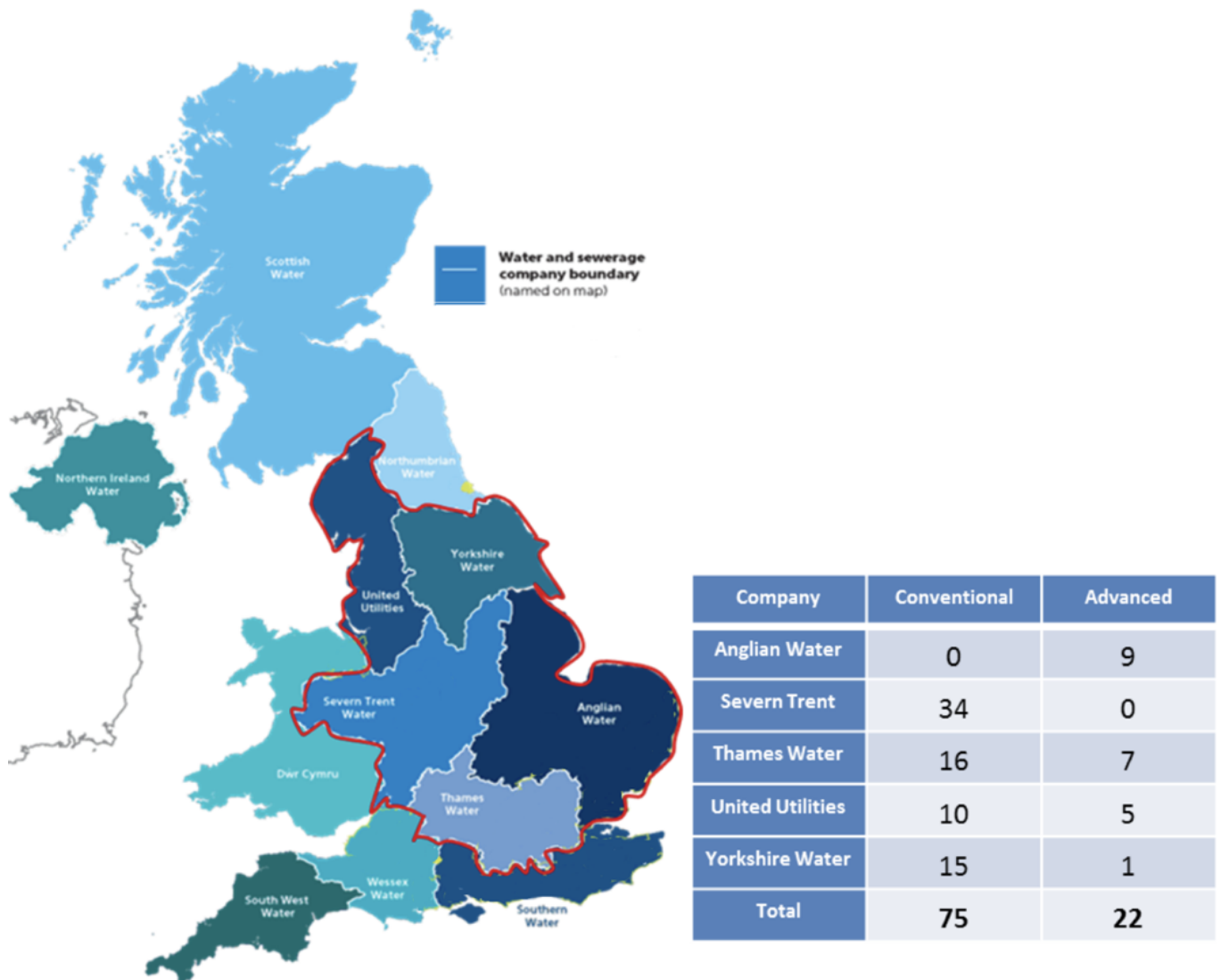


Figure 3-3 Geographical locations of the Water Utility companies providing AD data for analysis and the number of conventional and advance AD treatment sites within each company

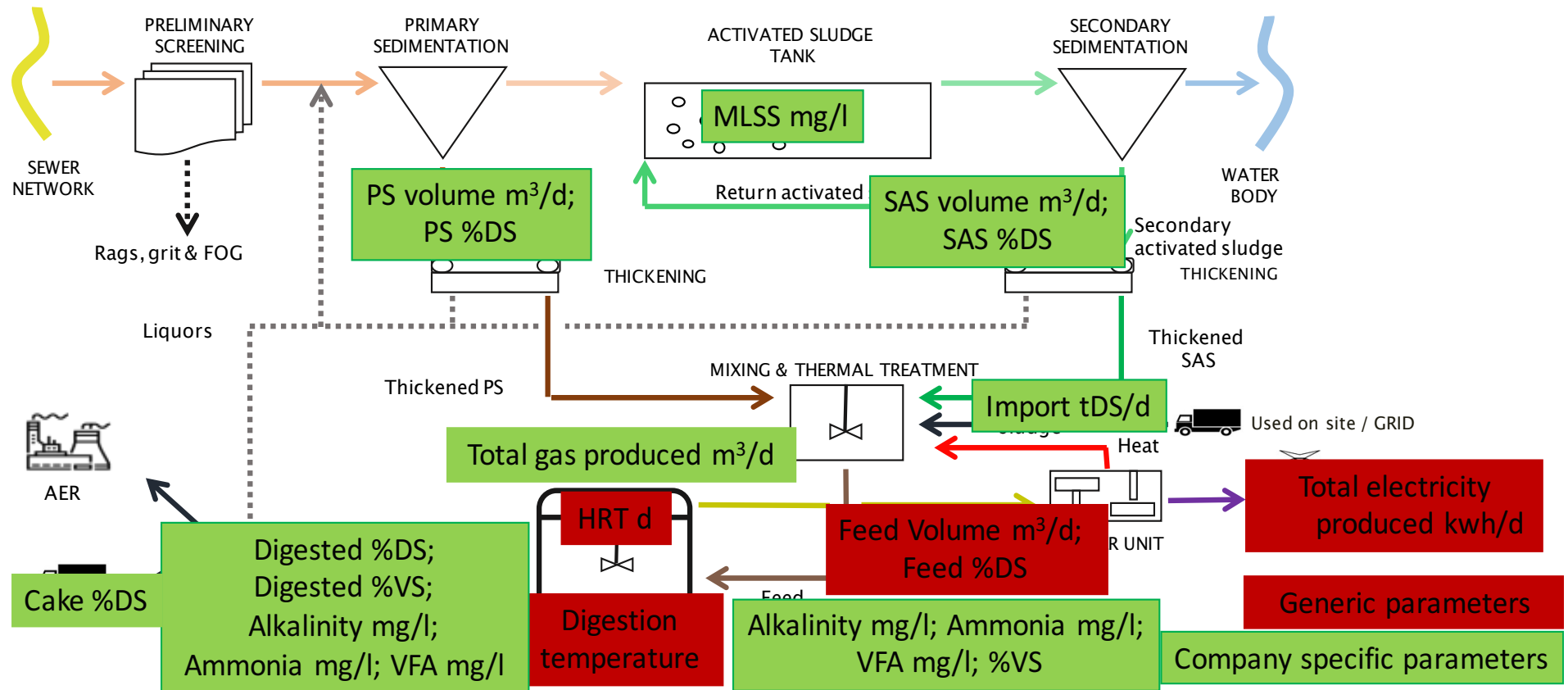


Figure 3-4 Typical WWTP flow diagram and operational variables recorded

Table 3-1 Data and parameters collected by each company (the time period for collected data is indicated in brackets for each company)

Datasets	Parameters	Company 1 (2013-2017)	Company 2 (2011-2016)	Company 3 (2014-2016)	Company 4 (2009-2016)	Company 5 (2014-2017)
Number of sites collected	Conventional site	34	18	10	15	0
	Advanced site	0	5	0	1	2
	Other advanced technologies	0	0	5	0	7
Process Monitoring dataset (Daily continuous variables)	Temperature (°C)	✓	✓	✓	✓	✓
	Hydraulic retention time (day)	✓	✓	✓	✓	✓
	Dry solids (%)	✓	✓	✓	✓	✓
	Volatile solids (%)		✓	✓		
	Feed Volume (m ³ /d)	✓	✓	✓	✓	✓
	Primary:SAS ratio (%)		✓			
	pH			✓		✓
	Volatile fatty acids (mg/l)					✓
	Alkalinity (mg/l)					✓
	Ammonia (mg/l)					✓
	Biogas data (m ³ /d)	✓	✓	✓	✓	
	CHP data	✓	✓	✓	✓	✓
	SAS age (day)		✓		✓	
	<i>E. coli</i>		✓		✓	✓
	Wastewater treatment features(ASP, biological filters, SBRs etc.)			✓	✓	✓
Categorical Variables	Sludge imports			✓	✓	✓
	Indigenous and import sludge ratio		✓		✓	✓
	Domestic and industry ratio			✓	✓	
	Sludge treatment features (conventional, THP, HpH, EEH, EH)	✓	✓	✓	✓	✓
	Digester number	✓	✓	✓	✓	✓
	Individual digester volume	✓	✓	✓	✓	✓
	Total digester volume	✓	✓	✓	✓	✓
	Mixing types	✓	✓	✓	✓	✓
	Quality of mixing				✓	✓
	Chemical dosing		✓	✓	✓	✓
	Dosing method		✓	✓	✓	✓
	Chemical used		✓		✓	✓
	Final dewatering features (centrifuge or others)		✓	✓	✓	✓
	Digester last cleaned			✓	✓	✓
	No. of staff on site involved in digester operation				✓	✓

Conventional site: MAD without pretreatment; Advanced site: MAD with pre-treatment process, including: Thermal hydrolysis process (THP), Heating, Pasteurisation and Hydrolysis (HpH) process; Enhanced Enzymatic Hydrolysis (EEH), and Enzymatic Hydrolysis (EH) process.; SAS: surplus activated sludge ASP: activated sludge process; SBRs: Sequencing biological reactor; CHP: combined heat and power electricity generation data

3.3.2. Analytical survey of sludge composition

The literature review of the effects of sludge macromolecular composition on the AD process identified four potentially influential groups of organic macromolecules that determine the CH₄ potential of primary and activated sludge. These include: fibre content, carbohydrates, proteins and fats. Three water companies (Company 1, 2 and 5) were selected, to undertake a programme of sample collection for compositional analysis of these major components of sludge organic matter. Eight MAD sites were selected based on their performance, data quality, pretreatment type, and geographic location. Four conventional MAD sites, representing high (Conventional Site 1), moderate (Conventional Site 5 and 31), and low (Conventional Site 38) observed BY were selected. Conventional Site 38 is a prepasteurisation site (70 °C for 1 h), however, this is shown to have no effect on BY (see Section 2.3.8) therefore, this site was considered to be an equivalent process to the other conventional sites in terms of BY. The selected advanced-treatment processes included: two THP sites (THP Site 4 and 7), one HpH site (HpH Site 3), and one EEH site (EEH Site 2), and these were all representative of average advanced MAD biogas performance. The number and types of processes from each company are listed in Table 3-2.

Table 3-2 Number and types of processes sampled in each company for detailed sludge compositional analysis

Company	Number and types of works
Company 1	2 conventional (Conventional Site 1 and 5)
Company 2	1 THP and 2 conventional (THP Site 4, Conventional Site 31 and 38)
Company 5	3 advanced (THP Site 7, EEH Site 2 and HpH Site 3)

Different sampling points were used for conventional and advanced works due to the different configurations of sludge mixing and treatment processes. A summary of the sampling locations is provided in Table 3-3 and pictures showing the sampling point locations are provided in Appendix 3-2. Sludge samples were collected on six occasions at intervals of 6-8 weeks, on 09/05/2018 – 11/05/2018; 25/06/2018 – 27/06/2018; 13/08/2018 – 15/08/2018; 15/10/2018 – 17/10/2018; 21/11/2018 – 23/11/2018 and 20/02/2019 – 22/02/2019.

Table 3-3 Sampling locations of conventional and advanced digestion sites for detailed sludge compositional analysis

Type of AD process	Sampling points
Advanced	1. Thickened raw mixed sludge 2. Post hydrolysis sludge 3. Post digestion sludge (before centrifuge dewatering)
Conventional	1. Digester feed sludge (after thickening) 2. Post digestion sludge (before dewatering)

Samples of each type of sludge were collected in four 500ml wide-necked high-density polyethylene (HDPE) to provide an approximate overall volume of 2 L of sludge. Each HDPE container was labelled with a permanent marker to indicate the site name, sampling point and date of sampling. Fresh, representative samples of sludge were obtained by opening the sampling valve and allowing the material to run to waste for several minutes before collection. The samples were stored in the dark in insulated polystyrene boxes filled with ice packs for transport. They were received at Imperial College London by the end of the day, where they were stored in a refrigerator (2 - 8 °C) until despatch to two contracted commercial laboratories for analysis. All treatment works were visited within a three-day cycle and, on the fourth day, a next day delivery courier was arranged to transport the samples to the laboratory.

Two accredited commercial laboratories (1. Sciantec Analytical, a division of Cawood Scientific Ltd, Stockbridge Technology Centre, Cawood, North Yorkshire. YO8 3SD and 2. NRM Laboratories, a division of Cawood Scientific Ltd, Coopers Bridge, Braziers Lane, Bracknell, Berkshire. RG42 6NS) were used for the following composition testing:

1. Macromolecular composition of sewage sludge (Sciantec Analytical Services): crude protein, total oil, sugar as sucrose, starch, neutral detergent fibre, acid detergent fibre, acid detergent lignin, cellulose, and hemicellulose.
2. Chemical composition (NRM laboratories): pH, total solids, total nitrogen, nitrate nitrogen, molybdenum (Mo), selenium (Se), manganese (Mn), cobalt (Co), nickel (Ni), cadmium (Cd), chromium (Cr), iron (Fe), lead (Pb), phosphorus (P), potassium (K), magnesium (Mg), copper (Cu), zinc (Zn), sulphur (S), calcium (Ca), sodium (Na) and ammonia nitrogen.

Total nitrogen (TN), ammonium- nitrogen ($\text{NH}_4\text{-N}$) and nitrate-nitrogen ($\text{NO}_3\text{-N}$) were determined by standard Dumas methods and EPA-600/4-79-020 method 350.1, respectively (USEPA, 1983). Protein was estimated by multiplying the organic nitrogen (TN minus $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$) by 6.25 (Mariotti and Mirand, 2008). Fats were analysed by standard method 5520E procedure (APHA, 2005). Fibre content were analysed by Van Soest (1991) fibre analysis methods to determine cellulose, hemicellulose, and lignin. The remaining carbohydrates were estimated by subtracting the amount of protein, fats and fibres from the VS content. Total elemental concentrations were determined by aqua-regia digestion and inductively coupled plasma optical emission spectrometry (ICP-OES) (Jackson *et al.*, 1986; EPA, 1996). A summary of the analysis methods used by the external laboratories is provided in Appendix 3-3.

The dry solids (DS) and volatile solids (VS) contents, and chemical oxygen demand (COD) were measured in the Imperial College laboratory following standard methods (APHA, 2005).

Dry solids and volatile solids (APAH, 2005): As-received samples were homogenised and a representative sub-sample (approximately 20 g) was taken in a suitable tray. The weight was

accurately recorded before and after drying in an oven at 105°C +/- 5°C to determine the 'Oven Dry Matter' as a % weight loss. The drying time was at least 12 hours and samples were reweighed to ensure complete dryness. The dried residue was transferred to a cool muffle furnace, which was subsequently programmed to increase the temperature to 550°C, and ignition was continued for 1 h. The ignited sample was cooled in a desiccator. The ignition (30 min), cooling, and desiccator and weighing steps were repeated until the weight change was less than 4%. Duplicate determinations should agree within 5% of their average weight. Volatile solids were calculated as the difference between both measurements.

Chemical oxygen demand (APHA, 2005): sludge samples were heated with potassium dichromate - sulphuric acid reagent containing silver sulphate catalyst. The dichromate residue was measured by colorimetric titration against ferrous (iron II) ammonium sulphate (FAS) with an indicator of ferroin (1,10-phenanthroline and ferrous (iron II) sulphate). The colour change at the end point is from blue-green to reddish-brown which persists for at least 1 minute. The procedure adopted the following steps. Test tubes and caps were washed with 20% sulphuric acid before use to prevent contamination. The sludge sample was homogenised using a hand mixer for one minute. Approximately 0.5 to 1 g of raw sample was collected and diluted with deionised water to about 200 to 1000 fold, depending on the sample DS concentrations. The diluted sludge sample (2.5ml) was placed into the test tube and potassium dichromate solution was added, followed by the careful addition of sulphuric acid. The cap was sealed and the tubes were inverted several times to completely mix the sample and reagents. The tubes were placed into heating block preheated to 150°C in a fume cupboard and refluxed for 2 hours 15 minutes. The sludge sample was cooled to room temperature and the contents were transferred to a conical flask for titrating. Ferroin indicator (2 drops) was added and the sample was stirred rapidly on a magnetic stirrer while titrating with standardised FAS. A reagent blank containing the equivalent volume of deionised water to the sludge sample was examined following the same procedure. Sludge samples were analysed in triplicate. The COD of the sludge sample was calculated by:

$$\text{COD as mg O}_2/\text{L} = \frac{(A-B) \times M \times 8000}{\text{mL sample}} \quad (3.1)$$

Where:

A is the volume (mL) of FAS used for the blank; B is the volume (mL) of FAS used for the sludge sample; and M is the molarity of FAS.

3.4. Calculations

Several important WWT and AD variables were calculated based on the operational data collected from the water companies.

The BY indicates the amount of biogas generated from per t of DS fed a digester, which is considered as one of the most important indicators of AD process performance (Weiland, 2010), and was calculated by:

$$\text{Biogas yield} = \frac{\text{Total gas (m}^3\text{/d)}}{\text{Total sludge feed (tDS/d)}} \quad (3.2)$$

The specific biogas yield (SBY) is a measure of biogas produced per t VS supplied to the digester, which is also a useful process performance indicator parameter, but subject to the availability of VS in the dataset.

$$\text{Specific biogas yield} = \frac{\text{Total gas (m}^3\text{/d)}}{\text{Tonnes of VS feed (t VS/d)}} \quad (3.3)$$

The electricity yield (EY) is a measure of the electricity generated per t of DS supplied to the digester, and was calculated by:

$$\text{Electricity Yield} = \frac{\text{Total electricity (kwh/d)}}{\text{Total sludge feed (tDS/d)}} \quad (3.4)$$

Similarly, the specific electricity yield (SEY) is a measure of electricity generated per t of VS, and was calculated by:

$$\text{Specific electricity yield} = \frac{\text{Total electricity (kwh/d)}}{\text{Tonnes of VS feed (t VS/d)}} \quad (3.5)$$

The Van Kleeck volatile solids reduction (VSR) was calculated by (VanKleeck. 1945):

$$\text{VSR}_{\text{VK}} = 1 - (\text{VS}_b \times (1 - \text{VS}_f) / \text{VS}_f - (1 - \text{VS}_b)) \quad (3.6)$$

Where VSR_{VK} represents fractional VSR calculated by Van Kleeck equation, and VS_f and VS_b represents the fractional VS in the feed sludge and after digestion, respectively. This equation assumes zero fixed solids reduction and grit accumulation in the bottom of the digesters. Additionally, the fixed solids input and fixed solids output were assumed to be equal.

The mass balance VSR was calculated by:

$$\text{VSR}_{\text{MB}} = ((\text{DS}_f \times \text{VS}_f) - (\text{DS}_b \times \text{VS}_b)) / (\text{DS}_f \times \text{VS}_f) \quad (3.7)$$

Where VSR_{MB} represents fractional VSR calculated by the mass balance equation, and VS_f and VS_b represent the fractional VS in the feed sludge and after digestion, respectively. Similarly, DS_f and DS_b represent the fractional DS in the feed sludge and after digestion, respectively.

Activated sludge age was calculated by:

$$\text{Sludge age} = \frac{\text{mass of sludge in activated sludge tank}}{\text{mass of sludge taken out everyday}}$$

$$= \frac{\text{MLSS (mg/l)} \times \text{AST volume (m}^3\text{)} \times 10^{-6}}{\text{SAS volume (m}^3\text{/d)} \times \text{SAS DS (\%)} \times 10^{-2}} \quad (3.8)$$

Where:

MLSS is the mixed liquor suspended solids (mg/l) during the activated sludge process;

AST volume is the tank volume (m³) of the activated sludge process;

SAS volume is the volume of SAS (m³/d) which has been taken out from the activated process tank each day;

SAS DS is the concentration of the surplus activated sludge which has been taken out from the activated process tank.

Primary:SAS ratio (%) was calculated by:

$$\begin{aligned} \text{PS ratio} &= 1 - \frac{\text{SAS mass (tDS/d)}}{\text{Total sludge feed to digester (tDS/d)}} \\ &= 1 - \frac{\text{SAS volume (m}^3\text{/d)} \times \text{SAS DS (\%)} \times 10^{-2}}{\text{Feed volume (m}^3\text{/d)} \times \text{Feed DS (\%)} \times 10^{-2}} \end{aligned} \quad (3.9)$$

Where:

SAS volume is the volume of SAS (m³/d) removed from the activated process tank each day;

SAS DS is the concentration of the surplus activated sludge (%) removed from the activated sludge process tank;

Feed volume is the volume of sludge fed into the digesters (m³/d);

Feed DS is the concentration of the mixed primary and secondary sludges (%) fed into the digesters.

The equation assumes all import sludge to a WWTP is primary sludge.

3.5. Statistical Analysis

3.5.1. General description of and stages in the statistical analysis

The overall approach to data merger and statistical analysis is shown in Figure 3-1. The IBM SPSS Statistics 21 programme was used to complete the statistical analysis calculations. A descriptive analysis and screening process was applied to the conventional and advanced AD datasets to remove extreme outliers and to convert the variables into a consistent format based on monthly average values. Boxplots were used to identify and removed extreme outliers. The points that were recorded as high leverage (leverage value>0.2) and highly influential (Cook's Distance values>1) were recorded and removed. Monthly average values were calculated for the range of operational parameters routinely measured at each WWTP and potentially important to BY and EY, including:

HRT, temperature and DS. This approach allowed the maximum data capture and provided representative performance data for each site by removing short term fluctuations in the process variables and also associated with the HRT.

Descriptive and preliminary statistical analyses of biogas and electricity data in relation to other operational parameters were conducted to check the quality of data and that it provided meaningful interpretation in terms of AD performance response. These preliminary statistical tests included: (1) Post-hoc tests and analysis of variance (ANOVA) to determine the significance of specific parameter comparisons; (2) A correlation analysis to test the significance of the relationships between process and sludge physico-chemical conditions and BY and EY; and (3) A cluster analysis test (Mooi & Sarstedt, 2014) for operational sewage sludge treatment AD data visualisation. The conventional and advanced AD sites were separated for the cluster analysis. The cluster analysis was applied to assess the effects of 3 principal AD operational variables, including: temperature, HRT and DS, on the BY and EY, using monthly average data.

Multiple regression analysis was used to assess the combined and interactive effects of multiple parameters, and hence provide quantitative coefficients for each predictor on the target dependent variables. The predictors were centred before fitting into the model, to evaluate interaction effects, by subtracting the mean value from each variable (Aiken *et al.*, 1991). Centring is the process of selecting a reference value (in this case it was the mean value) for each predictor, and hence a meaningful zero value for each predictor can be included in the model (Kraemer and Blasey, 2004). The zero value of each predictor represents the overall average operational condition, and the major operational factors influencing the process can be then quantified based on the changes in digester performance relative to zero. Backward elimination and forward selection methods were used to identify predictor variables in the multiple regression models.

The multiple regression was constructed based on the order of:

- (1) Linear regression;
- (2) Curvilinear and non-linear regression (quadratic/cubic and log transformed data);
- (3) Adding interaction terms between significant predictors.

Several multiple regression models were constructed based on all the possible combinations of available parameters. Three main models for digester performance, indicated by BY or EY, were developed for conventional, THP and enzymic MAD processes. Two sub-models were also tested to assess the effects of the main process control parameters (temperature, HRT and DS) on VSR and sludge dewaterability, indicated by final sludge cake DS.

The multiple regression models were validated against both specific WWTP datasets used in model development as well as independent datasets, which were collected subsequently and were not

used to develop the model. The conventional MAD model was also applied to the THP MAD sites to predict the AD performance of sludge pretreated by THP. The electricity generated from AD biogas through CHP was used to cross-check the gas data and calculate a BY based on electricity (BY_e), to enable calibration of the model for a specific site. Finally, the results from the sludge composition analysis were used to calculate the theoretical BY (BY_c) of the examined sites based on the destruction of the different organic fractions in sludge after AD, and the associated CH_4 production values presented in Figure 3-1(b). This provided an alternative approach to calibrate the AD model for specific sites, based on the combined data for all sites used in model development, that was also independent of the recorded operational information.

3.5.2. Screening process and descriptive analysis

The screening process of the raw data and descriptive analysis of the dataset was carried out simultaneously using the Explore function in SPSS. The screening process aimed to detect potential outliers in the dataset. According to Hawkins (1980), “An outlier is an observation that deviates so much from the other observations as to arouse suspicions that it was generated by a different mechanism.” Outliers present in the AD dataset could be caused by incorrect recorded measurements and data entry errors. The presence of outliers could potentially increase error variance and reduce the confidence in the statistical analysis. Furthermore, outliers could reduce the normality of distribution, and seriously influence the regression coefficients (Osborne and Overbay, 2004). In this analysis, boxplots generated during the Explore analysis procedure were used to identify extreme outliers. The boxplot method is a straightforward way to detect outliers (Frigge *et al.*, 1989). Data points more than 1.5 box-lengths from the edge of the box were classified as an outlier and indicated by a circular dot, and points greater than three box-lengths away from the edge of the box were defined as extreme points and were identified with an asterisk (Figure 3-5). The edges of the box were designated as the 25%ile and 75%ile boundaries of the data (Frigge *et al.*, 1989); only extreme points in the boxplots were removed. The descriptive analysis provided a summary of the data collected for each site and included: the range of the data, valid number of the data (n), mean, median, standard deviation, minimum, maximum, and 25% and 95% percentile values. Final summary tables for each site were generated after the extreme outliers were removed.

3.5.3. ANOVA analysis with post-hoc test

One-way analysis of variance (ANOVA) was used to determine the statistical significance of differences between two or more independent groups. The assumptions for ANOVA and means comparison are as follows (Pallant, 2010):

1. There are no outliers in any of the groups to be compared;
2. The data for each group is normally distributed;

3. The groups have equal variances (homogeneity of variances).

One-way ANOVA is a relatively robust to violations of normality, and it is possible for the assumption of normality to be violated and still provide valid results (Maxwell and Delaney, 2004). Sawilowsky and Blair (1992) showed that for fairly skewed distributions the violation of normality does not compromise ANOVA if sample sizes are large and the groups are similarly skewed. Furthermore, the ANOVA test is fairly robust to deviations from normality for equal or almost equal sample sizes (Lix and Keselman, 1996).

The assumption of homogeneity of variances was examined by Levene's Test (Levene, 1960). A significance level of $P < 0.05$ indicated violation of the assumption and, under these circumstances, a Welch ANOVA was performed (Welch, 1947). Post-hoc tests were conducted for groups showing statistically significant effects by one-way ANOVA. A Tukey post-hoc test was used when the assumption of homogeneity of variances was supported, alternatively the Games-Howell test was applied if the assumption was violated (Armstrong *et al.*, 2000).

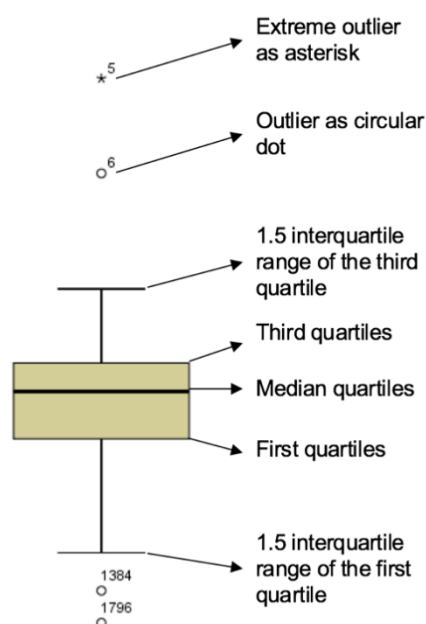


Figure 3-5 Using boxplot to identify extreme outliers

3.5.4. Cluster analysis

Cluster analysis is a generic name for a variety of mathematical methods used to identify similar groups of objects (or cases, observations) in datasets (Romesburg, 2004). It is a useful technique to identify homogenous groups and make classifications which simultaneously take the effects of several variables into account. The objective of the cluster analysis was to identify groups with similar digestion conditions and assign them into clusters. The general cluster analysis steps (Mooi &

Sarstedt, 2014) are shown in Figure 3-6. The first step in the procedure was to select which process variables to examine as this has a critical impact on the data interpretation. The principal digestion operational variables, namely, temperature, HRT and DS, were used to visualise the effect of digestion conditions on AD performance.

The second step was to select the most appropriate clustering procedure. There are numerous approaches available for cluster analysis, for example, the statistical package, SPSS, contains three of the main methods, including, hierarchical, non-hierarchical (*k-means*), and two-step clustering (which is a combination of the hierarchical and non-hierarchical approaches). The number of clusters to be derived from the data is predetermined before analysis. A small number of clusters is more straightforward to interpret compared to a larger number, but loses the ability to identify subtle differences between segments. The Two-step clustering procedure was applied here because it automatically selects the optimal cluster number, based on a cluster evaluation algorithm, the silhouette coefficient, which represents the succinct graphical representation of how well each object has been classified (Rousseeuw, 1987; Thinsungnoena *et al.*, 2015). Two-step clustering is also capable of analysing mixed variables (both continuous and categorical) measured on different scales. A minimum sample size was a requirement for valid analysis results, and Mooi and Sarstedt (2014) recommended a minimum sample size of 2^m , where m represents the number of clustering variables. However, the sample size of the data included in the analysis of the AD process was far larger (After screening, the total number of conventional MAD monthly operational data records was equivalent to: 3294 rows) than the minimum required for cluster analysis. The final step in the procedure was the validation and interpretation of the solution generated by the cluster analysis.

3.5.5. Multiple regression analysis

3.5.5.1. General Linear Models procedure in SPSS

The General Linear Model (GLM) procedure in SPSS was used for the multiple regression analysis of data. A major advantage of this modelling approach is that continuous and categorical variables and their interaction effects can be included, and dummy variables for these factors can be automatically generated (SPSS Handbook, 2013).

3.5.5.2. Test of assumptions

There are eight assumptions associated with multiple regression analysis:

Assumption #1: There should be only one dependent variable that is measured on a continuous scale. If the dependent variable is ordinal, ordinal regression should be carried out instead of multiple regression.

Assumption #2: The model should have two or more independent variables that can be either categorical or continuous.

Assumption #3: Independence of observation. The independence of observation suggests that the successive residuals are not correlated, which means there is no autocorrelation. Autocorrelation increases the variance of residuals as well as the variance of estimated coefficients (Wang and George, 2003). Autocorrelation was examined by the Durbin-Watson test; the range of the test is 0 to 4, and test values from 1.5-2.5 are considered as independent of errors (Durbin and Watson, 1951).

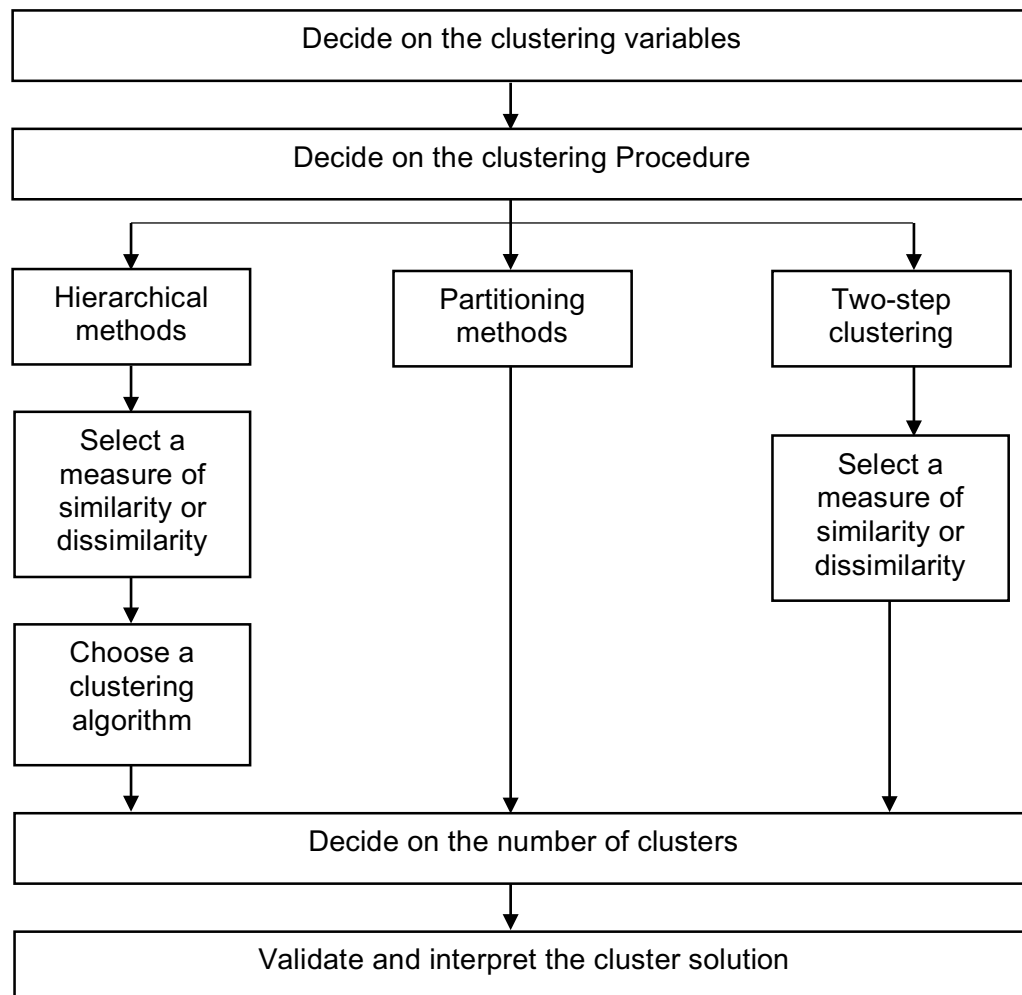


Figure 3-6 Cluster analysis steps (Mooi & Sarstedt, 2014)

Assumption #4: Linear relationship. There should be a linear relationship between dependent variables and each of the independent variables. This was checked by visually inspecting the scatter plots of variables.

Assumption #5: Homoscedasticity of residuals. Homoscedasticity of residuals means there is equal error of variance where the variance of errors is the same across all levels of the independent variables (Osborne and Jason 2002). This was checked by visual examination of the scatter plots of the studentized residuals against the unstandardized predicted values. A randomly and evenly distributed residual point around the horizontal line of 0 indicates a homoscedasticity of residuals.

Assumption #6: No multicollinearity. Multicollinearity occurs when multiple factors in the model not only correlate to the dependent variable, but also to each other. Therefore, under these conditions, it is difficult to identify which predictors contribute to the variance explained by the dependent variable, as well as the statistical issues associated in calculating multiple regression models. The statistical issues involve the large variances of the least squares estimators of coefficients of variables. Furthermore, the signs of the estimators may disagree with the known theoretical properties of the variables due to the collinearity problem with other variables (Mansfield and Helms, 1982). Multicollinearity was examined by inspection of the tolerance value, and values less than 0.1 suggested the presence of multicollinearity between explanatory variables. The recommended approach to multicollinearity is to remove the variables with tolerance less than 0.1 and test the model with and without these variables (Landau and Everitt, 2004).

Assumption #7: No significant outliers, high leverage points or highly influential points. These points can reduce the accuracy of the prediction as well as the statistical significance of the variables. The extreme outliers were detected by inspection of boxplots (see Section 3.5.2). A high leverage point can be defined by the leverage value greater than 0.5 (Pallant, 2010), and a highly influential point is a point with a Cook's distance value greater than 1 (Cook and Weisberg, 1982). Here, data points with both a high leverage value and Cook's distance value were removed.

Assumption #8: Normal distribution of the residuals. Multiple regression is fairly robust to the normality of the residuals and the normal distribution of residuals was examined by the predicted probability (P-P) plot of standardized residuals. Points aligned along the diagonal line indicated a normal distribution of residuals (Pallant, 2010).

Assumption #1, #2 were initially tested using BY (m^3/tDS), EY (kWh/t DS), or VSR (%) as continuous dependent variables, and the potential independent variables, which included monitored physico-chemical parameters, for example: Temperature ($^{\circ}\text{C}$), DS (%), and HRT (d). The independent variables were all numeric and measured on a continuous scale. Therefore, assumptions #1 and #2 are not violated.

The remaining six assumptions were divided into three major parts to simplify the testing process. Part 1: Assumption: #7; Part 2: Assumption: #3, #4, and #6; Part 3: Assumption: #5, #8; Part 1 was examined at the beginning of the regression process; Part 2 was tested during the predictor selection procedure and Part 3 was assessed for the final multiple regression model.

3.5.5.3. Selection of the predictors

A predictor selection procedure based on backward elimination and forward selection methods was applied to identify statistically significant predictors from the total number of independent predictors available. Backward elimination contains all the possible predictors in the first stage and the least significant and multicollinear variables are removed one by one until no insignificant predictors or

multicollinearity variables in the model are reached. Forward selection adds the significant predictors in descending order of their individual significance test by a Linear Relationship Test until the newly added-in predictor is not significant in the model. The threshold of statistical significance for identifying and selecting a predictor in the regression model was set to $P < 0.05$, to ensure that all possible predictors were considered. The final model was selected based on the adjusted R^2 value and the statistical significance. The adjusted R^2 is a measure of the goodness-of-fit of a multiple regression model, and is a modified version of R^2 obtained by adjusting for the number of predictors in the model (Wesolowsky, 1976).

3.5.5.4. Measure of effective size

The effective size of the predictor can be quantified by η^2 , which provides a measure of the proportion of the total variance explained by the prediction variables. However, in SPSS, only partial η^2 is automatically generated along with the regression process. Partial η^2 is a similar measure in which the effects of other independent variables and interactions are partialled out (Richardson, 2011), which can be represented by Equation 3.9. The η^2 for the predictors was calculated manually (Equation 3.11) based on the sums of squares values in a test between subjects table in the SPSS output file.

$$\eta^2 = SS_{\text{effect}} / (SS_{\text{effect}} + SS_{\text{error}}) \quad (3.10)$$

Where,

SS_{effect} is the sums of squares for the studied parameter.

SS_{error} is the sums of squares for the errors.

$$\eta^2 = SS_{\text{effect}} / SS_{\text{total}} \quad (3.11)$$

Where,

SS_{effect} is the sums of squares for the parameter studied.

SS_{total} is the total sums of squares for all parameters, errors and interactions.

3.6. Summary of the Multiple Regression Models

Several models were generated depending on the different variables recorded. The models were divided into two main groups, based on: (1) all data integrated models (Conventional, THP and Enzymic MAD model) across all companies, for BY and EY, and (2) parameter based models. All data integrated models used the generic operational condition parameters recorded by the participant companies, namely, temperature, HRT and DS feed, to construct the conventional and advanced MAD models of BY or EY. The parameter-based models were formulated using the additional information available, from smaller datasets recorded by certain companies (but, which

was not available for all companies), and this generated several sub-models of BY, EY, VSR and digested sludge dewaterability (Figure 3-7).

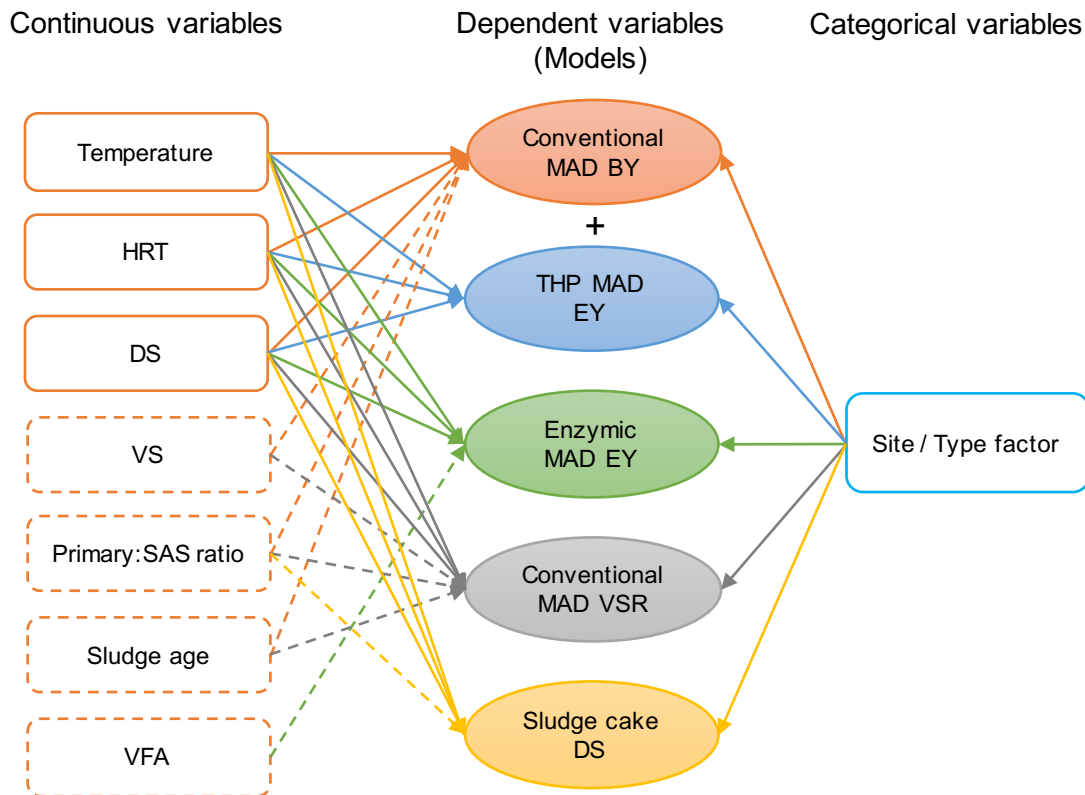


Figure 3-7 Summary of the statistical models (solid boxes: generic parameters; dashed boxes: company specific parameters)

3.7. User interface

A user interface was developed based on these models to enable operators to use the model to evaluate local AD process performance and provide recommending practical measures for process improvement and optimisation under different MAD processes. The detailed user manual and screenshot of the interface was presented in Appendix 3-4. The interface contains five sections, including temperature, DS, HRT, energy balance and model validation, which can provide a recommended operational condition to achieve the target performance set by the operator. Furthermore, the net energy balance of AD system will be calculated to assess the effects of changing operational conditions. The application of the user interface will be discussed in details in the Section 9.4.1.

Chapter 4.Descriptive and Preliminary Statistical Analysis of Biogas and Electricity Yield Data in Relation to Other Operational Parameters

4.1. General Descriptive Statistics

4.1.1. Conventional digestion sites

Site and company overall average values for the conventional MAD operational variables were calculated from the monthly mean data and are summarised in Figures 4-1 to 4-4, Table 4-1 and Appendix 4-1. The mean BY for conventional MAD sites (66 sites were included in the analysis) was 398.7 m³/t DS, which was entirely consistent with typical values reported for sewage sludge MAD (Bachmann *et al.* 2015). The overall mean DS of input raw sludge was 4.5%, the mean HRT was 21.2 days and the mean temperature was 35.9 °C. Company 2 had the highest average primary digestion temperature (36.8 °C). However, it had the lowest average BY (336.2 m³/t DS). Company 1 operated at the highest average HRT (23.6 days) with an above average BY (428.9 m³/t DS). Company 3 and 4 had an equivalent, and overall the highest, BY (441 m³/t DS), and had the lowest average digestion temperature (34.6 °C) and highest average DS input value (4.9%), respectively. The individual mean BY for each site was arranged in descending order for each company (Figure 4-1), and was in the range of 150 to 700 m³/t DS. Biogas yield varied considerably between full-scale sites (Figure 4-1), which was explained due to actual differences in AD process performance as well as relative differences in site data recording. In order to capture the maximum amount of data recorded, but also minimise the impact of poor-quality data recording on the analysis, the overall mean BY was maintained at approximately to 400 m³/t DS, representing the typical BY for MAD of sewage sludge (Bachmann *et al.* 2015). Furthermore, a novel multiple regression analysis approach was developed, described in Section 5.2, to take account of potential differences between site data recording, and to explain possible the variation caused by local data measurement and other unrecorded factors at specific sites. The site average DS feed, HRT and temperature were arranged to follow the same order as the BY data, but no apparent or direct trends were observed between the overall site average BY and HRT or temperature (*P* values for the correlation between the independent variable and BY were 0.14 and 0.77, respectively) (Figure 4-2, 4-3 and 4-4). However, a weak statistically significant negative correlation was observed between site average DS and BY (*P* = 0.03, *R*²=0.07), suggesting that DS is potentially one of the key factors controlling the process. The absence of any apparent major association between BY and these principal digestion conditions

may be expected due to the complexity of full-scale MAD processes and because digestion performance depends on multiple interacting factors (Appels *et al.*, 2008; Alepu *et al.*, 2016; Nielsen *et al.*, 2017). Therefore, more detailed correlation analysis was performed, using the monthly average data set, to scrutinise and test the statistical significance of these relationships further (Section 4.3).

Table 4-1 Summary of overall company mean values of the principal parameters collected by all of the water companies for managing conventional MAD process control

Company	DS feed (%)		HRT (days)		Temperature (°C)		BY (m ³ /t DS)	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Company 1	4.3	1.2	23.6	7.8	35.7	1.8	428.9	190.6
Company 2	4.7	1.0	20.0	5.2	36.8	2.4	336.2	126.7
Company 3	4.2	0.9	20.8	5.5	34.6	2.6	441.8	192.6
Company 4	4.9	1.2	19.0	6.3	34.9	1.9	441.4	188.5
Overall	4.5	1.1	21.2	6.9	35.9	2.2	398.7	176.3

BY- biogas yield; DS feed – dry solids feed; HRT – hydraulic retention time; SD – standard deviation. Note: Company 5 did not operate any conventional MAD sites

4.1.2. Advanced digestion sites

Site and overall average values for advanced MAD operational variables (2014 - 2016) were also calculated from the monthly data and are summarised in Figures 4-5 to 4-11 and Appendix 4-2 and 4-3. The mean EY for 18 advanced AD sites and BY for 10 advanced sites (Company 5 did not measure biogas flow) was 888.6 kWh/t DS and 456.4 m³/t DS, respectively. The mean DS and VS of THP hydrolysed digester feed sludge were 7.7 and 78.9%, respectively, and the mean HRT and temperature at THP sites were: 21.0 days and 38.6 °C, respectively. Enzymic sites typically operated at lower average DS feed, temperature and longer average HRT compared to THP sites, with values for each of these parameters equivalent to 5.3 %, 37.1 °C, and 23.0 days, respectively. The enzymic sites had slightly higher EY, 921.0 kWh/t DS, and BY, 482.4 m³/t DS, compared to THP sites, which had EY and BY values equivalent to 859.7 kWh/t DS and 447.8 m³/t DS, respectively. For THP sites, no apparent or direct trend was found between the site average DS feed, HRT and temperature data and EY (*P* values for the correlation between the independent variables and BY were 0.39, 0.36 and 0.91, respectively). Similarly, no apparent or direct trend was found between HRT and temperature, in relation to EY at enzymic sites (*P* values for the correlation between the independent variable and EY were 0.42 and 0.26, respectively). However, positive statistically significant correlations were observed between site average DS and VFA, in relation to EY (*P* = 0.02, *R*² = 0.51 and *P* = 0.02, *R*² = 0.69, respectively), and a negative statistically significant correlation was found between site

average pH and EY ($P = 0.01$, $R^2 = 0.63$). Monthly average data were used for further detailed scrutiny of the correlations between the main factors controlling BY and EY, and these results are discussed in Section 4.3.

4.2. Overall Patterns in Mean Digestion Performance by Year and Season

The overall patterns in digestion performance were assessed by year and season to determine whether there were any changes in overall process behaviour during the period of data collection. ANOVA and the plot of annual mean data for conventional BY showed there was no statistically significant difference in overall mean BY in relation to year ($P = 0.175$). Nevertheless, a modest, but consistent increase in BY was observed for 2011 to 2016, suggesting a systematic, albeit relatively small, improvement in overall mean process performance had occurred during this period (Figure 4-12). EY increased significantly in 2016 compared to the values in 2014 and 2015 at THP MAD sites ($P = 0.001$ and 0.023 , respectively). By contrast, no statistically significant differences were observed in DS feed, HRT and digestion temperature between 2016 and the previous two years when data was available for THP MAD sites. Therefore, the increased EY in THP MAD sites could be attributed to other factors, such as improved management, changes in sludge characteristics or CHP engine efficiency (Weiland, 2010; Li *et al.*, 2018). No statistically significant difference was observed for enzymic EY ($P = 0.075$) between years. However, the P value was close to the significance limit of 0.05 , and increases of 10% and 16% in EY were observed in 2015 compared to 2014 and 2017, respectively (Figure 4-12). There were no statistically significant differences in DS feed, HRT and digestion temperature between 2014, 2015 and 2016 for enzymic MAD sites ($P = 0.591$, 0.122 and 0.080 , respectively). However, considerably more data was available in 2015 (113 rows of data) compared to 2014 and 2016, which had 72 and 37 rows of data, respectively. Therefore, the apparent variation in the annual mean EY data for enzymic sites could partly be explained by the recording frequency.

Mesophilic anaerobic digestion is a managed process operated above ambient temperatures, and, ANOVA and the plot of seasonal mean data for THP EY and enzymic EY were consistent and not significantly different between seasons ($P = 0.466$ and 0.543 , respectively) (Figure 4-13). By contrast, seasonal differences were apparent in the digester performance at conventional MAD sites and BY increased significantly during the cooler winter period compared to the summer season (Figure 4-13), suggesting that lower ambient temperatures were associated with increased BY. There were also statistically significant differences observed in digestion temperature and HRT between seasons (Figure 4-14). As may be expected, digestion temperature was significantly increased in the summer period (overall mean for all sites = $36.5\text{ }^{\circ}\text{C}$) compared to the cooler winter season ($35.3\text{ }^{\circ}\text{C}$), however, HRT was reduced in summer (20.9 d) compared to winter operation (21.8 d).

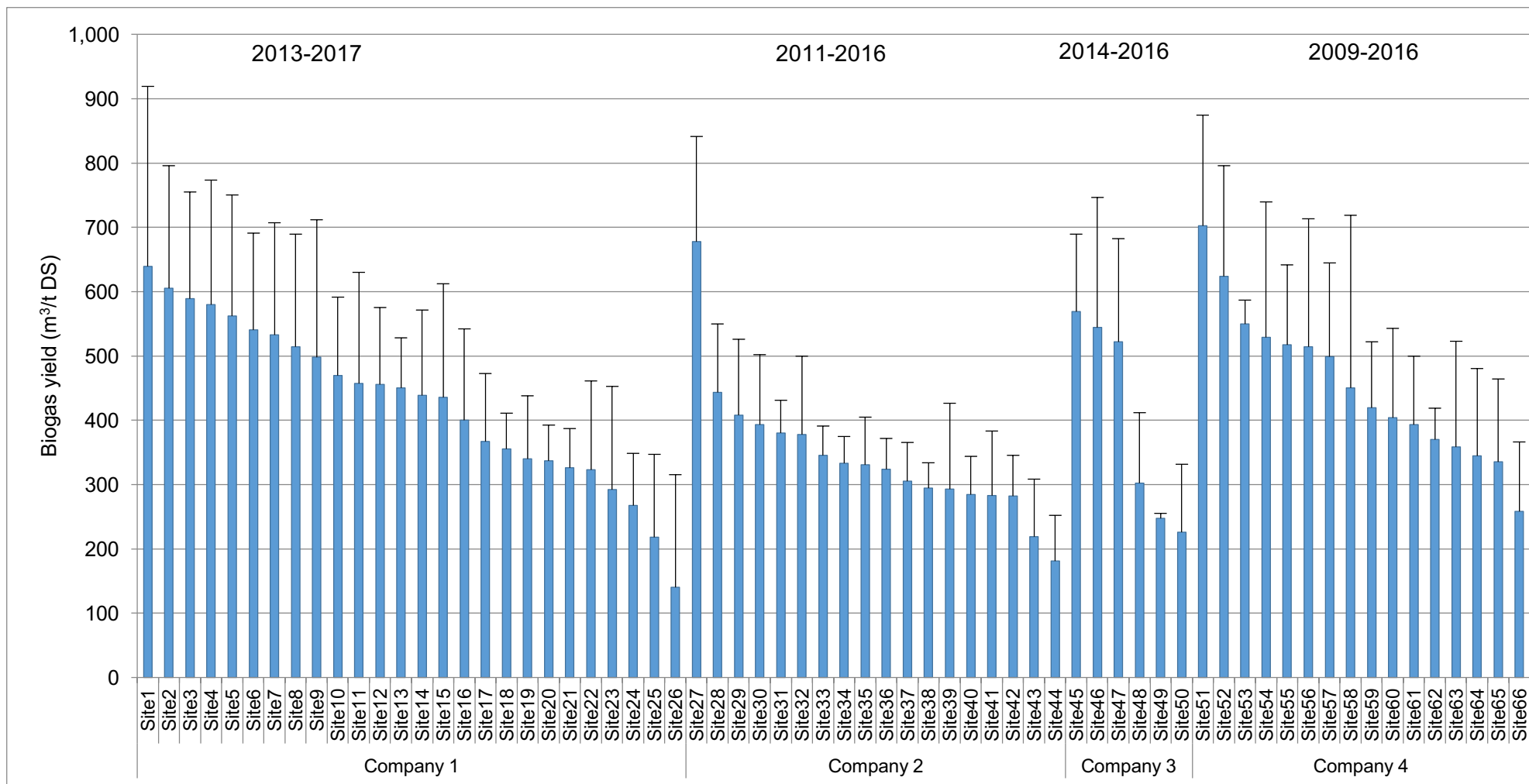


Figure 4-1 Mean biogas yield for individual conventional MAD sites, the period of data collection for each Company is also shown. Note: Company 5 did not operate any conventional MAD sites (Error bar: standard deviation)

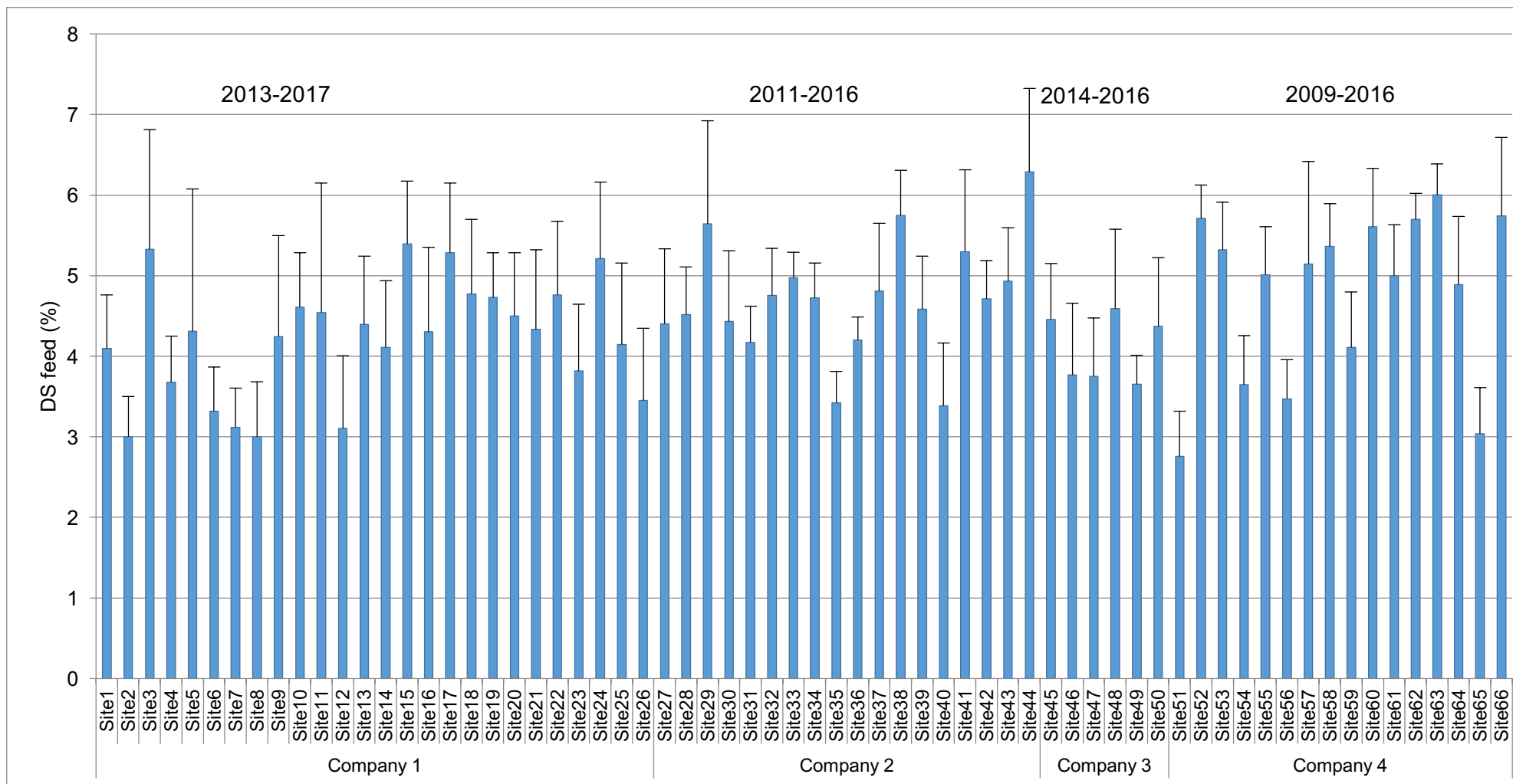


Figure 4-2 Mean DS feed for individual conventional MAD sites, the period of data collection for each Company is also shown. Note: Company 5 did not operate any conventional MAD sites (Error bar: standard deviation)

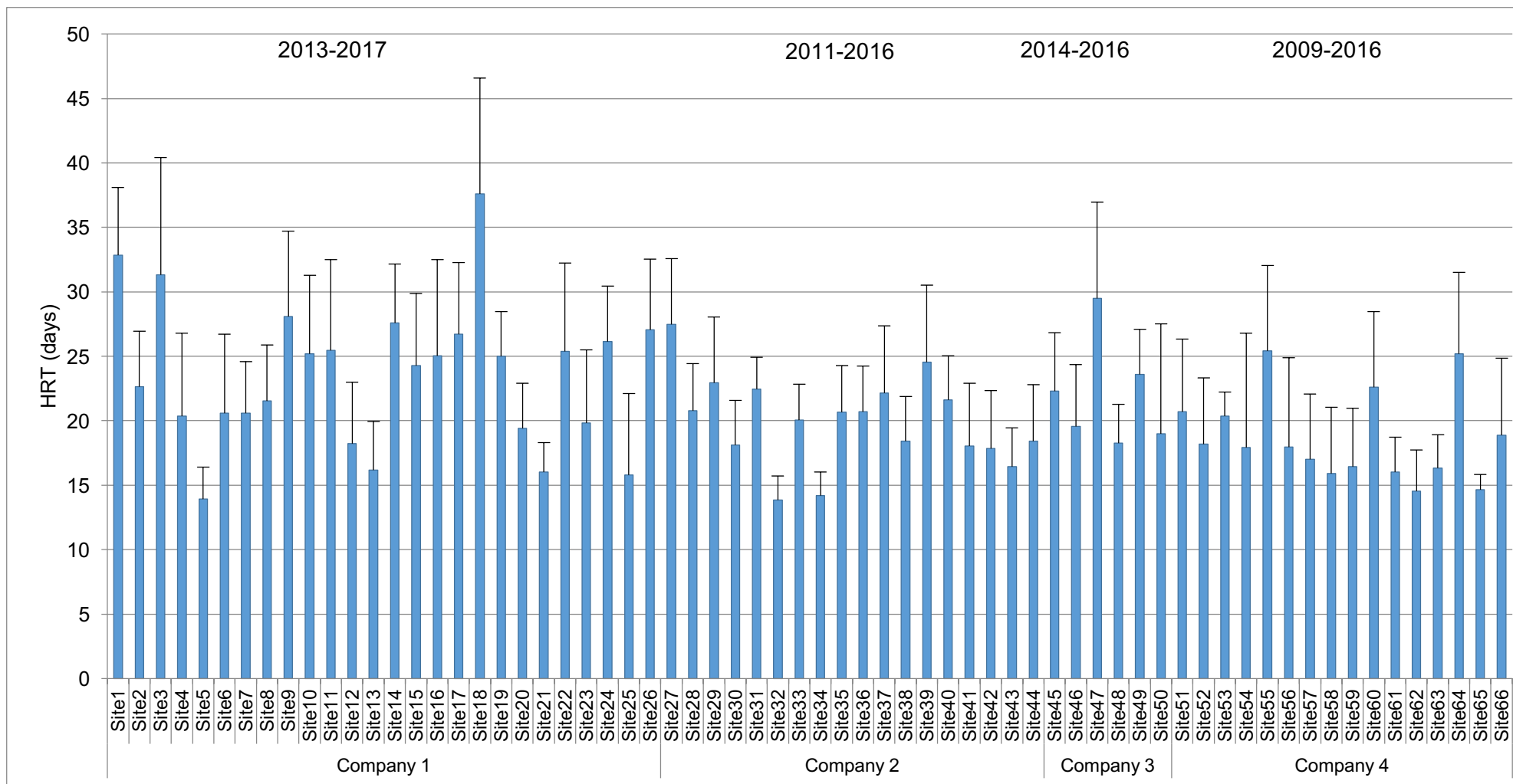


Figure 4-3 Mean HRT for individual conventional MAD sites, the period of data collection for each Company is also shown. Note: Company 5 did not operate any conventional MAD sites (Error bar: standard deviation)

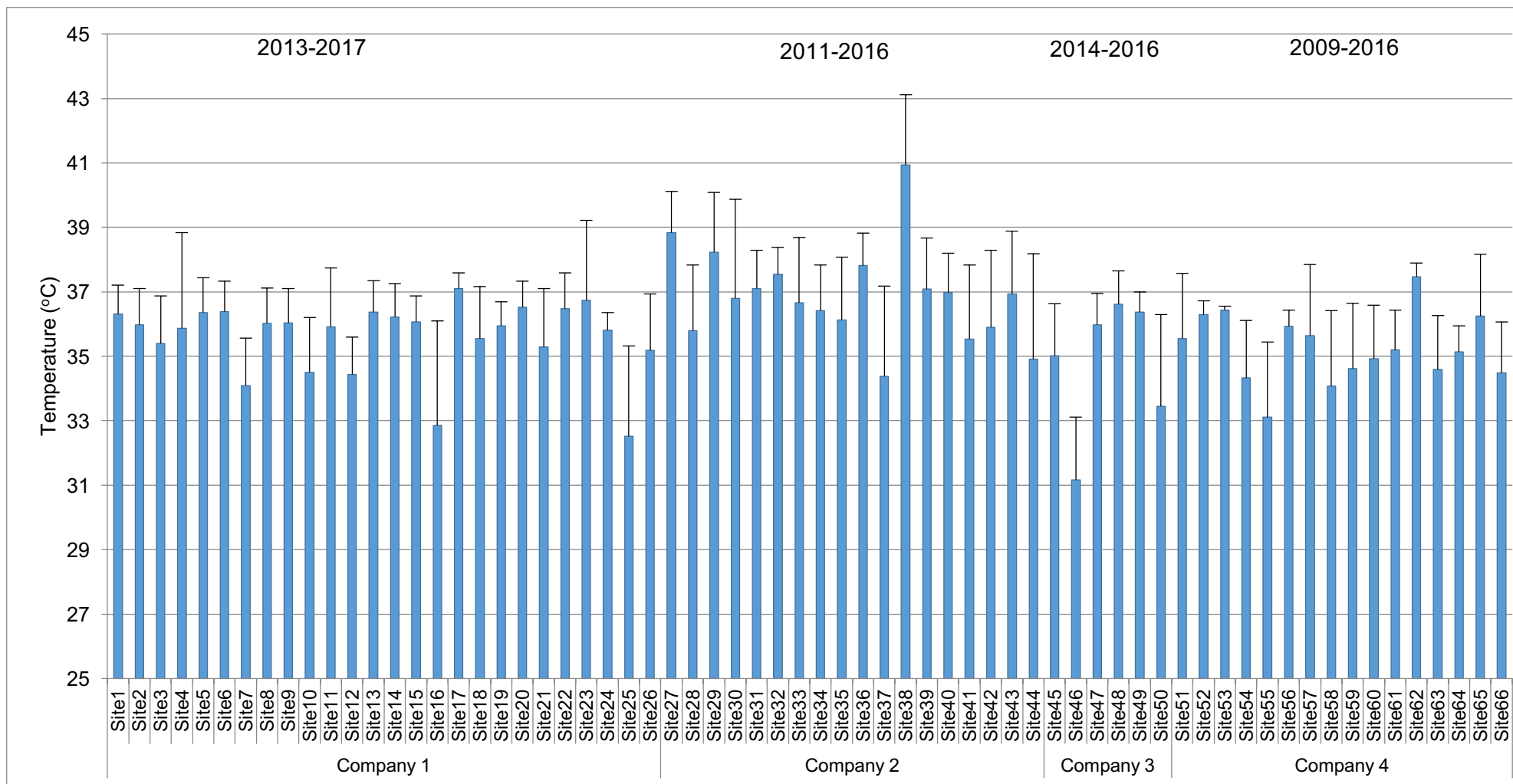


Figure 4-4 Mean temperature for individual conventional MAD sites, the period of data collection for each Company is also shown. Note: Company 5 did not operate any conventional MAD sites (Error bar: standard deviation)

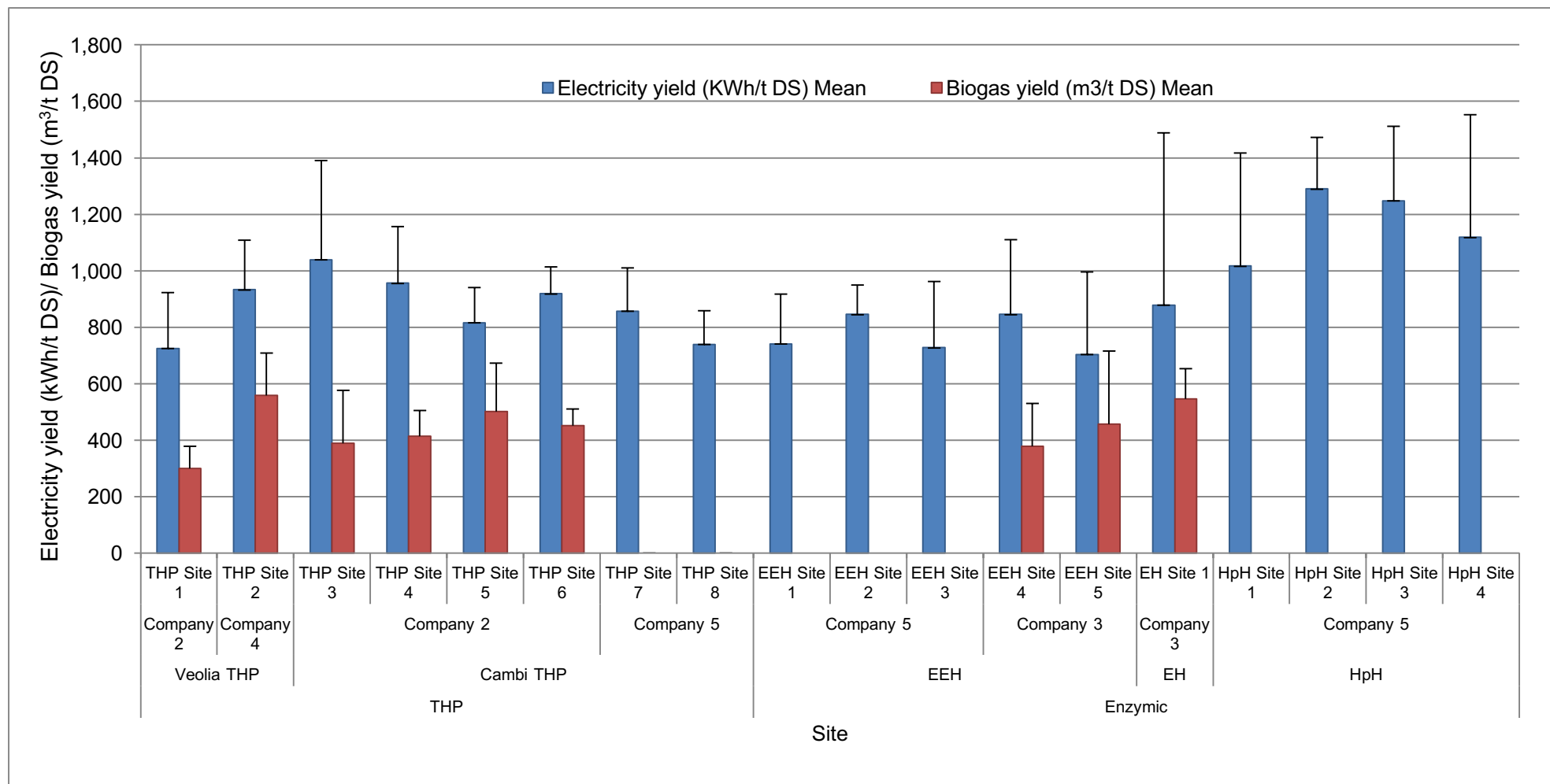


Figure 4-5 Mean electricity yield and biogas yield for individual advanced MAD sites. Note: Company 1 did not operate any advanced MAD sites and blanks indicate the data were not recorded (Error bar: standard deviation)

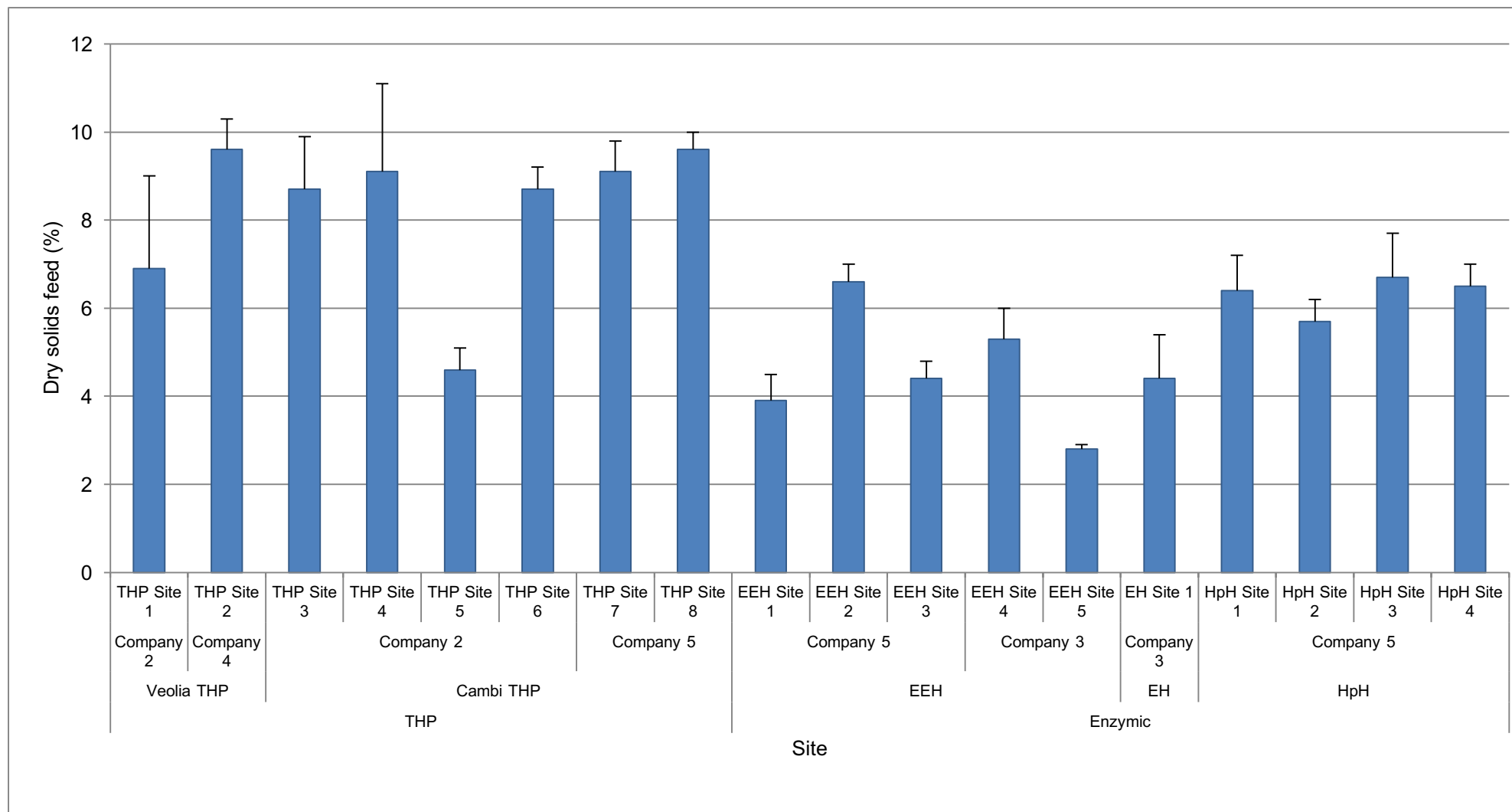


Figure 4-6 Mean DS feed for individual advanced MAD sites. Note: Company 1 did not operate any advanced MAD sites (Error bar: standard deviation)

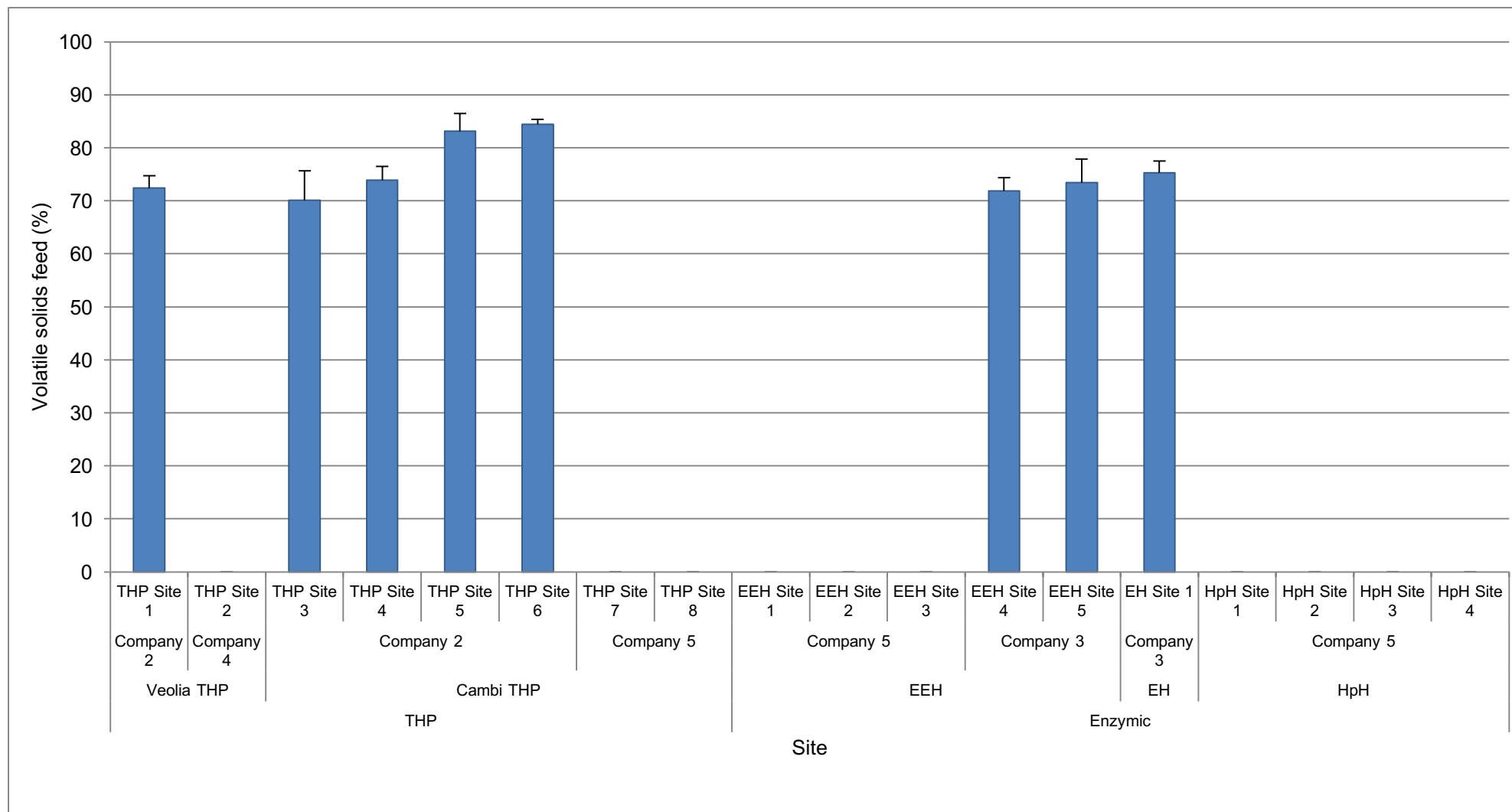


Figure 4-7 Mean VS feed for individual advanced MAD sites, Note: Company 1 did not operate any advanced MAD sites and blanks indicate the data were not recorded (Error bar: standard deviation)

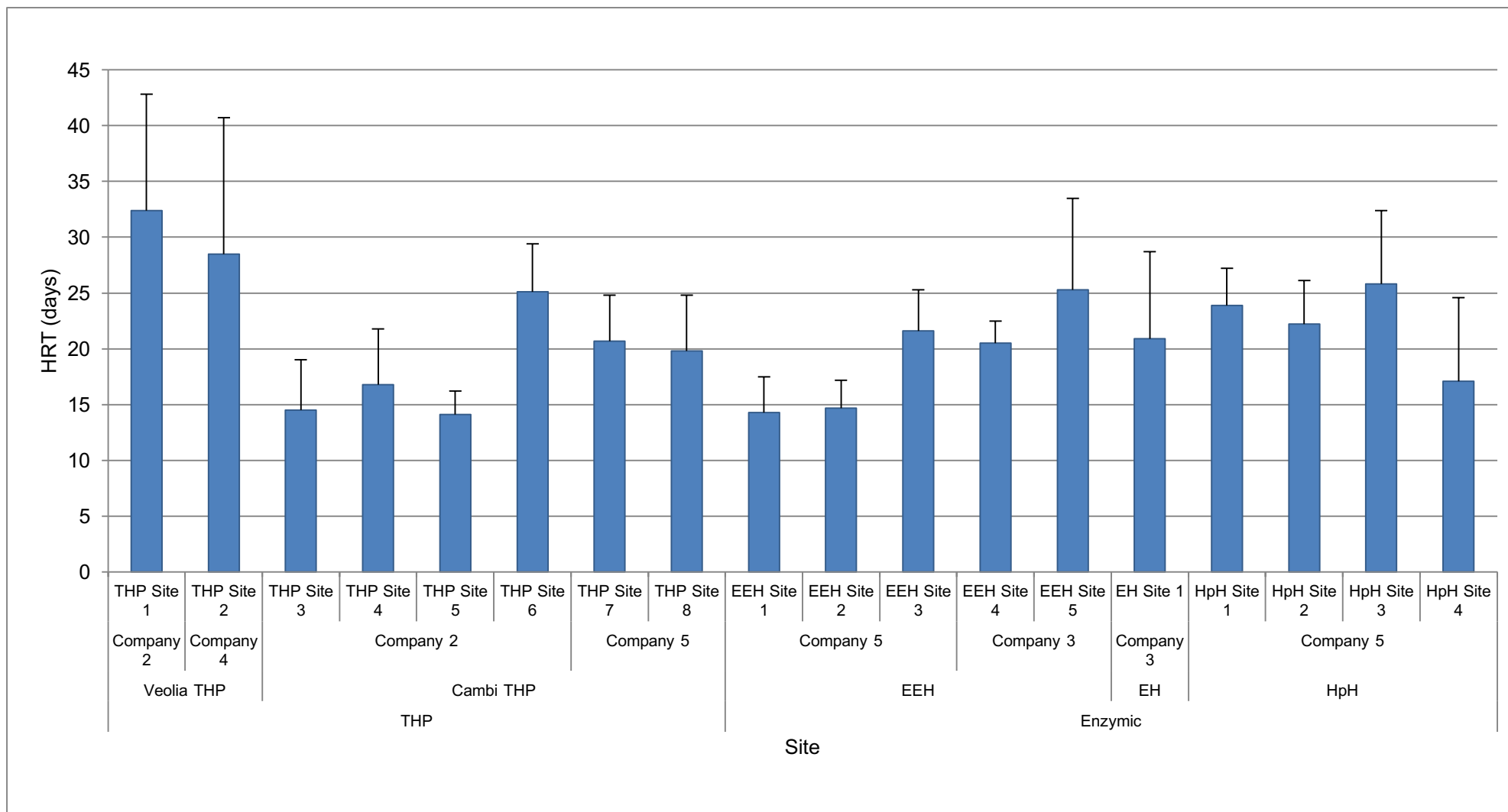


Figure 4-8 Mean HRT for individual advanced MAD sites, Note: Company 1 did not operate any advanced MAD sites (Error bar: standard deviation)

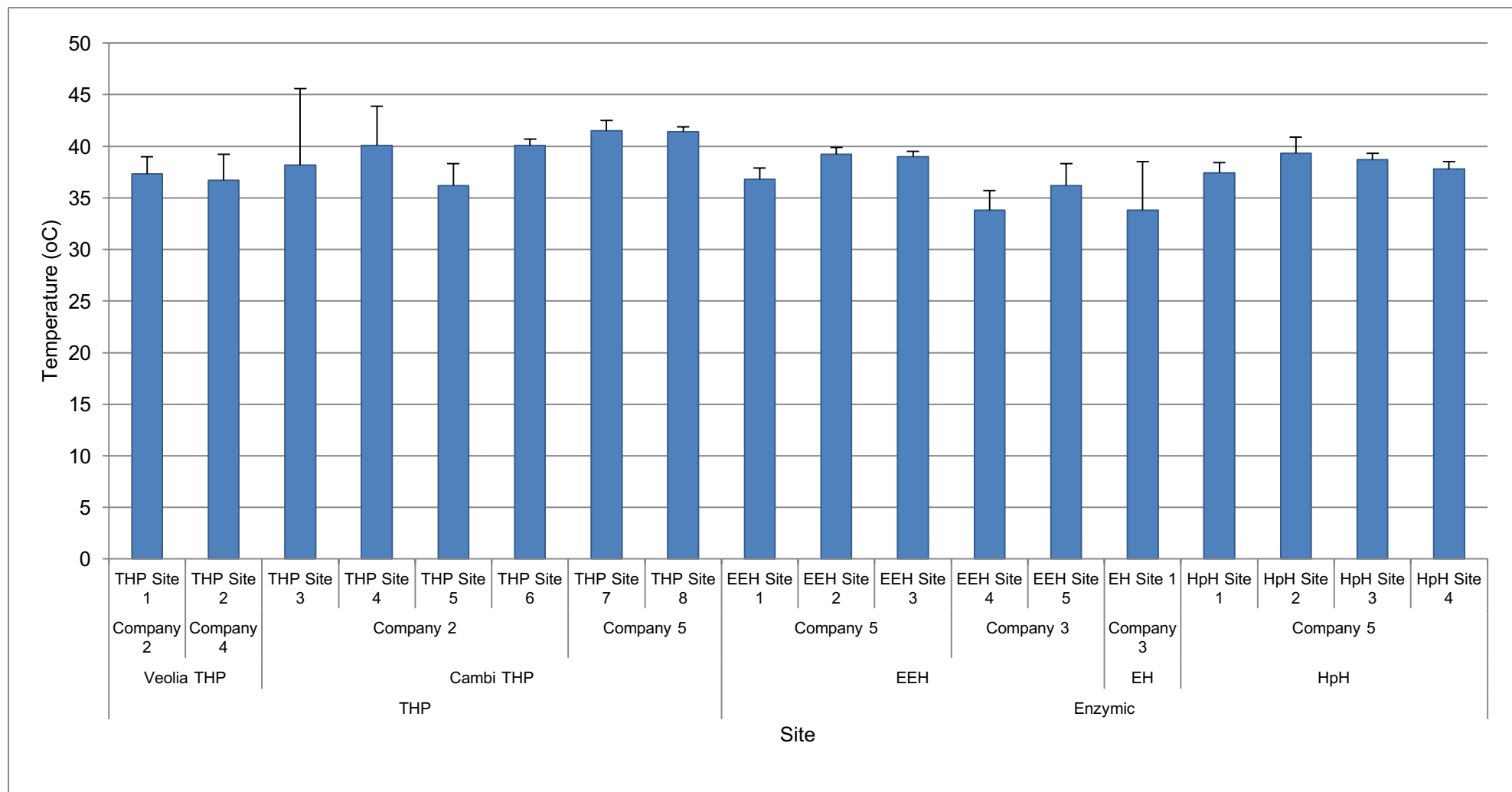


Figure 4-9 Mean Temperature for individual advanced MAD sites, Note: Company 1 did not operate any advanced MAD sites (Error bar: standard deviation)

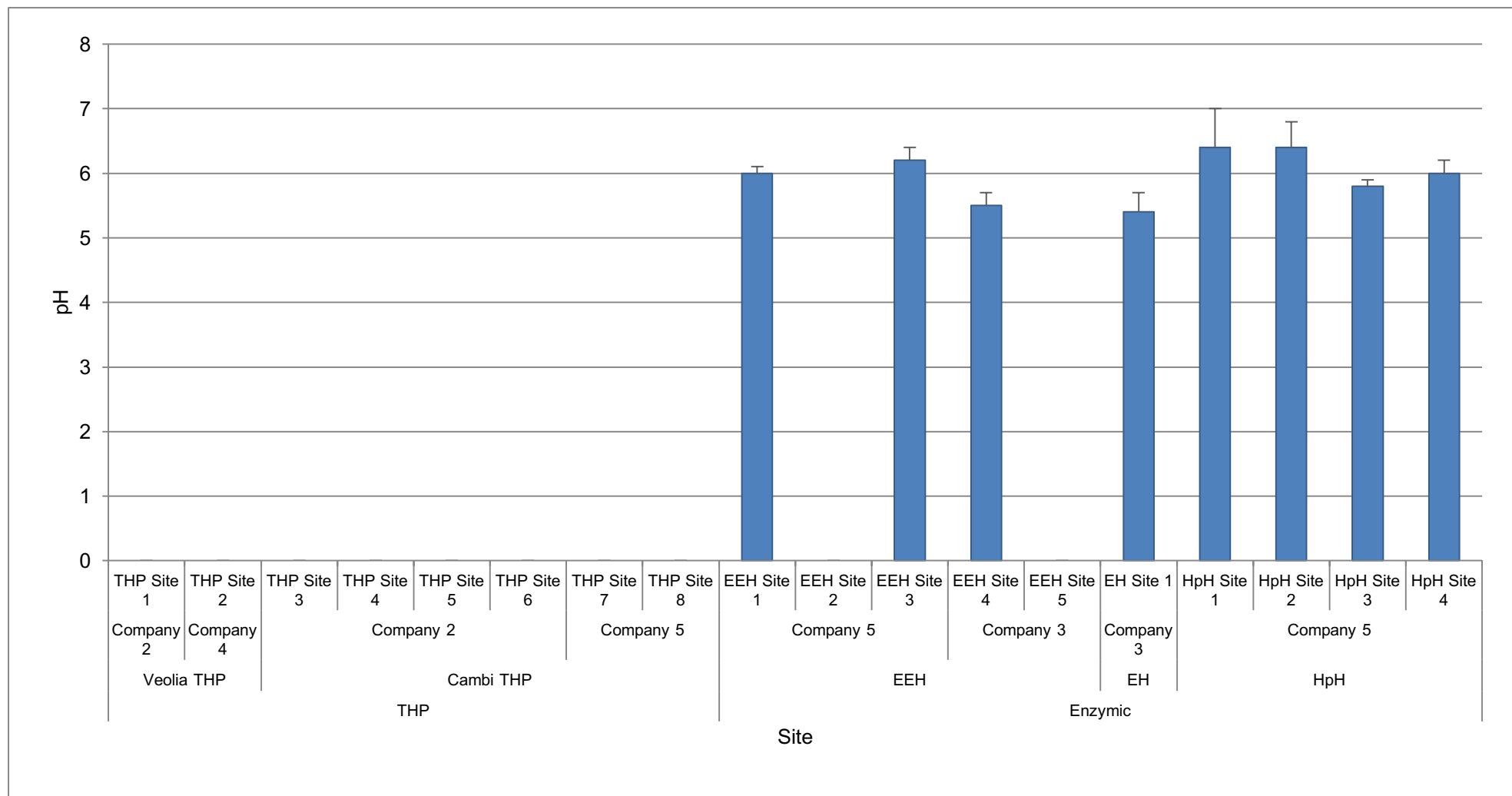


Figure 4-10 Mean pH feed for individual advanced MAD sites, Note: Company 1 did not operate any advanced MAD sites and blanks indicate the data were not recorded (Error bar: standard deviation)

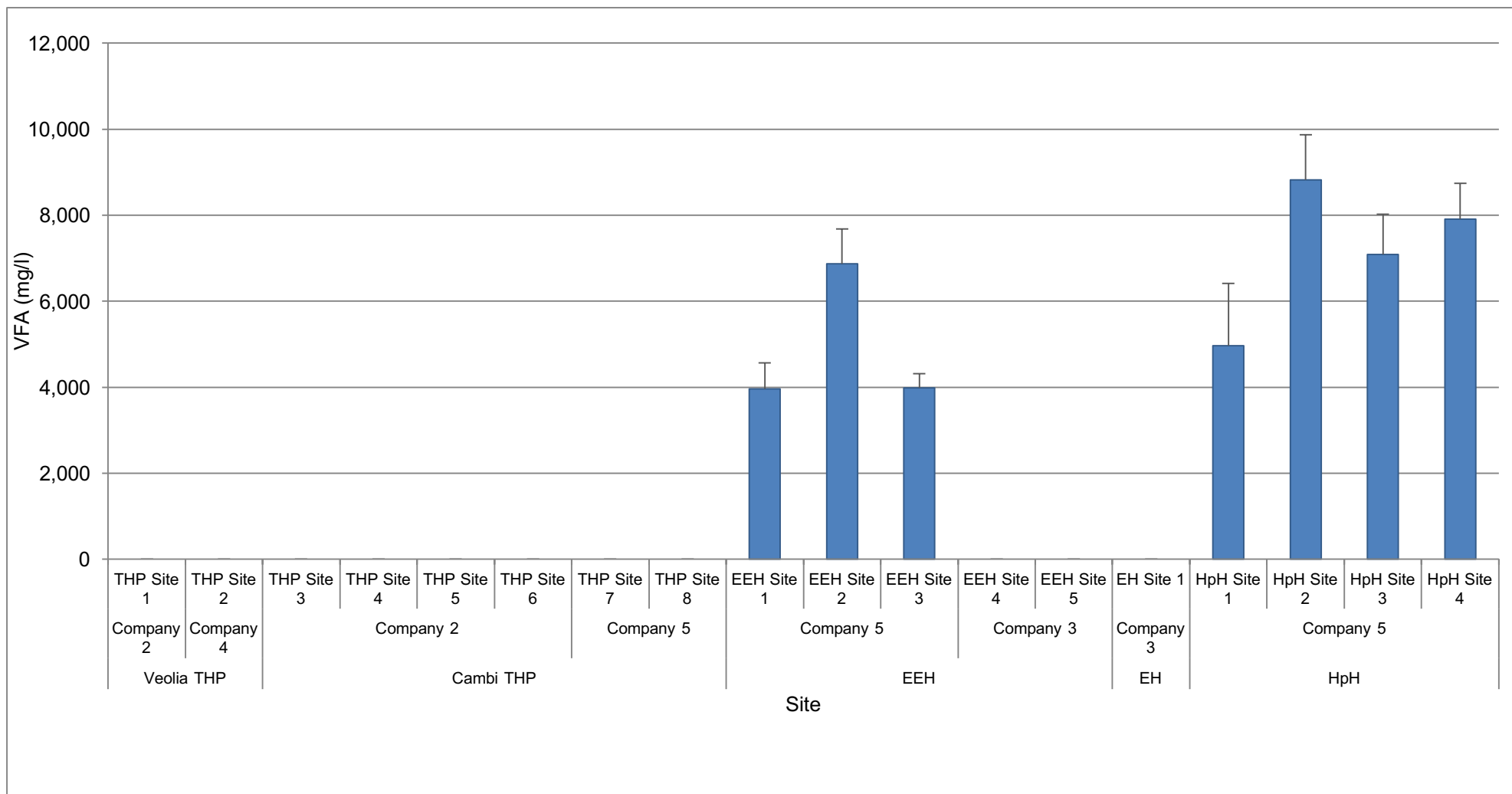


Figure 4-11 Mean VFA feed for individual advanced MAD sites, Note: Company 1 did not operate any advanced MAD sites and blanks indicate the data were not recorded (Error bar: standard deviation)

ANOVA of seasonal mean data for DS feed, indicated the feeding concentration was consistent and not significantly different between seasons ($P = 0.248$), with an overall mean value equivalent to 4.5%. The VS feed data from Company 2 indicated that there was a statistically significant increase in the winter period compared to the warmer summer period, although the magnitude was relatively small (76.4% compared to 75.1%, respectively) (Figure 4-14). Therefore, the improved BY in winter could be attributed to the combined effect of both increased HRT and VS feed observed in conventional MAD sites.

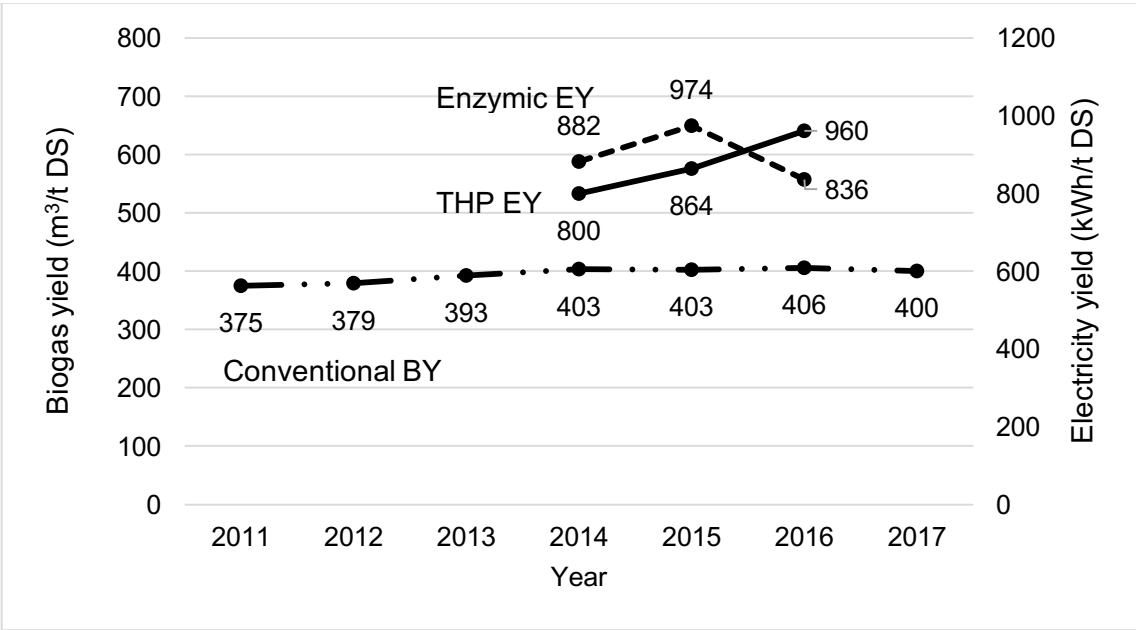


Figure 4-12 Mean annual conventional BY, THP EY and enzymic EY

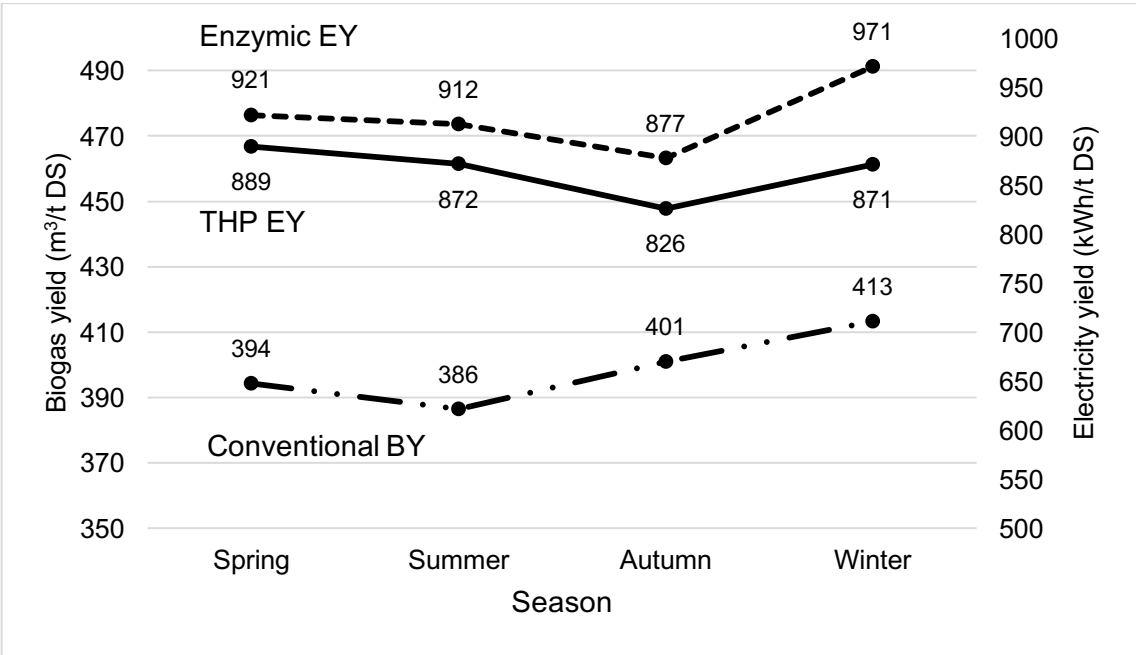


Figure 4-13 Mean conventional BY, THP EY and enzymic EY in relation to season

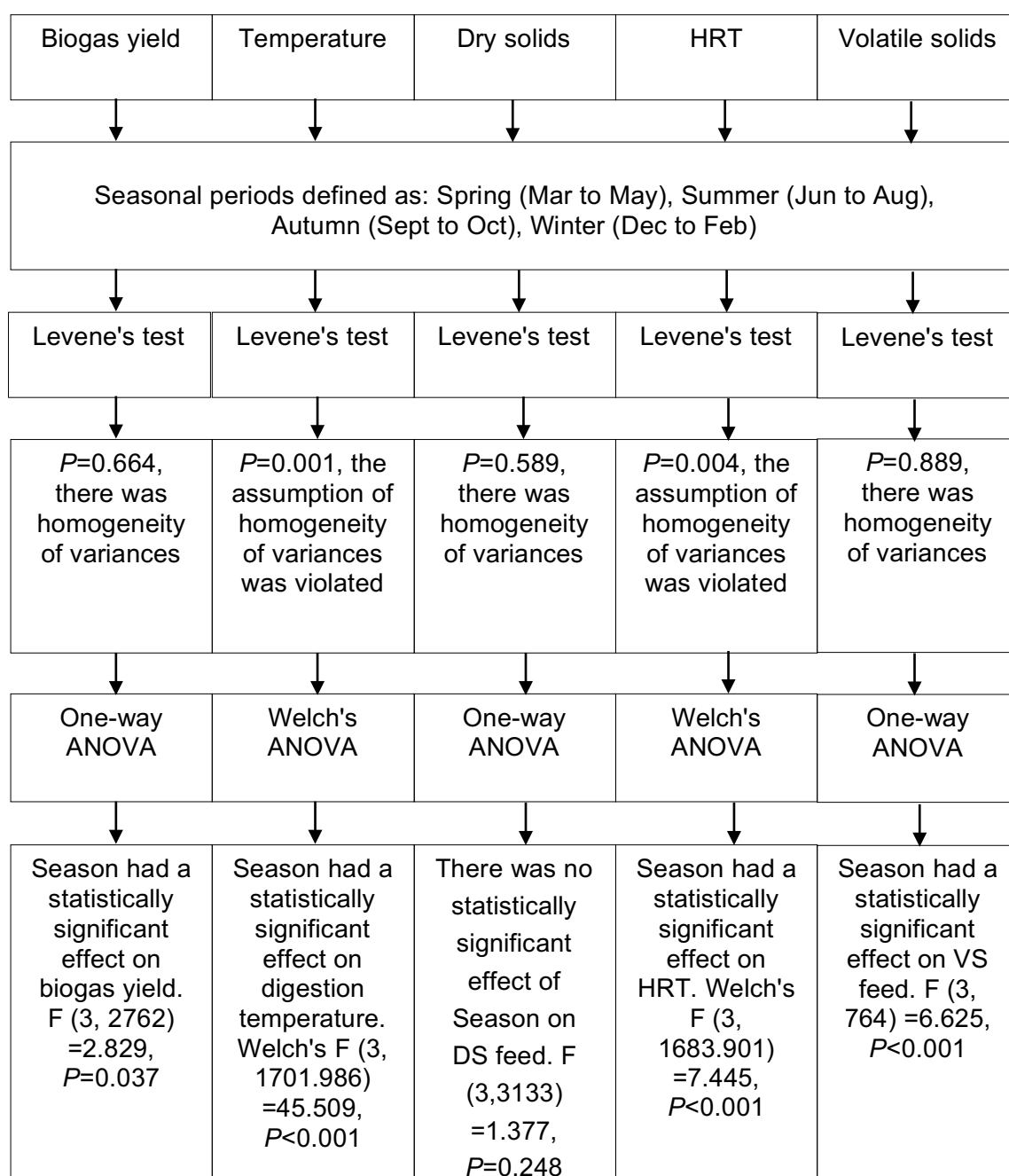


Figure 4-14 Analysis of variance flow diagram for effects of season on biogas yield and digestion conditions (note: VS data was from company 2 only)

4.3. Correlation Between Digestion Performance and Physico-chemical Variables

The linear correlation analysis and the corresponding scatter plots of the possible input parameters against conventional BY, THP EY and enzymic EY, based on monthly average data, are shown in Table 4-2 and Appendix 4-4 to 4-6, respectively. The results of conventional MAD sites test (Table 4-2) indicated that HRT, VS feed, primary SAS ratio and VSR were statistically significant ($P < 0.05$) and positively associated with BY. By contrast, DS feed was statistically significant ($P < 0.001$) and negatively associated with BY at conventional MAD sites and is consistent with the overall site

average data. No statistically significant effect was detected between temperature and BY at conventional MAD sites, and this may be explained because, in practical circumstances where a wide range of other contrasting and interacting conditions are represented, as is the case here for 66 conventional MAD treatment sites, and where the temperature range represents a relatively narrow operating range, temperature may not have a significant main effect on BY. By contrast, in simple laboratory experiments with linear, controlled temperature conditions, BY is shown to increase in relation to temperature, and this is particularly observed when the range of temperature treatments deliberately include suboptimal values (Wang *et al.*, 2019). Furthermore, the linear correlation analysis does not take the overall effects of multiple parameters or non-linear relationships into account, and hence the effect of temperature on BY may not be detected. Therefore, cluster analysis was performed to evaluate the interrelationships between, and influence of, principal operational parameters collected by all of the water utility companies, including temperature, DS and HRT, on digester performance (Section 4.4).

The results of THP MAD sites test (Table 4-2) indicated that both DS feed and temperature were statistically significant ($P < 0.05$) and positively associated with EY. Similarly, DS feed, temperature and VFA feed were also identified as positively statistically significant factors ($P < 0.05$) controlling EY at enzymic MAD sites (Table 4-2). In contrast to conventional MAD sites, the positive effect of DS feed at advanced sites could be attributed to the pretreatment which improved AD capacity to treat higher solids input as discussed latter in Section 4.5.

4.4. Cluster Analysis

4.4.1. Introduction to cluster analysis

Cluster analysis was introduced to assess the effects of three critical AD operational variables: HRT, DS feed and temperature, on BY and EY, using monthly average data. This approach enabled the visualisation of the data distribution of these principal operational variables and how their interrelationships influenced the digestion performance.

4.4.2. Conventional MAD

A two-step cluster analysis technique was applied to the monthly average data for the operational variables for 66 conventional MAD sites, which automatically selected the optimal cluster number, based on a cluster evaluation algorithm, the silhouette coefficient, as described in Section 3.5.4. The cluster analysis divided the digestion conditions into four separate groups according to the HRT, DS feed and temperature values (Figure 4-15). The majority of the data points were distributed between clusters 2 and 4, and the proportion of the total data set allocated to these clusters was equivalent to 34% and 31%, respectively (Table 4-3). The mean BY varied for the data clusters representing the different digestion conditions, shown in Figure 4-15. Cluster 2 and 4 had similar mean HRT and

temperature values, equivalent to approximately 19.0 days and 36.5 °C, respectively. However, Cluster 4 had lower DS feed (3.5%) compared to Cluster 2 (5.3%), but the mean BY (442.1 m³/t DS) for Cluster 4 was 22% higher compared to Cluster 2. Therefore, the results confirmed the correlation analysis and showed that increasing DS feed reduced the BY, consistent with the generally reported observation that higher DS concentrations can negatively influence CH₄ production, as discussed in Section 2.3.2.

Table 4-2 Linear correlation tests between physico-chemical parameters and digester performance indicators (BY and EY) at conventional, THP and enzymic MAD sites

Target variable	Parameter	Number of data	Significance (<i>P</i>)	R ²	Positive or negative correlation (+/-)
Conventional BY	DS feed	2726	<0.001	0.071	-
	HRT	2708	<0.001	0.029	+
	Temperature	2702	0.362	0.000	+
	VS feed	804	<0.001	0.017	+
	Primary SAS ratio	825	0.001	0.013	+
	Sludge age	844	0.092	0.003	+
	VSR	542	<0.001	0.056	+
THP EY	DS feed	183	0.001	0.064	+
	HRT	199	0.512	0.002	+
	Temperature	195	0.036	0.022	+
Enzymic EY	pH feed	138	0.810	0.000	-
	VFA feed	187	<0.001	0.140	+
	DS feed	203	0.002	0.047	+
	HRT	199	0.560	0.002	-
	Temperature	198	0.006	0.038	+

Cluster 3 data was also represented by a relatively high mean DS feed (5.0%), which was similar to Cluster 2, but in this case the mean HRT and temperature were lower, and equivalent to 17.9 days and 32.6 °C, respectively. Statistical comparison of Cluster 2 and 3 showed there was no statistically significant difference in the mean BY between these digestion conditions (*P* = 0.59). Thus, the cluster analysis suggested that the DS of the feed sludge had a dominant effect on the BY and that, by contrast, temperature had a relatively weaker effect across the range of temperature conditions recorded at operational digestion sites. The mean DS feed and temperature in Cluster 1 were similar to the overall average values for conventional MAD sites (Table 4-1), and were equivalent to 4.6% and 35.9 °C, respectively. However, Cluster 1 had the highest mean BY (442.1 m³/t DS), and this was probably explained due to the extended HRT (32.1 days) represented by Cluster 1, which allows a greater conversion of complex organic matter to CH₄ and CO₂ (Parkin and Owen, 1986).

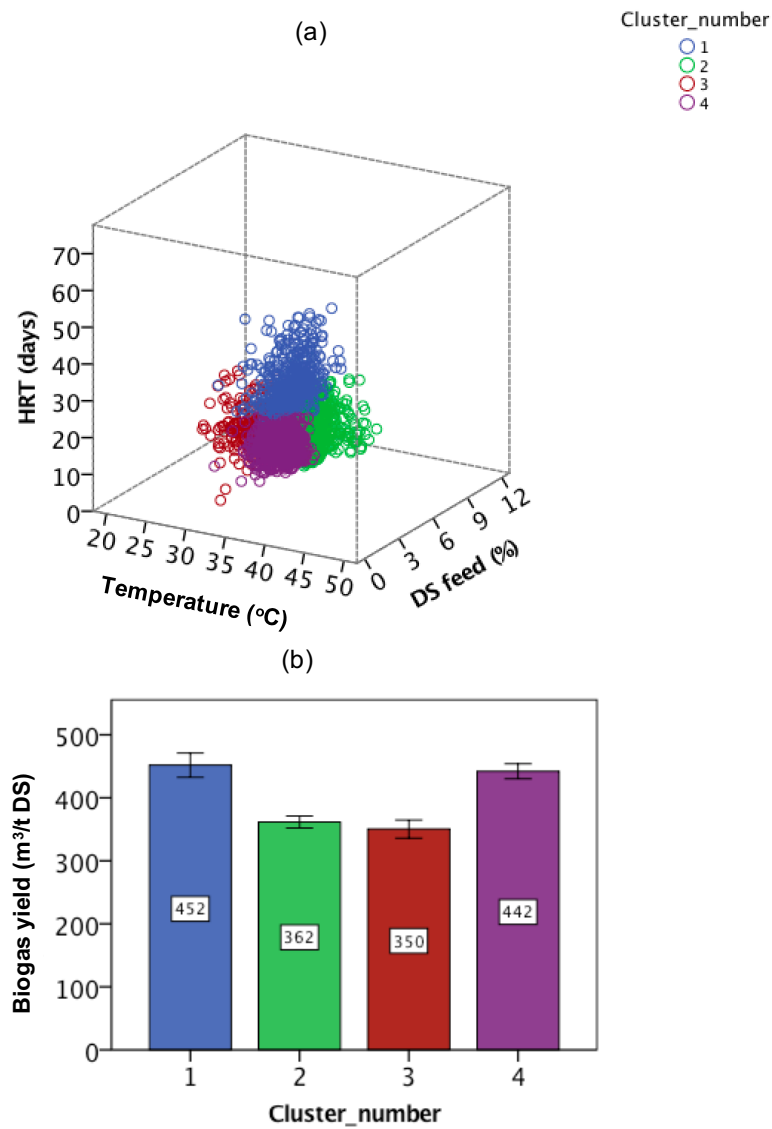


Figure 4-15 Clusters representing HRT, DS feed and temperature data distribution patterns and their corresponding BY for the conventional MAD dataset (Error bar: 95% confidence interval)

Table 4-3 Mean values for the DS feed, HRT, temperature and biogas yield represented by different clusters for conventional MAD sites

Cluster	Cluster size (% of total data represented)	DS feed (%)	HRT (days)	Temperature (°C)	Biogas yield (m ³ /t DS)
1	19	4.6	32.1	35.9	451.9
2	34	5.3	19.5	37.2	361.5
3	16	5.0	17.9	32.6	350.4
4	31	3.5	18.4	36.2	442.1
Overall	100	4.5	21.2	35.9	398.7

Note: The total number of mean monthly data is equivalent to 2929

4.4.3. THP MAD dataset

The monthly average values of operational variables from 8 THP MAD sites were divided into 3 clusters according to their HRT, DS feed and temperature (Figure 4-16). Cluster 1 had the highest mean EY (912 kWh/t DS), and contained approximately 60% of the total THP data. This Cluster represented digesters operating at high DS and temperature, equivalent to mean values of 9.2% and 40.4 °C, respectively (Figure 4-16 and Table 4-4). Cluster 3, by contrast, had lower mean EY (776.1 kWh/t DS) corresponding to the lowest mean HRT, DS feed and temperature values, which were equivalent to: 21.0 days, 4.6% and 36.1 °C, respectively. Cluster 2 accounted for 11.6% of the total data for THP sites, and represented a relatively extreme set of conditions corresponding to an extended HRT, but with relatively low temperature. Thus, the mean HRT was almost twice the mean value for Cluster 1, at 41.7 days (Cluster 1 = 20.2 days), however, the temperature was reduced to 36.9 °C (Cluster 1 = 40.4 °C). Interestingly, no statistically significant effect of digestion conditions ($P = 0.82$) on EY was detected between Cluster 1 and 2 and this may be explained because a potentially negative effective of temperature on EY was possibly offset by the extended HRT in Cluster 2. Therefore, the results clearly showed that the performance of THP sites was strongly influenced by digestion conditions, and also that similar EY can be achieved with different combinations of HRT, DS feed and temperature.

4.4.4. Enzymic MAD dataset

Similarly, two-step cluster analysis technique was applied to the monthly average data for the operational variables for 10 enzymic MAD sites, and enzymic MAD data were grouped into 3 clusters according to their HRT, DS feed and temperature (Figure 4-17). Cluster 1 had the smallest overall mean EY (771.4 kWh/t DS), DS (4.1%) and temperature (33.2 °C), but the longest HRT (47.9 days). The positive effect of HRT on EY was possibly offset by the reduced temperature and DS in Cluster 1. Most of the data fell within clusters 2 and 3, which accounted for 42.1% and 48.2% of the total data, respectively (Figure 4-17 and Table 4-5). Cluster 3 had the highest overall mean EY (1030 kWh/t DS) corresponding to the highest mean DS feed (6.5%) and temperature (38.4 °C). There was a 22% decrease in EY for Cluster 2 compared to Cluster 3, and this was explained because Cluster 2 corresponded to a lower DS feed (mean = 4.2%) and also a modest reduction in mean temperature, equivalent to 2 °C, relative to Cluster 3. Therefore, the results of the cluster analysis suggested that enzymic MAD EY responded to changing DS feed concentration in a similar manner to THP MAD sites. A further scrutiny of the site distribution showed that HpH sites were dominant in Cluster 3 (72%) (Table 4-6), and therefore overall HpH were the highest performing enzymic MAD processes.

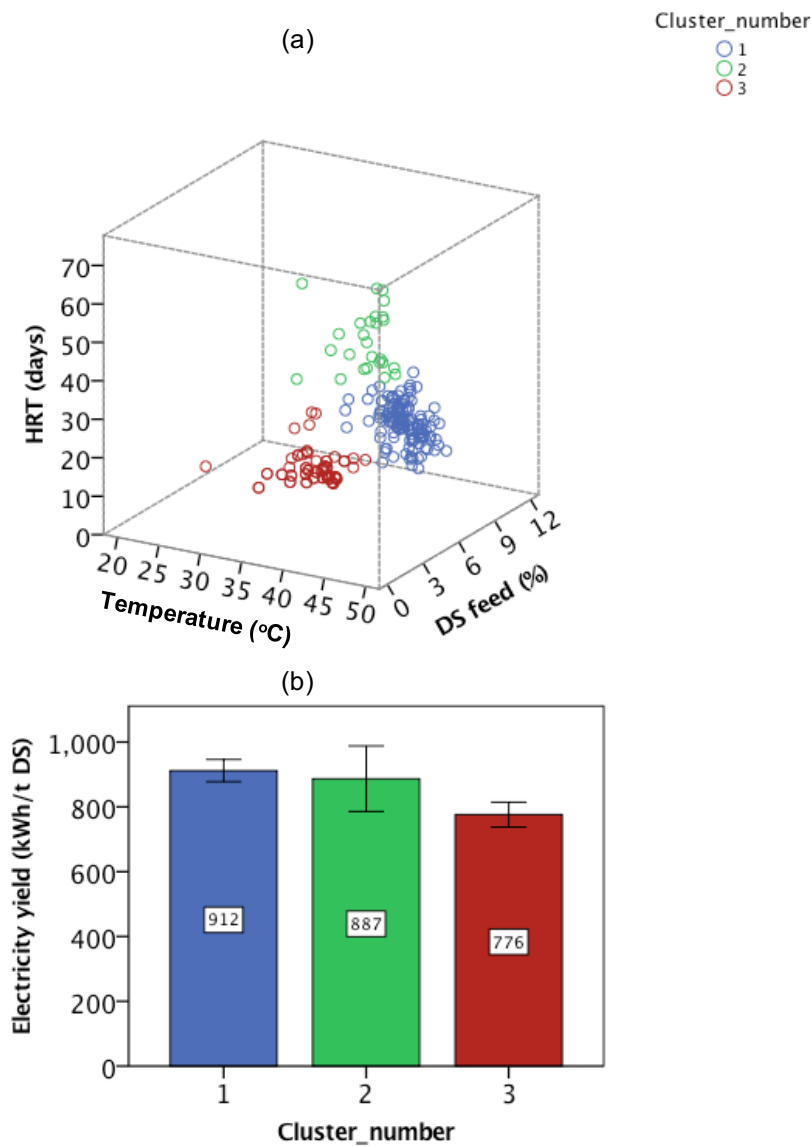


Figure 4-16 Clusters representing HRT, DS feed and temperature data distribution patterns and their corresponding EY for the THP MAD dataset (Error bar: 95% confidence interval)

Table 4-4 Mean values for DS feed, HRT, temperature and electricity yield represented by different clusters for THP MAD sites

Cluster	Cluster size (% of total data represented)	DS feed (%)	HRT (days)	Temperature (°C)	Electricity yield (kWh/t DS)
1	57.3	9.2	20.2	40.4	911.8
2	11.6	8.4	41.7	36.9	886.8
3	31.1	4.6	15.1	36.1	776.1
Overall	100	7.7	21.0	38.6	859.7

Note: The total number of mean monthly data is equivalent to 225

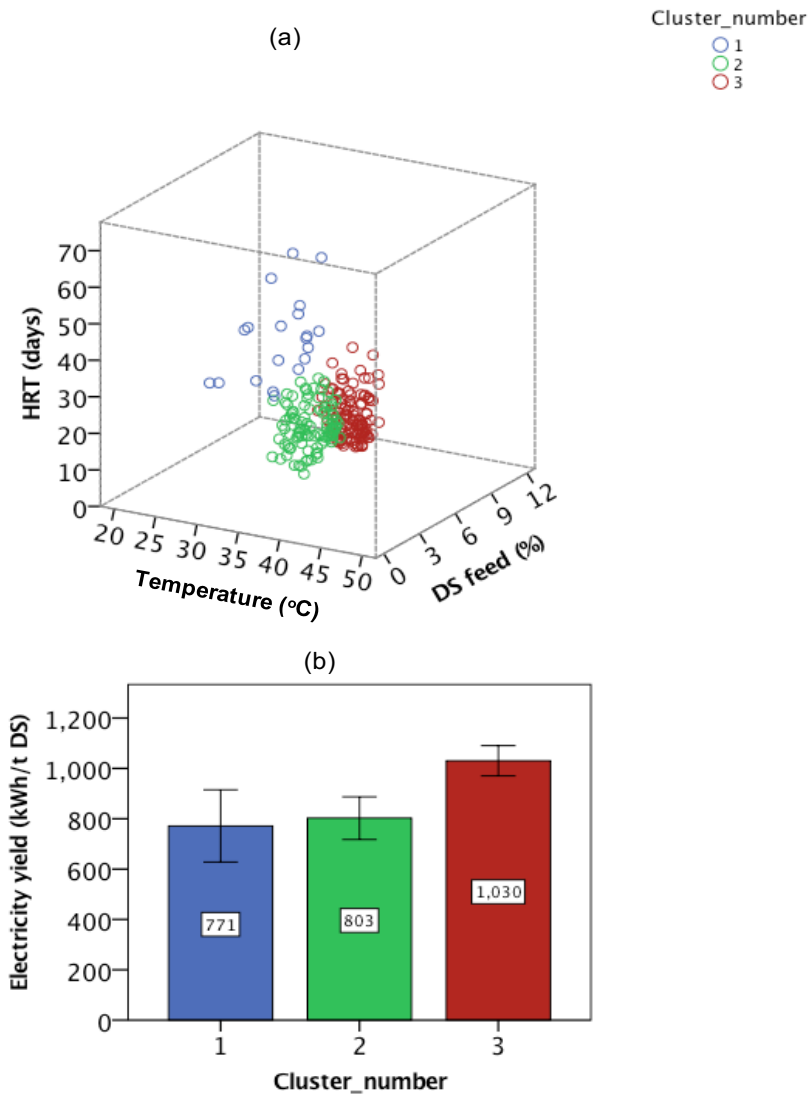


Figure 4-17 Clusters representing HRT, DS feed and temperature data distribution patterns and their corresponding EY for enzymic MAD dataset (Error bar: 95% confidence interval)

Table 4-5 Mean values of DS feed, HRT, temperature and electricity yield represented by different clusters for enzymic MAD sites

Cluster	Cluster size (% of total data represented)	DS feed (%)	HRT (days)	Temperature (°C)	Electricity yield (kWh/t DS)
1	9.6	4.1	47.9	33.2	771.4
2	42.1	4.2	20.7	36.6	802.5
3	48.2	6.5	20.3	38.4	1030.3
Overall	100	5.3	23.0	37.1	921.0

Note: The total number of mean monthly data is equivalent to 228

Table 4-6 Number of mean monthly data values for different enzymic treatment types in each cluster

Cluster	1		2			3		
Enzymic type	EEH	EH	EEH	EH	HpH	EEH	EH	HpH
Count	15	7	70	22	4	29	2	79

4.5. Discussion

Chapter 4 provides a comparison of the operational conditions represented at the different AD sites and presented a preliminary statistical analysis of BY and EY data in relation to other key operational parameters. The results illustrate the variable performance of both conventional and advanced MAD processes across the Industry in relation to site, year and season (Figure 4-1, 4-5, 4-11 and 4-13). From a statistical analysis point of view, the wide range of performance could be helpful to detect the main significant operational parameters that influence the process. The variation of performance could be attributed to actual differences in the AD process reflecting the effects of different operational conditions, improved management, sludge characteristics or CHP engine efficiency, as well as the relative differences in site data recording. For example, the overall mean BY increased during the winter season at conventional MAD sites, compared to other seasons and this corresponded with an increase in mean HRT and VS feed. We suspected this behaviour could be attributed to the conservation of easily digestible organics in the sewer during the cooler winter period, as this was consistent with the sludge sampling results which showed higher VS contents during cooler months at conventional MAD sites in Figure 7-3 (a) (Section 7.3).

In general, no apparent or direct trends were observed between most of the site average operational parameters in relation to BY and EY at conventional and advanced MAD sites. However, further detailed linear relationship testing of monthly average data detected several significant predictors of BY and EY. However, the contribution of individual predictors was small and could not effectively describe the variation observed in overall digestion performance. The weak association between digestion performance and the principal digestion conditions of full-scale processes is explained due to the complexity of industrial level MAD and because digestion performance depends on multiple interacting factors. Furthermore, simple linear relationships cannot account for differences in site variation caused by the interaction between different operating parameters or other unmeasured factors, such as process microbiology, feed properties and engineering controls. Nevertheless, the results of conventional MAD site tests (Table 4-2) showed that DS feed was statistically significant ($P < 0.001$) and negatively associated with BY. This result is consistent with the overall site average data, which showed that higher DS concentrations in feed sludge negatively influenced biogas production (Section 4.1). Increasing DS causes an exponential rise and decrease in sludge viscosity

and diffusive coefficient, respectively (Liao, *et al.*, 2014; Zhang *et al.*, 2015), which reduces the mixing efficiency and contact between substrate and microbial population (Parkin and Owen, 1986). Therefore, higher DS concentrations in feed sludge can negatively influence the CH₄ yield (Jolis, 2008). By contrast, HRT, VS feed, primary SAS ratio and VSR were statistically significant ($P < 0.05$) and positively associated with BY at conventional MAD sites. Adequate retention maintains a sufficient quantity and concentration of bacteria in the digester for the efficient conversion of complex organic matter to CH₄ and CO₂ (Parkin and Owen, 1986), and hence longer HRT is beneficial for biogas production. Similarly, the higher VS feed concentrations and primary SAS ratio improved the organic content and biodegradability of the mixed sludge (Bolzonella *et al.*, 2005; Batstone and Jensen, 2011; Mottet *et al.*, 2010), and hence have positive impacts on the biogas production.

In contrast to conventional MAD sites, the positive effect of DS feed at advanced sites could be attributed to the pretreatment which improved the sludge digestibility through the destruction of cellular material and long chain organic fatty acids (Gurieff *et al.* 2011). Additionally, it changes the sludge rheology by reducing the viscosity, and hence increases AD capacity to treat higher solids inputs (Appels *et al.*, 2008; Whitlock *et al.*, 2009; Zupančič and Grilc 2012) as discussed in Section 2.3.8. VFA feed was an additional measured parameter, which was statistically significant and positively associated with EY at enzymic sites ($P < 0.001$). VFA is an intermediate product during AD and is produced by the acidogenesis step of the process, where simple monomer products from hydrolysis are converted into VFAs (Madsen *et al.*, 2011). VFA accumulation also provides a stability indicator of AD, as discussed in Section 2.3.5; accumulated VFAs indicate a biological imbalance due to methanogen inhibition reducing the effective consumption rate (Franke-Whittle *et al.*, 2014). Enzymic pretreatment can be considered as a multi-stage AD system where the hydrolysis and acidogenesis processes are encouraged to proceed as far as possible prior to the conventional MAD process, thus assisting in overall process stability (Werker *et al.*, 2007). In this case, high VFA concentrations in the feed sludge indicated an efficient pretreatment process to convert the suspended organic material into soluble intermediate products, which are readily consumed by methanogens in the MAD process (Trisakti *et al.*, 2017). This explains why higher VFA feed benefitted the biogas production in the MAD stage.

Temperature was statistically significant and positively associated with EY at THP and enzymic sites ($P = 0.036$ and 0.006 , respectively). Digestion temperature has a significant influence on the physico-chemical and biological conditions within the digester environment. The major benefits of increasing digestion temperature are enhancing biological and chemical reaction rates, increasing solubility of the organic compounds and hence improving digestion performance (Boušková *et al.*, 2005; Appels *et al.*, 2008; Kim and Lee, 2016). However, no statistically significant effect was detected between temperature and BY at conventional MAD sites, and this may be explained because of the limitation of simple linear relationship tests and the relatively narrow range of operational temperatures found at full-scale sites compared to laboratory experiments, as discussed in Section 4.3.

Cluster analysis evaluated the combined effects of principal operational parameters collected by all of the water utility companies, including temperature, DS and HRT, on digester performance. The cluster results demonstrated that DS feed, HRT and temperature influenced BY and EY to a different extent, and similar process performances could be also achieved with different combinations of these principal operational parameters. Furthermore, visualisation of the data distribution for different AD process conditions demonstrated the potential effects and importance of the principal operational parameters on the AD process. However, cluster analysis could not effectively separate the effect of individual parameters or provide quantification of their effects on digestion performance.

Chapter 5. Multiple Regression Analysis

Results

5.1. Introduction to Multiple Regression Analysis

Multiple regression analysis was used to assess the combined and interactive effects of multiple parameters and to provide quantitative coefficients for each predictor on the target dependent variables. The model incorporated a categorical site factor to model the full-scale AD process and explain the variation associated with local data measurement and other unrecorded factors at specific sites. The major operational factors influencing the process were quantified based on the relative changes in digester performance by using the centred predictors as described in Section 3.5.1. Multiple regression analysis was used to assess the significance of all the possible input parameters against conventional BY, THP EY and enzymic EY. Several sub-models were also constructed to evaluate the effects of the possible input parameters on VSR and digested sludge dewaterability for conventional MAD.

5.2. Conventional MAD Model

Temperature, HRT and DS feed were identified in the preliminary analysis (Chapter 4) as statistically significant factors controlling BY, and data for each of these variables were routinely collected by all of the Water Utility companies. To test whether the magnitude of the effects varied as the values changed, quadratic, cubic and natural logarithm terms were included to assess the impact on the statistical description of operational data (Table 5-1). The adjusted R^2 of the different models was relatively consistent and equivalent to approximately 51%. However, the contribution of process predictors (HRT, temperature and DS) to the total variation explained in BY increased from 6.2% to 8.2% using a natural logarithm model (Table 5-1). The interactive effects of the predictors were also examined in the natural logarithm model, and a statistically significant ($P = 0.021$) interaction between HRT and DS feed was detected. This model (Model 4, Table 5-1), based on log transformation and including an interaction term for HRT and DS, was therefore selected as the best empirical solution to predict the BY of conventional MAD. The complete form of Model 4 was written as follows:

Model 4: Biogas yield = $230.9 * (\ln(\text{Temperature}) - 3.6) + 136.2 * (\ln(\text{HRT}) - 3.0) - 224.8 * (\ln(\text{DS}) - 1.5) + 75.5 * ((\ln(\text{HRT}) - 3.0) * (\ln(\text{DS}) - 1.5)) + \text{site factor}$

Where, Biogas yield is $\text{m}^3/\text{t DS}$, Temperature is $^{\circ}\text{C}$, HRT is the hydraulic retention time in d, and DS is the feed dry solids content in %.

Figure 5-1 (a) shows the relationship between temperature and the relative changes in BY when all other predictors were held constant for the natural logarithm Model 4. The effect of temperature on BY was positive and approximately linear for the operational range of temperatures obtained at conventional MAD sites. As may be expected, the BY increased following a diminishing pattern with increasing HRT. However, the effect of HRT on BY also depended on the DS concentration, thus, HRT had almost no effect on BY at low DS concentrations (for example at approximately 1%), but the influence of HRT on BY increased as the DS concentration was raised (Figure 5-1 b).

Table 5-1 Conventional mesophilic anaerobic digestion models

Model	Adjusted R ²	Covariates; coefficients indicated in brackets	Effective size (η^2 %)	Total variation explained by predictor
1. Linear	0.51	HRT_c (5.54) Temperature_c (6.80) DS input_c (-47.6) Site factor	HRT_c (2.23%) Temperature_c (0.46%) DS input_c (4.54%) Site factor (42.7%)	7.2%
2. Quadratic and Cubic	0.52	HRT_c (6.44) Temperature_c (7.99) DS input_c (-45.4) HRT_c squared (-0.44) Temperature_c squared (-0.40) DS input_c squared (10.3) HRT_c cubed (0.01) Temperature_c cubed (-0.09) DS input_c cubed (-1.92) Site factor	HRT_c total (3.25%) Temperature_c total (0.48%) DS input_c total (2.46%) Site factor (43.1%)	6.2%
3. Natural Logarithm	0.52	Ln(HRT)_c (137.1) Ln(Temperature)_c (232.5) Ln(DS)_c (-221.6) Site factor	Ln(HRT)_c (2.58%) Ln(Temperature)_c (0.46%) Ln(DS)_c (5.2%) Site factor (42.5%)	8.2%
4. Adding interaction	0.52	Ln(HRT)_c (136.2) Ln(Temperature)_c (231.0) Ln(DS)_c (-224.8) Ln(HRT)_c* Ln(DS)_c (75.5) Site factor	Ln(HRT)_c (2.55%) Ln(Temperature)_c (0.46%) Ln(DS)_c (5.31%) Ln(HRT)_c* Ln(DS)_c (0.11%) Site factor (42.4%)	8.4%

HRT_c: hydraulic retention time centred with mean; Temperature_c: digestion temperature centred with mean; DS input_c: digester feed dry solids concentration centred with mean; Ln(Temperature)_c: log transferred digestion temperature centred with mean; Ln(HRT)_c: log transferred hydraulic retention time centred with mean; Ln(DS)_c: log transferred digester feed dry solids concentration centred with mean; Site factor: is a categorical variable for site.

The operationally monitored parameters had a statistically significant influence on BY, nevertheless, major uncertainties remain about the factors controlling MAD performance at different sewage sludge treatment sites. Thus, more than 40% of the total variation in BY was attributed to the categorical factor in the model explaining the overall effects of site. This variation could be attributed to other factors not included in the monitored operational parameters that may also influence the AD process, for example, digester mixing efficiency, sludge characteristics and asset age. We also suspect that variation in the quality of gas flow monitoring could be a major, if not the most important, factor explaining the inherent variation observed in BY data between different sites (Figure 4-1). Critically, up to approximately 40% of the total variation observed in the operational data could not be explained by either the monitored process parameters, or the categorical site factor.

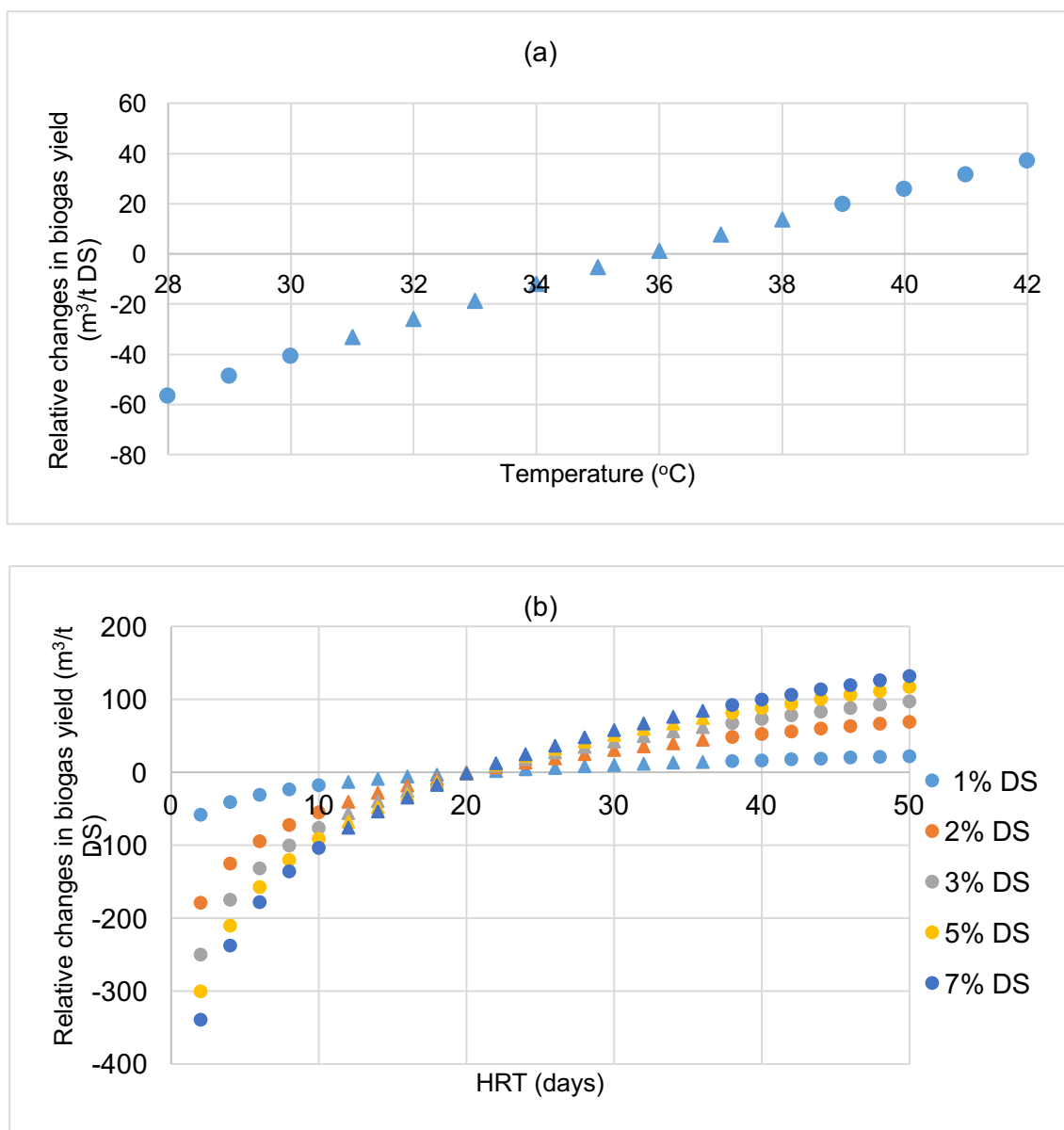


Figure 5-1 Effect of (a) digestion temperature and (b) HRT, on the relative change in biogas yield at different DS feed concentrations for conventional MAD model (Model 4) Triangle symbols: 90% data range

5.3. THP MAD Model

The THP MAD model was generated based on EY data collected at 8 THP sites from Company 2, 4 and 5. Backward elimination and forward selection methods were used to select the significant predictors from temperature, HRT, and DS feed, based on the routinely collected variables at THP sites (Table 3-1). Temperature and HRT were identified as statistically significant factors controlling EY ($P = 0.02$ and < 0.001 , respectively). The modelling process followed the same procedure as for the conventional MAD model and linear, quadratic and natural logarithm terms were included to assess the impact on the statistical description of operational data (Table 5-2). The adjusted R^2 was equivalent to approximately 30% and the contribution of process predictors (HRT and temperature) to the total variation explained in EY varied from 7.6% to 14.6%. The quadratic model (Model 6, Table 5-2) with HRT quadratic term, had the highest adjusted R^2 (0.32) and total predictor contribution (14.6%), was selected as the best empirical solution to predict EY performance of the THP MAD process. The complete form of Model 6 is written as follows:

Model 6: Electricity yield = $17.3 * (\text{Temperature} - 38.9) + 15.8 * (\text{HRT} - 21.9) - 0.48 * (\text{HRT} - 21.9)^2$ + site factor

Where, Electricity yield is kWh/t DS, Temperature is °C, and HRT is the hydraulic retention time in d.

Figure 5-2 (a) shows the relationship between temperature and the relative changes in EY when all other predictors were held constant for the Model 6. The temperature had a positive linear effect on EY, and followed a similar pattern to the effect of temperature on BY of the conventional MAD process. The EY increased following a quadratic relationship with increasing HRT, and attained a maximum EY value at around 38 days. The maximum HRT for the 90% of the data range was at 41.7 days, which was close to the HRT value that gave the maximum EY. Therefore, for the majority of data at THP MAD sites, the model could provide a reasonable description of HRT on EY, and the negative effect of HRT on EY after 38 days could be explained as an artefact caused by the quadratic term for HRT.

The categorical factor explaining the overall effects of site contributed more than 20% of the total variation in EY. Model 6 had a smaller adjusted R^2 compared to the conventional MAD model, equivalent to 0.32. On the one hand, the amount of data used in developing the THP model (8 sites) was significantly smaller compared to the conventional MAD model which was based on 66 sites. On the other hand, this may also be explained due to the way EY data are collected, because not all gas flow to CHP was accounted for at the advanced digestion sites and CHP engines also vary in electrical generation efficiency.

Table 5-2 THP mesophilic anaerobic digestion models

Model	Adjusted R²	Covariates; coefficients indicated in brackets	Effective size (η^2 %)	Total variation explained by predictor
5. Linear	0.27	HRT_c (7.28) Temperature_c (17.7) Site factor	HRT_c (5.64%) Temperature_c (1.94%) Site factor (26.3%)	7.6%
6. Quadratic	0.32	HRT_c (15.8) Temperature_c (17.3) HRT_c squared (-0.48) Site factor	HRT_c total (12.9%) Temperature_c total (1.71%) Site factor (28.3%)	14.6%
7. Natural Logarithm	0.30	Ln(HRT)_c (232.6) Ln(Temperature)_c (706.1) Site factor	Ln(HRT)_c (7.76%) Ln(Temperature)_c (2.26%) Site factor (28.0%)	10.0%
8. Adding interaction	0.31	Ln(HRT)_c (255.4) Ln(Temperature)_c (669.4) Ln(HRT)_c* Ln(Temperature)_c (789.0) Site factor	Ln(HRT)_c (8.60 %) Ln(Temperature)_c (1.98%) Ln(HRT)_c* Ln(Temperature)_c (1.05%) Site factor (28.5%)	11.6%

HRT_c: hydraulic retention time centred with mean; Temperature_c: digestion temperature centred with mean; DS input_c: digester feed dry solids concentration centred with mean; Ln(Temperature)_c: log transferred digestion temperature centred with mean; Ln(HRT)_c: log transferred hydraulic retention time centred with mean; Ln(DS)_c: log transferred digester feed dry solids concentration centred with mean; Site factor: is a categorical variable for site.

5.4. Enzymic MAD Model

The enzymic model was generated based on the data collected from 10 EEH, EH and HpH sites operated by Company 3 and 5. Temperature, HRT, DS feed and VFA feed concentrations were available and routinely collected for enzymic treatment sites (Table 3-1). Linear, natural logarithm and natural logarithm with interaction models were tested to determine which factors were important in influencing EY. No statistically significant relation between HRT and EY was detected by multiple regression analysis ($P = 0.56$), therefore HRT was removed from the model in this case. Temperature, DS feed and VFA feed were found to be statistically significant ($P = 0.049$, 0.02 and <0.001 , respectively) (Table 5-3). In contrast to the conventional and THP MAD models, there were no significant quadratic terms observed in the enzymic model. Furthermore, a categorical variable for treatment type, rather than a site factor was used to represent the different forms of enzymic pretreatment (EEH, EH, and HpH). Using treatment type, in place of a site factor, as the categorical variable increased the total variation in the data explained by the continuous predictors in the model by almost 60%, from 9.0% to 14.0%. This was explained because the frequency of recording varied considerably between specific sites, and some were only poorly represented in the data, whereas

pooling the data by treatment type provided a more consistent approach to differentiating the effects of unmeasured variables on EY between the different processes. The Model 11, based on log transformation and including an interaction term for temperature and VFA feed was selected as the best empirical model with the highest adjusted R^2 (0.32) and total predictor contribution, equivalent to 14.0% of the total observed variation in the data. The complete form of Model 11 is written as follows:

$$\text{Model 11: Electricity yield} = 1068.3 * (\text{Ln}(\text{Temperature}) - 3.6) + 273.2 * (\text{Ln}(\text{VFA}) - 8.6) - 411.4 * (\text{Ln}(\text{DS}) - 1.6) + 1485.8 * ((\text{Ln}(\text{Temperature}) - 3.6) * (\text{Ln}(\text{VFA}) - 8.6)) + \text{site factor}$$

Where, Electricity yield is kWh/t DS, Temperature is °C, VFA is the digester feed volatile fatty acids in mg/l, and DS is the feed dry solids content in %.

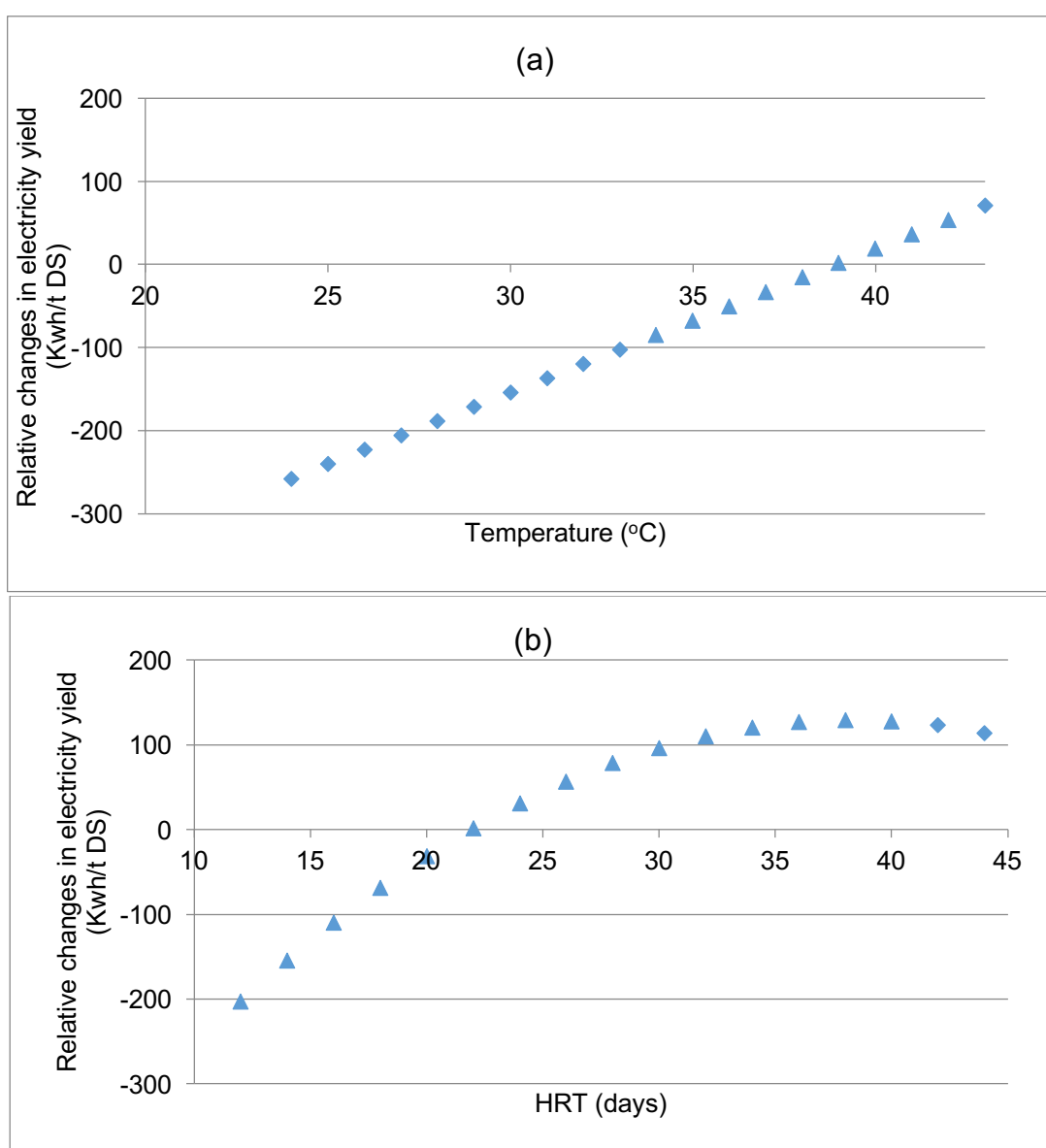


Figure 5-2 Effect of (a) digestion temperature and (b) HRT, on the relative change in electricity yield for THP MAD model (Model 6). Triangle symbols: 90% data range

Table 5-3 Enzymic mesophilic anaerobic digestion models

Model	Adjusted R²	Covariates; coefficients indicated in brackets	Effective size (η^2 %)	Total variation explained by predictor
9. Linear	0.31	VFA_c (0.06) DS_c (-67.7) Temperature_c (21.9) <i>Type factor</i>	VFA_c (6.1%) DS_c (3.8%) Temperature_c (1.57 %) <i>Type factor (18.6%)</i>	11.4%
10. Natural Logarithm	0.30	Ln(VFA)_c (319.3) Ln(DS)_c (-393.5) Ln(Temperature)_c (790.1) <i>Type factor</i>	Ln(VFA)_c (6.38%) Ln(DS)_c (4.16%) Ln(Temperature)_c (1.65%) <i>Type factor (21.2%)</i>	12.2%
11. Adding interaction	0.32	Ln(VFA)_c (273.2) Ln(DS)_c (-411.4) Ln(Temperature)_c (1068.3) Ln(Temperature)_c* Ln(VFA)_c (1485.8) <i>Type factor</i>	Ln(VFA)_c (4.55 %) Ln(DS)_c (4.74%) Ln(Temperature)_c (2.84%) Ln(Temperature)_c* Ln(VFA)_c (1.85%) <i>Type factor (18.2%)</i>	14.0%

VFA_c: volatile fatty acids feed centred with mean; Temperature_c: digestion temperature centred with mean; DS input_c: digester feed dry solids concentration centred with mean; Ln(Temperature)_c: log transferred digestion temperature centred with mean; Ln(HRT)_c: log transferred hydraulic retention time centred with mean; Ln(DS)_c: log transferred digester feed dry solids concentration centred with mean; Ln(VFA)_c: log transferred volatile fatty acids feed centred with mean; Type factor: is a categorical variable for enzymic treatment type

Figure 5-3 (a) shows the relationship between DS feed and the relative changes in EY for the natural logarithm Model 11. The EY decreased following a diminishing pattern with increasing DS. The effect of temperature on EY was positive and approximately linear for the operational range of temperatures obtained at enzymic MAD sites. The effect of temperature on EY was depended on the VFA feed concentration, thus, temperature had almost no effect on EY at low VFA concentrations (for example at approximately 3000 mg/l), but the influence of temperature on EY increased as the VFA concentration was raised (Figure 5-3 b).

The categorical factor explaining the overall effects of enzymic types contributed 18% of the total variation in EY. The overall prediction accuracy of Model 11 was relatively poor and similar to the

THP MAD model, and this could be attributed to similar factors as discussed in Section 5.3, such as small datasets available in model generation and the variable CHP engine efficiency.

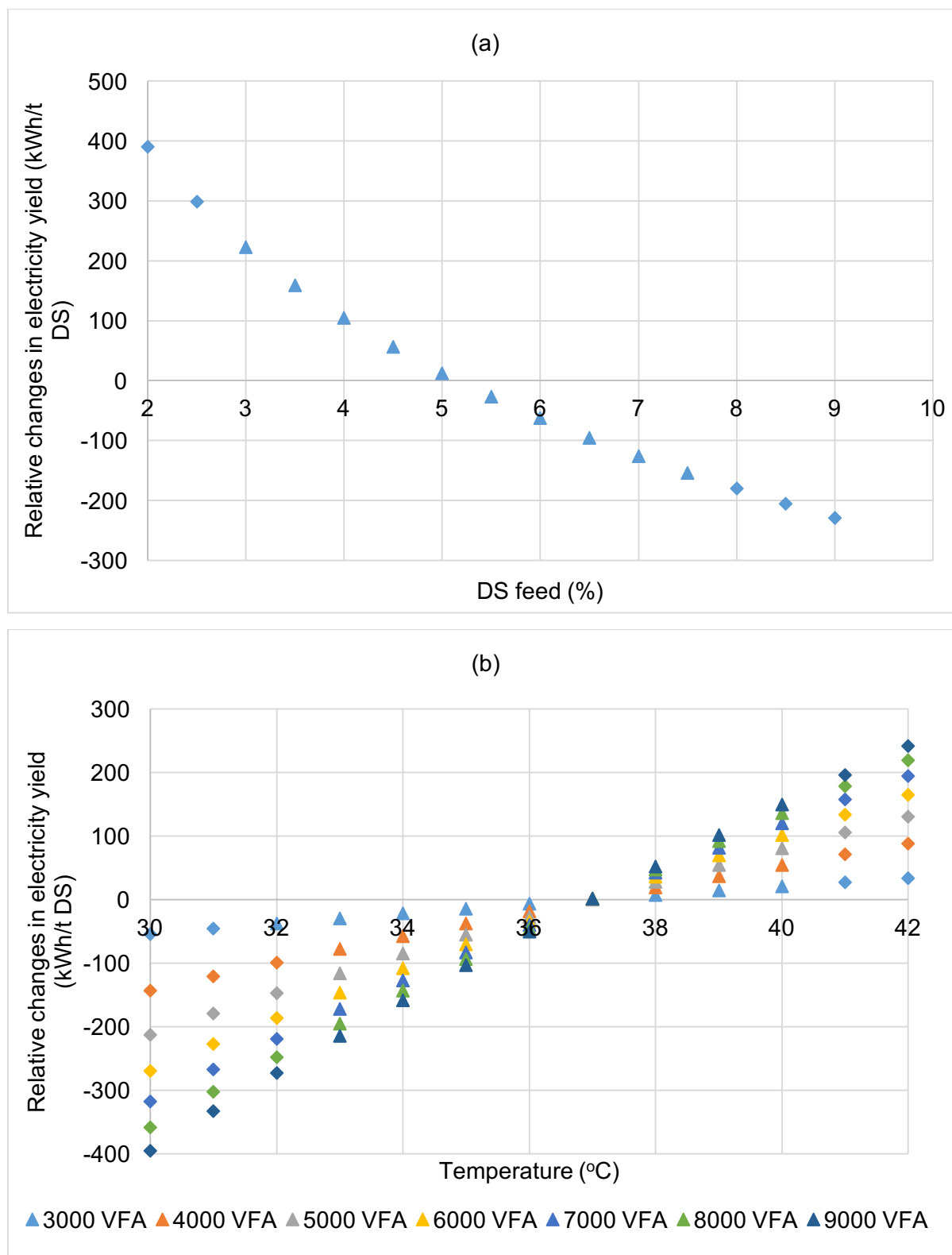


Figure 5-3 Effect of (a) DS feed and (b) digestion temperature, on the relative change in electricity yield at different VFA feed concentrations (mg/l) for enzymic MAD model (Model 11). Triangle symbols: 90% data range

5.5. Parameter Based Conventional MAD Model

Additional operational parameters (sludge age, primary:SAS ratio and VS feed) were measured at conventional MAD sites by specific companies (see Table 3-1). This allowed a number of different parameters to be tested in addition to the main variables of HRT, DS feed and temperature, that were collected by all companies. Therefore, several, secondary conventional MAD models were generated based on data from the sites that collected these additional parameters. A similar modelling procedure was followed, described in Section 5.2, but in this case the data sets were smaller (Figure 5-4). Thus, the main conventional MAD Model 4 contained 2648 rows of data from 66 conventional MAD sites. The effect of sludge age on BY was evaluated using 814 rows of data from 22 conventional MAD sites (Model 12). However, sludge age was not a significant predictor ($P = 0.471$) for the conventional MAD model (Model 12). Therefore, Model 12 contained the same significant predictors as Model 4, generated in Section 5.2, and both models had similar adjusted R^2 values, equivalent to 0.56 and 0.52, respectively (Table 5-4). The continuous predictors representing the controllable digestion conditions in Model 4 explained a larger proportion of the total variation in the data (8.4%) in BY compared to Model 12. Therefore, Model 4 was provided the best empirical model description of BY for the conventional MAD process.

Sludge age, primary:SAS ratio and VS feed data, were collected at 18 conventional MAD sites operated by Company 2. Therefore, Model 13 integrated all of these additional parameters and showed that both sludge age and primary:SAS ratio were not significant predictors of BY ($P = 0.591$ and 0.230 , respectively). The only additional significant predictor in the model was VS feed ($P = 0.018$), and Model 13 had slightly higher adjusted R^2 of 0.59 compared to Model 4, 0.52, respectively. Therefore, Model 13 is an alternative conventional MAD model when VS data is available (Table 5-4). The complete form of Model 13 is written as follows:

Model 13: Biogas yield = $238.5 * (\text{Ln}(\text{Temperature}) - 3.6) + 70.7 * (\text{Ln}(\text{HRT}) - 2.9) - 134.5 * (\text{Ln}(\text{DS}) - 1.5) + 163.6 * (\text{Ln}(\text{VS}) - 4.3) + 114.1 * ((\text{Ln}(\text{HRT}) - 2.9) * (\text{Ln}(\text{DS}) - 1.5)) + \text{site factor}$

Where, Biogas yield is $\text{m}^3/\text{t DS}$, Temperature is $^{\circ}\text{C}$, HRT is the hydraulic retention time in d, DS is the feed dry solids content in %, and VS is the feed volatile solids content in %.

Figure 5-5 shows the relationship between temperature, VS, HRT, DS and the relative changes in BY in Model 13. The effect of temperature, HRT and DS on BY were similar to those presented in Model 4. The VS feed was an additional predictor, and its effect on BY was positive and approximately linear for the operational range of VS obtained at conventional MAD sites. Nevertheless, major uncertainties remain about the factors responsible for MAD performance at different sewage sludge treatment sites since the categorical variable for site accounted for as much as 47% of the total variation in the data, compared to a total of 6.3% for the continuous variables (Table 5-4). A comparison of the prediction accuracy between Model 4 and 13 at selected

conventional MAD sites will be presented in Chapter 6.

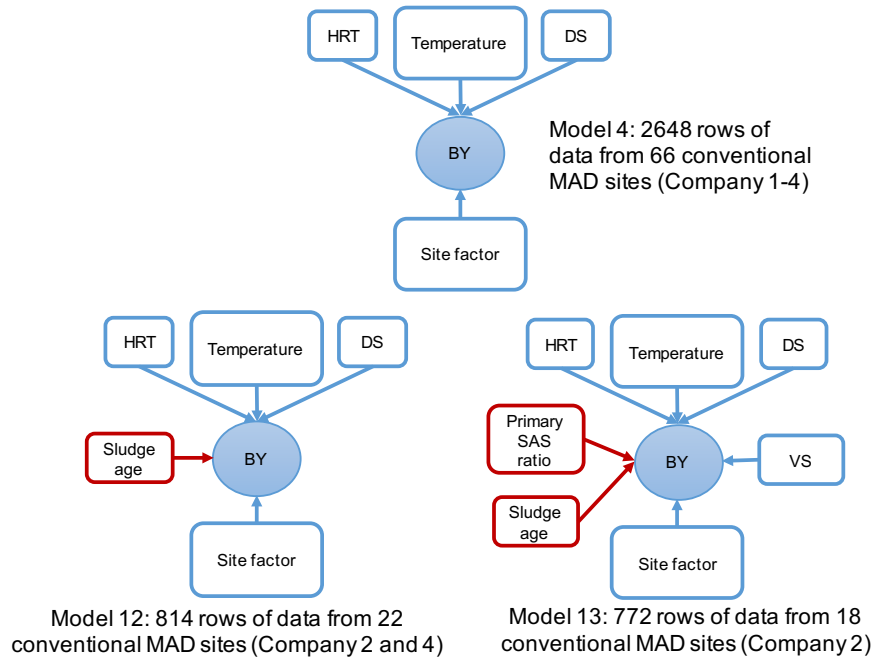


Figure 5-4 Conventional mesophilic anaerobic digestion models with additional parameters (Blue box: significant predictor; red box: available but insignificant parameter)

Table 5-4 Conventional mesophilic anaerobic digestion models with additional predictors (Model 12 and 13)

Model	Adjusted R^2	Covariates; coefficients indicated in brackets	Effective size (η^2 %)	Total variation explained by predictor
12.	0.56	Ln(HRT)_c (59.1) Ln(Temperature)_c (261.1) Ln(DS)_c (-188.6) Ln(HRT)_c* Ln(DS)_c (249.2) <i>Site factor</i>	Ln(HRT)_c (0.60%) Ln(Temperature)_c (0.98%) Ln(DS)_c (3.14%) Ln(HRT)_c* Ln(DS)_c (0.57%) <i>Site factor (51.6%)</i>	5.3%
13.	0.59	Ln(HRT)_c (70.7) Ln(Temperature)_c (238.5) Ln(DS)_c (-134.5) Ln(VS)_c (163.6) Ln(HRT)_c* Ln(DS)_c (114.1) <i>Site factor</i>	Ln(HRT)_c (1.37%) Ln(Temperature)_c (1.67%) Ln(DS)_c (2.76%) Ln(VS)_c (0.35%) Ln(HRT)_c* Ln(DS)_c (0.17 %) <i>Site factor (47.6%)</i>	6.3%

Ln(Temperature)_c: log transferred digestion temperature centred with mean; Ln(HRT)_c: log transferred hydraulic retention time centred with mean; Ln(DS)_c: log transferred digester feed dry solids concentration centred with mean; Ln(VS)_c: log transferred digester feed volatile solids concentration centred with mean; Site factor: is a categorical variable for site

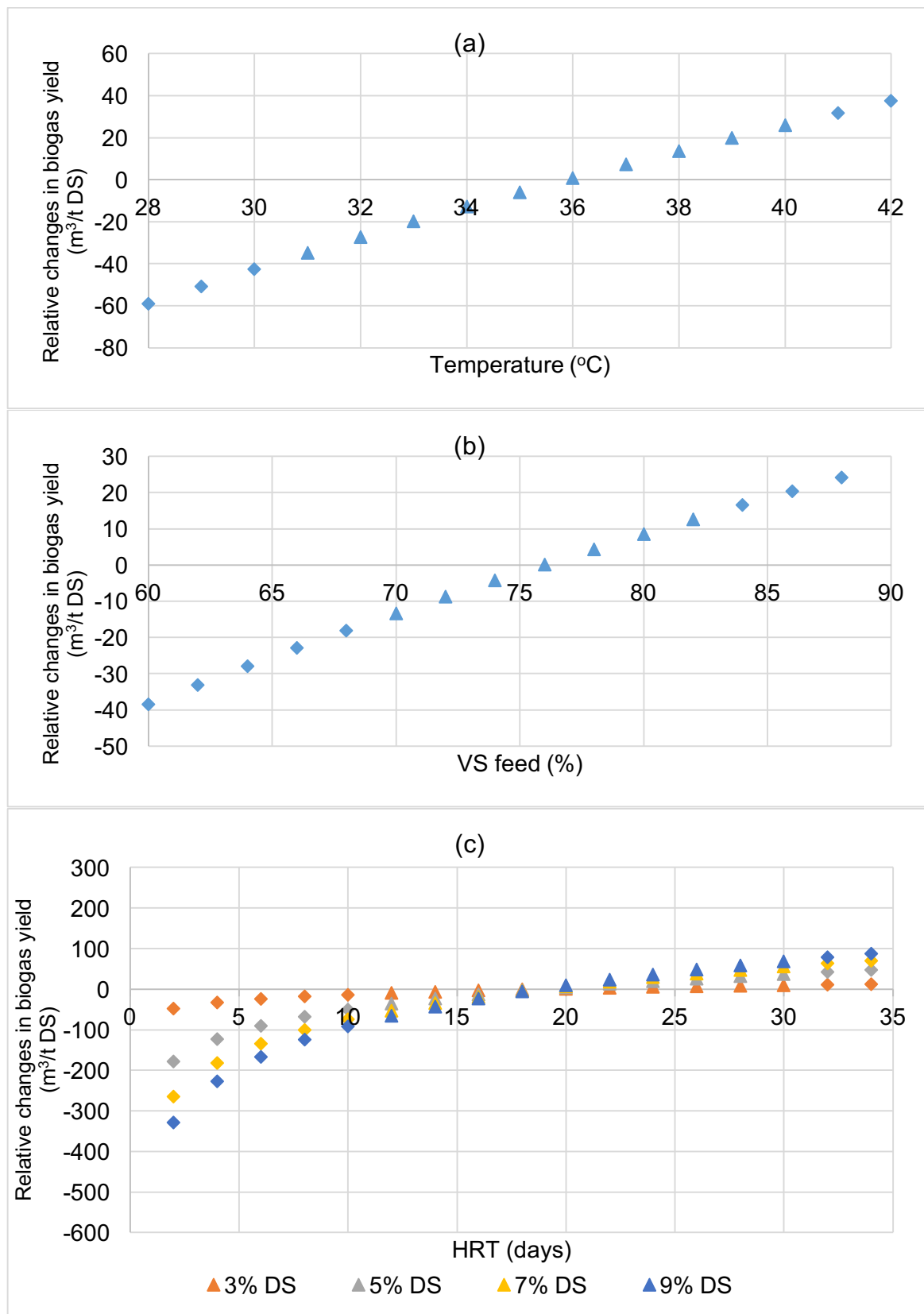


Figure 5-5 Effect of (a) temperature (b) VS feed and (c) HRT, on the relative change in biogas yield at different DS feed concentrations for conventional MAD model (Model 13). Triangle symbols: 90% data range

5.6. Conventional MAD Volatile Solids Reduction Model

The VSR was calculated by both the Van Kleeck equation (VanKleeck. 1945) and mass balance method (see Section 3.4) The VS feed and HRT were identified as statistically significant factors controlling VSR calculated by Van Kleeck equation, whereas VS feed, DS feed and temperature were statistically significant when calculated by mass balance method. Furthermore, the statistical prediction ($R^2 = 0.54$) was improved by Van Kleeck (Model 14) compared to mass balance (Model 15, $R^2 = 0.48$) (Table 5-5). The calculation of Van Kleeck VSR also required fewer parameters (VS) compared to mass balance (VS and DS). Therefore, Model 14 may be more suitable to predict VSR at conventional full-scale MAD sites. The complete form of Model 14 is written as follows:

Model 14: Volatile solids reduction = $5.8 * (\text{Ln}(\text{HRT}) - 2.9) + 113.5 * (\text{Ln}(\text{VS}) - 4.3) + 8.9 * (\text{Ln}(\text{Temperature}) - 3.6) + \text{site factor}$

Where, volatile solids reduction is %, HRT is the hydraulic retention time in d, and VS is the feed volatile solids content in %.

Table 5-5 Conventional mesophilic anaerobic digestion VSR models (Model 14 and 15)

Model	Adjusted R^2	Covariates; coefficients indicated in brackets	Effective size (η^2 %)	Total variation explained by predictor
14. VSR (Van Kleeck) model	0.57	Ln(VS)_c (113.5) Ln(HRT)_c (5.84) Ln(Temperature)_c (8.89)	Ln(VS)_c (16.6%) Ln(HRT)_c (1.5%) Ln(Temperature)_c (0.3%) Site factor (38.0%)	18.4%
15. VSR (mass balance) model	0.47	Ln(VS)_c (53.9) Ln(DS)_c (31.1) Ln(Temperature)_c (19.1)	Ln(VS)_c (2.50%) Ln(DS)_c (11.5%) Ln(Temperature)_c (0.70%) Site factor (34.7%)	14.7%

Ln(Temperature)_c: log transferred digestion temperature centred with mean; Ln(HRT)_c: log transferred hydraulic retention time centred with mean; Ln(DS)_c: log transferred digester feed dry solids concentration centred with mean; Ln(VS)_c: log transferred digester feed volatile solids concentration centred with mean; Site factor: is a categorical variable for site

Figure 5-6 shows the relationship between HRT, VS feed and the relative changes in VSR for Model 14. The VSR increased following a diminishing pattern with increasing HRT. The effect of VS feed and temperature on VSR was positive and approximately linear for the operational range of VS feed and temperature obtained at conventional MAD sites. VSR provides an alternative indicator of digestion performance and is statistically significantly correlated to BY as shown in Table 4-2 (Section 4.3). Indeed, the model adjusted R^2 of VSR Model 14 and conventional MAD Model 4 were

approximately similar and equivalent to 0.57 and 0.52, respectively. More than 30% of the total variation in VSR was attributed to a categorical factor explaining the overall effects of site, and this could be attributed to similar site factors influencing BY in conventional MAD Model 4 (Section 5.2), such as digester mixing efficiency, sludge characteristics and asset age.

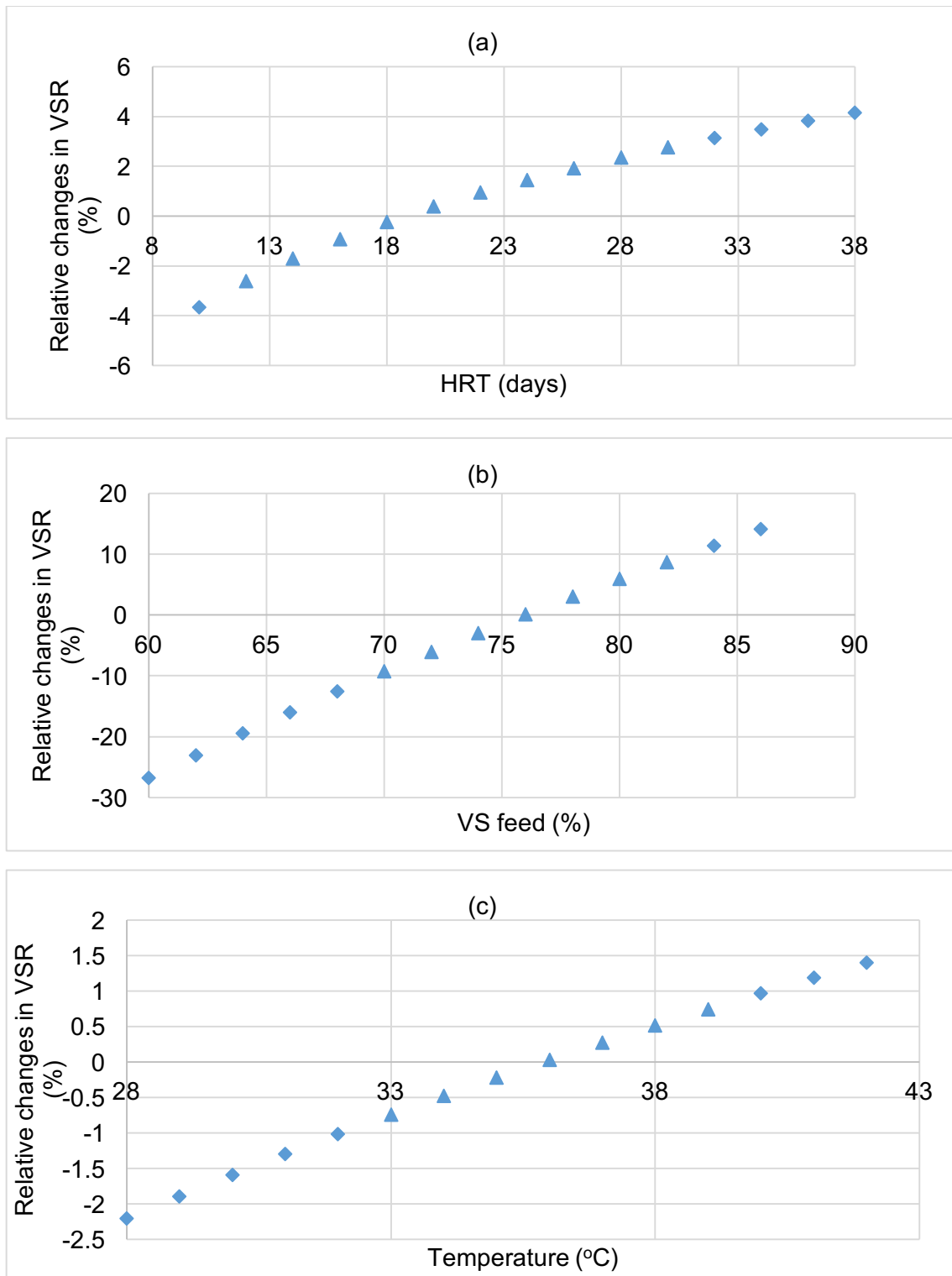


Figure 5-6 Effect of (a) HRT and (b) VS feed and (c) temperature, on the relative change in volatile solids reduction for conventional MAD VSR model (Model 14). Triangle symbols: 90% data range

5.7. Dewaterability Model

The DS concentration values in digested sludge cake after final dewatering were used as an indicator to evaluate the effects of the conventional MAD process on sludge dewaterability. The DS values in digested sludge cake were available in Company 2 and 4. Therefore, the dewaterability model (Model 16) was first generated based on 1104 rows of data from Company 2 and 4, containing basic operational parameters of HRT, DS feed, and temperature. Then, a further sub-dataset was scrutinised in more detail and included the additional parameters of primary:SAS ratio from Company 2 based on 776 rows of data (Model 17) (Figure 5-7).

Temperature and DS feed were identified as statistically significant factors controlling sludge cake DS in Model 16 ($P = 0.032$ and < 0.001 , respectively), and the primary:SAS ratio was identified as an additional significant predictor ($P < 0.001$) in Model 17. The adjusted R^2 was equivalent to approximately 30% in both models and the total total variation explained by the continuous predictors increased from 1.7% in Model 16 to 2.4% in Model 17 (Table 5-6). Therefore, the Model 17 with highest proportion of the total variation explained by the continuous predictors, based on temperature, DS feed and primary:SAS ratio, was selected as the best empirical description of sludge cake DS for conventional MAD. The complete form of Model 17 is written as follows:

Model 17: Sludge cake DS = $-0.14 * (\text{Temperature} - 36.1) + 0.38 * (\text{DS} - 4.7) + 0.03 * (\text{PSR} - 61.4)$ + site factor

Where, Sludge cake DS is %, Temperature is °C, DS is the digester feed dry solids content in d, and PSR is the primary sludge ratio in %.

Figure 5-8 (a) shows the relationship between temperature and the relative changes in sludge cake DS when all other predictors were held constant for the dewaterability Model 17. Temperature had a negative linear effect on sludge cake DS, which contrasted to the positive effect of temperature on BY and EY found in the previous models. The DS feed and primary:SAS ratio had positive linear effects on sludge cake DS (Figure 5-8), and this was consistent with the results reported by Gebreeyessus and Jenicek (2016) that primary sludge had better dewaterability compared to SAS.

The operationally monitored parameters accounted for a relatively small proportion in the total variation in sludge cake DS, and more than 20% of of the total variation was attributed to the categorical factor in the model explaining the overall effects of site. Up to approximately 70% of the total variation observed in the operational data could not be explained by either the monitored process parameters, or the categorical site factor. Thus, the dewaterability model may not reliably predict specific sludge cake DS concentrations after dewatering, but it can provide insight of how upstream processes affect the final sludge dewaterability. The large amount of unaccounted variation may also be associated with other factors more directly influencing the dewatering process,

such as, digested sludge characteristics, sludge dewatering equipment types and dewatering polymer dosage.

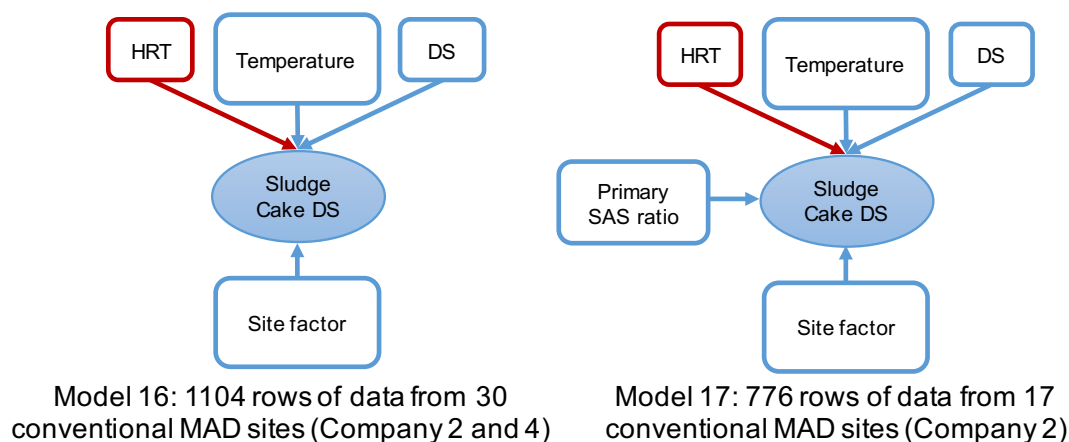


Figure 5-7 Conventional mesophilic anaerobic digestion dewaterability models (Blue box: significant predictor; red box: available but insignificant parameter)

Table 5-6 Conventional mesophilic anaerobic digestion dewaterability model (Model 16 and 17)

Model	Adjusted R^2	Covariates; coefficients indicated in brackets	Effective size (η^2 %)	Total variation explained by predictor
16. Dewaterability	0.34	Temperature_c (-0.11) DS_c (0.64)	Temperature_c (0.30%) DS_c (1.36%) Site factor (29.5%)	1.7%
17. Dewaterability	0.30	Temperature_c (-0.14) DS_c (0.38) PSR_c (0.03)	Temperature_c (0.56%) DS_c (0.50%) PSR_c (1.36%) Site factor (27.1%)	2.4%

Temperature_c: digestion temperature centred with mean; DS input_c: digester feed dry solids concentration centred with mean; PSR_c: primary:SAS ratio centred with mean; Site factor: is a categorical variable for site

5.8. Discussion

5.8.1. Conventional, THP and enzymic MAD models

The multiple regression analysis examined the statistical significance and behaviour of combined and interactive effects of multiple process control parameters on digester performance. In general, the individual main effects of the parameters on digester performance were similar to those identified by linear correlation analysis (Section 4.3). For example, temperature and HRT both had statistically significant positive effects on digester performance in both conventional and advanced MAD models.

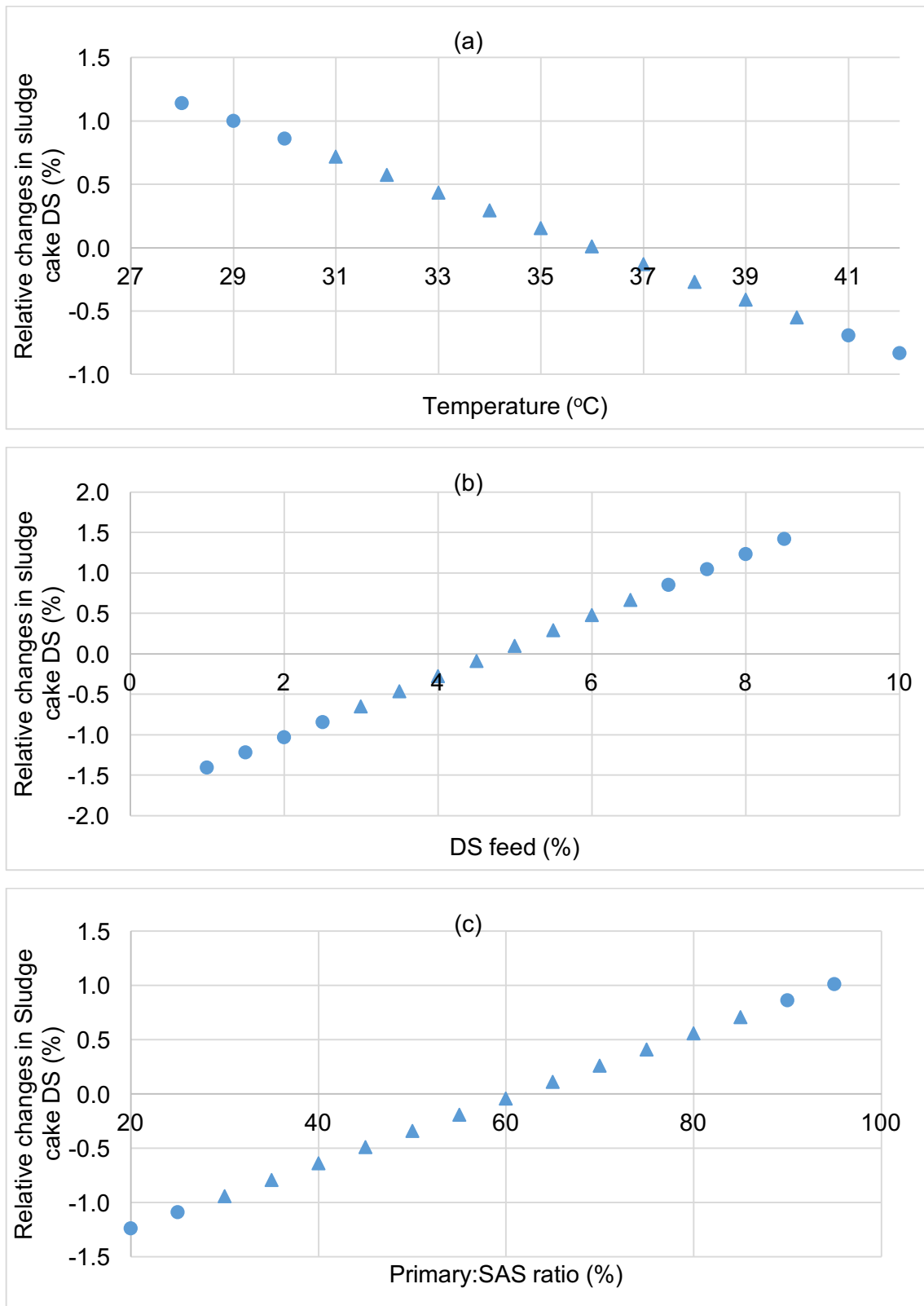


Figure 5-8 Effect of (a) temperature (b) DS feed and (c) primary:SAS ratio, on the relative change in sludge cake DS for conventional MAD dewaterability model (Model 17). Triangle symbols: 90% data range

In contrast to the linear correlation analysis results (Section 4.3), which showed EY increased with DS feed, this parameter had a negative effect on EY in both THP and enzymic MAD models (Model

6 and 11, respectively). This response may be explained due to the statistical effect of including a categorical factor for site (THP) or process (enzymic) to constrain the effect of DS on EY to an individual site or process type. For example, Figure 5-9 (a) shows that EY increased positively as DS feed rose across all THP MAD sites. However, at the individual site level (Figure 5-9 b), a negative effect of DS feed on EY was observed. Similarly, a positive trend was observed for the effect of DS on EY for the enzymic MAD sites, but, here also, a negative impact of DS feed on EY was observed for individual enzymic treatment types (Figure 5-10). This could be attributed to the increased DS caused an exponential rise in sludge viscosity, which impaired the mixing, and hence reduce microbial access to substrates within AD system (Liao, *et al.*, 2014; Zhang *et al.*, 2015). Therefore, although, sludge pretreatment improved overall AD capacity enabling greater solids input, increasing the DS concentration reduced the overall EY, thus a balance needs to be struck between the benefit of pretreatment and the DS concentration to avoid exceeding the treatment capacity at individual THP sites or enzymic treatment types.

VFA feed was an additional significant predictor for enzymic MAD model, which was routinely recorded at enzymic MAD sites. Increasing VFA concentration indicated more effective pretreatment and the presence of more soluble organic substrates available for biogas production (Werker *et al.*, 2007) (see Section 4.5).

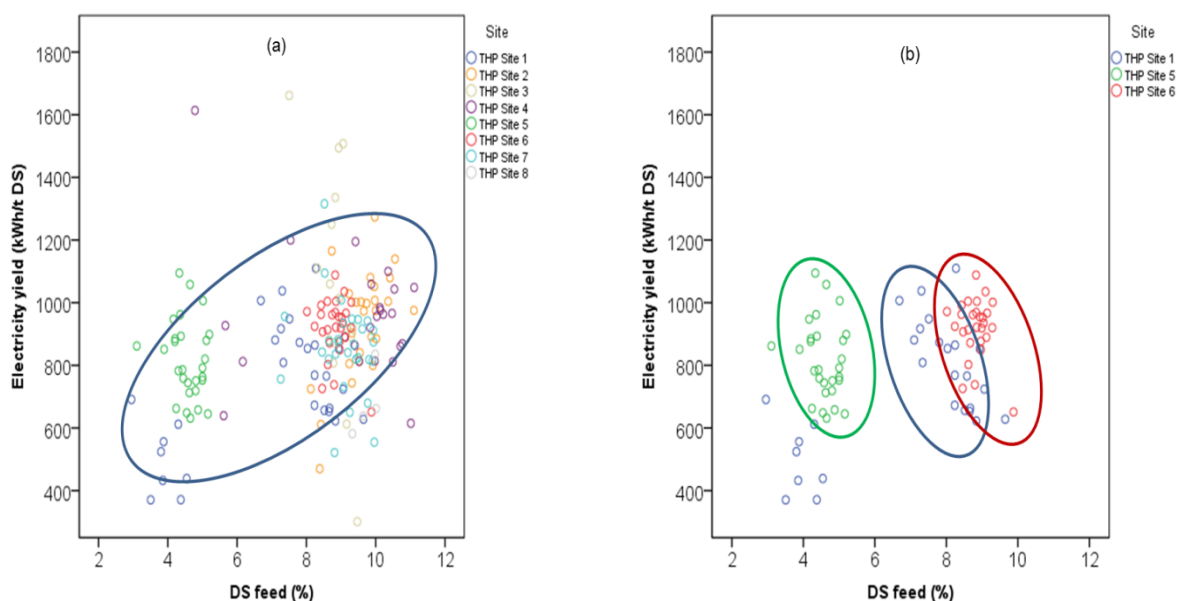


Figure 5-9 Relation between EY and DS feed (%) for THP MAD (a) positive overall effect of DS across all THP sites, and (b) negative effect of DS at individual sites

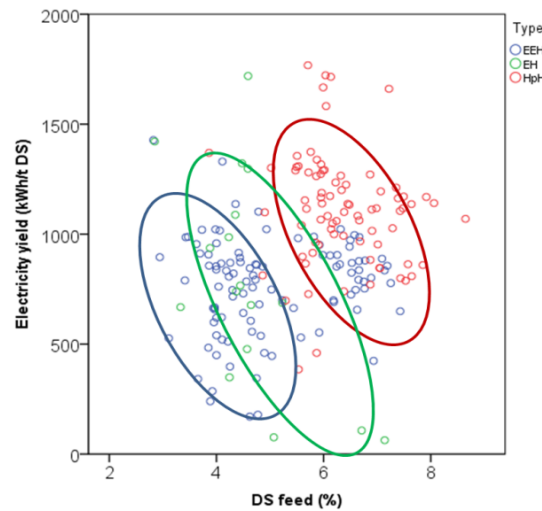


Figure 5-10 Relation between EY and DS feed (%) for enzymic MAD

5.8.2. Sludge age and primary:SAS ratio

Short sludge age and high primary:SAS ratio are beneficial to CH_4 yield as discussed in Section 2.3.7. However, no statistically significant effect of sludge age and primary:SAS ratio were detected by multiple regression analysis (Section 5.5). Inspection of the data showed that, in the practical situation, primary sludge:SAS ratio and sludge age are significantly correlated to some extent ($R^2 = 0.18$, $P < 0.001$) (Figure 5-11). This behaviour may be expected, since, at operational WWTPs, SAS production increases with reducing sludge age (Barber, 2014) and, in turn, this reduces the primary:SAS ratio. Therefore, unlike a laboratory environment where these sludge properties can be factorially controlled in AD experiments, a small sludge age coupled with a high primary:SAS ratio, which are favourable for raising the overall BY in AD (Ometto *et al.*, 2017), are unlikely to be achieved in the real operational situation. Thus, the opposing effects of sludge age and primary:SAS ratio on BY in AD could explain why they were found to have no overall significant effect on BY when modelling full-scale operational AD process data.

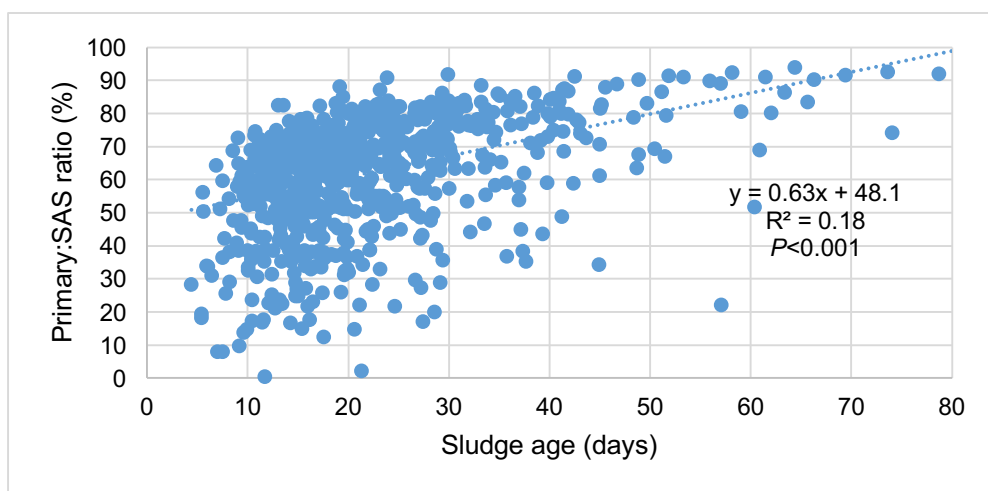


Figure 5-11 Relationship between sludge age and primary:SAS ratio at conventional MAD sites (Company 2)

5.8.3. Volatile solids reduction model

VSR is considered as an alternative indicator of digestion performance at conventional MAD sites (MRRDC, 2017). Different approaches, including the Van Kleeck equation and mass balance equation, were applied to calculate the VSR. However, different values of VSR were obtained by the Van Kleeck and mass balance equations ($R^2 = 0.18$) (Figure 5-12). The differences observed between the two models were probably attributed to the method of VSR calculation. Thus, Van Kleeck is based on a steady-state representation of VSR (Switzenbaum *et al.*, 2003) and can reduce the potential variation observed in mass balance calculations. The Van Kleeck equation also assumes that fixed solids are conserved during the digestion process (VanKleeck, 1945), however, this assumption may not always be valid at full-scale sites. For example, grit accumulation, improper mixing and sampling can result in apparent fixed-solids losses (Switzenbaum *et al.*, 2003). By contrast, the mass balance calculation is directly influenced by the mismatch between spot measurements of feed sludge and digestate at full-scale sites due to the retention time, and mixing of sludge of different age and properties in the process, such that the feed and digested sludge properties do not directly correspond. This may also explain why different significant predictors were detected in the Van Kleeck and mass balance VSR models (Table 5-5) and why a larger proportion of the total variation in VSR ($R^2 = 0.57$) was obtained with the Van Kleeck model (Model 14) compared to the mass balance model (Model 15) ($R^2 = 0.47$). Indeed, further analysis showed that Van Kleeck VSR was statistically significantly correlated to BY ($P < 0.001$), whereas no statistically significant relation was observed with mass balance VSR ($P = 0.453$). Thus, overall the results showed that Van Kleeck VSR provided a more representative indication of digester performance compared to the mass balance approach.

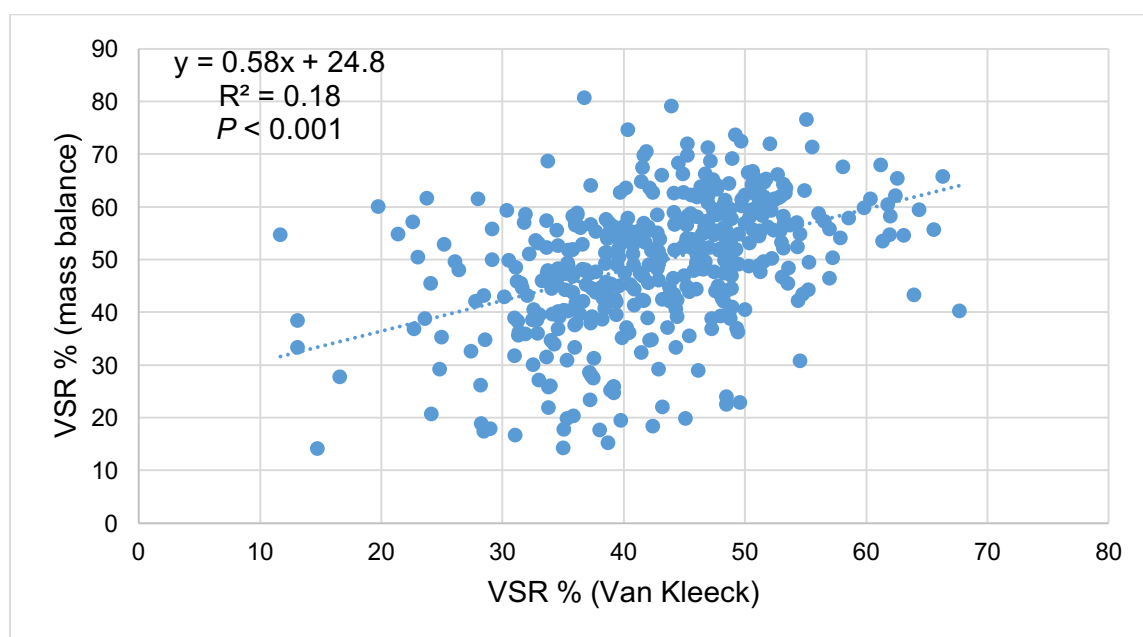


Figure 5-12 Relationship between VSR calculated by Van Kleeck and mass balance equations

5.8.4. Dewaterability model

A large amount of variation in the dewaterability, digestate DS data could not be accounted for by the tested explanatory variables in the dewaterability model (Model 17) relating to the AD process. This was as expected, because dewaterability is also influenced by other factors more directly relating to the dewatering process, such as the sludge dewatering equipment type and polymer type and dose rate (see Section 2.5). Mechanical dewatering devices, such as belt presses and centrifuges, have different operating principles and shear intensities, which greatly affect the efficiency of sludge dewatering (To *et al.*, 2016). In addition, polymer dosage is one of the main parameters influencing sludge rheological and structural properties, and the optimum polymer dose can depend on multiple factors, such as sludge characteristics, dosing points and sludge dewatering equipment types (Dieudé-Fauvel and Dentel, 2011; Minall *et al.*, 2014). Indeed, as these direct factors controlling sludge dewaterability were not considered in the dewaterability model (Model 17), it was not intended as a tool for predicting the DS concentration in digested sludge cake, but the aim was to examine the influence of digestion conditions on the dewaterability of digested sludge.

Sludges with high flocculated biological content, included activated or digested sludge from AD, are potentially challenging to mechanically dewater, due to their high compressibility and gel-like water retention capacity (Curversa *et al.*, 2009). These properties are partly attributed to the presence of surface charges, which are due to the biological nature and the presence of weakly charged extracellular polymeric substances (Minall *et al.*, 2014). Extracellular polymer is produced by bacteria particularly within the activated sludge process (Kang *et al.*, 1989) and sludge with high levels of ECP is particularly difficult to dewater (Houghton *et al.*, 2000). This behaviour is consistent with the dewaterability model reported here, which showed that increasing primary:SAS ratio in digester feed was associated with improved sludge dewaterability. Furthermore, the statistical analysis showed that digestion temperature had a statistically significant ($P < 0.001$), negative effect on sludge dewaterability. This suggested that dewaterability may be negatively affected to some extent by improved digestion conditions in terms of increased BY. A possible hypothesis to explain this behaviour is that improved destruction of readily degradable organic substrates actually raised the proportion of the less digestible SAS fraction in the final digested sludge, and hence reduced the overall dewaterability to some extent.

Chapter 6. Model Validation

6.1 General Model Validation Procedures

The conventional MAD models (Model 4 and Model 13), THP MAD model (Model 6), enzymic model (Model 11), VSR model (Model 14), and sludge dewaterability model (Model 17) were validated against both specific WWTP datasets used in model development and independent datasets which were collected subsequently and were not used to develop the model, in most cases. In this procedure, the model was recalibrated for each calendar year (or part year) by shifting the predicted values of BY, VSR or final digested sludge cake DS to find the best fit to calculate a specific site factor for the data for each WWTP selected as examples for model validation purposes.

6.2 Conventional MAD Sites

The conventional MAD model (Model 4), integrating the main operational data collected by all of the companies (temperature, HRT and DS feed), and the parameter based conventional MAD model (Model 13) also including VS as an additional parameter based on data provided by Company 2, were validated against specific, selected WWTP datasets.

Sites 37 (2011 – 2016), 38 (2013 – 2016) and 42 (2011 – 2016), with typical average observed BY values of approximately 400 m³/t DS (CIWEM, 1996; Bachmann *et al.* 2015), were selected as good examples for model validation using the data which was already represented in the model development. The results showed that both models provided an effective description of BY for all sites, especially Site 42 (Model 4: $R^2 = 0.68$, $P < 0.00$, and Model 13: $R^2 = 0.65$, $P < 0.001$) and Site 37 (Model 4: $R^2 = 0.52$, $P < 0.001$, and Model 13: $R^2 = 0.49$, $P < 0.001$) (Figure 6-1 a). A slightly poorer statistical description ($R^2 = 0.35$, $P < 0.001$) was provided for Site 38 using Model 4, although the general pattern in process performance at the site was followed. However, an improved prediction for Site 38 ($R^2 = 0.57$, $P < 0.001$) was obtained using Model 13 for the same site. Therefore, as would be expected, the accuracy of predictions may vary between sites using different models and for different time periods at the same site.

Additional data were collected for Site 1, 31 and 38 between 2016 – 2019 as part of the sludge composition study (Chapter 7) and were used for model validation as independent datasets, collected subsequent to the main data pool that was used to produce the model (Figure 6-1 b). The results showed that the predicted BY values generally followed the overall patterns in measured operational monthly average data for Site 38 (Model 4: $R^2 = 0.63$, $P < 0.001$, and Model 13: $R^2 = 0.61$, $P < 0.001$) and Site 31 (Model 4: $R^2 = 0.61$, $P < 0.001$, and Model 13: $R^2 = 0.61$, $P < 0.001$). However, where the site biogas measurement was excessive and unrepresentative of the expected AD operational range, for example, 300 – 440 m³/t DS (CIWEM, 1996; Bachmann *et al.*, 2015), this

default approach to internal site calibration using local BY data may not provide a good description of the actual process BY, as shown for Site 1 ($R^2 = 0.32$, $P = 0.003$). Thus, if local site recording instruments are measuring values outside the typical range for full-scale MAD, this will be reflected in the predicted BY (Figure 6-1 b).

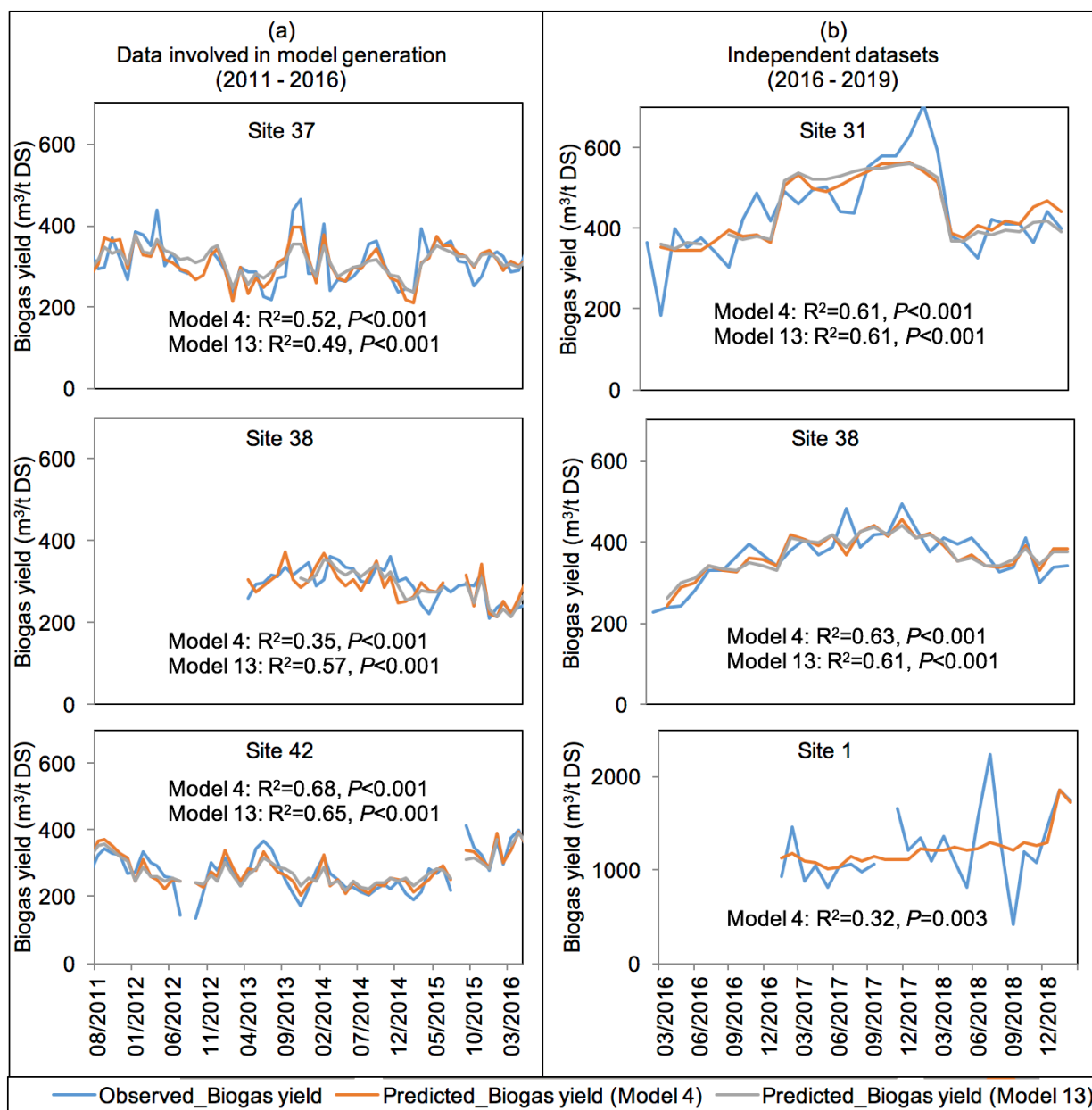


Figure 6-1 Conventional MAD model validation (Model 4 and Model 13) showing the time series of observed and predicted monthly average biogas yield for (a) Site 37, 38 and 42 for the data involved in model generation, and (b) Site 31, 38 and 1 for independent datasets

6.3 THP MAD Sites

THP Site 1 (2014 -2016), 5 (2014 -2016) and 4 (2014 -2016) were selected as examples of low, average and good EY for model validation using the original data from the model development. The results showed the accuracy of the predictions for Model 6 varied between sites, and good prediction

results were obtained for THP Site 1 ($R^2 = 0.60$, $P < 0.001$) and 5 ($R^2 = 0.48$, $P < 0.001$). An apparent trend of improving EY was also observed for these two sites between 2014 and 2016 (Figure 6-2 a), and this response was consistent with the results from ANOVA shown in Section 4.2 (Figure 4-12). A poorer statistical description ($R^2 = 0.15$, $P = 0.074$) was provided for THP Site 4 (Figure 6-2 a). Additional data were collected for THP Site 4 (2016 -2019) as an independent dataset for model validation (Figure 6-2 b). However, the agreement between the model and measured data was still relatively poor with a R^2 value, equivalent to 0.14, compared to the original data collected between 2014 – 2016 for this site. THP pretreatment significantly increases the intensification of the MAD process, and improves digestibility of SAS, nevertheless, the overall average performance in terms of BY was relatively similar to the conventional process (Table 4-1 and Appendix 4-2). Therefore, a hypothesis was proposed that the effects of conventional and THP MAD on BY, are essentially equivalent and can be represented by the same process coefficients for temperature, HRT and DS, and that THP, therefore, simply represents different ranges of these values within the same overall BY process response profile. To test whether THP MAD could be explained by the same kinetic parameters as conventional MAD, Model 4 was applied to the THP dataset with biogas recording (THP Sites: 1 – 6) to predict BY. Figure 6-3 shows the observed and predicted BY results obtained by the conventional MAD Model 4 (calibrated each year following the internal default site approach using local BY data) for the THP sites covering the period of data collection between 2014 to 2016. The results showed the conventional MAD Model 4 gave a good overall description of BY at THP MAD sites ($P < 0.001$, $R^2 = 0.72$). Therefore, the results from the multiple regression analysis confirmed that AD of sludge preconditioned by THP is fundamentally similar to standard MAD and that the effects of temperature, HRT and DS feed on the BY of sludge receiving THP pretreatment were equivalent to those operating in the conventional MAD process, albeit at a higher range.

6.4 Combined Conventional-THP MAD Model

The results demonstrated the BY of THP and conventional MAD responded in similar manner to the statistically significant operational variables identified in Model 4 (temperature, HRT and DS feed). Therefore, the THP data were pooled together with the conventional MAD dataset to generate a combined conventional-THP model (Model 18) following the procedure described in Section 5.2. A comparison of both models (Table 6-1) showed that the adjusted R^2 values of Model 18 and Model 4 were similar and equivalent to approximately 51%. Both models contained the same statistically significant independent variables and had similar individual parameter coefficients. The THP dataset (THP Sites: 1 – 6) and data from the conventional Sites: 37, 38 and 42 were used to test BY prediction by Model 18 (Figure 6-4). The results showed that Model 18 gave a good overall description of BY at both THP ($R^2 = 0.73$, $P < 0.001$) and conventional MAD sites ($R^2 = 0.59$, $P < 0.001$). Therefore, Model 18 can be used as a practical application model for both full-scale conventional and THP MAD processes.

The complete forms of Model 4 (based on conventional only MAD sites) and Model 18 (based on combined data from conventional and THP sites) are shown below for comparisons:

Model 4: Biogas yield = $230.9 * (\ln(\text{Temperature}) - 3.6) + 136.2 * (\ln(\text{HRT}) - 3.0) - 224.8 * (\ln(\text{DS}) - 1.5) + 75.5 * ((\ln(\text{HRT}) - 3.0) * (\ln(\text{DS}) - 1.5)) + \text{site factor}$

Model 18: Biogas yield = $265.3 * (\ln(\text{Temperature}) - 3.6) + 133.7 * (\ln(\text{HRT}) - 3.0) - 216.4 * (\ln(\text{DS}) - 1.5) + 61.7 * ((\ln(\text{HRT}) - 3.0) * (\ln(\text{DS}) - 1.5)) + \text{site factor}$

Where, Biogas yield is m³/t DS, Temperature is °C, HRT is the hydraulic retention time in d, and DS is the feed dry solids content in %.



Figure 6-2 THP MAD model validation (Model 6) showing the time series of observed and predicted monthly average electricity yield for (a) THP Site 1, 5 and 4 for the data involved in model generation, and (b) THP Site 4 with independent data

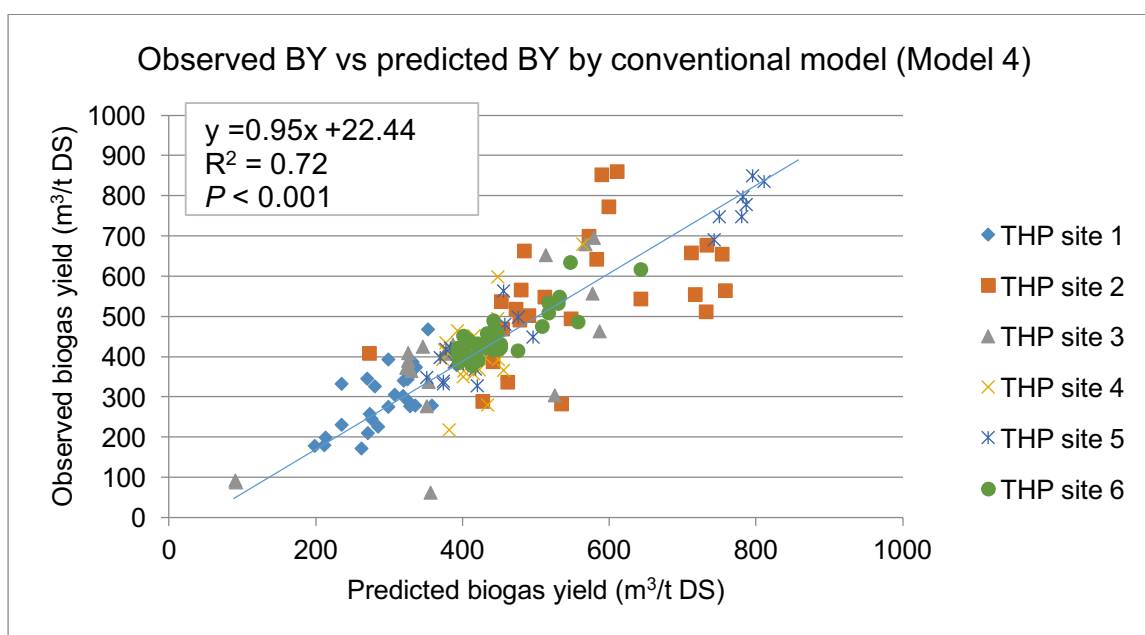


Figure 6-3 Observed biogas yield at THP MAD sites relative to values predicted by conventional MAD Model 4

Table 6-1 Conventional and combined conventional-THP mesophilic anaerobic digestion model coefficients

Model	Coefficients				Predictor total variation explained	Adjusted R ²
	Ln(Temperature)_c	Ln(HRT)_c	Ln(DS)_c	Ln(HRT)_c* Ln(DS)_c		
Conventional (Model 4)	230.9	136.2	-224.8	75.5	8.4%	51.6%
Combined conventional-THP (Model 18)	265.3	133.7	-216.4	61.7	7.7%	50.7%

6.5 Enzymic Model

The EEH Site 2 (2014 -2016) and HpH Site 2 were selected as typical performance examples for enzymic model validation using the data which was already represented in the model development (Figure 6-5). A categorical variable for treatment type, rather than a site factor, was used in the multiple regression analysis in this case to increase the proportion of variation explained by the continuous predictors. This was because, the frequency of data reported was highly variable between sites, and some were only poorly represented in the dataset (Section 5.4). This approach reduced the degrees of freedom associated with the categorical factor to two, since only 3 larger categorical groups of data were represented by treatment type, thus, the categorical variable had a smaller influence on EY whilst maintaining a similar overall R². Fundamentally, both methods (i.e. using either 'site' or 'process' as the categorical variable) generated a universal coefficient for each

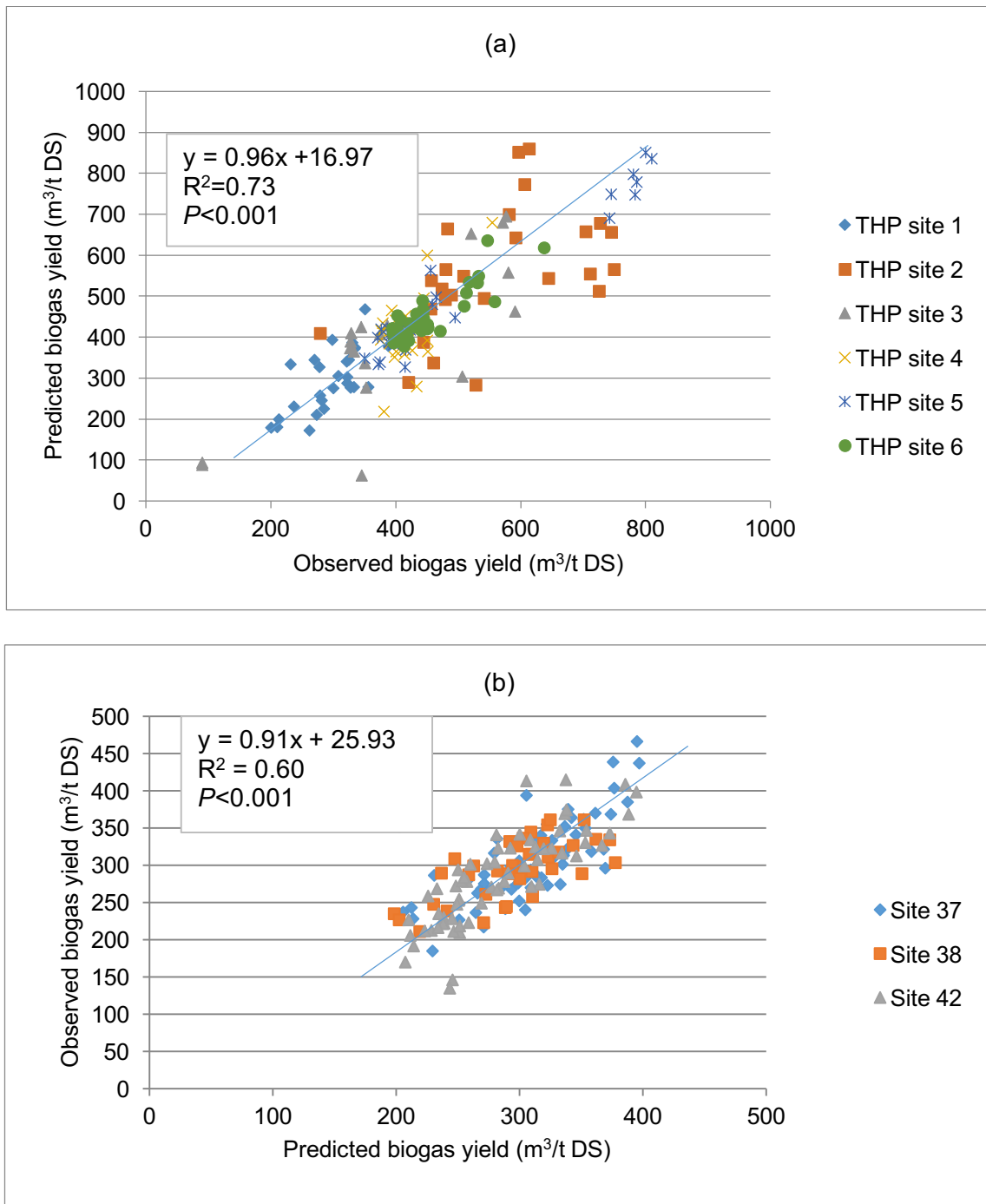


Figure 6-4 Observed biogas yield relative to values predicted by Model 18: (a) THP MAD sites and (b) conventional MAD sites

continuous predictor applied for all groups in the model. Therefore, the default site calibration method (Section 6.1) was still valid for calibrating the model to the performance of individual enzymic MAD sites. The overall R^2 of the enzymic model (Model 11) was 0.32, which was relatively poorer compared to the conventional MAD model, nevertheless, Model 11 provided a reasonable description of EEH site performance at the selected site (EEH Site 2) with R^2 and P values equivalent to 0.65 and < 0.001 , respectively. The validation for the selected HpH site gave slightly poorer $R^2 = 0.36$ and $P = 0.006$. The dataset used to develop the enzymic model was much smaller compared

to the conventional MAD model (Model 18). Therefore, the range of values represented for the different explanatory and dependent variables was likely to be considerably reduced compared to the much larger conventional MAD dataset. The poorer description of EY provided by the enzymic model could therefore be explained by statistical limitations in the actual variability in the data, constraining the ability to detect significant relationships between the different factors. Furthermore, it may also suggest that the other unmeasured factors had dominant effects on EY and that, by contrast, the principal operational parameters used in enzymic model had a relatively weaker effect at some specific sites.

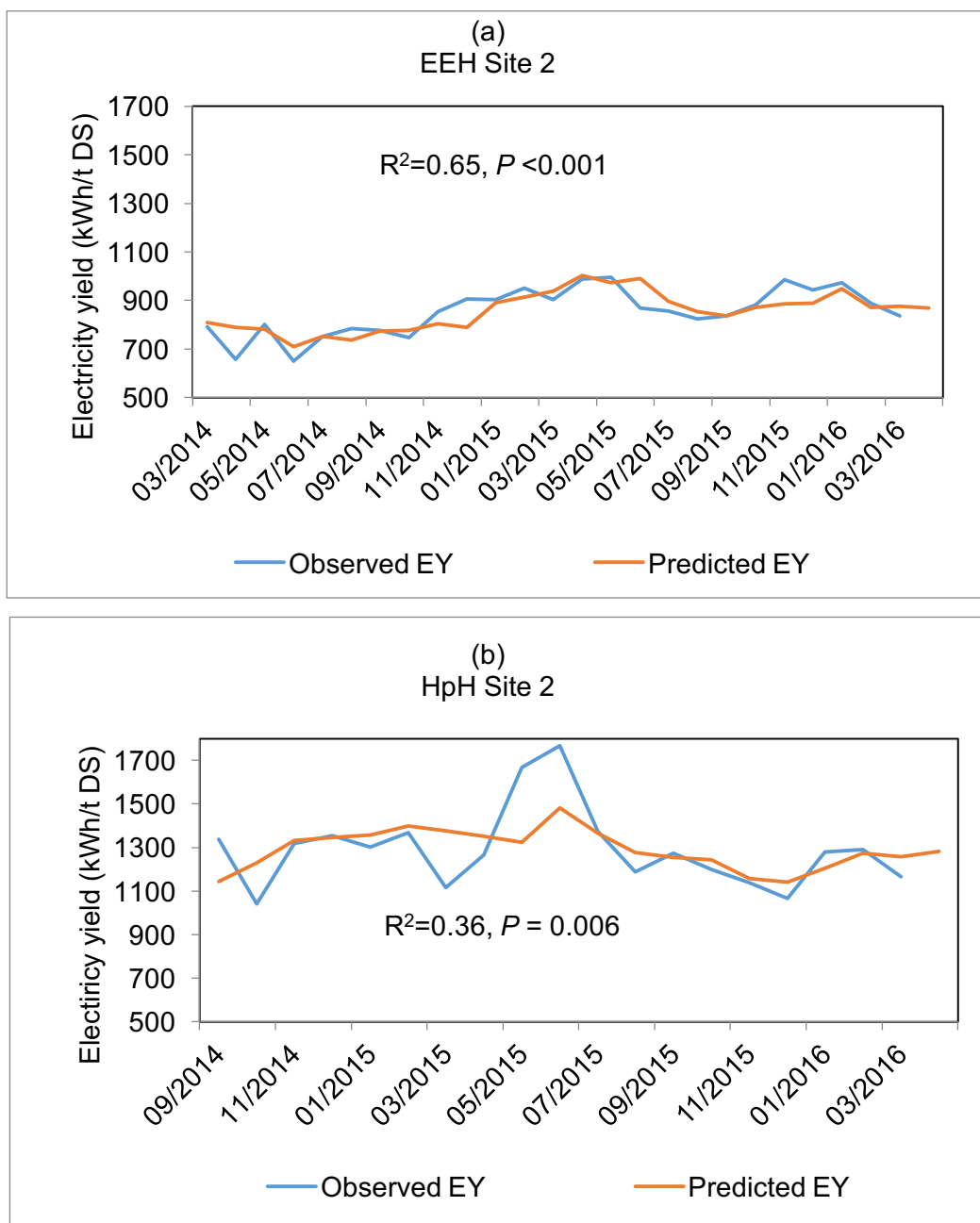


Figure 6-5 Enzymic MAD model validation (Model 11) showing the time series of observed and predicted monthly average electricity yield for (a) EEH Site 2, and (b) HpH Site 2 for the data involved in model generation

6.6 Volatile Solids Reduction Model

Site 37 (2011 – 2016), 38 (2013 – 2016) and 42 (2013 – 2016) were used for VSR model (Model 14) validation using the data which was already represented in the model development (Figure 6-6 a). Site 31 (2016 – 2019) and 38 (2016 – 2019) were used for model validation as independent datasets collected subsequent (Figure 6-6 b).

The results showed the model (Model 14) gave a good description of VSR for Site 37 and 42 with $R^2 = 0.46$ and 0.59 , respectively and $P < 0.001$ in both cases. By contrast, the description of VSR data already represented in the model development for Site 38 was not statistically significant ($R^2 = 0.05$, $P = 0.24$) the. This may be explained mainly due to the unusual variations of VSR observed in 2014, where a marked decrease in VSR was observed from 45% to 30%. In general, Site 38 had a consistent VSR of approximately 40% as observed subsequently from 2015 to 2019. The accuracy of predicting the independent dataset for VSR collected between 2016 to 2019 was substantially improved ($R^2 = 0.26$, $P = 0.002$) for Site 38. A good description of VSR was also observed for Site 31 ($R^2 = 0.59$, $P < 0.001$) using the independent dataset collected from 2016 to 2019. Therefore, in overall assessment, Model 14 provided generally reliable descriptions of VSR for different conventional MAD sites.

6.7 Dewaterability Model

Site 38 (2013 – 2016) and 42 (2011 – 2016) were used for dewaterability model (Model 17) validation using the data which was already represented in the model development (Figure 6-7 a). Site 31(2016 – 2019) and 38 (2016 – 2019) were used for model validation as independent datasets that were collected subsequently (Figure 6-7 b). The results showed that the dewaterability model could not provide a good description of the specific sludge cake DS for Site 38 for either dataset (original data: $R^2 = 0.13$, $P = 0.021$; independent data: $R^2 = 0.08$, $P = 0.122$). A better prediction was observed for Site 42 and 31 with $R^2 = 0.33$ and 0.41 , respectively, and $P < 0.001$ in both cases. The differences in the ability to determine dewaterability could be attributed to factors other than the AD process conditions, such as digested sludge characteristics, polymer dose and type of mechanical dewatering process that more directly influence dewatering, which were not included in the AD operational parameters. In general, the dewaterability model could not reliably predict sludge cake DS concentrations and, consequently, it was not taken further to model application analysis. However, it was not specifically intended for this purpose, rather, the relationships between dewaterability and digestion conditions were examined to provide insight into how upstream WWT and sludge digestion processes may influence final sludge dewaterability, and this objective was achieved.

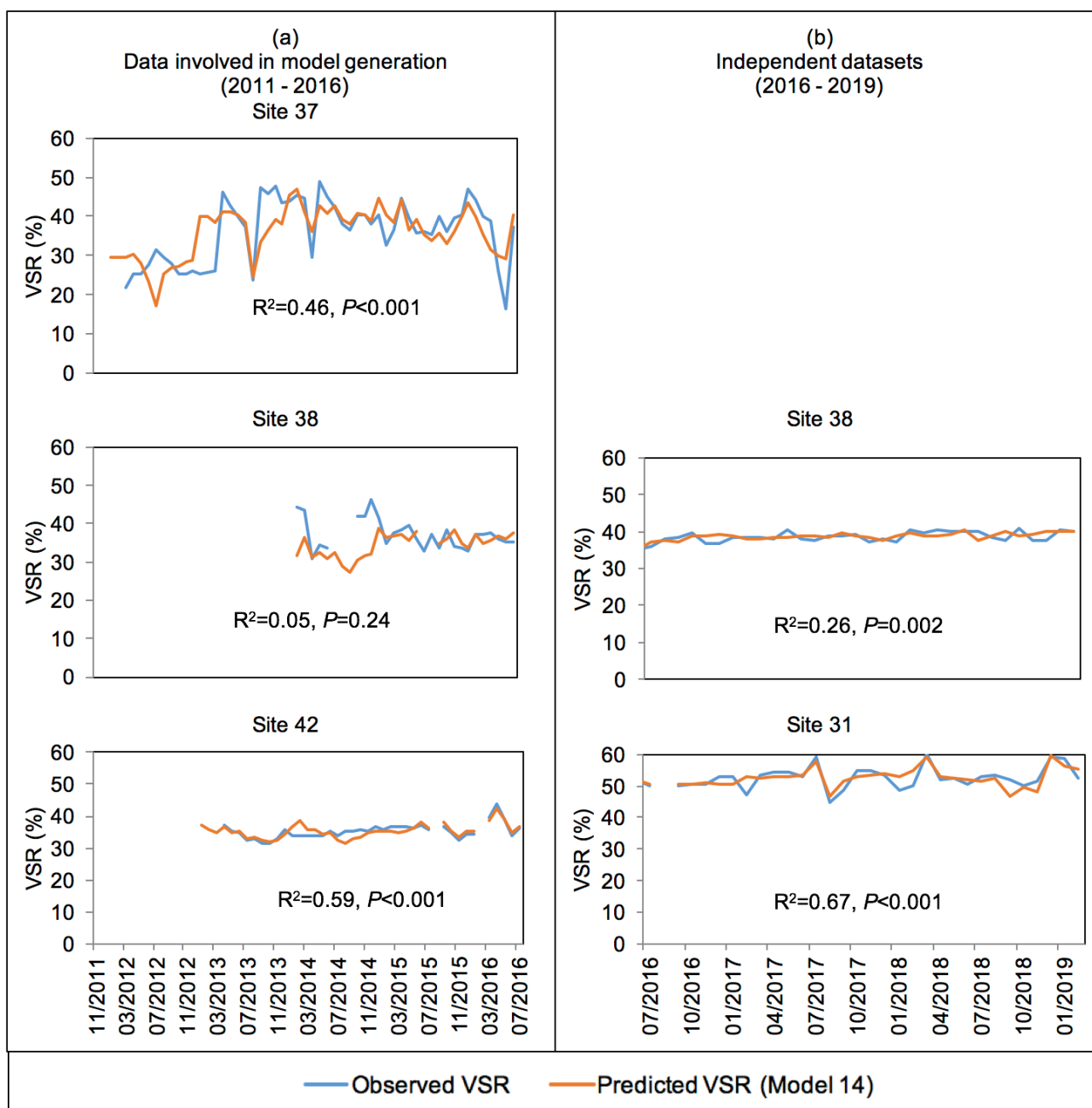


Figure 6-6 Conventional MAD VSR model validation (Model 14) showing the time series of observed and predicted monthly average VSR for (a) Site 37, 38 and 42 for the data involved in model generation, and (b) Site 31 and 38 for independent datasets

6.8 Model Boundary Conditions and Stability

Model 18 and Model 11 provide a practical, operationally defined tools to assess the performance of both conventional and advanced, enzymic MAD processes. However, the models are empirically derived based on data from full-scale operational sites and, therefore, strictly, are applicable and limited to within the boundary of process conditions represented. The minimum, maximum, 5% and 95% percentile operational range of conditions for the combined conventional-THP and enzymic

dataset are summarised in Table 6-2. Thus, the most reliable descriptions of conventional-THP and enzymic MAD processes should be obtained within the 5%-95% percentile data boundary range.

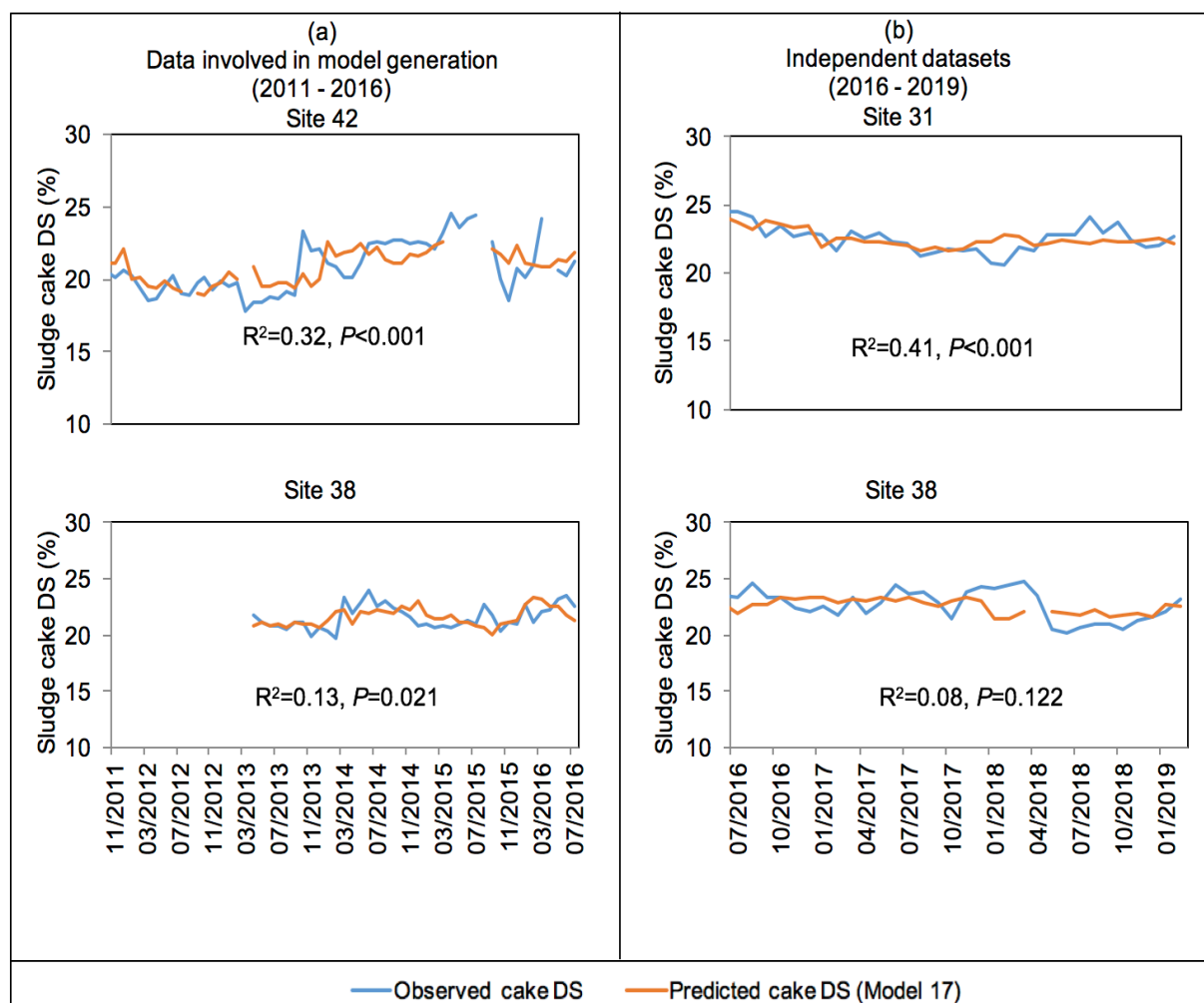


Figure 6-7 Conventional MAD dewaterability model validation (Model 17) showing the time series of observed and predicted monthly average sludge cake DS for (a) Site 38 and 42 for the data involved in model generation, and (b) Site 31 and 38 for independent datasets

The accuracy of empirical statistical models is strongly dependent on the quality of the datasets used to derive the model. At full-scale operational WWTP, many different types of parameters are measured and recorded and the quality of this is well known to vary considerably within the industry. Indeed, this is likely to be a major reason for the large proportion of unexplained variation in BY data observed in the multiple regression models developed here (Table 5-1). To test the influence of uncontrolled variability in the data, possibly linked to the recording process, random generated data were inserted to test the model stability. The random generated parameters were created using Excel RANDBETWEEN function within 90% of the data range listed in Table 6-2. The randomly generated values were added into the original datasets until the structure of the original model broke down, indicated by the drop out of predictors ($P > 0.05$) that were significant in the original model.

Up to 50 rows of random data numbers per site (representing the average frequency of data recording for each site in the combined conventional-THP MAD datasets) were inserted into the original dataset. Table 6-3 shows the changes in P value of the principal operational variables for the combined conventional-THP MAD model (Model 18) as the proportion of randomly generated data was increased. The results showed that the stability of the model was maintained with the addition of >50% of random data. At this point, temperature was approaching the threshold of statistical significance with a P value equivalent to 0.04, but this independent variable was no longer statistically significant ($P = 0.204$) at approximately 70% of additional random data. Figure 6-8 shows the changes in the adjusted R^2 , total variation explained by the continuous predictors and the coefficients of the principal operational variables for Model 18, as the proportion of randomly generated data was increased. As may be expected, the values decreased exponentially as the percentage of random data increased, and this can be attributed to the dilution effect caused by additional random data.

Random data were also inserted into enzymic MAD dataset and were evenly distributed in different enzymic pretreatment groups. The adjusted R^2 , total variation explained by the predictors and the coefficients of the principal operational variables for the enzymic MAD model decreased approximately linearly as the proportion of random data increased (Figure 6-9). The adjusted R^2 decreased to about the same extent (slope = -0.30) in both the combined conventional-THP and enzymic MAD models as the amount of random data was increased. Temperature was the least dominant predictor and was the first to be removed from both models as the proportion of random data increased (Table 6-3 and 6-4). This was consistent with the relatively smaller proportion of the total variance in the data which was explained by this independent variable compared to the other factors represented in both models (Table 5-1 and 5-3). However, the stability of the enzymic model structure decayed to a greater extent in response to the introduction of random data compared to Model 18, and the point at which structural breakdown of the model occurred was at approximately 25% of additional random data, when the P value for temperature increased above the threshold of statistical significance ($P = 0.068$) (Table 6-4). This was explained because Model 11 was generated based on a relatively small dataset containing only 182 rows of data. By contrast, Model 18 was generated based on 2838 rows of data and the results show that larger datasets increase statistical robustness and provide greater tolerance to possible unreliable data entry and reporting.

Table 6-2 Boundary data limits for the combined conventional-THP MAD and enzymic MAD datasets used to develop Model 18 and 11

Model	Parameter	Mean	Minimum	Maximum	Percentile 05	Percentile 95
Combined conventional +THP-MAD model	Temperature (°C)	36.0	11.5	45.2	31.6	39.5
	HRT (days)	21.2	1.8	53.3	12.7	35.0
	DS feed (%)	4.7	0.7	11.1	2.7	7.0
Enzymic MAD model	Temperature (°C)	37.1	25.0	42.1	32.9	40.0
	DS feed (%)	5.3	2.4	8.6	3.4	7.4
	VFA feed (mg/l)	5627	1363	10556	2187	9175

Table 6-3 Effect of increasing the proportion of random data on the stability of the combined conventional conventional-THP MAD model (Model 18) as indicated by the P values of the predictors

Percentage of additional random data (%)	<i>P</i> value			
	HRT	Temperature	DS feed	HRT*DS feed
0 (original model)	<0.001	<0.001	<0.001	0.038
18	<0.001	0.001	<0.001	0.004
35	<0.001	0.011	<0.001	0.002
53	<0.001	0.042	<0.001	0.007
70	<0.001	0.204	<0.001	0.001

Table 6-4 Effect of increasing the proportion of random data on the stability of the enzymic MAD model (Model 11) as indicated by the P values of the predictors

Percentage of additional random data (%)	<i>P</i> value			
	Temperature	VFA feed	DS feed	Temperature*VFA feed
0 (original model)	0.008	0.001	0.001	0.030
5	0.013	0.001	0.003	0.042
10	0.007	<0.001	0.002	0.104
15	0.027	<0.001	0.002	0.052
20	0.039	<0.001	0.006	0.037
25	0.068	<0.001	0.016	0.058
30	0.095	<0.001	0.040	0.049

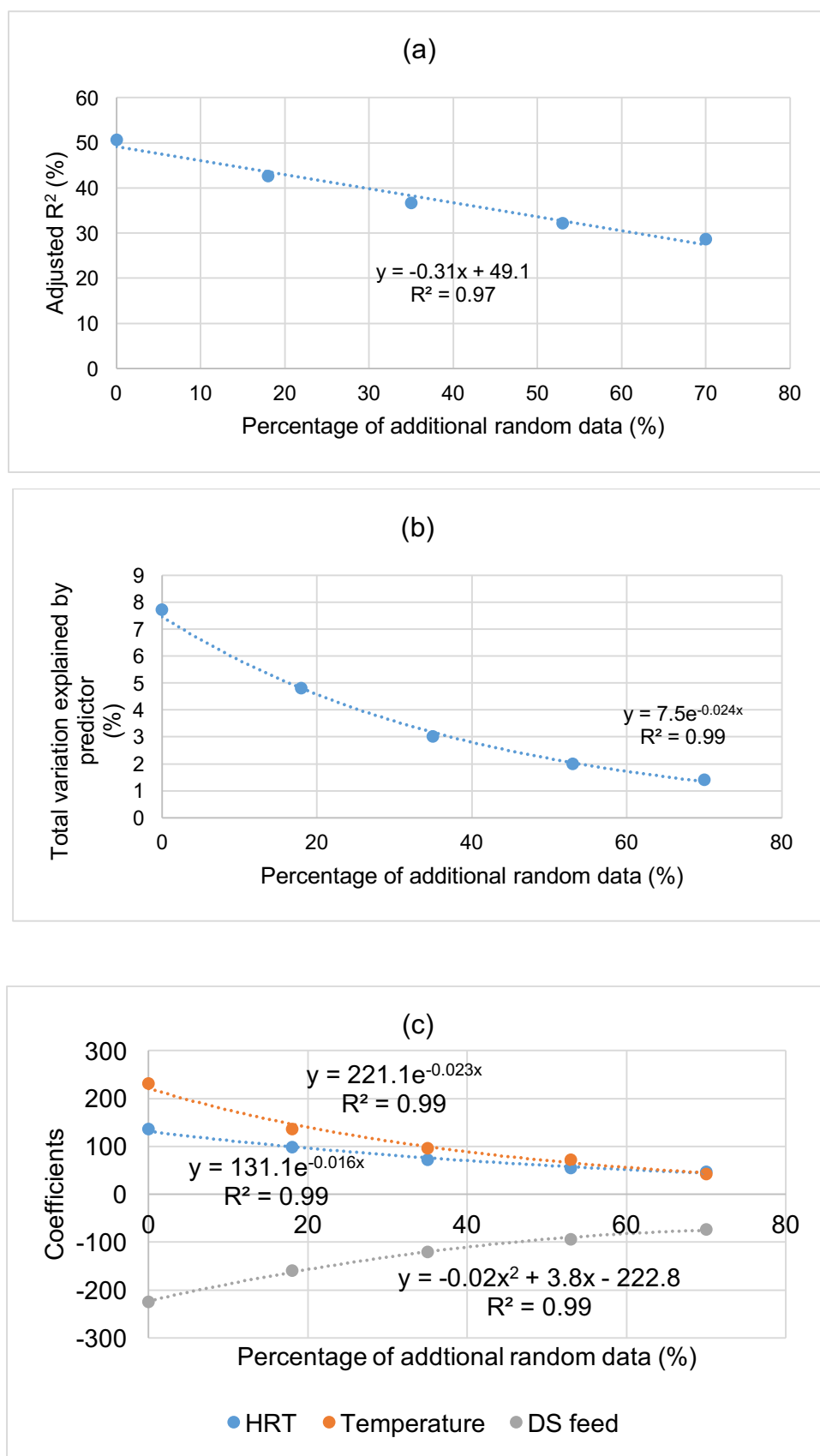


Figure 6-8 Effect of increasing the proportion of random data on the changes of (a) adjusted R^2 and (b) effective size of the predictors, and (c) principal predictors' coefficients of the combined conventional conventional-THP MAD model (Model 18)

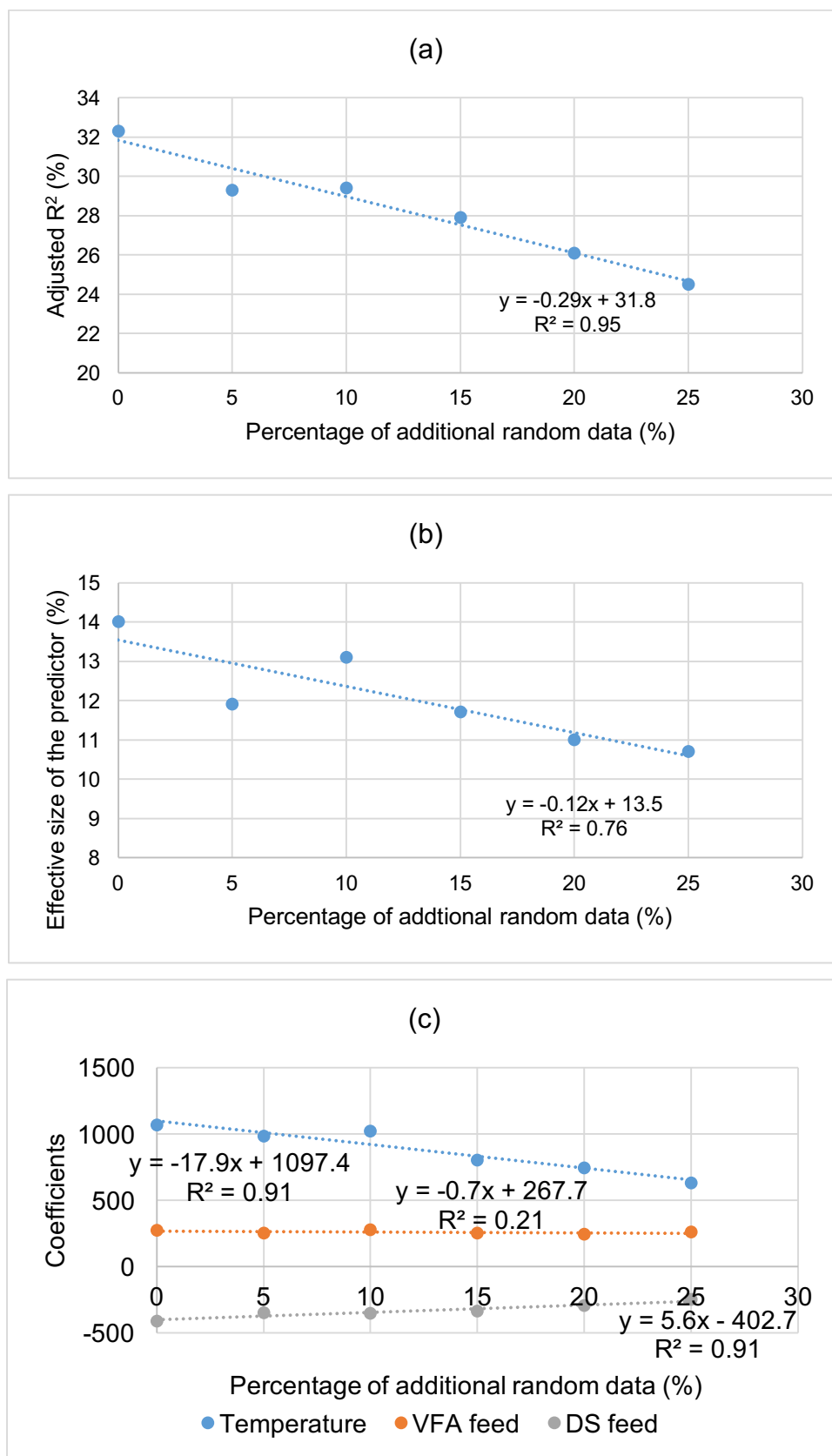


Figure 6-9 Effect of increasing the proportion of random data on the changes of (a) adjusted R^2 and (b) effective size of the predictors, and (c) principal predictor coefficients of the enzymic MAD model (Model 11)

6.9 Summary

This Chapter validated the multiple regression models against both specific WWTP datasets used in model development, and independent datasets which were collected subsequently and were not used to develop the model. The model validation results showed that conventional MAD model (Model 4) could effectively predict the BY of conventional MAD sites with reasonable data recording. Furthermore, the BY of THP and conventional MAD responded in a similar manner to the statistically significant operational variables identified in Model 4 (temperature, HRT and DS feed). Therefore, a combined conventional-THP model (Model 18) was generated, integrating data for both process types from all of the participating UK Water Utility companies. The combined conventional-THP model (Model 18) could effectively predict the BY of both conventional ($P < 0.001$, $R^2 = 0.59$) and THP sites ($P < 0.001$, $R^2 = 0.73$). The enzymic MAD model (Model 11) had relatively low overall R^2 (0.32), but it could still effectively describe the general patterns of EY for the selected validation sites, such as EEH Site 2 with R^2 and P values equivalent to 0.65 and < 0.001 , respectively.

The R^2 of the validation results depended on two factors: (1) the extent to which the coefficients of the principal operational parameter explained the BY, and (2) the frequency of model calibration to adjust the site factor (intercept) to determine the absolute BY value. The model was calibrated yearly in the model validation, and therefore assumed that the influence of unmeasured factors was consistent for each of these periods. Therefore, the R^2 values presented in the model validation may only be used as a guide to the model description of operational data. The results showed that the model effectively predicted the BY of sites demonstrating stable operating conditions, for example, Site 38 (Figure 6-1 Model 4). However, under such stable operation the deviation in BY is relatively limited, therefore the apparent R^2 obtained for the relationship between observed and expected results may be relatively modest (for example, $R^2 = 0.35$ in this case). The most reliable predictions of BY will be obtained for sites with characteristically stable operating conditions and where the site factor is consistent and captures the effects of other relevant management factors (for example, stable sludge composition, mixing efficiency, primary sludge ratio) that are not explained by the continuous variables in the model.

Furthermore, a model stability test demonstrated these models (Model 11 and 18) were robust to the possible entry and reporting of unreliable data at full-scale MAD sites. Therefore, the results demonstrated that the models were reliable and could be practically applied to predict the variations of BY at full-scale AD plants.

Chapter 7. Mass Balance and Contribution of Organic Macromolecule Energy Substrates in Sewage Sludge to the Biogas Yield of the AD Process

7.1 Introduction

The biodegradability and CH₄ production of sewage sludge in full-scale MAD processes is affected by multiple, complex physical, chemical and biological factors as discussed in Section 2.3 and 2.4. The operational conditions of temperature, HRT and DS feed have statistically significant impacts on digestion performance which have been represented in multiple regression models, as presented in Chapter 5. However, sites with similar operating conditions do not necessarily demonstrate the same performance, and part of the reason for these differences could be attributed to the variation in sludge composition (Weiland, 2010; Li *et al.*, 2018). Indeed, a categorical factor was used to explain the variation between sites in the models, nevertheless, the description of the process performance could still vary between sites as observed in the model validation (Section 6.2). The effect of sludge composition on AD performance was therefore investigated by conducting a comprehensive sampling and analysis programme to measure the macromolecular (protein, fat, carbohydrate and fibre contents) and inorganic elemental content. The experimental procedures and strategy for site selection are described in Section 3.3.2. Eight sites were selected for sample collection with representative examples of each of the main AD treatment types, including: conventional, THP and enzymic processes. Sludge feed and digestate composition were compared during the course of 6 sampling events, over a period of approximately one year (see Section 3.3.2).

7.2 Volatile Solids Reduction

Volatile solids is simple measurement of the total of organic matter content in sludge. The volatile solids reduction (VSR) by conventional MAD processes was strongly linked to the VS available in the sludge feed. For example, Site 38 had both the smallest VS in the feed (72.6%) and VSR (39.9%), by contrast, Site 1 had the largest VS in the feed (78.2%) and also VSR (52.0%) (Figure 7-1). Pretreatment by THP improved overall sludge digestibility and reduced the dependency of VSR on the VS content in the feed. Thus, THP Site 4 and 7 had a relatively small VS feed concentration (75.4% and 77.2%, respectively), but achieved a similar or larger VSR (50.4% and 53.7%, respectively) compared to conventional AD processes supplied with feed sludge with high VS values (Site 1 and 31) (Figure 7-1). However, pretreatment by EEH and HpH gave no apparent increase in

VSR, and EEH Site 2 and HpH Site 3 had the same VSR (38.3%) compared to conventional MAD Site 38 with the lowest VS input. To test the statistical significance of VSR and VS available in the sludge feed further, more detailed correlation analysis was performed, using the data from each sampling event from all sampling sites. The results showed a weak, albeit statistically significant positive effect ($R^2 = 0.15$, $P = 0.015$) of VS feed on VSR across all the sampling sites (Figure 7-2).

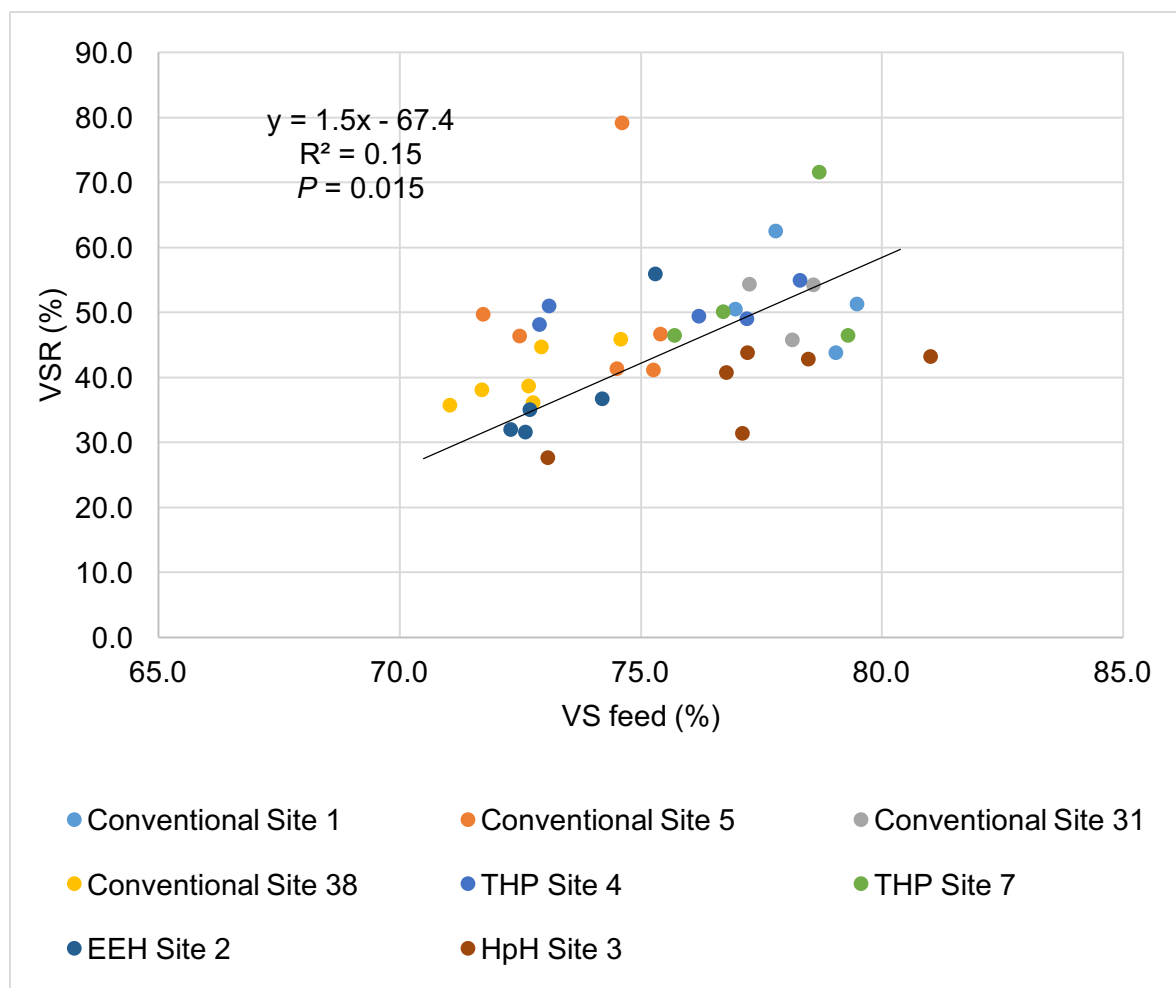


Figure 7-1 Relationship between VS feed and VSR for all sampling sites

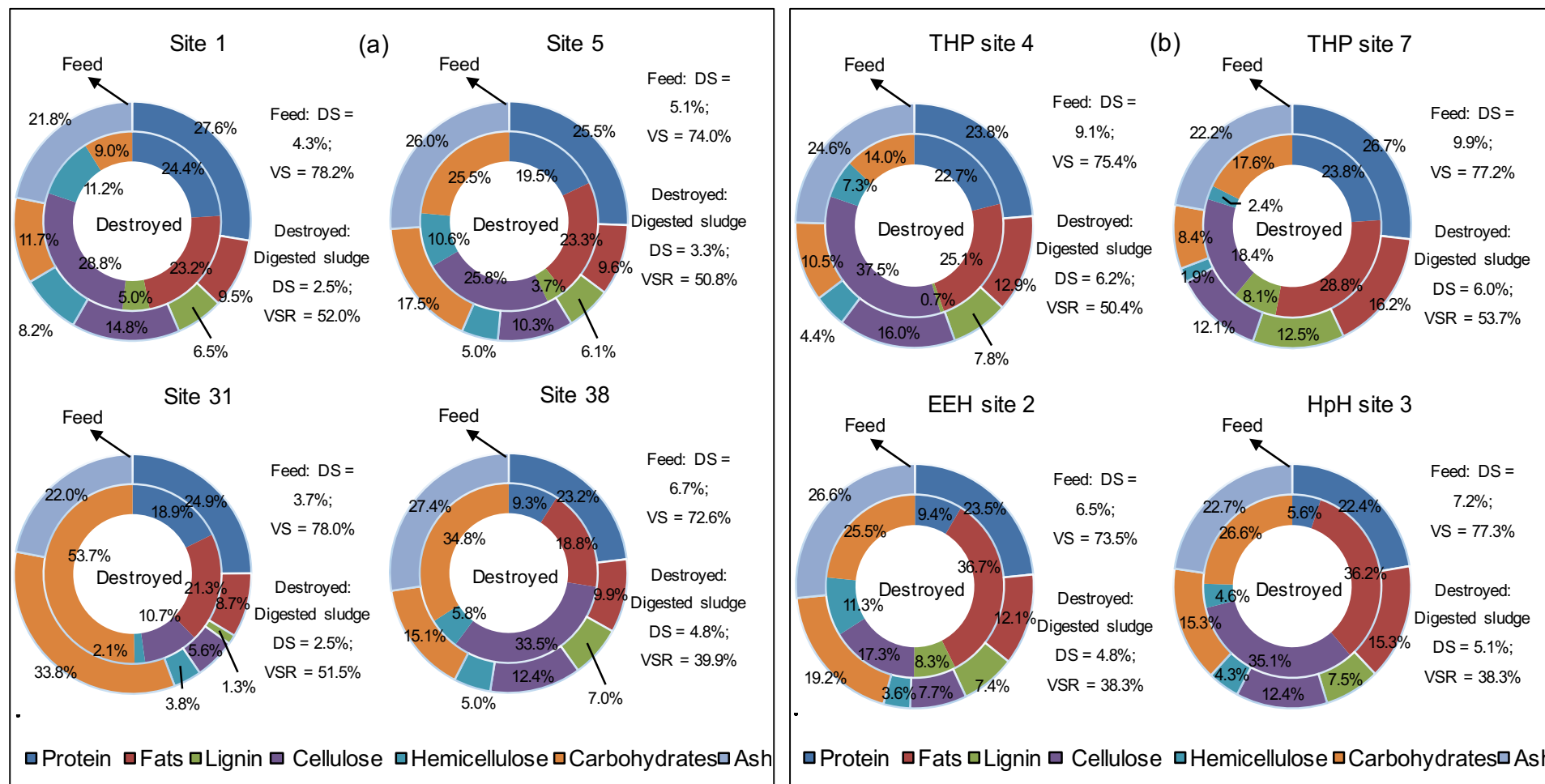
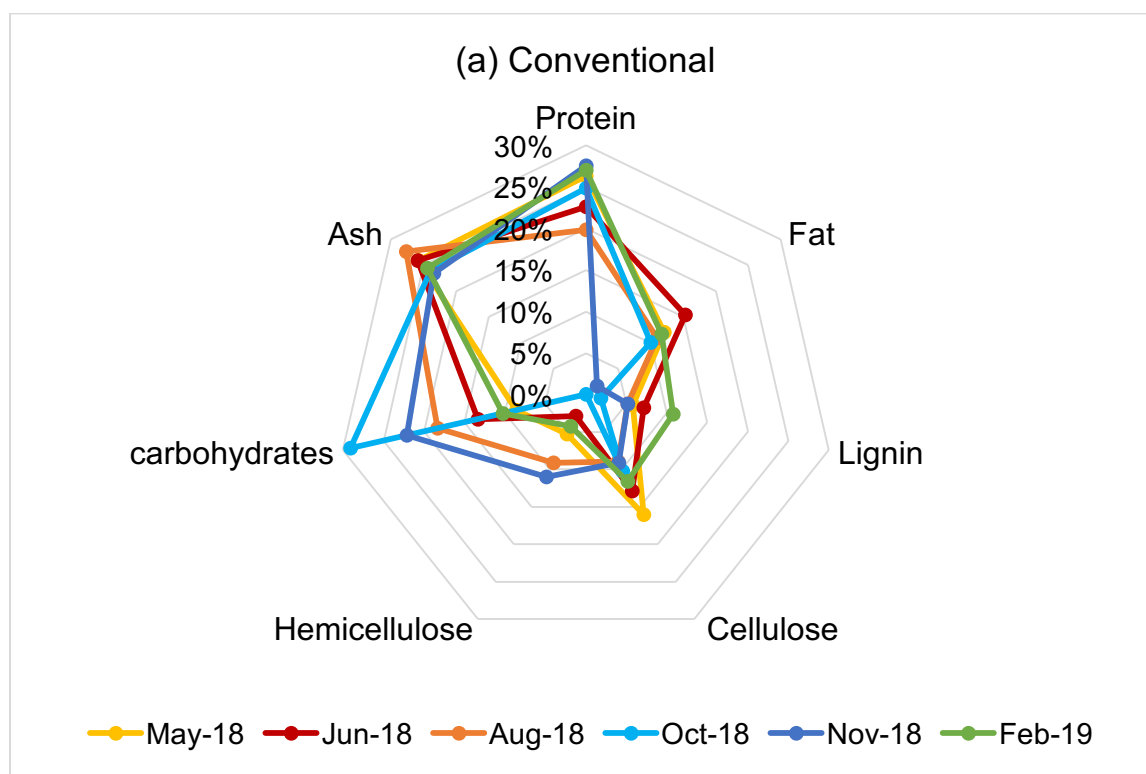


Figure 7-2 Composition of organic and ash fractions in sludge feed (% DS basis) and corresponding destroyed fractions (% VS basis) by anaerobic digestion for (a) Conventional Site 1, 5, 31 and 38; (b) THP Site 4, 7, EEH Site 2 and HpH Site 3 (outer circle: sludge feed; inner circle: fraction destroyed by digestion)

7.3 Temporal Patterns in Feed Sludge Composition

The size of major organic energy substrate and ash fractions in feed sludge (% DS basis) for conventional, THP and enzymic sites at different sampling times is shown in Figure 7-3, and the detailed data for individual sites at each sampling time are summarised in Appendix 7-1. In general, the proportions of each energy substrate in feed sludge followed a similar pattern at both conventional and advanced MAD sites, but increased amounts of high CH₄ yielding substrates (fat and protein) and VS contents available in the feed sludge were observed for specific sampling events. For example, protein and fat, typically accounted for a significant proportion of the organic matter in feed sludge and varied between 19 - 27% DS and 2 - 22% DS, respectively. Similarly, the VS content available in the feed sludge varied from 71 to 81%. Therefore, variations in BY observed at different sites or the same site at different times could be attributed to the specific composition of the feed sludge, which is examined in detail in Section 7.4.



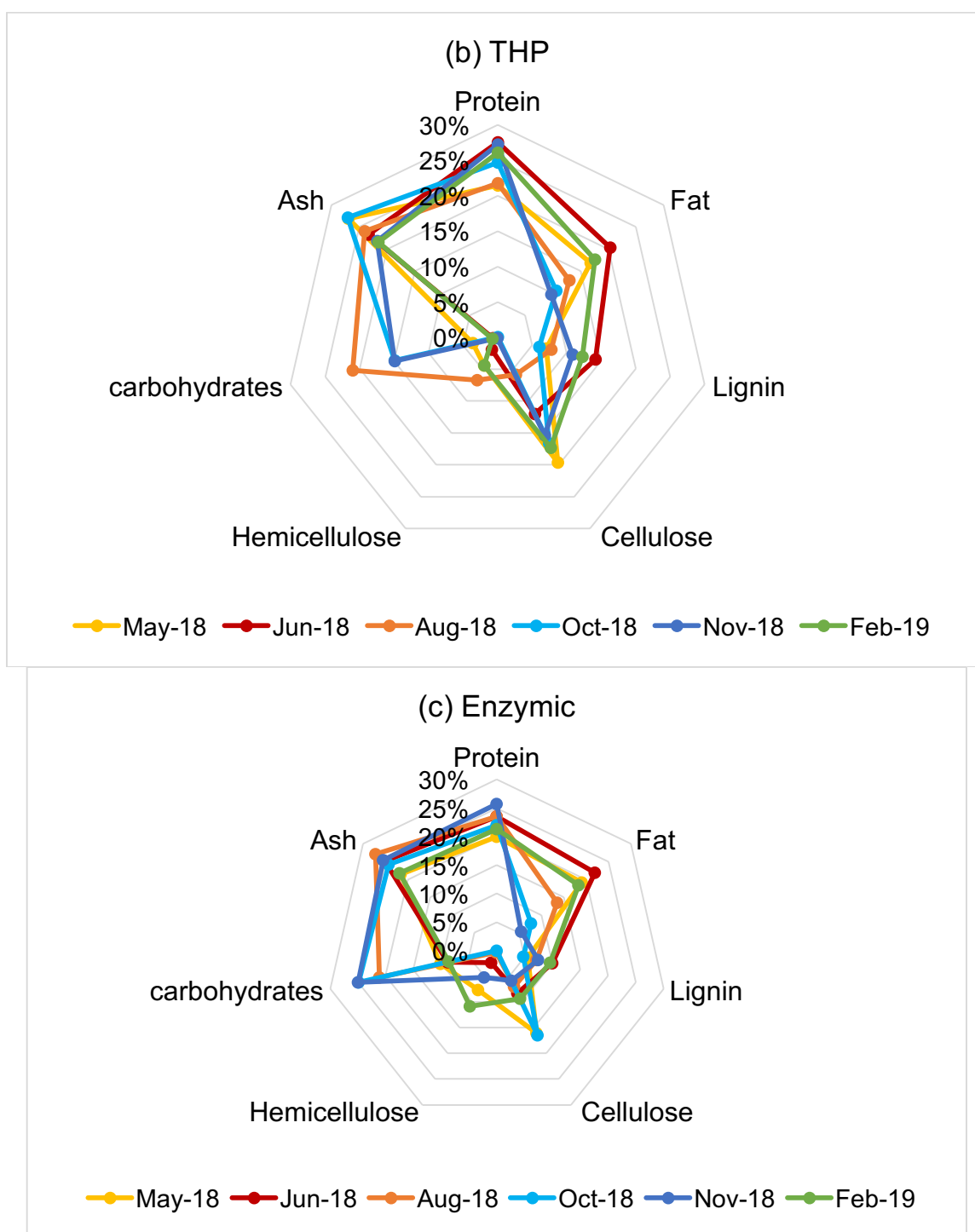


Figure 7-3 Mean composition of organic and ash fractions in sludge feed (% DS basis) for (a) conventional, (b) THP and (c) enzymic MAD processes at different sampling times

7.4 Macromolecular Composition

The proportions of each substrate in the feed sludge and the destroyed fractions during AD on a VS basis were calculated and are summarised in Figure 7-1. Site average organic fraction composition of digested and feed sludge are summarised in Table 7-1 and Appendix 7-2, respectively. Macromolecules comprising the fibre fraction (hemicellulose and cellulose) and carbohydrates have

a similar CH₄ yield, approximately equivalent to 400 m³/t DS (Table 2-2), thus, variations in BY at different sites mainly depended on the fractions of higher CH₄ yielding substrates, such as fat (CH₄ yield = 1014 m³/t DS) and protein (CH₄ yield = 496 m³/t DS), and also the total amount of VS available in the sludge feed. For example, Site 38 had the smallest observed BY during the sampling period compared to the other conventional MAD sites examined. This was consistent with the smallest VS content (72.6% DS) and fractions of fat (18.8%) and protein (9.3%) destroyed, compared to the sludge samples collected from the other sites. By contrast, the highest BY was observed for Site 1 with the largest VS content (78.2%) in the feed sludge, which also had high fractions of fat (23.2%) and protein (24.4%) destroyed during AD.

To further test the statistical significance of sludge feed composition on BY, more detailed correlation analysis was performed, using the data from each sampling event. Positive, statistically significant correlations were observed between BY and sludge feed VS and VSR for the 4 sampling sites with reasonable biogas recording (BY >700 m³/t DS was removed) ($P = 0.038$, $R^2 = 0.24$ and $P = 0.004$, $R^2 = 0.42$, respectively) (Figure 7-4), and this was consistent with the average site observation that higher VS feed can positively influence BY. However, no significant relation was detected between BY and sludge feed protein or fat content, or the fractions of protein and fat destroyed. Estimated P values for the correlation between each of these high CH₄ yield substrates in the feed and destroyed during AD, in relation to BY for the 4 sites with reasonable biogas recording were 0.729 and 0.758 for protein, and 0.279 and 0.674 for fat, respectively. Thus, the content of individual energy substrate macromolecules apparently has no detectable influence on BY, at least at the frequency of spot sampling followed here. However, further scrutiny of the energy substrate composition results was undertaken and a sludge composition BY_c was derived for each sampling event based on the substrates destroyed during AD, and the theoretical CH₄ yield and content in the biogas (Table 2-2). The detailed calculated BY_c values for each sampling event are summarised in Appendix 7-3. A positive and highly statistically significant correlation ($P = 0.005$) was found between BY_c and the observed BY for the sites with reasonable biogas recording (Figure 7-5). Therefore, observed BY could be described by the BY_c with a reasonable degree of confidence based on the composition of energy substrates measured in sludge. This approach also provided a BY value to evaluate the effect of VS and macromolecular composition on digester performance for advanced MAD sampling sites where there was no biogas recording (THP Site 7, EEH Site 2 and HpH Site 3).

Table 7-1 Mean dry solids (%), volatile solids and major organic energy constituents in digested sludge (% DS basis) for Conventional Site: 1, 5, 31, 38; THP Site: 4, 7; EEH Site 2 and HpH Site 3 (n = 6)

Site	DS	VS	Protein	Fat	Lignin	Cellulose	Hemicellulose	Carbohydrates
Conventional Site 1	2.5	59.8	33.5	0.0	9.1	6.9	8.4	8.1
Conventional Site 5	3.3	56.4	28.7	7.2	11.0	4.2	3.9	13.2
Conventional Site 31	2.5	62.3	28.3	7.7	8.5	7.8	6.7	16.8
Conventional Site 38	4.8	61.0	28.7	12.7	9.9	4.8	14.1	7.3
Conventional average	3.3	59.9	29.8	6.9	9.6	5.9	8.3	11.4
THP Site 4	6.2	58.4	23.6	8.3	12.9	5.2	3.5	8.2
THP Site 7	6.0	60.8	29.4	8.0	16.1	7.1	2.7	4.9
THP average	6.1	59.6	26.5	8.2	14.5	6.1	3.1	6.6
EEH Site 2	4.8	60.9	28.4	8.7	8.8	5.4	4.6	17.0
HpH Site 3	5.1	66.5	29.1	8.4	12.1	5.0	5.0	10.8
Enzymic average	5.0	63.7	28.8	8.5	10.4	5.2	4.8	13.9

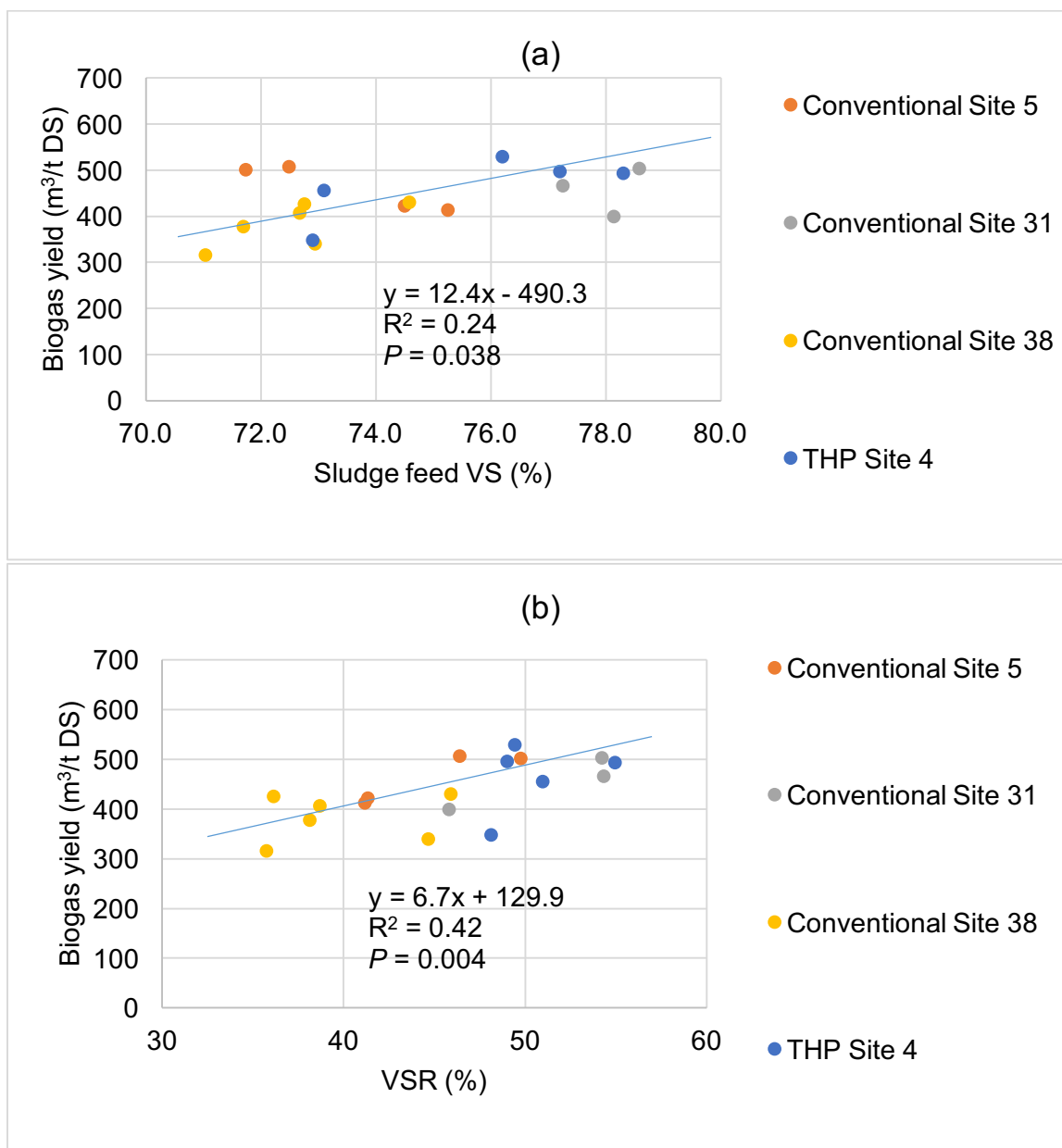


Figure 7-4 Relationship between (a) sludge feed VS, and (b) VSR on BY for the sampling sites with reasonable biogas recording ($\text{BY} < 700 \text{ m}^3/\text{t DS}$)

For example, HPH Site 3 had a higher BY_c ($335.2 \text{ m}^3/\text{t DS}$) during the sampling period (05/2018 – 02/2019) compared to EEH Site 2 ($282.0 \text{ m}^3/\text{t DS}$), and this was attributed to the higher VS content in feed sludge at HPH Site 3 (77.3%) compared to EEH Site 2 (73.5%). The two enzymic sites (EEH Site 2 and HPH Site 3) also exhibited a unique pattern of low protein (5.6% - 9.4%), but high fat (36.2% - 36.7%) destruction during the MAD process (Figure 7-1), and this may be attributed to the smaller protein, but larger fat fractions measured in the feed sludge compared to the conventional sites. Similarly, THP Site 7 had higher VS content (77.2%), and fractions of fat (28.8%) and protein (23.8%) destroyed during MAD, compared to THP Site 4. As may be expected, therefore, THP Site 7 concomitantly had a larger BY_c ($397.3 \text{ m}^3/\text{t DS}$) compared to THP Site 4 ($372.0 \text{ m}^3/\text{t DS}$).

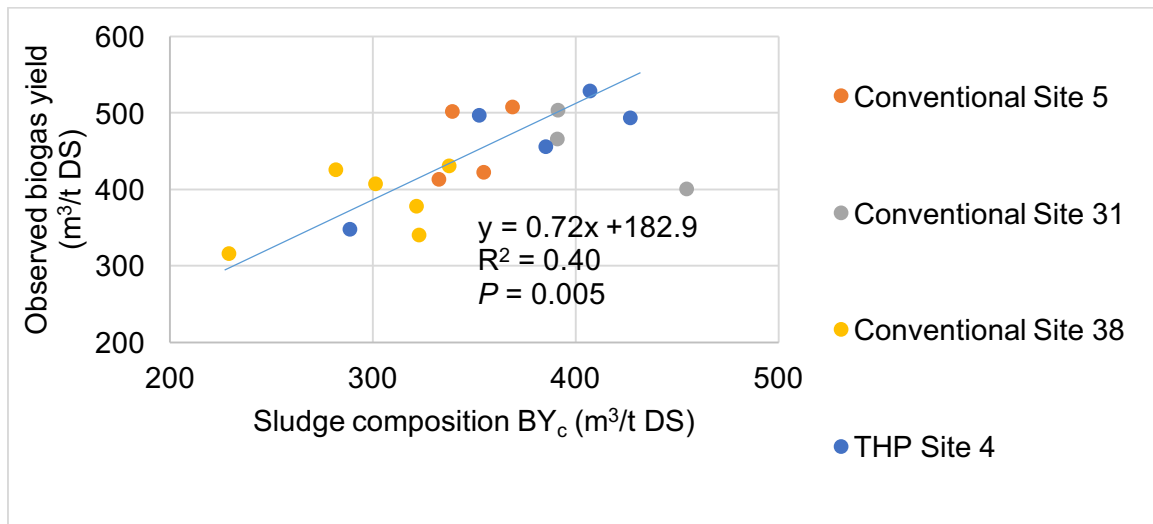


Figure 7-5 Relationship between sludge composition BY_c and observed BY for the sampling site with reasonable biogas recording ($BY < 700 \text{ m}^3/\text{t DS}$)

7.5 Inorganic Elemental Composition

Sewage sludge also contains inorganic elements from industrial and domestic sources and the mean values of feed and digested sludge samples for the different treatment types are presented in Table 7-2, and the detailed data are summarised in Appendix 7-4. Heavy metals can be present in significant concentrations in municipal sewage sludge, including Cu, Zn, Pb, Cd, Hg, Cr, Co, Fe, Ni, and Mo (Altaş, 2009). Many heavy metals are part of essential enzymes and are required at low concentrations for anaerobic biological reactions (Takashima and Speece, 1989). However, excessive amounts of heavy metals can lead to inhibition or toxicity (Li and Fang, 2007). The concentrations of potentially toxic elements (PTEs) (eg. Zn, Cu, Pb and Cd) were within the normal low ranges expected as a consequence of effective source control measures (Smith, 2009), and the results were consistent with other sludge composition surveys reported within the last 10 years (Table 7-2). Zinc and Cu were present in the largest amounts in sludge; the most significant inputs of these elements to the wastewater system arise from diffuse domestic sources, such as body care products and plumbing systems (Smith, 1995).

Iron salts are used as a precipitant for chemical phosphorus removal (CPR) to remove P from wastewater, generating sludge rich in P and Fe. As expected, Fe and P are present in the largest amounts in sludge generated by CPR processes (for example: Conventional Site 31; THP Site: 4, 7 and EEH Site 2). There are contradictory reports of the influence of CPR on AD performance (Grigoropoulos *et al.*, 1971; Ghyoot and Verstraete, 1997; Gosset *et al.*, 1978; Dentel and Gosset, 1982; Lagrotta, 2015) as discussed in Section 2.3.6. This is because the impact of Fe-dosing on sludge characteristics and AD performance may depend on the location where Fe is introduced into the wastewater treatment process. For example, it is reported to have a beneficial effect on CH_4 production if supplied in primary WWT as suggested by Lagrotta (2015).

Table 7-2 Mean inorganic elemental composition of feed and digested sludge after conventional, THP and enzymic MAD (n = 6)

Element	Conventional feed	Conventional digested	Conventional increase (%)	THP feed	THP digested	THP increase (%)	Enzymic feed	Enzymic digested	Enzymic increase (%)	Typical ranges
Total N (% DS)	5.1	7.6		5.2	7.7		5.9	8.2		1.1 – 7.5 ^{a,e}
NH ₄ -N (mg/l)	539.9	985.2		826.3	2206.8		1551.3	1870.5		
Fe (% DS)	3.8	5.4	40.7	4.8	7.8	61.1	3.6	5.1	41.5	1.4 - 6.9 ^b
Ca (% DS)	3.5	4.4	26.9	2.5	4.2	68.1	2.8	3.9	41.3	
P (% DS)	2.4	3.3	39.6	2.4	4.0	64.9	2.2	3.1	40.7	1.5 – 3.2 ^b
S (% DS)	1.0	1.3	34.7	1.1	2.0	77.6	1.4	1.9	40.6	0.8 – 1.3 ^b
K (% DS)	0.4	0.6	43.1	0.2	0.3	59.9	0.2	0.3	46.2	0.07 – 0.49 ^c
Mg (% DS)	0.4	0.5	34.5	0.2	0.3	56.5	0.2	0.3	40.6	0.12 -0.43 ^c
Na (% DS)	0.3	0.3	10.4	0.1	0.2	56.4	0.2	0.4	47.0	0.02 – 0.53 ^c
Zn (mg/kg DS)	617.9	687.4	11.2	447.1	729.1	63.1	556.5	773.0	38.9	556.3 – 740.1 ^d
Cu (mg/kg DS)	401.9	477.5	18.8	202.0	345.1	70.8	265.2	370.4	39.7	401.6 – 607.0 ^{d,e}

Table 7-2 continued

Mn (mg/kg DS)	234.0	322.8	38.0	298.3	417.3	39.9	190.7	267.7	40.4	75.2 – 959.7 ^f
Pb (mg/kg DS)	48.0	64.0	33.5	27.1	45.3	67.0	42.1	60.7	44.3	60.0 – 74.4 ^{d,e}
Ni (mg/kg DS)	22.1	32.6	47.6	18.1	29.5	63.0	24.0	34.2	42.7	23.0 – 30.2 ^{d,e}
Cr (mg/kg DS)	31.1	41.3	32.8	28.1	48.3	72.1	34.8	50.3	44.6	37.8 – 79.0 ^d
Mo (mg/kg DS)	5.9	7.9	34.6	4.9	7.9	61.0	7.4	10.4	40.8	0 – 25 ^c
Co (mg/kg DS)	4.7	7.0	48.8	4.8	7.4	53.4	3.8	5.3	40.2	1.5 – 16.7 ^f
Se (mg/kg DS)	2.8	4.0	42.3	1.8	3.1	69.5	2.8	3.9	40.5	3.4 – 53.6 ^f
Cd (mg/kg DS)	1.0	1.4	45.4	0.6	1.1	65.5	0.8	1.2	41.1	0.8 – 1.2 ^{d,e}
Overall increase			34.3			62.9			41.8	

a. Total N of raw and digested sludge (Rigby *et al.*, 2016); b. Survey of feed and digested sludge from 4 WWTPs in Belgium (Dewil *et al.*, 2009); c. Survey of digested sludge from 6 WWTPs in Swaziland, Southern Africa (Mtshali *et al.*, 2014); d. Survey of digested sludge from 75 WWTPs in UK, selected data from 2010 to 2017 (Liu, 2018); e. Survey of raw sludge from 28 WWTPs in UK (Jones *et al.*, 2014); f. Results of a 61 randomly taken sewage sludge samples within European Countries (Gawlik, 2012).

During AD, VSR increases the concentrations of conserved elements. Digested sludge collected from the THP MAD process showed the largest overall increase in elemental concentrations, equivalent to 62.9%, compared to enzymic and conventional MAD which increased the overall average concentration by 41.8% and 34.3%, respectively. The rise in concentrations of conservative elements demonstrated the effectiveness of pretreatment at advanced sites to some extent, which increased the VSR during MAD process, and hence resulted in a higher concentration of the conservative elements in digested sludge. Thus, THP MAD sites had the largest overall VSR, equivalent to 51.9%, however, enzymic and conventional MAD, had overall VSR values equivalent to 38.0% and 46.7%, respectively, and therefore, in these cases the increase in total elements in digested sludge could not related to a simple function of the average VSR.

7.6 Chemical Oxygen Demand

The results from the determination of DS and VS contents and the COD of separate subsamples of feed and digested sludge from the selected sampling sites samples are summarised in Table 7-3 and Appendix 7-5. Good agreement was observed between DS measured at the external laboratory (NRM) and the supplementary measurements performed in the Roger Perry Laboratory at Imperial College London ($R^2 = 0.97$; slope coefficient = 0.975) (Figure 7-6). The mean COD input and output of the digester for conventional and enzymic sites were 62 and 40 g/l, respectively, and 108 and 64 g/l, respectively. THP sites had higher COD input and output values compared to conventional and enzymic sites, equivalent to 128 and 82 g/l, respectively. The higher COD in feed sludge for the THP sites was reflected in the DS concentrations and the results reflected the higher solids content supplied in feed sludge to THP digestion systems, as discussed in Section 2.3.8. The mean DS feed values for THP, enzymic and conventional sites were 9.4%, 6.7% and 4.6%, respectively, and a statistically significant linear relationship ($R^2 = 0.89$, $P < 0.001$) was observed between COD feed and DS feed values (Figure 7-7).

Site overall average COD removal efficiency values were calculated from the difference in feed and digested sludge COD at each sampling time and are summarised in Table 7-3. The mean COD removal efficiency for conventional MAD sites was 36.4%. The THP and enzymic MAD sites had higher mean COD removal efficiency compared to conventional sites, and their values were equivalent to 40.6% and 40.5%, respectively. Therefore, pretreatment by THP and enzymic methods not only increased the solids contents in sludge feed to the digesters, but also improved overall COD removal efficiency at advanced MAD sites. The COD removal efficiency represented the overall destruction of organic substrates during the AD process. As expected, it was statistically significant ($P = 0.013$) and positively associated with BY for the sites with reasonable biogas recording (Figure 7-8). Furthermore, a statistically significant, positive correlation was observed between the COD removal efficiency and VSR for all sampling sites ($P < 0.001$, $R^2 = 0.37$) (Figure 7-9).

Table 7-3 Site average DS, VS and COD in feed and digested sludge

Site	DS feed (%)	DS digested (%)	VS feed (%)	VS digested (%)	COD feed (g/l)	COD digested (g/l)	COD removal efficiency (%)
Conventional Site 1	3.6	2.4	78.3	59.8	46	33	35.1
Conventional Site 5	4.8	3.2	74.0	56.4	70	40	39.3
Conventional Site 31	3.3	2.5	78.2	62.3	43	31	34.8
Conventional Site 38	6.6	4.7	72.6	61.0	89	58	35.4
Conventional average	4.6	3.2	75.8	59.9	62	40	36.4
THP Site 4	8.9	6.1	75.4	58.4	114	79	38.4
THP Site 7	9.9	5.9	77.2	60.8	142	85	42.8
THP average	9.4	6.0	76.3	59.6	128	82	40.6
EEH Site 2	6.4	4.7	73.4	60.9	102	62	39.1
HpH Site 3	7.1	5.1	77.3	66.5	115	66	41.7
Enzymic average	6.7	4.9	75.4	63.7	108	64	40.4

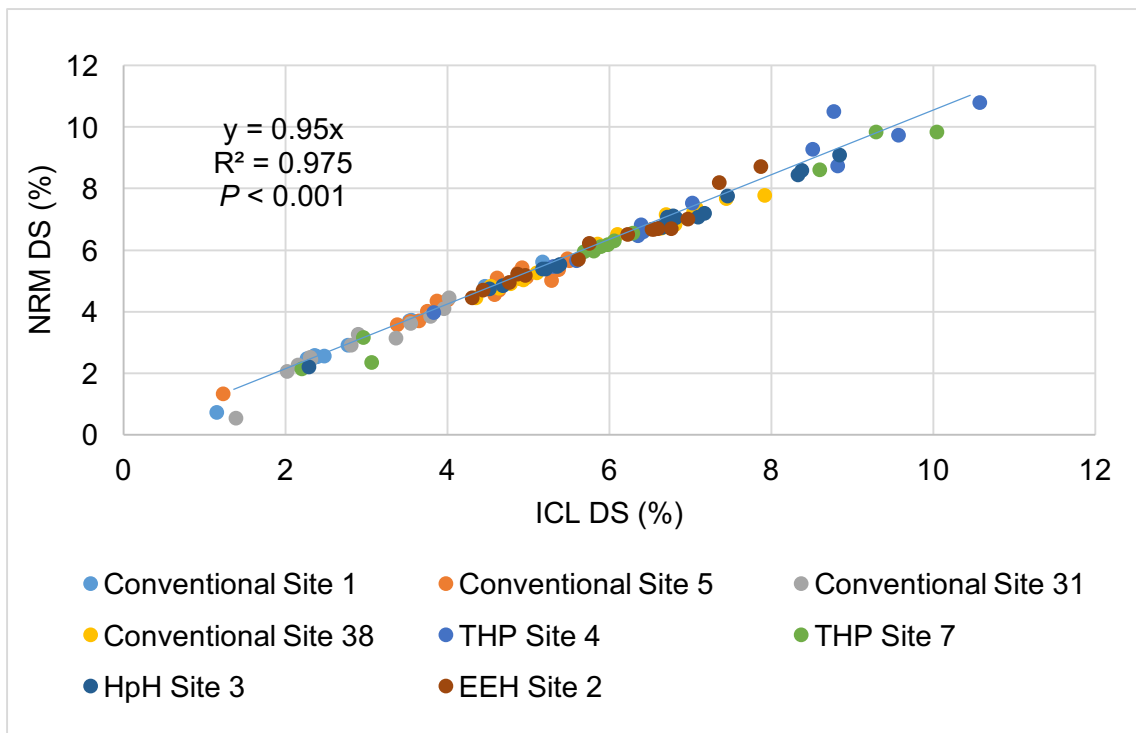


Figure 7-6 Relationship between DS measured at the external laboratory (NRM lab) and the supplementary internal measurements of DS at Imperial College London (ICL) laboratory for all sampling sites

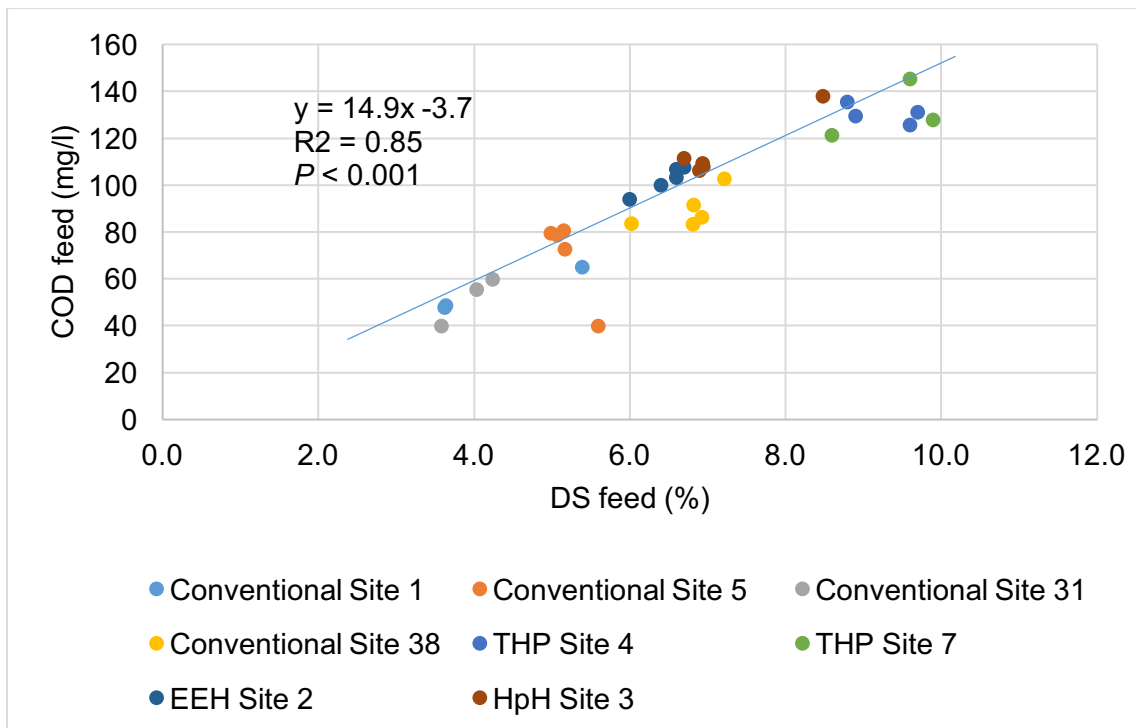


Figure 7-7 Relationship between COD content and DS of feed sludge for all sampling sites

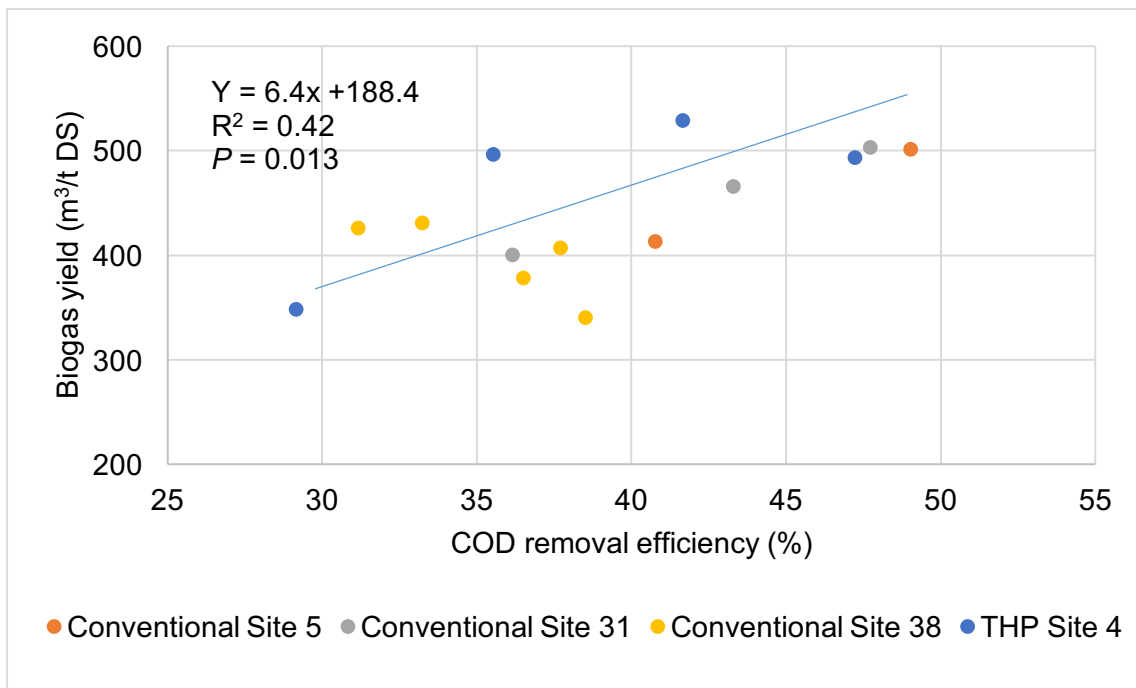


Figure 7-8 Relationship between BY and COD removal efficiency for the sampling sites with reasonable biogas recording ($\text{BY} < 700 \text{ m}^3/\text{t DS}$)

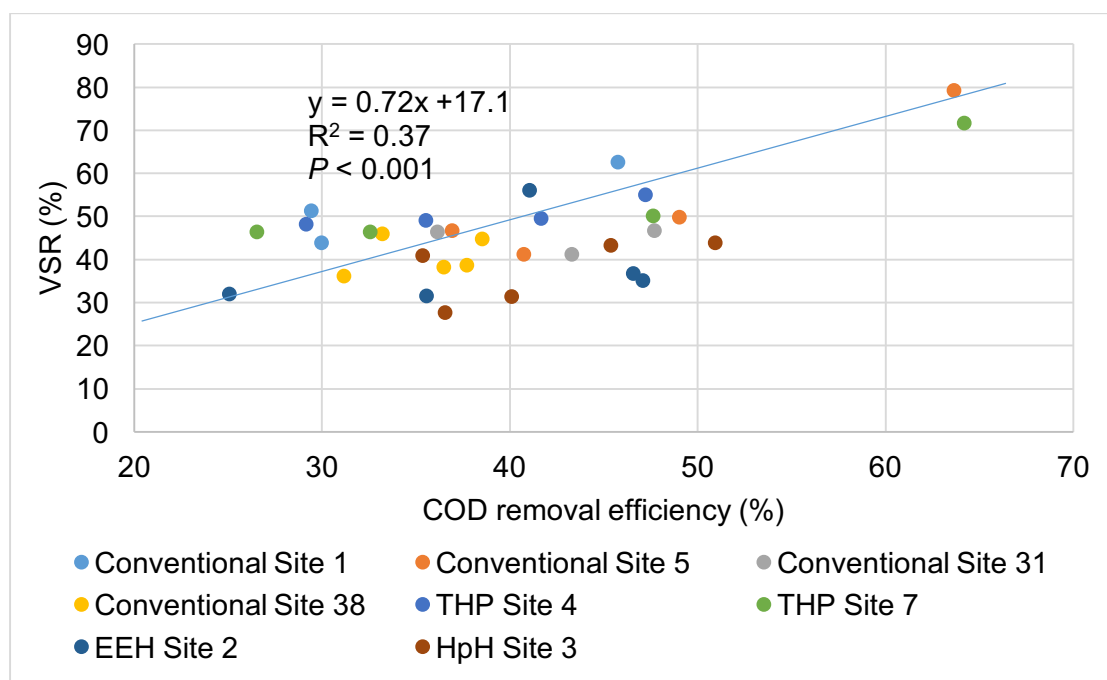


Figure 7-9 Relationship between VSR and COD removal efficiency for all sampling sites

7.7 Summary

The effects of temperature, HRT and DS feed on full-scale AD performance can be represented by a multiple regression model approach (Chapter 5). However, sites with similar operating conditions may have different BY performance, and part of the reason for this may be attributed to variation in

feed sludge composition (Weiland, 2010; Li *et al.*, 2018). The effect of sludge composition on the AD process was therefore investigated by measuring the macromolecular contents (protein, fat, carbohydrate and fibre contents) of feed and digested sludge from the eight selected MAD sites representing different levels and types of operational performance (Conventional: high, Site 1; moderate, Site 5 and 31; and low, Site 38; THP Site 4 and 7; Enzymic: EEH Site 2 and HpH Site 3). No significant relation was detected between BY and sludge feed composition. However, a compositional BY_c value was derived for each sampling event based on the substrates destroyed during AD, and the theoretical CH_4 yield and content in the biogas. A positive and highly statistically significant correlation ($P = 0.005$) was found between BY_c and the observed BY for the sites with reasonable biogas recording. Therefore, observed BY could be described by the BY_c with a reasonable degree of confidence based on the composition of energy substrates measured in sludge. This provided another approach to model calibration that was independent of the standard operational data recording. Furthermore, it also provided a BY value to evaluate the effect of VS and macromolecular composition on digester performance for advanced MAD sampling sites where there was no biogas recording (for example, THP Site 7, EEH Site 2 and HpH Site 3). A comprehensive analysis of inorganic elemental composition, including the heavy metals, such as, Cu, Zn, Pb, and Cd, and the major nutrients, including: N, P, K and Mg, were also completed, and the results were consistent with other sludge composition surveys reported within the past 10 years.

Chapter 8. Model Calibration and Testing the Impact of Process Conditions on AD Performance

8.1. Introduction

Several multiple regression models were developed to describe the relationships between the principal operational parameters on digestion performance, indicated by BY, EY and VSR (Model 18, 11 and 14, respectively). The model coefficients for the continuous operational parameters represent the relative changes in process response, based on a zero centring approach of the variables. The specific site conditions were represented by a categorical factor that was calibrated by shifting the predicted values of BY, EY or VSR to find the best fit to calculate a specific site factor in the model validation process (Chapter 6). This default calibration approach accounted for the effects of other factors that are not explained by the continuous variables in the model, and enables the operators to apply the model to evaluate the local AD performance. However, if the local data recording is not representative, the predicted values will also reflect this and will not estimate the actual performance of the process. For example, a poor description of BY was observed for Conventional Site 1 in Section 6.2, and this could be attributed to unrepresentative biogas measurement on site (in this specific case underestimation of sludge feed to the digesters was suspected as the cause (see Section 8.2.1)). Therefore, alternative calibration methods, integrating the routinely measured electricity data from CHP plant and macromolecular composition results from sludge sampling (Section 7.4), were developed in Section 8.2.1. This enables the model to be applied universally to any full-scale, stable, MAD process, by adopting one or more calibration options. In this Chapter, the influence of digestion conditions on process performance has been evaluated to demonstrate the overall effect of principal operational parameters on BY, EY and VSR within the typical, normal operating range of full-scale MAD processes.

8.2. Combined Conventional +THP-MAD Model (Model 18, Section 6.4)

8.2.1. Model calibration

The default approach to internal calibration of the model to specific site conditions, using local BY data, was unable to estimate the actual BY of the process if the site biogas recording was unrepresentative (see Section 6.2). To solve this problem, alternative calibration methods, using electricity generation and sludge composition analysis, were developed.

Electricity was generated from AD biogas through CHP plant at all sites and, therefore, the amount of electricity can correspond to the amount of biogas produced (accounting for other uses, for example, combustion in a boiler and flaring). A strong linear relationship ($R^2 = 0.90$, $P < 0.001$) was found (Figure 8-1 a) between the electricity and BY for the period 2016 to 2019 for the sites selected for model calibration assessment (Conventional: 1, 31 and 38, and THP Site 4). However, Site 1 showed a very different gas-electricity profile compared to the other sites and both gas and electricity yields were significantly above the expected AD operational range, based on the sludge loading rate to the digesters reported by the operator for this site. Poor quality of gas data recording could be suspected of causing misalignment of gas and electricity data. However, in this case, the strong relationship between biogas and electricity yield ($R^2 = 0.90$, $P < 0.001$) observed here suggested that the gas measurement was reliable. Therefore, the large biogas (and electricity) yields were attributed to other factors and, in this case, we suspect that unreliable recording of sludge feed volumes is the most likely explanation. Operational sludge feed volumes are typically influenced by different sources, including indigenous (primary and SAS) and imported sludge, therefore, it is critical to ensure that all sludge feed inputs are included in the biogas/electricity yield analysis. Site 1 was removed from the correlation of biogas and electricity yield (Figure 8-1 b), nevertheless, the relationship remained highly statistically significant ($R^2 = 0.60$, $P < 0.001$). An electricity derived BY_e can be calculated based on a conversion factor of 2.14 for electricity generation from biogas (assuming electrical conversion efficiency = 35%, 1m^3 biogas = 2.14kWh electricity; Banks, 2009). Biogas-electricity generation conversion factors can therefore be used as an alternative approach for site specific calibration of the model.

Alternatively, a sludge composition BY_c was derived for each sampling site based on the substrates destroyed during AD, and the theoretical CH_4 yield and content in the biogas (see Section 7.4). This provided another approach to model calibration that was, independent of the standard operational data recording. The results (Figure 8-2) showed that sludge composition generally underestimated the BY to some extent compared to observed, predicted and electricity derived values, but particularly so for Site 1. In this case, the BY_c reflected the AD performance for the actual volume of sludge feed recorded at the site. Therefore, where known local issues exist with the absolute measurement of gas flow or feed volumes, for instance, sludge composition can be used depending on local circumstances to calibrate the model and cross-reference the data recording. Average site biogas yields using the different calibration methods are shown in Figure 8-2.

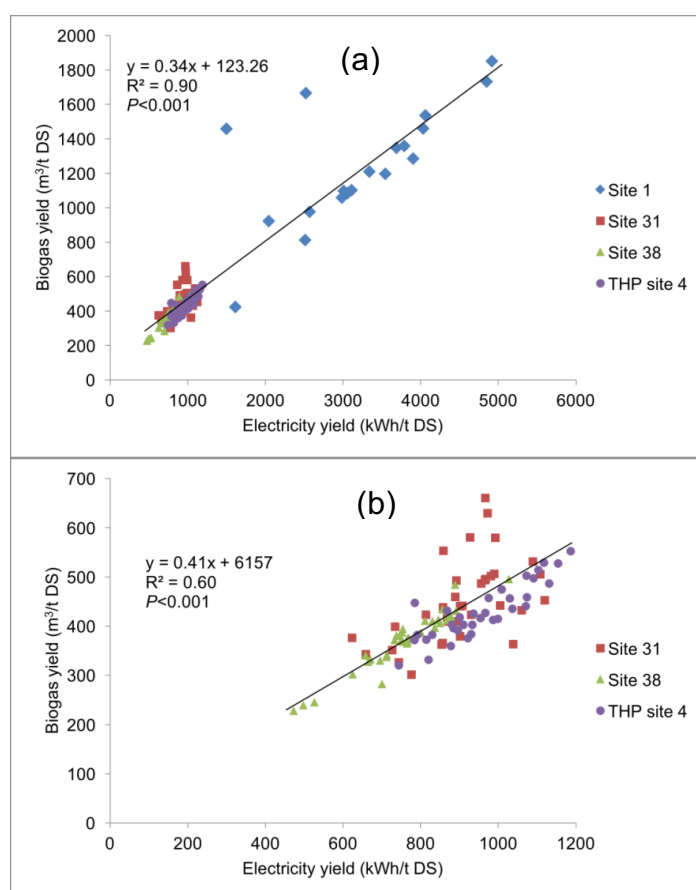


Figure 8-1 Relation between biogas yield and electricity yield for selected sampling sites (a) Conventional Site: 31, 38 and THP Site 4, and (b) Conventional Site: 31, 38 and THP Site 4

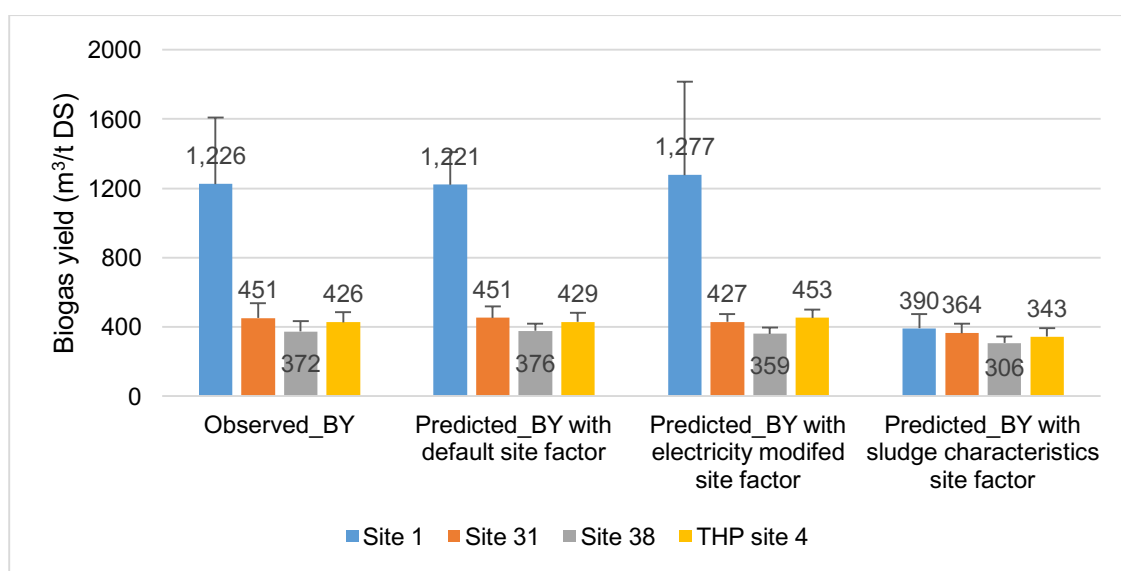


Figure 8-2 Comparison of average site biogas yield under default, electricity generation and sludge composition calibration methods for conventional Site 1, 31 38 and THP Site 4 (error bar: standard deviation)

8.2.2. Impacts of digestion conditions on process performance

The relationship between DS feed, HRT and the relative changes in BY represented by Model 18 are shown in Figure 8-3 at the average temperature recorded for conventional MAD sites, equivalent to 35.9 °C (Table 4-1). The results showed that BY decreased with increasing DS and that the magnitude of the response diminished with increasing HRT. Similarly, the effect of HRT on BY depended on the concentration of DS in the feed sludge, and increasing HRT had a stronger effect on BY at higher DS concentrations. For example, increasing HRT from 15 to 20 days raised the relative BY at 2.5% and 4.0 % DS by 28.1 m³/t DS and 36.4 m³/t DS, respectively. However, the magnitude of the response in BY to extending the HRT further from 20 to 25 days diminished at equivalent DS contents in the feed sludge to 21.8 to 28.3 m³/t DS, respectively (Table 8-1), due to the logarithmic relationship between HRT and BY. Thus, the model (Model 18) indicated that the overall increases in BY possible with 10 days more HRT were equivalent to approximately 50 and 65 m³/t DS for these DS contents in feed sludge, respectively. The relative changes in BY for THP MAD sites followed similar patterns to conventional MAD, but in this case, the operational conditions represented larger DS and longer HRT values (Table 8-1).

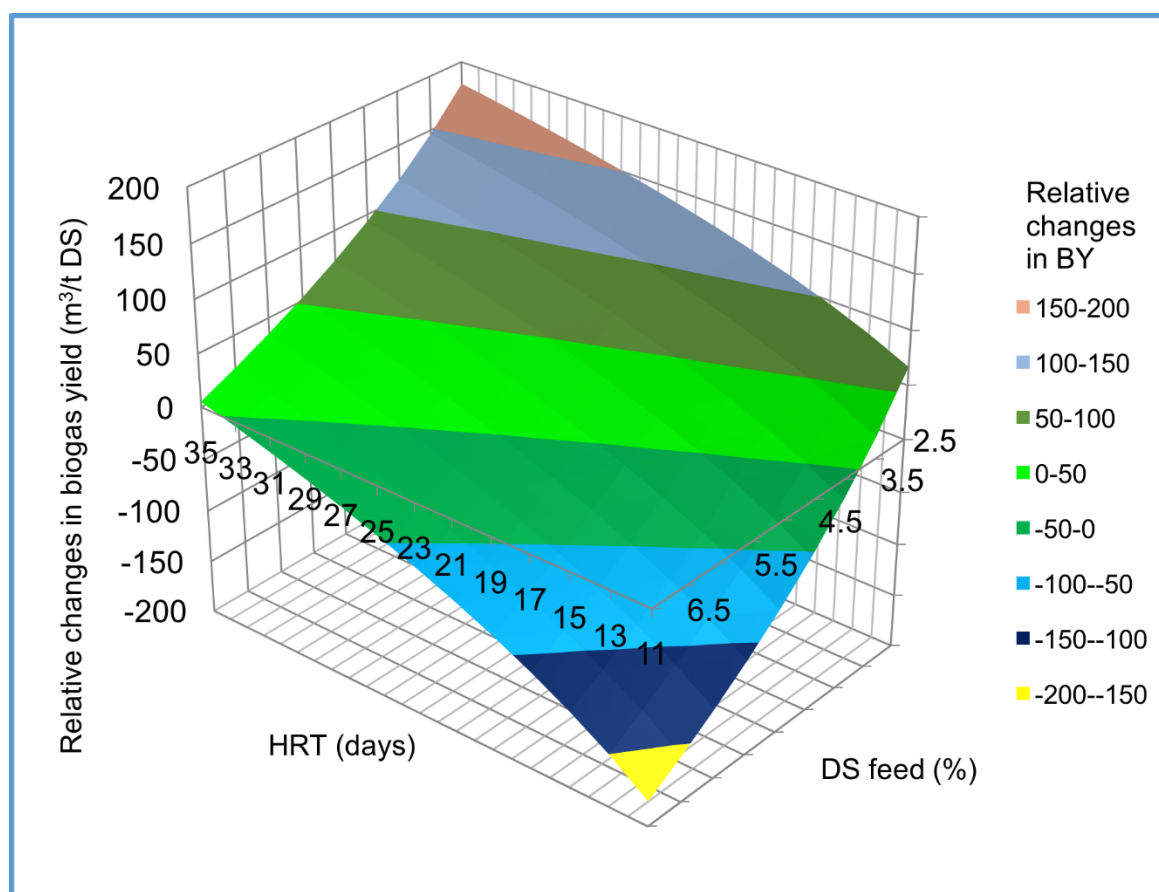


Figure 8-3 Relative changes in biogas yield in relation to HRT and DS feed at the overall mean conventional digestion temperature of 35.9 °C (Model 18) (HRT and DS data ranges limited to the 5 and 95 percentiles of monthly average operational values)

The effect of temperature and HRT on the relative changes in BY for Model 18 at the overall mean DS in feed sludge of 4.5% DS for conventional MAD (Table 4-1) is shown in Figure 8-4. The results showed that BY increased following a diminishing pattern as temperature rose. THP MAD processes operated at higher temperature and DS feed, compared to conventional sites, with mean values equivalent to 38.2 and 35.9 °C and 7.9 and 4.5%, respectively (Tables 4-1 and Appendix 4-3). However, the statistical analysis showed that the effect of temperature on BY was independent of DS, therefore, equivalent relative increases in BY with temperature may be expected, irrespective of the differences in DS feed to these processes. For example, a 3 °C lift in temperature increased BY by 23.1 m³/t DS relative to 33 °C, and by 21.2 m³/t DS relative to 36 °C (Table 8-2). For practical application, the changes of BY can be calculated by the user interface as listed in Appendix 3-4.

Table 8-1 Relative changes in biogas yield for different combinations of HRT and DS feed concentrations, reflecting conventional and THP MAD operating ranges (Model 18) (conventional range: bold values; THP range: underlined values)

DS feed (%) \ HRT changes (days)	2.5	4.0	5.0	6.5	8.0	10.0
	Relative changes in BY (m ³ /t DS)					
10 to 15	39.6	<u>51.4</u>	<u>57.0</u>	<u>63.5</u>	<u>68.7</u>	<u>74.3</u>
15 to 20	28.1	<u>36.4</u>	<u>40.4</u>	<u>45.1</u>	<u>48.8</u>	<u>52.7</u>
20 to 25	21.8	<u>28.3</u>	<u>31.3</u>	<u>35.0</u>	<u>37.8</u>	<u>40.9</u>
25 to 30	17.8	<u>23.1</u>	<u>25.6</u>	<u>28.6</u>	<u>30.9</u>	<u>33.4</u>
30 to 35	NA	19.5	21.7	24.1	26.1	28.2
35 to 40	NA	16.9	18.8	20.9	22.6	24.5

Note that bold and underlined values reflect conditions that may overlap in operational practice for conventional and THP MAD; NA, not applicable

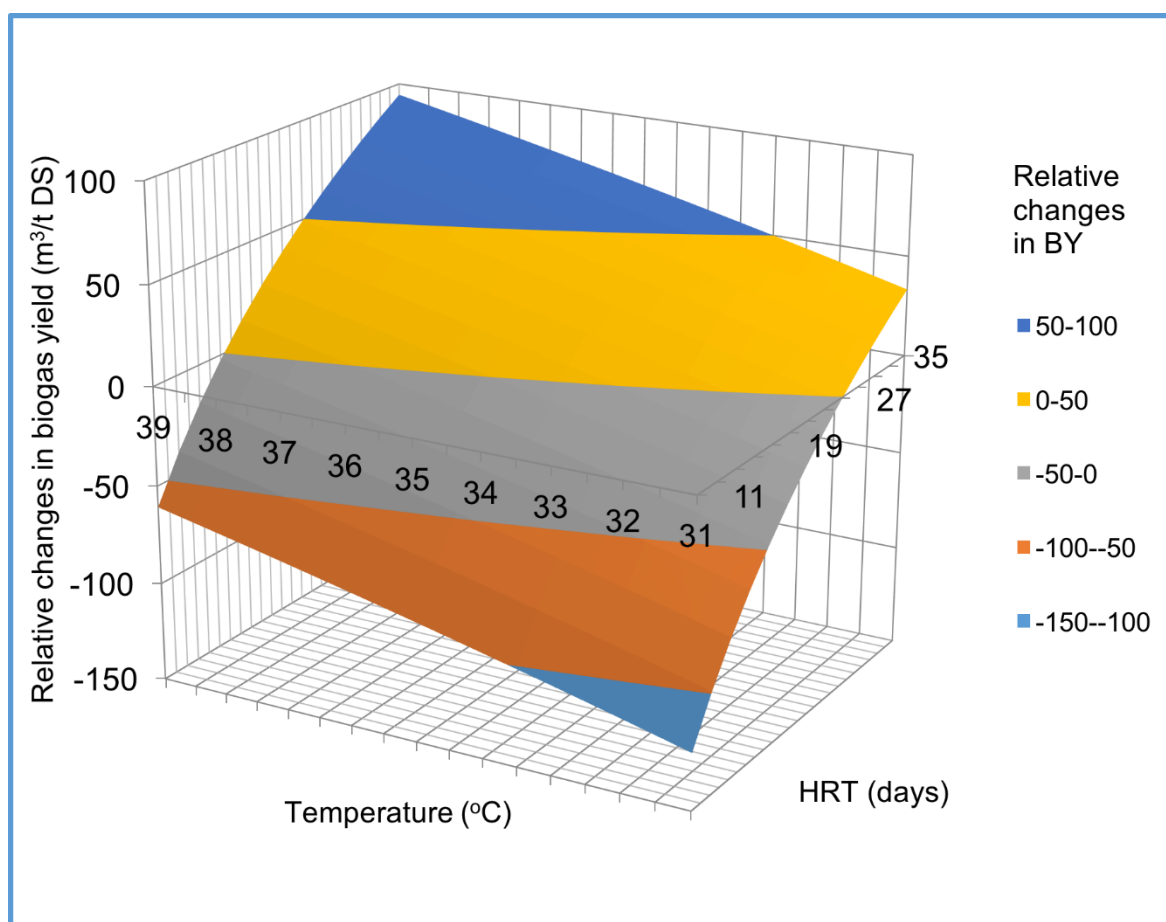


Figure 8-4 Relative changes in biogas yield in relation to HRT and temperature at the overall mean DS feed concentration for conventional digestion of 4.5 % (Model 18) (HRT and Temperature data ranges limited to the 5 and 95 percentiles of monthly average operational values)

Table 8-2 Relative changes in biogas yield for different temperature conditions, reflecting conventional and THP MAD operating ranges (Model 18)

Temperature change (°C)	30 to 33	33 to 36	36 to 39	39 to 42
Relative change in BY (m ³ /t DS)	25.3	23.1	21.2	19.7

8.3. Enzymic MAD Model (Model 11, Section 5.4)

The relationship between temperature, VFA feed and the relative changes in EY represented by Model 11 are shown in Figure 8-5 at the average DS feed concentration recorded for enzymic MAD sites, equivalent to 5.4% (Appendix 4-3). The results showed that EY increased with increasing VFA and that the magnitude of the response was greater with increasing temperature. Similarly, the effect of temperature on EY depended on the concentration of VFA in the feed sludge, and increasing temperature had a stronger effect on EY at higher VFA concentrations. For example, increasing

temperature from 35 to 37 °C raised the relative EY at 5500 and 7000 mg/l VFA by 60.4 and 80.3 kWh/t DS, respectively. However, the magnitude of the response in EY to lifting the temperature further from 37 to 39 °C diminished at equivalent VFA contents in the feed sludge to 57.2 and 76.1 kWh/t DS, respectively (Table 8-3), due to the logarithmic relationship between temperature and EY. Thus, the model (Model 11) indicated that the overall increases in EY possible with 4 °C higher temperature were equivalent to approximately 118 and 157 kWh/t DS for these VFA contents in feed sludge, respectively.

The effect of DS feed and temperature on the relative changes in EY at the overall mean VFA in feed sludge of 5500 mg/l for enzymic MAD (Model 11) is shown in Figure 8-6. The results showed that EY decreased following a diminishing pattern as DS rose. For example, a 1% rose in DS feed decreased EY by 83 kWh/t DS relative to 4.5%, and by 69 kWh/t/t DS relative to 5.5% (Table 8-4).

Table 8-3 Relative changes in electricity yield for different combinations of temperature and VFA feed concentrations, reflecting enzymic MAD operational ranges (Model 11)

VFA feed (mg/l)	3000	5500	7000	9000
Temperature changes (°C)	Relative changes of EY (kWh/t DS)			
33 to 35	11.0	64.0	85.0	107.0
35 to 37	10.4	60.4	80.3	101.1
37 to 39	9.8	57.2	76.1	95.7
39 to 41	9.3	54.4	72.3	90.9

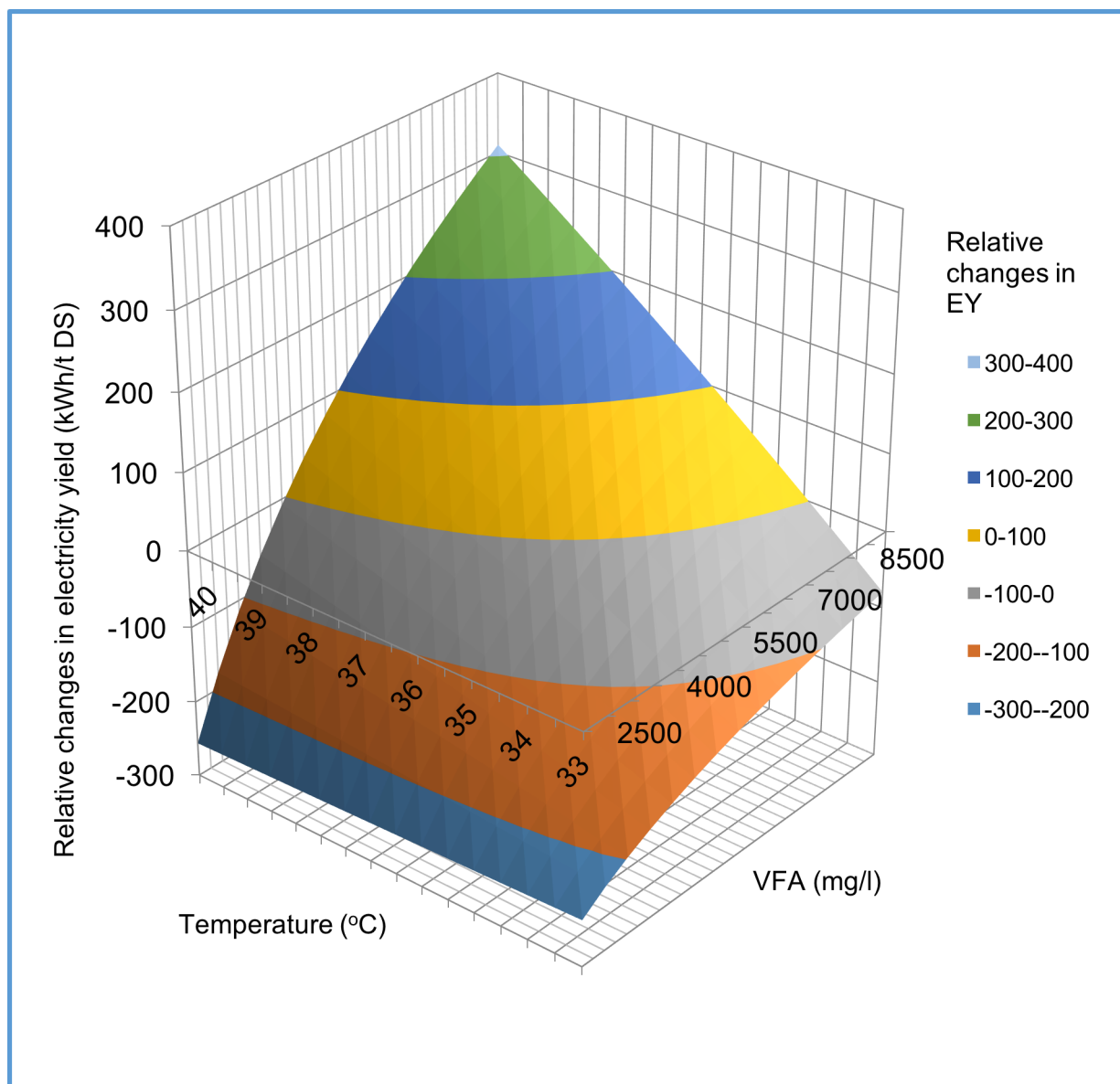


Figure 8-5 Relative changes in electricity yield in relation to temperature and VFA feed at the overall mean enzymic DS feed of 5.4% (Model 11) (temperature and VFA data ranges limited to the 5 and 95 percentiles of monthly average operational values)

Table 8-4 Relative changes in electricity yield for different DS conditions, reflecting enzymic MAD operating ranges (Model 11)

DS change (%)	3.5 to 4.5	4.5 to 5.5	5.5 to 6.5	6.5 to 7.5
Relative change in EY (kWh/t DS)	-103	-83	-69	-59

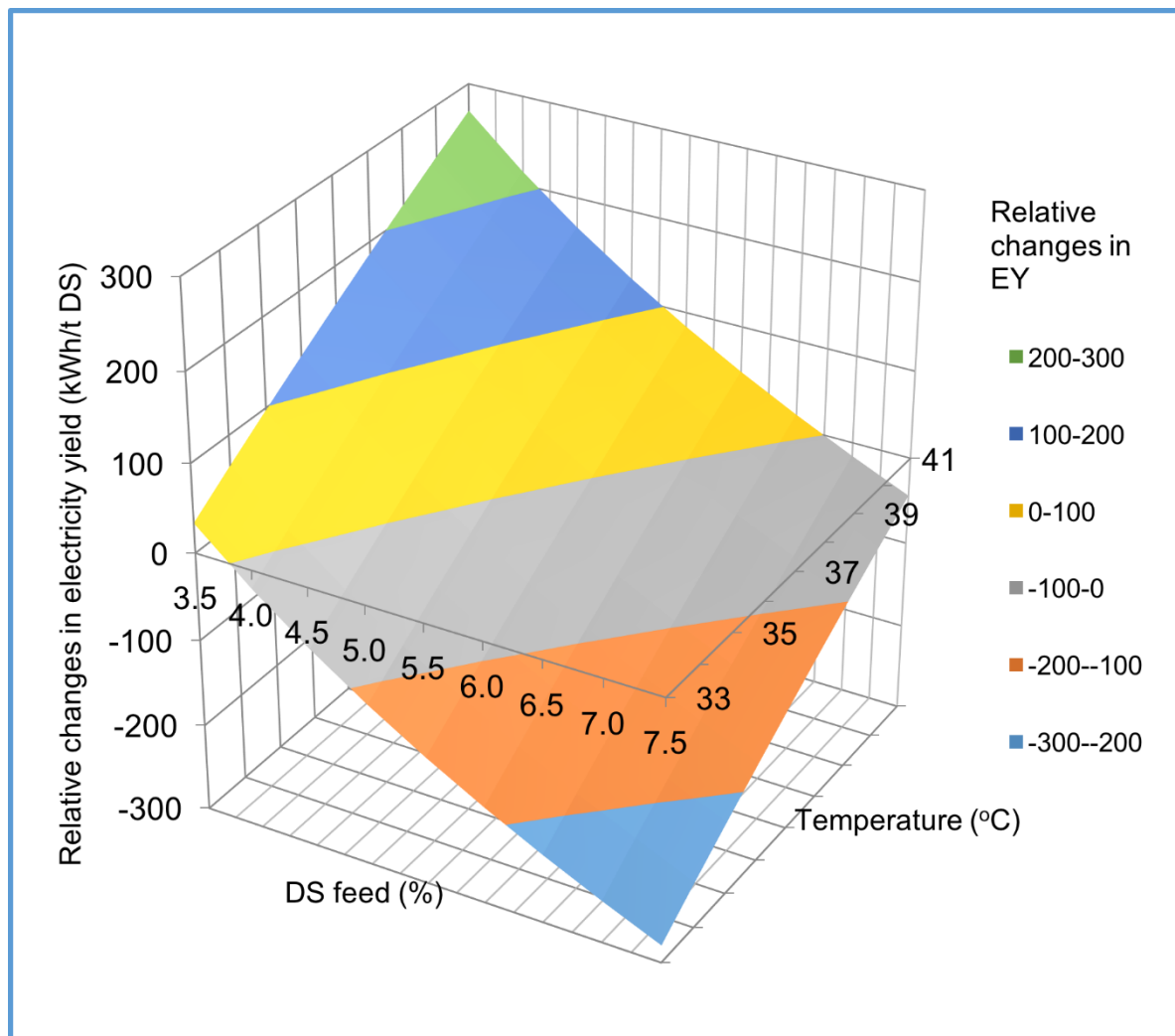


Figure 8-6 Relative changes in electricity yield in relation to temperature and DS feed at the overall mean VFA feed concentration for enzymic MAD of 5500 mg/l (Model 11) (temperature and DS feed data ranges limited to the 5 to 95 percentiles of monthly average operational values)

8.4. Conventional VSR Model (Model 14, Section 5.6)

The effect of HRT and VS feed on the relative changes in VSR for conventional MAD VSR Model 14 (Table 5-5) is shown in Figure 8-7, and the effect of temperature and HRT on the relative changes in VSR is shown in Figure 8-8. The results showed that VSR increased following a diminishing pattern as VS feed concentration and HRT rose. For example, a 2% lift in VS feed concentration increased VSR by 3.2% relative to 70% VS feed, and by 2.8% relative to 80% VS feed (Table 8-5). Similarly, a 5-day rise in HRT increased VSR by 2.4% relative to 10 days, and by 1.1% relative to 25 days (Table 8-5). Temperature had a small positive effect on VSR, and a 2 °C rise in temperature only increased VSR by 0.5%. Thus, the model (Model 14) indicated that within the 90% of operational data range, the maximum overall increases in VSR resulted from lifting VS feed, HRT and

temperature were 20.7%, 7.3% and 1.5%, respectively. This result was consistent with the largest contribution of VS feed in Model 14 (Table 5-5, Section 5.6).

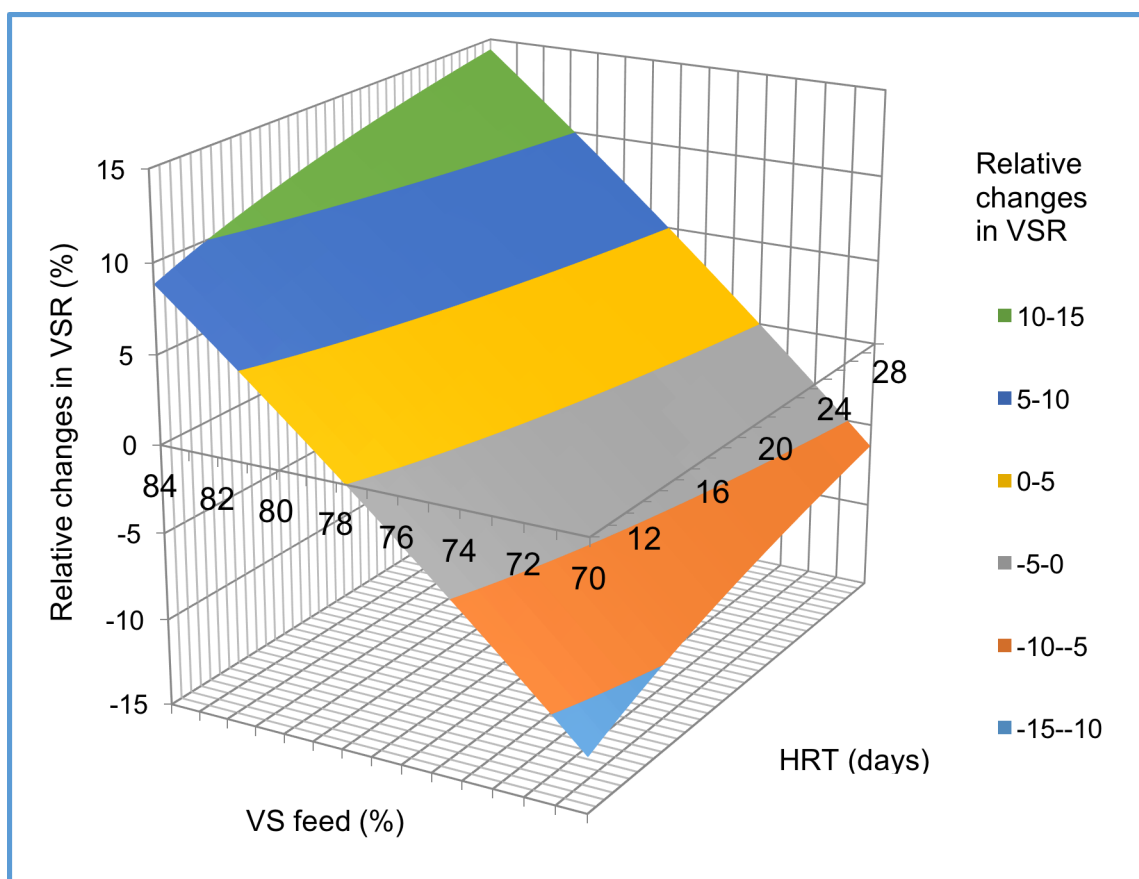


Figure 8-7 Relative changes in VSR in relation to HRT and VS feed concentrations (Model 14) (HRT and VS data ranges limited to the 5 and 95 percentiles of monthly average operational values)

Table 8-5 Relative changes in VSR for different VS feed and HRT conditions, reflecting conventional MAD operating ranges (Model 14)

VS feed change (%)	Relative change in VSR (%)	HRT change (days)	Relative change in VSR (%)	Temperature change (°C)	Relative change in VSR (%)
70 to 72	3.2	10 to 15	2.4	33 to 35	0.52
72 to 74	3.1	15 to 20	1.7	35 to 37	0.49
74 to 76	3.0	20 to 25	1.3	37 to 39	0.47
76 to 78	2.9	25 to 30	1.1	NA	NA
78 to 80	2.9	30 to 35	0.9	NA	NA
80 to 82	2.8	NA	NA	NA	NA
82 to 84	2.7	NA	NA	NA	NA
Overall changes	20.6		7.4		1.5

NA, not applicable

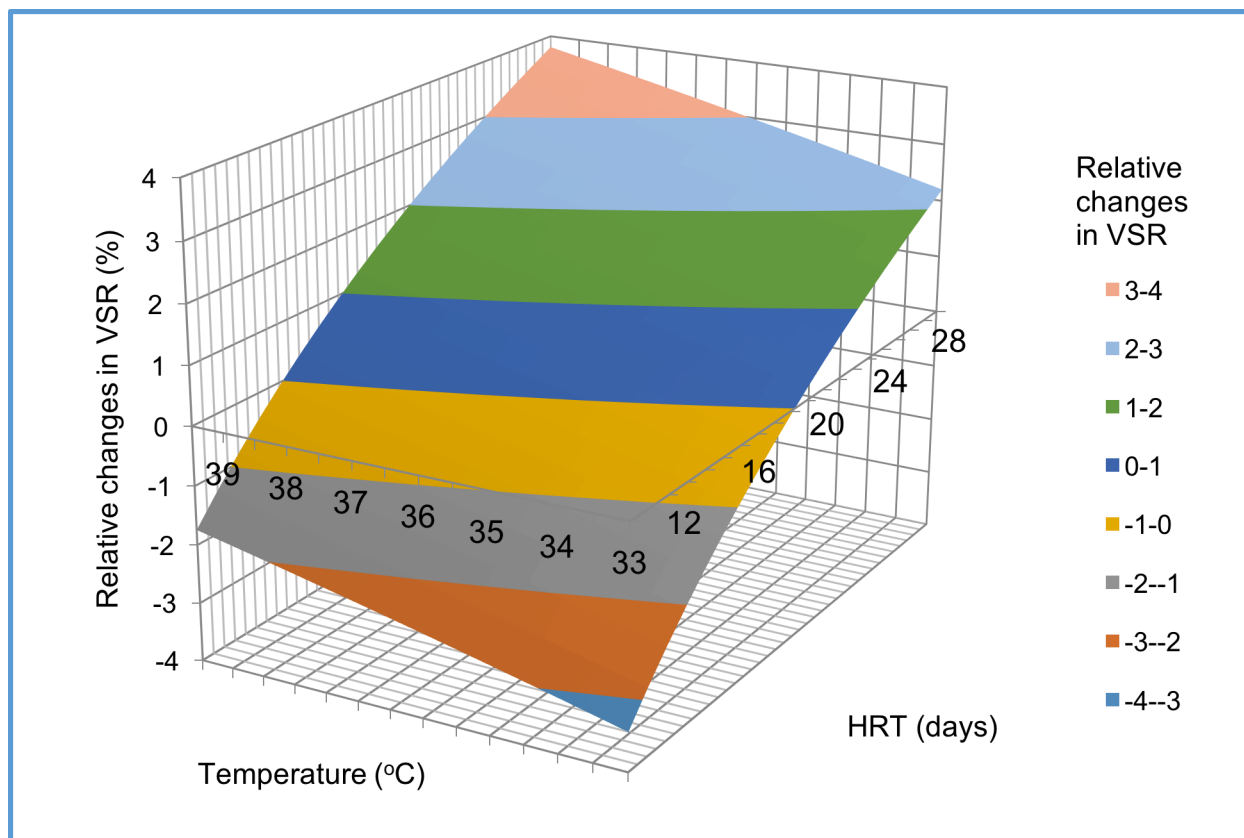


Figure 8-8 Relative changes in VSR in relation to temperature and HRT (Model 14) (Temperature and HRT data ranges limited to the 5 and 95 percentiles of monthly average operational values)

8.5 Summary

Chapter 8 tested the impact of process conditions on AD performance and provided examples of the effects of different model calibration methods on the predicted BY for the selected sites. The results showed that the magnitude of the effects of operational parameters on AD performance varied depending upon the digestion conditions. For example, extending the HRT by 5 days gave a relative increase in BY in the range of 17.8 to 63.5 m³/t DS under typical conventional MAD operational conditions (Table 8-1, Model 18). Similarly, a 3 °C rise in temperature gave a relative increase in BY of 19.7 to 25.3 m³/t DS within the operating digestion temperature range of 30 and 42 °C (Table 8-2, Model 18).

Three model calibration methods were applied using the local BY, electricity generation and sludge composition data to calculate the specific site factor to represent the effects of unmeasured parameters on digestion performance. The default, internal calibration method using local, site BY data is generally recommended for routine model calibration, however, it relies heavily on the availability of reliable gas flow measurements. Two additional calibration methods, using EY or sludge composition analysis are also proposed, and can be useful when local data issues exist with

the absolute measurement of gas flow or sludge feed volume, for instance. Electricity yield is an effective method to cross-check contemporary and historical gas data, and for model calibration, as electricity recording is reliable and routinely available at most AD sites operating CHP. Measuring the destruction of major organic substrate groups in sludge by AD requires additional sample collection and analysis and is not routinely conducted at WWTP. The results presented here indicate the value of this approach to characterise the potential performance of sludge AD systems; it can provide an independent approach to industry recorded operational data for model calibration and can be used, for instance, when there are multiple data recording issues at a particular site. For example, the BY_e was consistent with the observed and predicted BY results for Site 1, but both values were all larger than would appear reasonable for conventional MAD of sewage sludge (Bachmann *et al.* 2015) based on the DS loading rates to the digesters reported by the operator for this site. Therefore, unreliable recording of sludge feed volume to the MAD process was suspected as the cause of the unrepresentative biogas and electricity yield values obtained in this case. However, a BY_c based on sludge composition analysis and the recorded sludge volume at Site 1 was consistent with the typical performance of a conventional MAD process for sludge treatment.

Chapter 9. General Discussion

9.1 Introduction

The multiple regression analysis examined the statistical significance and behaviour of combined and interactive effects of multiple process control parameters on digester performance. In general, these models provided effective description of digester performance for most of the selected validation sites (Chapter 6). Different parameters were involved in model development for different MAD processes, and the differences and similarities between these for conventional, THP and enzymic MAD models on digester performance are discussed in Section 9.2. In addition to the routinely measured parameters, other factors, such as sludge composition, digester mixing and data quality could also influence the accuracy of the empirical statistical models, and these influences are discussed in Section 9.3.

The combined conventional-THP and enzymic MAD models (Model 18 and 11) can be applied universally to any full-scale MAD process, by adopting one or more calibration options to maximum BY or EY as described in Section 8.2.1. However, the highest BY or EY does not necessarily mean an optimised performance for full-scale AD as this may not correspond to the maximum biogas volume or the net energy output. The optimised strategies for maximum net energy at full-scale AD performance under different MAD processes are discussed in Section 9.4.

9.2 Comparison of Combined Conventional-THP and Enzymic MAD Model

Three multiple regression models (Model 4, 6 and 11) were developed to describe the performance of conventional, THP and enzymic MAD using the routinely measured parameters available at operational WWTPs (Chapter 5). The performance of THP MAD can be described by the same process coefficients of temperature, HRT and DS generated from conventional MAD model, and hence a combined conventional-THP MAD model (Model 18) was developed in Section 6.4, using the data from both conventional and THP MAD sites. Unlike the enzymic MAD, which can be considered as a multi-stage AD system, where the hydrolysis and acidogenesis stages are separated from the following acetogenesis and methanogenesis steps (Ariunbaatar *et al.*, 2014) (Section 2.3.8), THP pretreatment is a stand-alone system prior to conventional MAD. THP pretreatment breaks down cellular material and long chain organic fatty acids in sludge, which increases the soluble organic content and sludge digestibility (Gurieff *et al.*, 2011; Xue *et al.* 2015), and hence it may accelerate the rate of biogas production and reduce HRT, although the overall BY is generally similar compared to conventional MAD. For example, Xue *et al.* (2015) observed a 70% higher total biogas output from thermally hydrolysed sludge compared to sludge without

pretreatment, in a batch MAD (37 °C) process with 16.5% DS feed concentration and HRT equivalent to 10 days. After 25 days HRT, however, there was no discernible difference in the total gas production between raw and thermal hydrolysed sludge. Wilson *et al.* (2008) concluded that digestion performance with THP at 15 days HRT was equivalent to that without at 20 days. This behaviour is consistent with the combined conventional-THP model (Model 18) which showed the magnitude of the AD response to HRT was greater under higher DS feed concentrations (Figure 8-3). THP sites were operated at higher DS feed compared to conventional MAD (Table 8-1), which is possible due to the rheology changes in thermal hydrolysed sludge allowing higher solids input to the MAD process (Barber, 2016). Consequently, the magnitude of the response in BY to changes in the HRT was greater for THP MAD processes compared to conventional MAD operating at lower DS feed concentrations over an equivalent range of HRT. Hence, the model results confirmed that the THP MAD process can achieve the maximum BY with shorter HRT at a given sludge composition compared to conventional MAD.

The patterns in enzymic MAD performance were slightly different from conventional and THP MAD processes. The enzymic MAD is a multiple-stage AD system (Ariunbaatar *et al.*, 2014), and the resulting increased concentration of VFAs in the feed sludge to the methanogenesis stage of the process was detected as an additional significant predictor in Model 11 for digester performance, indicated by EY. The EY increased with VFA concentration, however, the significant interaction between VFA and temperature resulted in an increasing positive effect on EY at higher temperature (Figure 8-5). As discussed in Section 4.5, high VFA concentrations in the feed sludge indicate an efficient pretreatment process, and hence improved AD performance may be expected (Trisakti *et al.*, 2017). As was the case in the combined conventional-THP MAD model (Model 18), temperature had an approximately linear positive effect on digestion performance in the enzymic model (Model 11). The relationship between temperature and the relative changes in EY was converted into an equivalent BY value using a conversion factor of 2.1 as discussed in Section 8.2 (Banks, 2009) and summarised in Table 9-1. The effect of temperature on EY in the enzymic MAD process was similar to the combined conventional-THP MAD model when the VFA feed concentration was approximately 4000 mg/l (Table 9-1 and Figure 9-1); 90% of VFA feed values were between 2187 to 9175 mg/l, therefore, this represented towards the lower range of VFA feed concentrations in sludge (Table 6-2, Section 6.8). However, the response to temperature for the enzymic-MAD process increased to a greater extent in comparison to the conventional-THP model when the VFA concentration rose above 4000 mg/l.

The effect of temperature on BY represented in Model 18 was consistent with the reported improvement in BY observed with relatively small increases in temperature within the mesophilic range (Boušková *et al.*, 2005; Kim and Lee, 2016; Nielsen *et al.*, 2017). For example, Nielsen *et al.* (2017) showed that increasing digestion temperature from 35 to 39 °C in a lab-scale batch reactor raised the specific CH₄ yield by 20 ml CH₄ g/VS, which is equivalent to an increase in BY of

approximately 25 m³/t DS (at 60% CH₄ and 75% VS content); this is consistent with the increase in BY observed here for a similar temperature range, based on Model 18 (Table 6-1) for the full-scale combined conventional-THP MAD process (Figure 8-4; Table 8-2).

The combined conventional-THP MAD model (Model 18) showed that BY increased markedly with HRT below 20 days, but the magnitude of the effect of HRT decreased progressively particularly as HRT extended beyond 20 days (Figure 5-1 and 8-3). Alepu *et al.* (2016) reported a similar diminishing effect of HRT on CH₄ yield for sewage sludge in a lab-scale mesophilic anaerobic digester at 10, 20 and 30 days HRT, which gave corresponding CH₄ yields of 10, 110 and 160 ml/g COD, respectively. By contrast, for enzymic MAD, HRT had no significant effect on EY and was therefore not represented in the enzymic model (Model 11). The lack of an apparent response to HRT observed for enzymic MAD sites could be attributed to the higher operating average HRT (23 days) apparent for this process in practice compared to conventional and THP MAD sites (21 days). Therefore, HRT was already sufficient to achieve the maximum EY and there was insufficient variation in the operational HRT data to detect a significant effect of this important parameter on the performance of enzymic digestion processes. Consequently, there was no significant interaction between DS and HRT detected in the enzymic model (Model 11). The effect of DS feed on EY was converted to BY (using 2.1 conversion factor) to allow comparison with the conventional-THP MAD model (Model 18). This showed that the BY response to increasing DS in the enzymic model (Model 11) was equivalent to the effect of DS in the combined conventional-THP MAD model (Model 18) at a HRT of 25 days (Table 9-2; Figure 9-2). Thus, enzymic-MAD can be considered as a special case that was also represented by the combined conventional-THP model (Model 18), with extended HRT (23 days was average for enzymic processes).

Table 9-1 Relative changes in electricity yield and its equivalent biogas yield for different temperature conditions (Model 11 and 18)

Temperature change (°C)	30 to 33	33 to 36	36 to 39
Relative change in EY (kWh/t DS) (VFA = 4000 mg/l; Model 11)	53	49	46
Equivalent relative change in BY (kWh/t DS) (Model 11)	25	23	22
Relative change in BY (m ³ /t DS) (Model 18)	25	23	21

Table 9-2 Relative changes in electricity yield and its equivalent biogas yield for different DS conditions (Model 11 and 18)

DS change (%)	3.5 to 4.5	4.5 to 5.5	5.5 to 6.5	6.5 to 7.5
Relative change in EY (kWh/t DS) (Model 11)	-103	-83	-69	-59
Equivalent relative change in BY (m ³ /t DS) (Model 11)	-49	-40	-33	-28
Relative change in BY (m ³ /t DS) (HRT =25 days; Model 18)	-51	-41	-34	-29

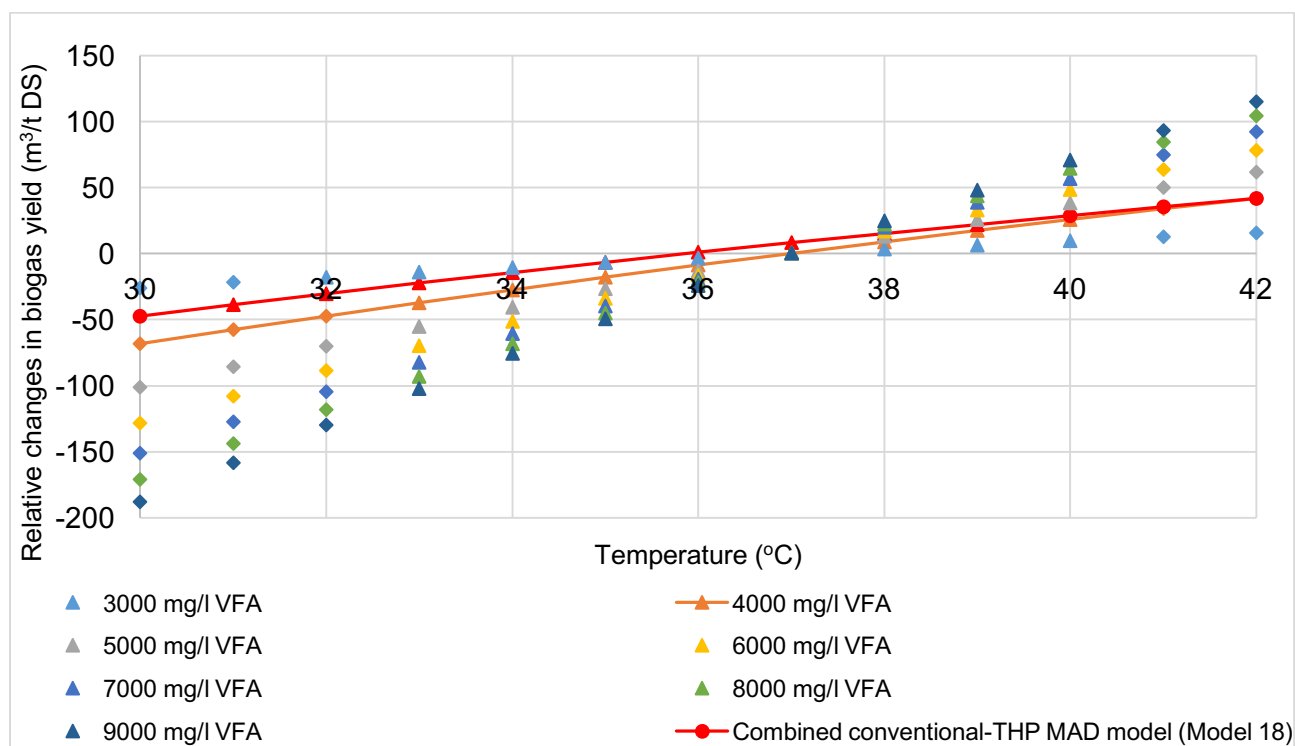


Figure 9-1 Effect of temperature on the relative change in biogas yield at different VFA feed concentrations for enzymic MAD model (Model 11) and combined conventional-THP MAD model (Model 18) without VFA interaction. Triangle symbols: 90% data range

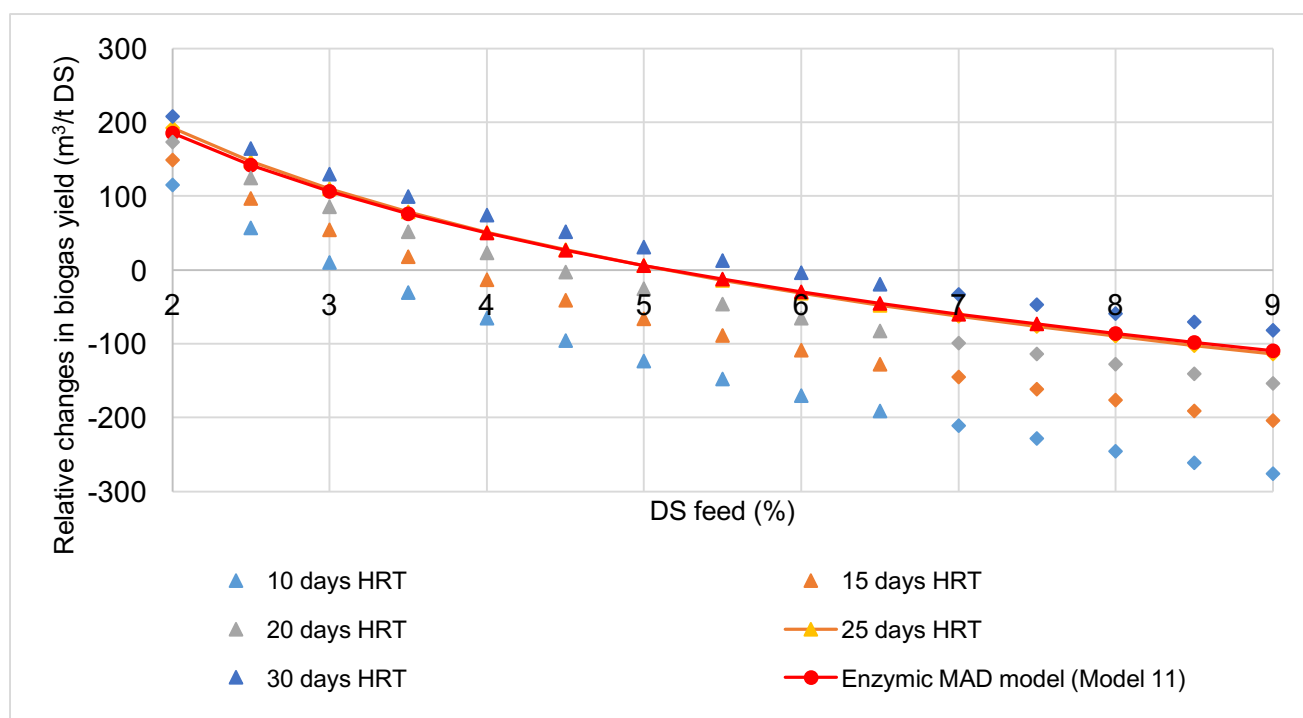


Figure 9-2 Effect of DS feed on the relative change in biogas yield at different HRT for combined conventional-THP MAD model (Model 18) and enzymic MAD model (Model 11) without HRT interaction. Triangle symbols: 90% data range

9.3 Other Factors Affecting Digestion Performance

9.3.1 Physical, chemical and biological factors

In general, BY was effectively predicted for conventional and THP MAD data as presented in Section 6.2 and 6.4. However, the prediction quality may vary between different sites and time periods (Figure 6-1). This is because the model (Model 18) uses 3 operationally monitored parameters as predictors and more than 40% of the total variation in BY was attributed to a categorical factor explaining the overall effects of other site conditions, not directly considered in the model. Therefore, sites with unstable operating conditions, for example, where input sludge characteristics change frequently, will require regular calibration of the model to adjust the site factor to take these changes into consideration. The most reliable predictions of BY should be obtained for sites with characteristically stable operating conditions and where the site factor is therefore expected to be consistent and capture the effects of other relevant management factors (for example, stable sludge composition, mixing efficiency, primary sludge ratio) that are not explained by the continuous variables in the model.

Sludge composition and primary:SAS ratio define the theoretical CH₄ yield and biodegradability of the feed sludge. Different substrates have varying CH₄ potentials and a significant correlation between the compositional derived BY_c based on the proportions of substrates destroyed during AD, and the observed BY was detected using sludge sampling data from 6 sampling events at 8 selected

sites (Section 7.4). The compositional derived BY_c from sludge sampling results provided an effective alternative to the default model calibration approach (based on local site BY data), which was independent of the standard operational data recording (see Section 8.2.1). This supplementary analysis is recommended to be routinely measured at full-scale sites to give operators a benchmark of theoretical performance and identify the potential recoding errors at local AD sites.

Under optimum digestion conditions, the typical CH_4 production for SAS is 190 - 240 Nm^3/t VS, which is much smaller compared to 315 - 400 Nm^3/t VS for primary sludge, under equivalent conditions (Bachmann *et al.* 2015). The BY of SAS is less than for primary sludge due to its biological origin and lower availability to the anaerobic biomass, because of the inherent resistance and EPS protection to lysis of living cells, and the large dimension of the flocs (Bolzonella *et al.*, 2005; Batstone and Jensen, 2011; Mottet *et al.*, 2010). However, no statistically significant correlation was detected between primary:SAS ratio and BY. This was explained because sludge age and primary:SAS ratio have opposing effects on BY, and SAS production increases with reducing sludge age at full-scale MAD sites. Therefore, sludge age was positively correlated with primary:SAS ratio (Figure 5-11), and their effects on BY tend to offset each other when modelling full-scale operational AD process data.

Digester mixing is also an important factor defining AD performance, but is difficult to measure and quantify at full-scale, and models of the AD process generally assume a perfectly mixed system (Kythreotou *et al.*, 2014). The effect of mixing is linked strongly to the DS in the feed sludge, as discussed in Section 4.1, and poorer mixing efficiency at high solids concentrations has been confirmed using tracer tests (Kamarád *et al.*, 2013). Effective mixing of sludge digesters is necessary to provide a uniform temperature and substrate concentration. Therefore, maximising AD performance can be achieved only by the simultaneous optimisation of multiple interacting parameters (Ward *et al.*, 2008). The approach followed here was to combine and capture all such 'known-unknown' variables within the categorical site factor of the MAD process models.

However, this approach was only partially successful, with the continuous and categorical variables together explaining approximately 60 % of the total variation in BY in the AD data (Model 18). Therefore, other major sources of variation are still to be identified and explored as a considerable amount of uncertainty in predicting AD performance and BY remains. For example, the microbial community involved in AD is very complex, however, it is well known that a stable population with the correct balance of the major groups responsible for the various stages of organic matter conversion by AD is essential (Narihiro *et al.*, 2015). This requires knowledge of the AD ecosystem to develop a fundamental understanding of how microbial community dynamics, interactions and functionality influence digester efficiency and stability (Vanwonterghem *et al.*, 2014). There have been several attempts to define the core AD microbiome, to identify the critical population responsible for the AD process, but only 28.0% to 36.4% of the total 16S rRNA gene sequences constituting the core population involved in digestion have been reported (Riviere *et al.*, 2009; Lee

et al., 2012). Furthermore, Mei *et al.* (2017) examined 90 full-scale digesters from 5 countries and concluded that AD microbiomes were influenced by the operating conditions (for example, pretreatment, temperature range, and salinity) and could be classified into eight clusters on this basis. Therefore, greater fundamental insight to the AD process may be possible from a combination of engineering and further meta-omics analysis.

9.3.2 Data quality

Besides the complex physical, chemical and biological properties discussed above, the accuracy of empirical statistical models is also strongly dependent on the quality of the datasets used to derive the model (Section 6.8). Data quality problems are a common issue in many industries. Poor data quality can reduce business productively, revenues, and lead to misguided business decision (Gao *et al.*, 2016). It has been reported in Section 6.8 that the overall model accuracy, indicated by adjusted R^2 , is effectively influenced by uncontrolled variability in the data, represented by randomly generated values in the original datasets. As may be expected, a significant decrease of prediction accuracy was also observed at individual sites with unrepresentative data measurement (Figure 6-1). Therefore, it is essential to increase the awareness and understanding of data quality, and hence maintain good data recording at full-scale sites. Based on the data collection, cleaning, and analysis experiences from 97 full-scale AD sites, four data quality criteria, specific for AD management can be identified (Gao *et al.*, 2016; Khushali, 2018):

- **Data accuracy**, refers to the agreement of results of observations to the true values or values accepted as being true (Gao *et al.*, 2016). At WWTPs, the data accuracy issue can be identified by comparing the measured values with their typical ranges from the published literature describing similar treatment process. Furthermore, the observed digester performance, in terms of BY, can be compared with electricity production or sludge compositional data to identify the potential measurement issues as shown in Section 8.2.1. Finally, a regular calibration of gas-flow and solids meters could also improve the data accuracy.
- **Data completeness**, is a quantitative measure to evaluate how many valid analytical data were obtained (Gao *et al.*, 2016). For example, a complete dataset for combined conventional-THP MAD model (Model 18) should include temperature, HRT, DS feed and BY data, and therefore, the amount of valid analytical data is defined by the parameter with lowest recording frequency. Temperature and feed volume data (used to calculate HRT) were usually recorded daily at most of the conventional and THP MAD sites, and therefore, more attention needs to be placed on the frequency DS feed recording, to ensure it captures the variation in DS concentrations at local AD sites.

- **Data consistency**, refers to data type and format and is defined as the absence of difference when comparing two or more representations of the same thing (Gao *et al.*, 2016; Khushali, 2018). For full-scale AD sites, data were collected and reported differently within and between companies. A consistent data format in a centralised data management system would largely improve analysis efficiency leading to improved model descriptions of the process.
- **Data accessibility**, indicates the ease of access to data by users (Khushali, 2018). There is no centralised dataset available at operational WWTPs, and an extensive amount of effort was necessary in this project to collect the data both within and between the different companies. Therefore, better data accessibility provided by a centralised dataset with a real-time access could benefit both future model development and operational management.

9.4 Optimisation Strategies

9.4.1 Combined conventional-THP MAD model (Model 18, Section 6.4)

Biogas yield is a widely used performance indicator to evaluate AD performance and, the research on AD optimisation has a tendency to focus on maximising BY or CH₄ yield by evaluating individual operational parameters (Nixon, 2016). For example, Dokulilová *et al.* (2018) showed a positive effect of raising the temperature of lab-scale digesters operated at 20 days HRT, and suggested the optimum temperature to achieve the maximum BY from sewage sludge AD was in the upper mesophilic temperature range (42 °C). Alepu *et al.* (2016) reported that the CH₄ yield of a lab-scale mesophilic anaerobic digester improved as HRT increased from 10 to 20 and 30 days, and recommended 30 days HRT as the optimum design criteria for sewage sludge AD to achieve maximum CH₄ yield. However, digester performance is controlled by the combined and interactive effects of multiple process parameters as indicated by the model generated based on full-scale MAD data (for example, Model 18). Therefore, selecting appropriate and corresponding HRT, DS feed and digestion temperature conditions is necessary to maximise the BY. Moreover, the highest BY does not necessarily correspond to the optimum performance of full-scale AD in terms of the overall maximum biogas volume. For example, if it is assumed that a 2000 m³ digester (operated at 20 days HRT and 5% DS with an optimum BY of 400 m³/t DS) produces 2000 m³ biogas per day, the model outputs and relative changes in BY presented in Table 8-1 (Section 8.2.2) show that increasing the daily feed volume from 100 m³/day to 133.3 m³/day would reduce the HRT from 20 to 15 days and BY from 400 to 358 m³/t DS, respectively. However, the total sludge feed would increase from 5.0 to 6.7 t DS/day. Therefore, despite a reduction in HRT and BY, the daily biogas flow would increase from 2000 to 2387 m³, equivalent to 19.4 %. Therefore, balancing the HRT, DS feed and total throughput of sludge solids is important to optimise the overall energy balance of the AD process, and the net biogas output due to a change in HRT can be calculated using Equation 9.1:

Net daily biogas gas (m³/day) = Biogas volume₁ – Biogas volume₂

$$\begin{aligned}
 &= \frac{DS}{100} \times \left[\frac{\text{Digester volume}}{HRT_1} \times BY - \frac{\text{Digester volume}}{HRT_2} \times (BY + x) \right] \\
 &= \frac{DS}{100} \times \text{Digester volume} \times \left[\frac{BY \times HRT_2 - HRT_1 \times (BY + x)}{HRT_1 \times HRT_2} \right] \quad (9.1)
 \end{aligned}$$

Where:

Biogas volume₁ (m³/day) = the volume of biogas produced when HRT is equal to HRT₁

Biogas volume₂ (m³/day) = the volume of biogas produced when HRT is equal to HRT₂

BY (m³/t DS) = the biogas yield when HRT is equal to HRT₁

x (m³/t DS) = the relative change of biogas yield when HRT is changed from HRT₁ to HRT₂

To test the net effect of increasing HRT and show if this produces a positive net biogas flow, the result of BY*HRT₂ – HRT₁*(BY+x) must be negative. To make it more practical to be applied at full-scale site by operators, the net biogas production and net energy production by changing the digestion conditions can be automatically calculated by the user-interface developed based on the model, and the screenshot of the above example was shown in Appendix 3-4.

Raising digestion temperature increases BY, but requires additional energy input. Increasing the temperature of 1 t of wet sludge by 1°C requires 4.18 kJ/kg = 1.16 kWh energy (assuming sludge has the same specific heat capacity as water), and the energy in 1m³ biogas (60% CH₄ content) is equivalent to 22 MJ = 6.1 kWh (Banks, 2009). Table 9-3 shows the energy required to heat 1 t of wet sludge to increase the digestion temperature by 2 °C, and net energy generated from biogas produced from 1 t of sludge at DS concentrations in the feed equivalent to: 2.7%, 4.5% and 7.9%, respectively. A sludge DS of 2.7% was selected to illustrate the effects of a lower range value on the process energy balance, as this represented the lower 5% percentile of monthly average operational DS data for conventional MAD sites. For this example, the results showed a negative net energy balance for temperatures higher than 39 °C (note that the heating efficiency of the system is not considered). Thus, at low DS concentrations in the feed sludge, there is a greater energy demand to heat larger volumes of water and the calorific output is reduced per wet t of sludge treated, compared to digesting sludge with larger DS contents. Therefore, for conventional MAD operated with low feed DS (< 2.7%), it is recommended that the temperature is maintained in the low to medium mesophilic temperature range (< 39 °C) to improve the energy balance of the system. This is consistent with the current operational strategies at conventional MAD sites, since 95% of temperature data was below 39 °C (Figure 5-1). The model results showed that a minimum DS of 3.0% was required to avoid a net negative energy balance for process operation above 39 °C. However, it is emphasised that these conditions only meet the heating demands of the system and do not take other energy consumption for process operation into consideration. Nielsen *et al.* (2017)

made a similar observation to the results presented here and showed that the additional energy required to raise the temperature from 35 to 42 °C was balanced by the extra quantity of gas produced for a digester supplied with raw sludge at 4% DS with 80% VS. Sites operated at higher DS feed, which included all THP processes, can be heated to the upper mesophilic temperature range (> 39 °C). Indeed, the model showed that a 2°C lift in temperature can generate an additional 1.3 and 4.1 kWh of energy per t of wet sludge at the average DS concentrations of 4.5 and 7.9% found in sludge feed to conventional and THP MAD processes, respectively. Thus, the results demonstrated the potential advantage of increasing the digestion temperature of conventional MAD processes operated at typical DS feed concentrations, and also supported current operational strategies of adopting high digestion temperatures at full-scale THP sites, to gain the overall maximum quantitative energy benefit. However, careful consideration of the balance between the other main process conditions of sludge DS content and HRT is necessary when selecting the operating temperature for conventional MAD treatment.

Table 9-3 Energy balance of conventional and THP MAD with increasing digestion temperature under different DS feed concentration regimes

Temperature increase (°C)	31 to 33	33 to 35	35 to 37	37 to 39	39 to 41
Energy required to heat 1 t wet sludge(kWh)	2.3	2.3	2.3	2.3	2.3
Net energy out from 1 t wet sludge in kWh (2.7% DS)	0.4	0.3	0.1	0.0	<i>-0.1</i>
Net energy out from 1 t wet sludge in kWh (3.0% DS)	0.7	0.6	0.4	0.3	<i>0.1</i>
Net energy out from 1 t wet sludge in kWh (4.5% DS)	2.3	<u>2.0</u>	<u>1.8</u>	<u>1.5</u>	<u>1.3</u>
Net energy out from 1 t wet sludge in kWh (7.9% DS)	5.7	<u>5.2</u>	<u>4.8</u>	<u>4.4</u>	<u>4.1</u>

Note that bold and underlined values reflect conditions that may overlap in operational practice for conventional and THP MAD; The italic values reflect extreme cases that fall outside of 90% operational data range. 2.7% DS is the lower 5% percentile range value of monthly average operational data for conventional MAD sites; 3.0% is the break point sludge feed DS for a positive net energy balance for MAD; 4.5% and 7.9% DS are the mean values of monthly average operational data for conventional and THP MAD sites, respectively.

9.4.2 Enzymic MAD model (Model 11, Section 5.4)

The enzymic MAD model described the effect of DS feed, VFA feed and temperature on EY. Similar to the combined conventional-THP MAD model, raising digestion temperature increased EY, but requires additional energy input. Table 9-4 shows the energy required to heat 1 t of wet sludge to increase the digestion temperature by 2 °C, and the net energy generated by 1 t of sludge at different VFA and DS feed concentrations. A digester feed VFA of 5500 mg/l was selected to illustrate the typical values for enzymatic pretreated sludge to the digester, which represented the mean concentration of VFA recorded in feed sludge for enzymic processes. For this example, the results showed a negative net energy balance for a DS feed lower than 3.5% (note that the heating efficiency of the system is not considered), equivalent to the lower 5% percentile of monthly average operational DS data for enzymic MAD sites. Similarly, a negative net energy balance was also observed at all DS feed levels when the pretreatment was less effective (VFA = 3750 mg/l). Thus, in rare case when the DS feed supplied to enzymic MAD processes is less than 3.5% or VFA is less than 3750 mg/l, a lower mesophilic temperature (< 35 °C) should be applied to improve the energy balance of the system. However, with typical or higher VFA values in the feed sludge (≥ 5500 mg/l) the process can be effectively operated in the upper mesophilic temperature range (> 39 °C) to maximise the BY and energy balance.

Raising DS feed concentration decreases EY, but increases the total throughput of sludge under the same HRT. The net electricity output due to a change in DS feed can be calculated using Equation 9.2:

$$\begin{aligned}
 \text{Net daily electricity (kWh/day)} &= \text{Electricity}_1 - \text{Electricity}_2 \\
 &= \frac{DS_1}{100} \times EY \times \frac{\text{Digester volume}}{\text{HRT}} - \frac{DS_2}{100} \times (EY - x) \times \frac{\text{Digester volume}}{\text{HRT}} \\
 &= \frac{\text{Digester volume}}{100 \times \text{HRT}} \times [DS_1 \times EY - DS_2 \times (EY - x)] \quad (9.2)
 \end{aligned}$$

Where:

Electricity₁ (kWh/day) = the amount of electricity produced when DS is equal to DS₁

Electricity₂ (kWh/day) = the amount of electricity produced when DS is equal to DS₂

EY (kWh/t DS) = the electricity yield when DS feed is equal to DS₁

x (kWh/t DS) = the relative change of electricity yield when DS is changed from DS₁ to DS₂

To test the net effect of increasing DS and show if this produces a positive net electricity benefit, the results of $EY(DS_1 - DS_2) + DS_2 x$ must be negative. For example, if it assumed that a 2000 m³ digester operated at 20 days HRT and 4.5% DS has an EY of *b* kWh/t DS, the daily electricity produced is equivalent to 4.5*b* kWh/day. The model outputs and relative changes in EY presented in Table 8-4

show that increasing DS feed concentration from 4.5 to 5.5% would reduce EY from b to $b-83$ kWh/t DS. Then, the daily electricity produced would become $5.5 \times (b - 83)$ kWh/day. A positive net energy balance is achieved when $4.5b - 5.5 \times (b - 83)$ is less than zero. Therefore, the effect of increasing DS feed from 4.5 to 5.5 % on the minimum EY necessary for a positive net energy output is 457 kWh/t DS. The minimum EY for a positive net energy output under different DS feed concentrations is summarised in Table 9-5. The results indicated that, as long as the current EY is larger than approximately 470 kWh/t DS (equivalent to a BY of 224 m³/t DS) and extended HRT condition is maintained (HRT $\geq$ 23 days), it is beneficial to increase the DS feed concentrations of enzymic MAD processes within the DS operational range from 3.5 to 7.5% as listed in Table 8-4. However, this minimum EY value is only intended to provide a general guide to show the potential for net energy improvement as, in practice, it is rare for enzymic MAD processes to operate at such a low EY. Furthermore, it is important to consider other local, site specific conditions, that were not included in the model, when designing process optimisation strategies and interventions.

Table 9-4 Energy balance of enzymic MAD with increasing digestion temperature under different VFA and DS feed concentration regimes

Temperature (°C)		33 to 35	35 to 37	37 to 39	39 to 41
Energy required to heat 1 t wet sludge (kWh)		2.3	2.3	2.3	2.3
3750 mg/l VFA feed	DS feed (%)	Net energy output from 1 t wet sludge in kWh			
	3.5	-1.3	-1.3	-1.4	-1.4
	4.5	-0.9	-1.0	-1.1	-1.2
	5.5	-0.6	-0.7	-0.8	-0.9
	6.5	-0.3	-0.4	-0.5	-0.6
	7.5	0.0	-0.2	-0.3	-0.4
5500 mg/l VFA feed	3.5	-0.1	-0.2	-0.3	-0.4
	4.5	0.6	0.4	0.3	0.1
	5.5	1.2	1.0	0.8	0.7
	6.5	1.8	1.6	1.4	1.2
	7.5	2.5	2.2	2.0	1.8
6500 mg/l VFA feed	3.5	0.4	0.3	0.1	0.0
	4.5	1.2	1.0	0.8	0.7
	5.5	2.0	1.8	1.5	1.4
	6.5	2.8	2.5	2.2	2.0
	7.5	3.6	3.2	3.0	2.7

3.5% and 7.5 % DS are the lower and upper 5 percentile range values of monthly average operational data for enzymic MAD sites, respectively; 5500 mg/l VFA is the mean value of monthly average operational data for enzymic MAD sites; 3750 mg/l is the minimum VFA feed concentration for positive net energy output at high DS feed for enzymic MAD; 6500 mg/l is the minimum VFA feed concentration to avoid negative net energy output at any DS feed for enzymic MAD

Table 9-5 Minimum EY for positive net energy output by increasing DS

DS change (%)	3.5 to 4.5	4.5 to 5.5	5.5 to 6.5	6.5 to 7.5
Relative change in EY (kWh/t DS)	-103	-83	-69	-59
Minimum EY (kWh/t DS) to increase DS for positive net energy balance	464	457	449	443

9.5 Summary

Through a comprehensive statistical analysis of full-scale AD data from a total number of 66 conventional, 8 THP and 10 enzymic MAD sites, the performances of both conventional and advanced MAD processes was shown to be statistically significantly correlated to the routinely measured operational parameters available at full-scale MAD sites. Further scrutiny of the models showed the effects of the operational parameters on digester performance could be cross-referenced to each other in these independently developed models. For example, the effects of temperature, HRT and DS feed on the BY of sludge receiving THP pretreatment were equivalent to those operating in the conventional MAD process, albeit at a higher range. Hence a combined conventional-THP MAD model (Model 18) was developed in Section 6.4. In addition, the enzymic-MAD can be considered as a special case that was also represented by the combined conventional-THP model (Model 18), with extended HRT (23 days was the average HRT for enzymic processes) (see Section 9.2). The behaviour of individual operational parameters (temperature and HRT), described by the model (Model 18), on digester performance was consistent with laboratory-scale tests reported by several authors (Boušková *et al.*, 2005; Kim and Lee, 2016; Alepu *et al.*, 2016; Nielsen *et al.*, 2017) (Section 9.2). Additional interaction effects, such as the HRT-DS (Model 18), and temperature-VFA interaction (Model 11), on digester performance were also detected in the models developed from full-scale AD data.

Furthermore, the effects of sludge pretreatment on AD performance were also captured in the models. Higher solids inputs and quicker reaction rates are reported by several authors (Wilson *et al.*, 2008; Xue *et al.*, 2015) for thermally hydrolysed sludge in the MAD process compared to conventional MAD, and this is attributed to changes in sludge rheology and soluble organic contents after THP (Barber, 2016; Gurieff *et al.*, 2011; Xue *et al.* 2015). This behaviour was captured by Model 18, where the magnitude of the response in BY to changes in the HRT was greater for THP MAD processes compared to conventional MAD operating at lower DS feed concentrations over an equivalent range of HRT. Therefore, THP MAD can achieve a similar BY compared to the conventional MAD process with shorter HRT. Similarly, the effects of enzymatic pretreatment was captured by the model (Model 11) by the significant interaction between VFA feed and temperature. Thus, the response of the enzymic-MAD process to temperature increased to a greater extent in

comparison to the conventional-THP system when the VFA concentration rose above 4000 mg/l, which was the case for 70% of the data recorded for enzymic MAD processes.

Although the relative changes in digester performance were effectively captured by the principal operational parameters, sites with similar operating conditions do not necessarily demonstrate the same performance, and part of the reason for these differences could be attributed to the variation in sludge composition (Weiland, 2010; Li, *et al.*, 2018). Several studies confirm that the BY is significantly and positively correlated to the VS content in crops, pig manure slurry, agro-industrial waste, and the organic fraction of municipal solid waste as discussed in Section 2.4.2 (Schievano *et al.*, 2008; Luna, 2011). The VS content in sewage sludge showed a similar positive effect on BY of MAD as shown in Figure 7-5 (Section 7-4), based on the sludge sampling results. However, no significant correlation was detected between BY and sludge feed macromolecular composition, such as protein and fat content (Section 7-4). Only a limited number of published results are available that consider the effect of sewage sludge composition on CH₄ yield (Mottet *et al.*, 2010; Astals *et al.*, 2013), and these have not found any significant impact of the macromolecular composition (protein, fat and carbohydrate) in feed sludge on CH₄ yield. However, further scrutiny of the substrate compositions of both feed and digested sludge showed the compositional derived BY_c, which is based on the proportions of substrates destroyed during AD, was significantly correlated to the observed BY (Section 7.4). The compositional derived BY_c also provided an alternative to the default model calibration approach (based on local site BY data), and is helpful in identifying possible data recording issues at full-scale AD sites (see Section 8.2.1 and 9.3.2).

Recommended optimisation strategies based on controlled laboratory tests tend to purely focus on the effect of individual parameters on BY or CH₄ yield, for example, see Section 9.4.1 (Alepu *et al.*, 2016; Dokulilová *et al.*, 2018). However, rather than simply focusing on BY, the quantitative benefits of balancing the principal operational parameters (temperature, HRT, DS, VFA) on AD net energy output was evaluated using the multiple regression models (Model 18 and 11) generated from full-scale AD data (Section 9.4). This demonstrated that the highest BY does not necessarily equate to an optimised performance for full-scale AD, based on overall net energy balance. For example, the maximum BY may be achieved at the lowest DS feed and longest HRT within the operational range of conditions represented at full-scale sludge AD sites. However, this combination of conditions does not correspond to the maximum overall biogas volume. Indeed, the optimum combination of HRT and DS feed to produce the maximum biogas volume is a function of local BY, and can be calculated from Equation 9.1, as discussed in Section 9.4. For enzymic sites, the minimum EY to avoid negative net energy output is approximately 470 kWh/t DS, which is a relatively low output value for the enzymic-MAD process.

Selecting an optimum operating temperature strongly dependent on DS feed concentration. Approximately 7.6 kWh of net energy can be generated per t of wet sludge at the average conventional MAD DS feed concentration (4.5%) by raising the process temperature within its 90%

data range (31 to 39 °C) at conventional MAD sites. The magnitude of the net energy response to temperature was greater under higher DS feed concentrations, thus, more than 18 kWh of net energy per t of wet sludge could be generated at the average THP DS feed concentrations (7.9%) by raising temperature within its 90% data range (33 to 42 °C). The maximum net energy produced by raising temperature (33 to 41 °C) in enzymic MAD sites with typical VFA feed concentrations (5500 mg/l) is approximately 8.5 kWh per t wet sludge at 7.5% DS feed, and this value could increase with VFA feed concentration, which depends on the effectiveness of the enzymatic pretreatment stage. The benefit of operating at high mesophilic temperature would be further improved in a modern AD system, where the waste heat from CHP is used for digester heating. For example, at the average conventional MAD DS concentration of 4.5%, an additional 3.6 kWh of energy per t of wet sludge would be generated by raising the process temperature from 39 to 41 °C for digesters heated by the waste heat. Since 95% of temperature data is below 39 °C at conventional MAD sites, this strategy would offer considerable potential to improve the overall net energy balance in a modern conventional MAD system with CHP.

The results emphasise the importance of balancing the DS feed, HRT and temperature to optimise the energy performance of the AD process. The combined conventional-THP (Model 18) and enzymic (Model 11) MAD models can thus be universally applied to enable operators to balance these principal operational parameters to devise practical measures and interventions to gain the maximum net energy output under different, full-scale, sewage sludge MAD processes.

Chapter 10. Conclusions and Recommendations

10.1. General Data Performance

1. A series of statistical tests and multiple regression modelling techniques were applied to large process monitoring data sets collected from 66 conventional, 8 THP and 10 enzymic MAD sludge treatment sites in five major UK water utility companies to quantify the significance of operational parameters controlling digester performance, indicated by BY, EY and VSR.
2. The mean BY for 66 conventional MAD sites included in the analysis was 398.7 m³/t DS, which was entirely consistent with typical values for sewage sludge MAD reported in the literature. The 90% data ranges for the major operating parameters of DS, HRT and temperature represented typical digestion conditions for conventional MAD sites in the UK and were equivalent to: 2.7 - 6.3%, 12.8 - 34.7 d and 31.6 - 38.9 °C, respectively. The mean BY for THP MAD was higher compared to the conventional process, and was equivalent to 447.8 m³/t DS. Thermal hydrolysis sites had wider 90 % operational ranges of DS, HRT and temperature compared to conventional AD, equivalent to: 4.2 - 10.5%, 11.7 - 45.2 d, and 32.8 - 42.4 °C, respectively. The enzymic MAD sites had slightly higher mean EY, 921.0 kWh/t DS, and mean BY, 482.4 m³/t DS, compared to THP MAD sites; the mean EY of THP-MAD was equivalent to 859.7 kWh/t DS. Enzymic MAD sites also recorded the VFA concentration of feed sludge, as an additional chemical operating parameter, and the 90% data ranges for VFA, DS, HRT and temperature were equivalent to: 2186.7 - 9175.5 mg/l, 3.1 - 7.4%, 12.3 - 44.3 d, and 32.9 - 40.0 °C, respectively.
3. In general, no apparent or direct trends were observed between most of the site average operational parameters in relation to BY and EY at conventional and advanced MAD sites. Further scrutiny of monthly average data detected several significant predictors of BY and EY. However, the contribution of individual predictors was small and could not effectively describe the variation observed in overall digestion performance. The weak association between digestion performance and the principal digestion conditions of full-scale processes was explained due to the complexity of industrial level MAD and simple linear relationships cannot account for differences in site variation caused by the interaction between different operating parameters or other unmeasured factors, such as process microbiology, feed properties, engineering controls and data recording.

10.2.Key Parameters for AD Control

1. A novel multiple regression analysis approach was developed, which integrated a categorical site factor to explain the variation associated with local data measurement and other unrecorded factors at specific sites. The major operational factors influencing the process were quantified based on the relative changes in digester performance by using a centred predictors approach.
2. Temperature, HRT and DS feed were the three main operational parameters collected by all water companies for MAD process control, and all had statistically significant main effects on the BY of conventional MAD. Temperature had a positive, approximately linear relationship with BY. The relationships between BY, and HRT and DS were described by logarithmic functions, and BY increased in relation to HRT and decreased in relation to DS. The effect of HRT on BY also depended on the concentration of the DS feed and increasing HRT had a stronger effect on BY at higher DS concentrations.
3. Sludge age and primary:SAS ratio were routinely collected at Company 2, but no statistically significant effects of sludge age or primary:SAS ratio on BY of conventional-MAD were detected by multiple regression analysis. A small sludge age coupled with a high primary:SAS ratio, which are favourable for raising the overall BY in AD, are unlikely to be achieved in the real operational situation as these process parameters are strongly and negatively correlated to each other ($P < 0.001$). Thus, the opposing effects of sludge age and primary:SAS ratio on BY in AD could explain why they were found to have no overall significant effect on BY when modelling full-scale operational AD process data.
4. The conventional MAD model (Model 4) was validated against independent datasets as well as selected site data involved in model development. In general, BY was effectively predicted for the selected conventional sites used in model validation with R^2 values in the range of 0.32 – 0.65. Model 4 also effectively predicted the BY for THP sites, with an overall R^2 of 0.72.
5. The effect of digestion temperature, HRT and DS feed in THP MAD sites on BY were equivalent to those operating in conventional MAD sites. Therefore, a combined conventional-THP MAD model (Model 18) was developed, integrating data for both process types from all of the participating UK water utility companies. Model 18 was tested against selected conventional (Site 37, 38 and 42) and THP (THP site 1-6) datasets, and the results showed a good overall description of BY was obtained for both conventional MAD ($P < 0.001$, $R^2 = 0.59$) and THP sites ($P < 0.001$, $R^2 = 0.73$).
6. Temperature, HRT, DS feed and VFA feed concentrations were routinely collected by Company 3 and 5 for enzymic treatment sites. However, not all the enzymic MAD sites had biogas measurement, and their AD performance was indicated by EY in this case (Model 11). The EY

decreased following a diminishing pattern with increasing DS. The relationships between EY, and temperature and VFA feed were described by logarithmic functions, and EY increased in relation to both temperature and VFA feed. The effect of temperature on EY in the enzymic MAD process was similar to the combined conventional-THP MAD model (Model 18) when the VFA feed concentration was approximately 4000 mg/l, however, the magnitude of the response in EY to temperature increased at higher VFA feed concentrations. By contrast, the multiple regression analysis showed there was no statistically significant effect of HRT on EY of the enzymic MAD process (Model 11). This behaviour may be attributed to the relatively small variation in the overall longer average HRT values (23 days) applied at enzymic-MAD sites, compared to other MAD processes; thus, no statistically significant response could be detected to this parameter at the operational range of HRT values observed in practice. Enzymic-MAD was shown to be a special case of the combined conventional-THP model (Model 18), as the performance of both systems was approximately equivalent when the HRT of the conventional-THP model was set to the representative mean value for the enzymic-MAD process (23 days).

7. Three model calibration methods were introduced using local site BY, EY and sludge composition, to adjust the categorical site factor, and thus represent the effects on digestion performance of site factors not represented as continuous variables in the model, and that were not routinely recorded at WWTP. The default, internal calibration method, based on local site BY data, is the generally recommended procedure for routine model calibration, however, this approach depends on the availability of reliable gas flow measurements. Two alternative calibration methods, using EY or sludge composition analysis, are also proposed, and can be useful when problems are suspected with the quality of local data recording on a particular site (eg biogas flow).
8. Two sub-models were constructed to evaluate the effects of digestion conditions on VSR and digested sludge dewaterability for conventional MAD. Volatile solids reduction provides an alternative indicator of digestion performance and was statistically significantly correlated to BY. It was described by Model 14, which showed that VSR increased following a diminishing pattern with increasing HRT. The effects of VS feed and temperature on VSR were positive and approximately linear for the operational ranges of VS feed and temperature obtained at conventional MAD sites. The dewaterability model (Model 17) could not reliably predict sludge cake DS concentrations, but it provided insight into how upstream wastewater treatment and sludge digestion processes may influence final sludge dewaterability. For example, sludge dewaterability was negatively affected to some extent by improved digestion temperature that led to increased BY. This may be explained because improved destruction of readily degradable organic substrates could potentially raise the proportion of the less digestible SAS fraction in the final digested sludge, and hence reduce the overall dewaterability to some extent.

10.3. Sludge Composition and Its Implications for AD

1. Eight MAD sites were selected for sample collection with representative examples of each of the main AD treatment types, including: conventional, THP and enzymic processes. Sludge feed and digestate composition were compared during the course of 6 sampling events, over a period of approximately one year. The average concentrations of major organic energy constituents of protein, fat, fibre and carbohydrates in all feed sludge samples were 24.8, 13.6, 27.2 and 14.9% DS, respectively. A comprehensive analysis of inorganic elemental composition, including the heavy metals, such as, Cu, Zn, Pb, and Cd, and the major nutrients, including: N, P, K and Mg, were also completed, and the results were consistent with other sludge composition surveys reported within the last 10 years.
2. Positive, statistically significant correlations were observed between BY and sludge feed VS, VSR and COD removal efficiency ($P = 0.038$, $R^2 = 0.24$; $P = 0.004$, $R^2 = 0.42$ and $P = 0.013$, $R^2 = 0.42$, respectively). However, no significant correlation was detected between BY and sludge feed macromolecular composition, such as protein and fat content, which is consistent with the results reported in the literature. Further scrutiny of the substrate compositions of both feed and digested sludge showed the compositional derived BY_c , which is based on the proportions of substrates destroyed during AD, was significantly correlated to the observed BY. The compositional derived BY_c provided an effective alternative to the default model calibration approach (based on local site BY data), which was independent of the standard operational data recording. This supplementary analysis is recommended to be routinely measured at full-scale sites to give operators a benchmark of the theoretical BY performance and identify potential recording errors in important process performance assessment metrics, such as gas flow, at local AD sites. Alternatively, a relatively simple and inexpensive biodegradability test, such as the BMP or respiratory test, may also be used to provide a benchmark of theoretical BY performance at local AD sites.

10.4. Ensure Good Data Quality

1. In addition to the complex effects of physical and chemical factors, the accuracy of empirical statistical models describing the AD process also depends on the quality of the datasets used to derive the model. Uncontrolled variability reduces the overall proportion of the total variation in the data that is explained by the model coefficients, indicated by adjusted R^2 . To examine the impact of this on model predictions, model stability was tested by including an increasing proportion of random data into the datasets. As would be expected, the R^2 decreased to an extent as more random data were added, but large amounts of random data, up to 70%, was necessary to materially change the coefficients and for them to lose statistical significance. Therefore, whilst data recording errors were very likely to be a factor defining the extent to which

the significance of process variables could be detected, the models showed a high degree of resilience to random data in terms of describing the actual process kinetics. Furthermore, as would be expected, site recorded data that were inaccurate, or unrepresentative could not be described effectively by the derived process model. The impact of such data on the overall model description was minimised by applying a categorical site factor for site and using the centred prediction approach to the statistical modelling. These statistical precautions and strategies were effective in providing confidence in the overall model quality, nevertheless, the importance of ensuring high standards of data recording cannot be overestimated. It is essential to increase awareness and understanding of data quality to ensure and maintain accurate data recording protocols at full-scale AD sites to support process modelling and management.

2. Four data quality criteria, specific for AD management are proposed, including: data accuracy, data completeness, data consistency and data accessibility. A regular calibration of gas-flow and solids meters is recommended to improve the data accuracy. The potential data recording errors may be identified by the compositional derived BY_c and electricity derived BY_e , which provided benchmarks of theoretical performance at local AD sites. Finally, providing better accessibility through a centralised database with a real-time access would benefit both future model development and operational management.

10.5. Maximum Energy Balance of the AD System

1. From the total of 66 conventional MAD sites included in the statistical analysis, 32 were operating below the typical, average BY of 400 m³/t DS, which demonstrated the potential scope for improving the performance of full-scale MAD processes. The model (Model 18) developed here is based on currently monitored operational parameters and can provide a useful decision support to increase the efficiency of sewage sludge AD. The model can be used to manipulate the BY within the range up to the operational average value equivalent to 400 m³/t DS and to larger values, where this may be feasible, for example, due to sludge composition or pretreatment, such as by THP process. However, simply focussing on increasing the BY of MAD does not necessarily lead to an overall improvement in process performance in terms of energy balance. For example, a sub-optimal BY with reduced HRT can increase the total process capacity, increasing the overall amount of biogas produced, and thus favouring an overall positive energy for the process, compared to digestion at higher BY. Raising the digestion temperature also increases BY, but requires additional energy input. The model results showed that positive energy benefits could be gained by increasing the operating temperature in the high mesophilic range >39 °C, provided that a minimum DS of 3.0% was supplied in the feed sludge, and that a greater energy surplus is obtained with increasing feed DS content, which is a significant advantage of THP pretreatment. Therefore, balancing the DS feed, HRT and temperature is necessary to optimise the BY and overall net energy balance of the AD system,

and the user-interface developed based on the model can assist operators to identify and devise practical optimisation strategies for application to full-scale MAD sites.

2. Enzymic-MAD is special case that is also represented by the combined conventional-THP model (Model 18), with extended HRT (23 days was average for enzymic processes). The model results showed that positive energy benefits could be gained at any DS feed level, provided that a minimum VFA of 6500 mg/l was supplied in the feed sludge. Under a typical VFA feed of 5500 mg/l, positive net energy output is maintained by increasing the operating temperature in the high mesophilic range $>39^{\circ}\text{C}$, if the system is operated at or above the minimum DS feed of 4.5%. The effect of increasing DS feed concentration on net energy output is a function of local EY (Equation 9.2); the minimum EY value to achieve a positive energy balance is approximately 470 kWh/t DS at a $\text{HRT} \geq 23$ days.

10.6.Recommendations for Future Research

The statistical modelling of AD data with monitored operational parameters and categorical factors for site explained approximately 10% and 40% of the total variation in BY, respectively (Model 18). Therefore, major uncertainties remain in understanding the factors controlling full-scale sewage sludge AD processes, and further research is necessary to identify other possible variables that play an important role in controlling the process and how to incorporate them into a practical operational model suitable for industrial application. The following recommendations for further work are proposed to overcome the current technical uncertainty and improve the ability to manipulate, control and optimise the AD process:

1. Full-scale implementation: A first step is to apply the model to develop optimisation strategies and implement these at selected full-scale MAD sites to practically demonstrate the benefits for the energy balance and operation of the process.
2. Improve data quality: The quality of the operational data used to derive the model explains part of the uncertainties in the models and there is probably considerable potential to further increase model accuracy and the variation in digester performance explained by the operational parameters. The current data screening methods, focused on the overall data range across all sites, effectively reduced the extreme values in the datasets. However, this approach cannot identify data recording errors within the typical AD operational boundary. The electricity or sludge composition calibrated model, specifically adopted for each WWTP included in the database, could be used to further screen the data and check the accuracy of the data recording.
3. Integrating sludge composition data into the model: No significant correlation was detected between BY and sludge feed macromolecular composition, and this may be explained as substrate degradability was highly related to the digestion conditions. However, a significant

correlation was detected between the observed BY and the sludge composition derived BY_c , based on the proportions of substrates destroyed during AD. The BY_c provided an effective alternative method for model calibration, and it is recommended that detailed energy substrate composition data is routinely measured at full-scale sites. In future, sludge composition data or a relatively simpler biodegradability test result may be integrated into the model as predictors, and hence the BY value could be directly estimated for specific AD sites without model calibration.

4. Effective digester volume: The HRT is one of the main operational parameters recorded by all companies, which has significant impact on digester performance. The HRT is positively correlated with digester volume, however, the accumulation of scum and grit reduces the effective digester volume overtime. The variation in the effective digester volume, due to different asset ages at full-scale sites, was not taken into account in this project. However, the effective digester volume could explain part of the variation in site performance and improved measurements of this metric would provide a better understanding of the direct effects of HRT on digester performance.
5. Mixing power input measurements: Effective mixing of digesters is essential to provide a uniform environment for sewage sludge AD. However, there is no mixing efficiency related parameter recorded at full-scale MAD sites. Mixing power input together with its mixing mode and mixer type could be easily and reliably recorded at full-scale MAD sites. These factors may be integrated into the model as predictors in the future to evaluate the impact of mixing efficiency on AD performance.
6. Better energy balance model: The optimisation strategies were developed based on an optimum overall net energy balance of the AD system. However, the net energy calculation was based on the energy difference between digester heating and biogas production at the MAD stage. The results showed a greater energy surplus was obtained with increasing feed DS content, especially at THP sites, which typically operated with a high DS feed. However, in practise, the extra energy consumed in THP pretreatment may remove the energy advantage of the improved energy balance specifically relating to the MAD process. Furthermore, the additional biogas produced in the unmixed post treatment tanks are already collected by some water companies to avoid fugitive emissions. Therefore, a more comprehensive energy balance model could be developed based on the current digester performance models to take the pretreatment energy consumption, waste heat from CHP, biogas collected from these post treatment stages, and heat loss and transfer efficiency, into account, to provide a more accurate assessment of the overall energy balance of the sewage sludge treatment system at full-scale MAD sites.
7. Better understanding of AD performance with site specific microbial community information: The microbial community involved in AD is very complex, however, it is well known that a stable

population with the correct balance of the major groups responsible for the various stages of organic matter conversion by AD is essential (Narihiro *et al.*, 2015). Indeed, the unexplained performance variations observed in the current process models could be partly explained by the variations in AD microbial communities at different sites. Therefore, measuring the microbial community structural dynamics and function could provide greater fundamental insight that is necessary to understand and explain AD performance. This could be achieved by building an AD microbiomes database mapping the operating parameters and corresponding process performance in each plant. The microbiomes in AD could be characterised by high-throughput sequencing technologies, such as 16S rRNA gene sequencing (Mei *et al.*, 2017). More effective site specific AD optimisation strategies could therefore be developed by incorporating microbial community information.

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Appendix

Appendix 3-1 Advanced mesophilic anaerobic digestion

Thermal hydrolysis process (THP)

There are several thermal hydrolysis vendors available on the market and Cambi™ THP and Veolia Bio Thelys™ are two major THP technologies that operate as a series of process batch tanks used at the Water Utilities in this study.

Cambi thermal hydrolysis process

The THP scheme of the Cambi system is shown in Figure A3-1; dewatered sludge cake (up to 17% DS concentration) is added to the pulper tank which is heated by recycled steam from the flash tank. The sludge is held in the pulper tank until the reactor is ready for batch processing. Preheated sludge is pumped into the main THP reactor and steam is injected to achieve a 170 °C and 6 bars pressure. After a minimum 20 minutes holding time, the sludge is fed into the flash tank where the rapid expansion causes the bacterial cells to rupture and the pressure is reduced to about 1.5 bar. The temperature is also reduced by flashing, and the condensed water from steam and added dilution water reduce the temperature of the sludge to approximately 37 °C. The solids concentration is reduced to 8 to 10 % for feeding into the anaerobic digester (Williams and Burrowes, 2016).

The total cycle time is approximately 74 minutes with 29 minutes for filling the solids and injecting steam into the reactor, 20 – 30 minutes holding time in the reactor, and 15 minutes blowdown to the flash tank (Williams and Burrowes, 2016).

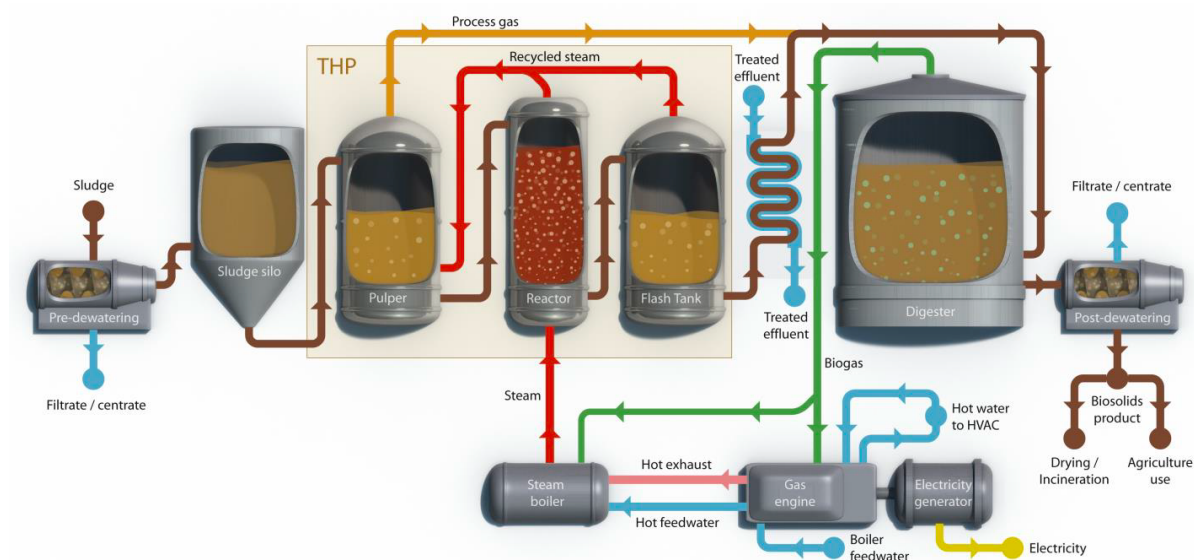


Figure A3-1 Schematic representation of the Cambi THP (Williams and Burrowes, 2016)

Veolia BioThelys thermal hydrolysis process

The Veolia BioThelys THP is similar to Cambi THP and also operates as a batch system with operating temperatures in the range 130 °C to 150 °C and pressures between 8 to 15 bars (Figure A3-2). BioThelys reactors operate in parallel pairs and the thermal energy from the flash steam is recovered from one reactor to the parallel unit (Ray, 2013). The sludge is held in the batch reactor for 30 minutes at the required temperature and pressure for thermal hydrolysis. After the reactor, the sludge is flashed into a buffer tank to partially cool, and is then fed through a heat exchanger with addition dilution water to cool and reduce the solids content as required by the downstream AD process (Abu-Orf and Goss, 2012).

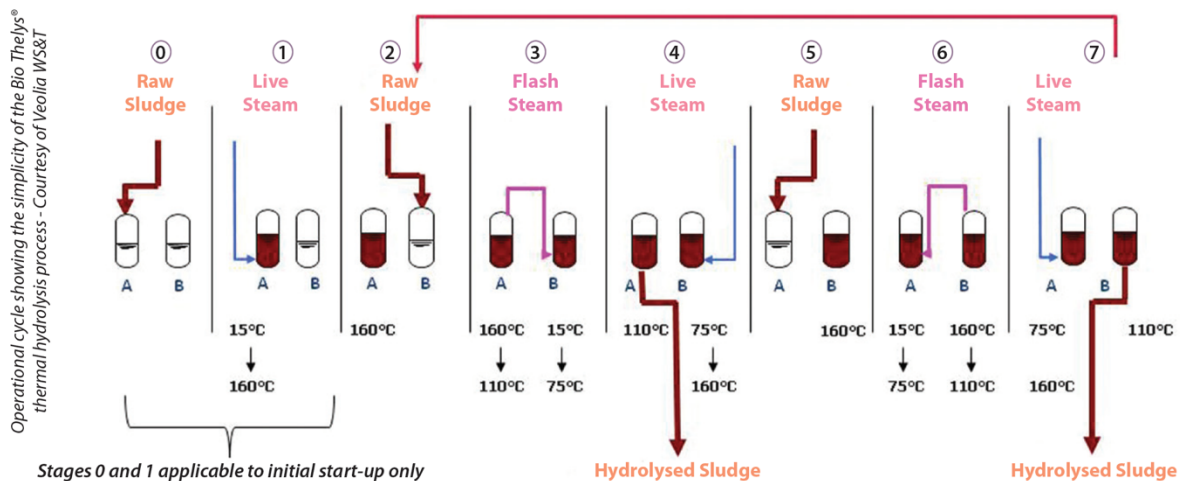


Figure A3-2 Schematic representation of the Veolia BioThelys THP (Ray, 2013)

Heating, pasteurisation and hydrolysis (HpH)

The HpH process was developed ‘in-house’ by Anglian Water, and includes a three-stage system shown in Figure A3-3 that:

- Pre-heats the blended feed sludge prior to pasteurisation;
- Pasteurises the sludge and achieves an “enhanced” sludge product and rapid pathogen kill;
- Pre-conditions (hydrolyses) the digester feedstock for optimum biogas production.

The HpH system comprises of four reactors: 1 reactor for pre-heating, followed by 2 reactors for pasteurisation and 1 reactor for hydrolysis. After the sludge temperature is raised to 42 °C in the pre-heating process, it is fed into the pasteurisation tanks to further raise the temperature to 57 °C by steam injection. The system operates an automated batching arrangement centring on the two pasteurisation tanks. Whilst one pasteurisation tank is filling and heating, the other is holding at temperature until pasteurisation is completed and the batch holding time of at least five hours is achieved. After the holding period the sludge is fed to the hydrolysis tank and the cycle is repeated. The sludge is held in the hydrolysis tank for 1 or 2 days at 38 - 42 °C, and is subsequently transferred

via a heat exchanger to reduce the temperature to 38 °C before being pumped slowly into the digester (Kubeshnee, 2014).

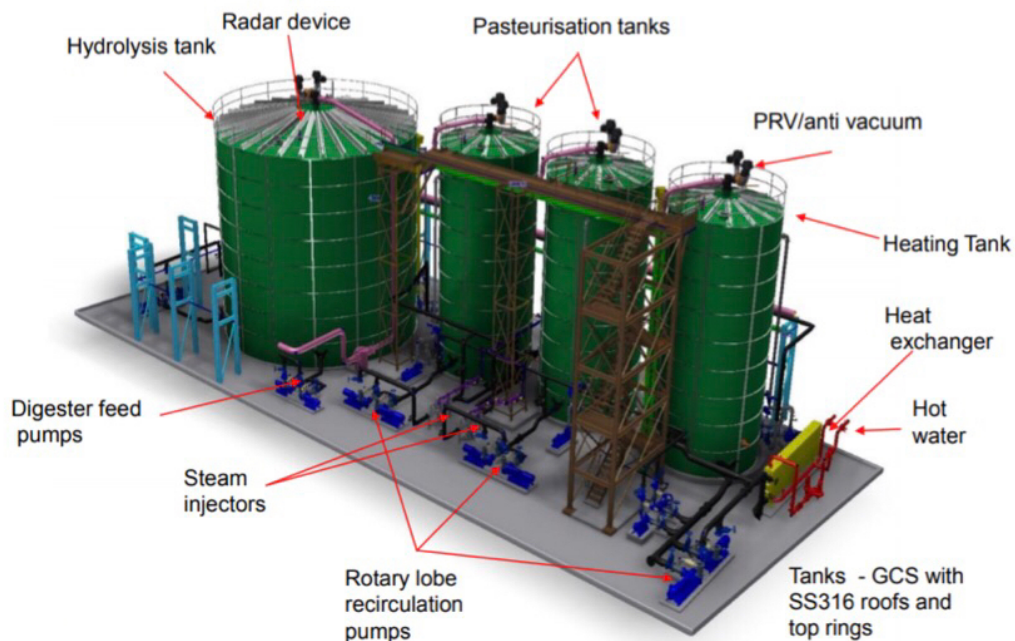
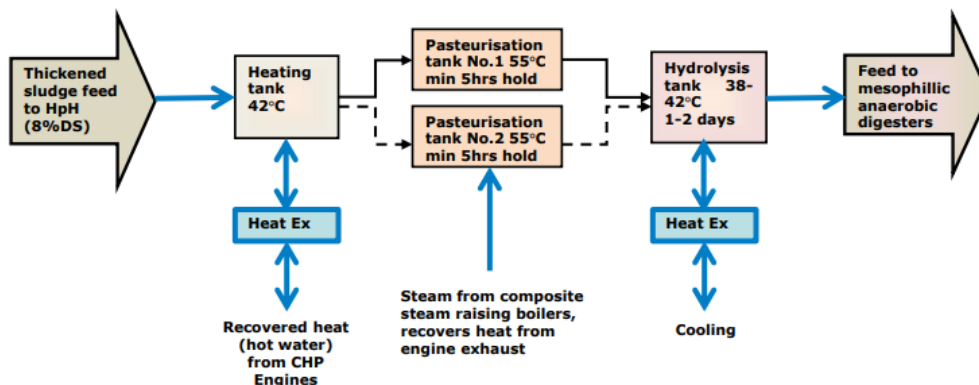


Figure A3-3 Heating, pasteurisation and hydrolysis process flow diagram (Kubeshnee, 2014).

Enzymic Hydrolysis (EH) and Enhanced Enzymic Hydrolysis (EEH) processes

United Utilities PLC together with Monsal Limited has established a biological pre-treatment strategy based on plug-flow, hydrolytic acidogenic pre-fermentation. Plug flow designs for acidogenic pre-fermentation aim to separate volatile fatty acid production from the methanogenesis stage providing overall stability benefits to the anaerobic digestion process (Sans *et al.*, 1994; Bolzonella *et al.*, 2005).

The standard EH process was designed with six equal volume well-mixed tanks in series for mesophilic pre-fermentation with an overall HRT of 2 - 3 days upstream of MAD (Figure A3-4) (Bungay and Abdelwahab, 2008). The EH process has been modified to achieve enhanced

treatment status for pathogen removal (ADAS, 2001), by the addition of a 55 °C pasteurisation tank with a minimum 5 hours holding time at the end of the process. The EEH comprises EH mesophilic pre-fermentation in plug-flow form tanks 1 to 3. A second external heat exchanger loop is included after tank 3 to increase the temperature of sludge in tank 4 to 55 °C. The sludge is heated and well-mixed in tank 4 and then 5/8 of its nominal volume is delivered to tanks 5 or 6, which are operated as parallel sequencing batch reactors (Werker *et al.*, 2007).

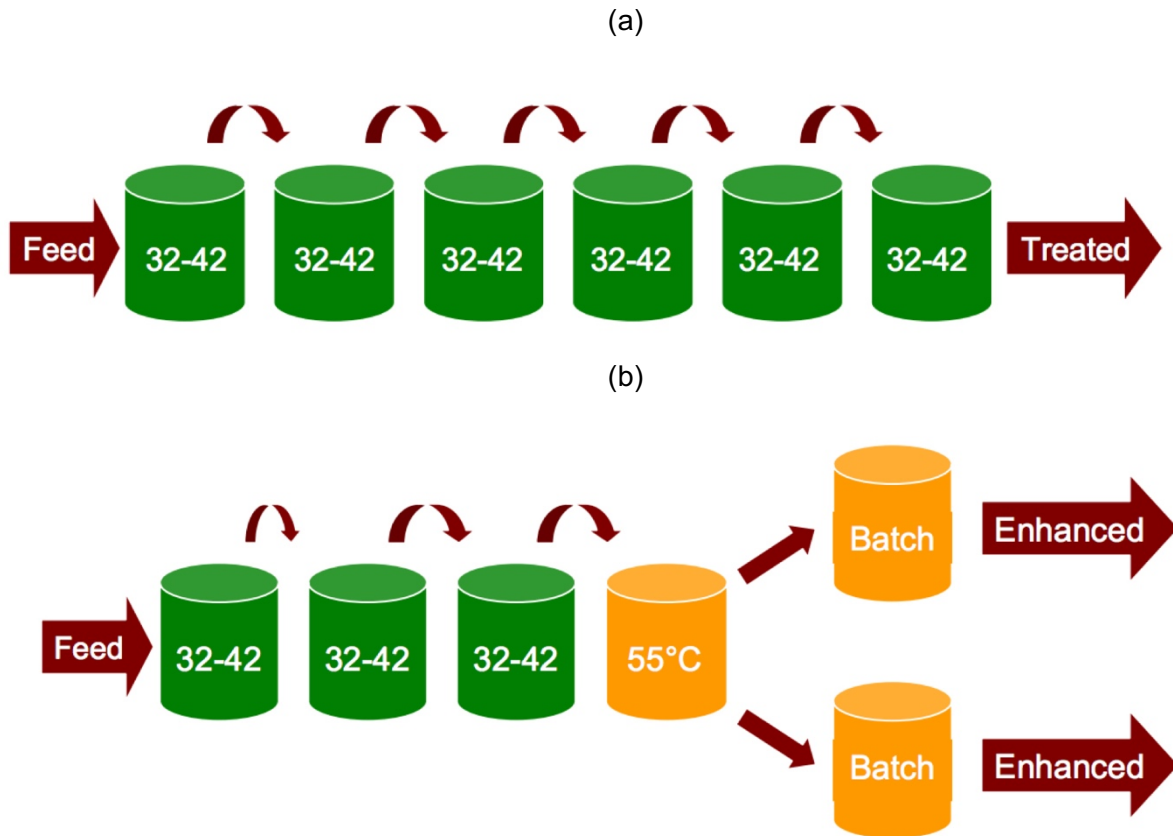


Figure A3-4 Sewage sludge anaerobic digestion by (a) conventional Enzymic Hydrolysis configuration, and (b) Enhanced Enzymic Hydrolysis configuration

Pasteurisation (PST) and autothermal thermophilic aerobic digestion (aTAD)

Alpha Biotherm has developed a prepasteurisation and aTAD pretreatment process designed for disinfection and conditioning pre-thickened sludge of 5.0 to 8.6% DS. In the pasteurisation process, sludge is pumped into the pasteurisation reactor and held at 70 °C and for 60 minutes with adequate mixing. Pasteurised sludge is passed through a heat exchanger to recover heat and to pre-heat the next batch of sludge (Ray, 2013; Terry and Graham, 2004).

Appendix 3-2 Sampling points for the selected sampling sites



Conventional Site 1 feed



Conventional Site 1 Digested



Conventional Site 5 feed



Conventional Site 5 Digested



Conventional Site 31 feed



Conventional Site 31 Digested



Conventional Site 38 raw



Conventional Site 38 feed



Conventional Site 38 Digested



THP Site 4 raw



THP Site 4 feed



THP Site 4 Digested



THP Site 7 raw



THP Site 7 feed



THP Site 7 Digested



EEH Site 2 raw



EEH Site 2 feed



EEH Site 2 Digested



HpH Site 3 raw



HpH Site 3 feed



HpH Site 3 Digested

Appendix 3-3 Summary of the analysis methods used by the external laboratories

pH (SCA, 1997): pH of the sample was measured by a pH meter. For sludge with more than 99% water content: the pH probe was placed in the sample for at least 30 s. For thickened sludge (97-98% water content): the sludge was settled for 1h or centrifuged for 5 min. The supernatant was collected and the pH probe was inserted for at least 30 s.

Total nitrogen, ammonium nitrogen and nitrate nitrogen (Ebeling, 1968; USEPA, 1983): The sample was weighed into a nitrogen (N) free foil parcel and dropped into a hot furnace, flushed with pure oxygen to produce rapid combustion. The combustion products were passed through filters and a thermoelectric cooler to remove water and then collected in a ballast tank and allowed to equilibrate. An aliquot of the gaseous mixture was passed through hot copper (Cu) to remove oxygen (O₂) and reduce nitrogen oxides (NO_x) to dinitrogen (N₂). Carbon dioxide and water were removed by chemical absorption and the remaining N was measured by a thermal conductivity cell. Protein content was calculated from the total N concentration using the multiplication factor 6.25 ((Mariotti and Mirand, 2008).

The determination of NH₄-N and NO₃-N is based on the formation of a diazo compound between NO₂⁻ and sulphanilamide, which couples with N-1-Naphthylethylenediamine dihydrochloride to give a red azo dye. The colour is measured at 540nm. In channel one, nitrate is reduced quantitatively to nitrite by cadmium metal in the form of an open tubular cadmium reactor (OTCR). The nitrite and reduced nitrate are therefore both measured as total oxidised nitrogen (TON). In channel two, nitrite is measured. Nitrate-N was determined by deducting the nitrite figure from the TON. In channel three, ammonium reacts with alkaline hypochlorite and phenol to form indophenol blue. Sodium nitroprusside acts as a catalyst in the formation of indophenol blue which is measured at 640nm. Precipitation of calcium and magnesium hydroxides is eliminated by the addition of a combined potassium sodium tartrate/sodium citrate complexing reagent.

Oil (Fats) (APAH 2005): The Oil content was measured by the continuous extraction of the sample with warm light petroleum ether (boiling range 40 - 60 °C). The solvent was removed by evaporation and the dry oil was weighed. The residual material is boiled in hydrochloric acid to release the bound fat and the digest is filtered with a filter aid and washed until neutral. The fat was retained by the filter paper and filter aid. After drying the residue was also extracted with light petroleum ether in an extraction tube.

Neutral detergent fibre (NDF) (Van Soest, 1991): Starch in the sample was converted to soluble sugars by the action of α-amylase after de-fatting of the sample with petroleum spirit. The residue was boiled with a neutral solution (sodium dodecyl sulfate) and the soluble fraction was separated. The insoluble matter remaining is designated NDF.

Acid detergent fibre (ADF) (Van Soest, 1991): Acid detergent fibre is defined as the organic material which remains insoluble after treatment with an acid detergent solution. The sample was weighed into an Ankom fibre bag and sealed. The sample was de-fatted using petroleum ether and digested with acid detergent (cetrimonium bromide) under controlled conditions. The mass of the resulting residue plus bag following drying was recorded. The bag and remaining residue were ashed at 510 °C and the difference in two mass is designated as ADF.

Acid detergent lignin (ADL) (Van Soest, 1991): Acid detergent removes cellular contents and other acid soluble material which interferes with the lignin determination. The residue from the ADF determination is primarily lignin and cellulose. The cellulose is dissolved using 72% sulphuric acid. The remaining residue consists of lignin and acid-insoluble ash, which was subtracted by ashing the residue at 510 °C.

Cellulose and hemicellulose (Van Soest, 1991): an estimate of the cellulose and hemicellulose content was calculated from the ADF, ADL and NDF fractions using the formulae provided in Table A3-1.

Table A3-1 Fibre content calculation

Substrate	Components:
Neutral detergent fibre	Cellulose + hemicellulose + lignin + mineral ash
Acid detergent fibre	Cellulose + lignin + mineral ash
Acid detergent lignin	lignin + mineral ash
Cellulose	NDF - ADF
hemicellulose	ADF - ADL

Aqua-regia soluble elements analysis (Jackson *et al.*, 1986; EPA, 1996): the sample was digested in an open vessel with concentrated hydrochloric and nitric acid (aqua-regia) using a temperature controlled digestion block. The formation of strong oxidising agents destroys organic matter and breaks down the mineral matrix of the sample. The elements dissolved in the acid were analysed by inductively coupled plasma mass spectrometry (ICP-MS).

Appendix 3-4 User interface

User manual

Green cell: Input box for parameters

Blue cell: Predicted results from the model

Red cell: The input parameter range is the recommended ranges for each parameter, and the cell will automatically turn red if the input value is outside this range. The 90% of data range for different anaerobic digestion process is included as a guidance for the model application, a more reliable prediction may be observed if the model is used within this range.

The Conventional-THP MAD model interface contains five spreadsheets, including biogas yield (BY) and energy balance, temperature, dry solids (DS) feed, hydraulic retention time (HRT), and model validation.

1. BY and energy balance

In the BY and energy balance spreadsheet, the model predicts the changes of BY and net energy balance for the system to switch from the current operational condition to the proposed operational condition.

Step 1: Define the current operational conditions by filling the current digester volume, temperature, HRT, DS feed and BY data into the green input box. The model will calibrate itself based on this condition.

Step 2: Define the proposed operational condition by filling the new temperature, HRT, DS feed concentration. The model will calculate the predicted BY, biogas production and net energy output.

2. Temperature

In the Temperature spreadsheet, the required digestion temperature to achieve a target BY can be predicted based on the input HRT and DS feed.

Step 1: Define the current operational condition by filling the current digester volume, temperature, HRT, DS feed and BY data into the green input box. The model will calibrate itself based on this condition.

Step 2: Input a target BY, and the required digestion temperature to achieve the target BY can be predicted based on the same HRT and DS feed content listed in the current operational conditions in step 1.

3. DS feed

In the DS spreadsheet, the required DS feed to achieve a target BY can be predicted based on different HRT and temperature.

Step 1: Define the current operational condition by filling the current digester volume, temperature, HRT, DS feed and BY data into the green input box. The model will calibrate itself based on this condition.

Step 2: Input a target BY, and the required DS to achieve the target BY can be predicted based on the same HRT and temperature listed in the current operational conditions in step 1.

4. HRT

In the HRT spreadsheet, the required DS to achieve a target BY can be predicted based on different DS and temperature.

Step 1: Define the current operational conditions by filling the current digester volume, temperature, HRT, DS feed and BY data into the green input box. The model will calibrate itself based on this condition.

Step 2: Input a target BY, and the required HRT to achieve the target BY can be predicted based on the same DS and temperature listed in the current operational condition in step 1.

5. Model validation

The model validation section is used to check if the model can accurately capture the changes of digester performance at the specific site.

Input the monthly or weekly average values of DS feed, digester temperature (°C), HRT and observed BY (m^3/t DS) into green cell. Input digester daily total feed volume and observed total biogas production for biogas volume prediction validation

The model can automatically calibrate itself based on the input operational condition and performance, and is set to recalibrated itself every year.

The model automatically calculates the predicted BY and generate a plot of observed biogas yield vs predicted biogas yield; and observed biogas production vs predicted biogas production.

BY and energy balance

Conventional + THP MAD biogas yield and energy balance				
Assest information				
Site				
Total tank Volume (m3)	2000			
Current Operational Conditions				
Digestion condition		Input range	Conventional MAD 90% range	THP MAD 90% range
DS feed%	5.0	DS:1-11 %	2.7 -6.3 %	4.2 - 10.4 %
HRT(days)	20.0	HRT:10-45 days	13 - 35 days	12 - 42 days
Temperature (oC)	38.0	Temperature:28-42 oC	31.6 - 38.9 oC	33.6 - 42.5 oC
Feed Volume (m3/day)	100.0			
Current biogas yield (m3/t DS)	400.0			
Current biogas production volume (m3/day)	2000.0			
		Model calibration factor based on current operational condition and biogas yield 414		
Proposed Operational Conditions		System Energy Balance		
Digestion condition		Energy input difference in digester heating (kWh)		
DS feed%	5.0	0.00		
HRT(days)	15.0			
Temperature (oC)	38.0	Energy output difference in biogas production (kWh)		
Feed Volume (m3/day)	133.3	2423.5		
Predicted		Net energy output (kWh)		
Predicted biogas yield (m3/t DS)	359.6	2423.5		
Predicted biogas production (m3/day)	2397.3			

Biogas yield and energy balance

In the BY and energy balance spreadsheet, the model predicts the changes of BY and net energy balance for the system to switch from the current operational condition to the proposed operational condition.

Step 1: Define the current operational conditions by filling the current digester volume, temperature, HRT, DS feed and BY data into the green input box. The model will calibrate itself based on this condition.

Step 2: Define the proposed operational condition by filling the new temperature, HRT, DS feed concentration. The model will calculate the predicted BY, biogas production and net energy output.

Assuming sludge has the same specific heat capacity as water, and the energy in 1m³ biogas (60% CH₄ content) is equivalent to 22 MJ = 6.1 kWh

Temperature

Conventional + THP MAD temperature				
Assest information				
Site				
Total digester volume (m3)	2000			
Current Operational Conditions				
Digestion condition		Input range	Conventional MAD 90% range	THP MAD 90% range
DS feed%	5.0	DS:1-11 %	2.7 -6.3 %	4.2 - 10.4 %
HRT(days)	20.0	HRT:10-45 days	13 - 35 days	12 - 42 days
Temperature (oC)	35.0	Temperature:28-42 oC	31.6 - 38.9 oC	33.6 - 42.5 oC
Feed Volume (m3/day)	100.0			
Current biogas yield (m3/t DS)	300.0			
Current biogas production volume (m3/day)	1500.0			
		Model calibration factor based on current operational condition and biogas yield 336	Temperature In the Temperature spreadsheet, the required digestion temperature to achieve a target BY can be predicted based on the input HRT and DS feed. Step 1: Define the current operational condition by filling the current digester volume, temperature, HRT, DS feed and BY data into the green input box. The model will calibrate itself based on this condition. Step 2: Input a target BY, and the required digestion temperature to achieve the target BY can be predicted based on the same HRT and DS feed content listed in the current operational conditions in step 1.	
Target biogas yield (m3/t DS)	350			
Predicted temperature (oC)	42.3			
Predicted biogas production (m3/day)	1750.0			

DS

Conventional + THP MAD DS feed				
Assest information				
Site				
Total digester volume (m3)	2000			
Current Operational Conditions				
Digestion condition		Input range	Conventional MAD 90% range	THP MAD 90% range
DS feed%	5.0	DS:1-11 %	2.7 - 6.3 %	4.2 - 10.4 %
HRT(days)	20.0	HRT:10-45 days	13 - 35 days	12 - 42 days
Temperature (oC)	35.0	Temperature:28-42 oC	31.6 - 38.9 oC	33.6 - 42.5 oC
Feed Volume (m3/day)	100.0			
Current biogas yield (m3/t DS)	300.0			
Current biogas production volume (m3/day)	1500.0			
		Model calibration factor based on current operational condition and biogas yield 336		
Target biogas yield (m3/t DS)	350			
Predicted DS feed%	4.0			
Predicted biogas production (m3/day)	1389.4			

Dry solids

In the DS spreadsheet, the required DS feed to achieve a target BY can be predicted based on different HRT and temperature.

Step 1: Define the current operational condition by filling the current digester volume, temperature, HRT, DS feed and BY data into the green input box. The model will calibrate itself based on this condition.

Step 2: Input a target BY, and the required DS to achieve the target BY can be predicted based on the same HRT and temperature listed in the current operational conditions in step 1.

HRT

Conventional + THP MAD HRT				
Assest information				
Site				
Total digester volume (m3)	2000			
Current Operational Conditions				
Digestion condition		Input range	Conventional MAD 90% range	THP MAD 90% range
DS feed%	5.0	DS:1-11 %	2.7 - 6.3 %	4.2 - 10.4 %
HRT(days)	20.0	HRT:10-45 days	13 - 35 days	12 - 42 days
Temperature (oC)	35.0	Temperature:28-42 oC	31.6 - 38.9 oC	33.6 - 42.5 oC
Feed Volume (m3/day)	100.0			
Current biogas yield (m3/t DS)	300.0			
Current biogas production volume (m3/day)	1500.0			
		Model calibration factor based on current operational condition and biogas yield 336		
Target biogas yield (m3/t DS)	350			
Predicted HRT (days)	28.6			
Predicted feed Volume (m3/day)	70.0			
Predicted biogas production (m3/day)	1225.8			

HRT

In the HRT spreadsheet, the required DS to achieve a target BY can be predicted based on different DS and temperature.

Step 1: Define the current operational conditions by filling the current digester volume, temperature, HRT, DS feed and BY data into the green input box. The model will calibrate itself based on this condition.

Step 2: Input a target BY, and the required HRT to achieve the target BY can be predicted based on the same DS and temperature listed in the current operational condition in step 1.

Model validation

Date	DS Feed (%)	HRT (days)	Digester Temperature (°C)	Digester Feed Total Volume (m³/d)	Observed Total Biogas Production (m³/d)	Observed BY (m³/t DS)	Predicted value without site factor	Difference between Predicted & Observed	Predicted BY (m³/t DS)	Predicted Total Biogas Production (m³/d)	Site factor
01/2011 Average							#NUM!		#NUM!	#NUM!	
02/2011 Average	5.1	15.5	32.9	287.0	5941.0		-92.93	92.93	234.75	3436.01	
03/2011 Average	4.7	15.1	37.0	291.9	5582.5	413.2	-45.64	458.81	282.04	3857.51	
04/2011 Average	4.9	13.3	37.4	328.5	4688.9	323.3	-68.65	391.92	259.03	4135.53	
05/2011 Average	4.7	16.0	37.4	308.0	4803.7	307.8	-36.84	344.65	250.83	4226.24	
06/2011 Average	4.6	13.1	37.6	330.2	4737.9	323.1	-58.69	381.84	268.99	4123.01	
07/2011 Average	4.4	14.3	37.4	305.1	3622.2	274.6	-35.64	310.25	292.04	3931.25	
08/2011 Average	4.0	17.0	38.4	267.9	3358.8	326.8	-15.22	311.61	342.90	3661.42	
09/2011 Average	4.3	19.2	38.9	241.7	3705.5	343.4	-21.72	321.68	349.39	3551.08	
10/2011 Average	4.2	18.6	36.6	284.4	3685.5	330.9	2.00	328.91	329.68	3974.05	
11/2011 Average	4.1	15.4	35.4	276.6	3762.8	322.9	-25.43	348.29	302.24	3460.31	
12/2011 Average	4.2	16.3	33.2	300.9	3314.3	271.7	-41.90	313.57	285.78	3652.77	327.68
01/2012 Average	4.6	17.3	33.5	287.4	3893.6	272.2	-49.78	322.01	248.04	3283.98	
02/2012 Average	5.1	29.3	34.3	207.0	3239.2	334.4	10.87	323.58	328.87	3408.68	
03/2012 Average	4.8	18.0	35.2	283.3	4041.5	301.5	-38.05	339.52	259.76	3499.08	
04/2012 Average	4.2	13.3	35.2	328.9	4100.5	293.9	-47.75	341.67	250.06	3426.84	
05/2012 Average	4.4	12.2	34.9	346.8	4096.3	258.8	-72.31	331.12	225.50	3401.57	
06/2012 Average	4.8	16.9	35.2	276.2	3221.3	254.7	-47.00	301.71	250.81	3301.80	
07/2012 Average	4.8	18.6	33.1	238.7	1042.8	146.0	-52.28	198.31	245.53	2815.04	
08/2012 Average	5.1	28.1	36.0	212.7	1619.3		#NUM!		#NUM!	#NUM!	
09/2012 Average	5.0	19.6	32.9	307.0	2219.3	135.0	-54.88	189.91	242.94	3728.66	
10/2012 Average	4.9	15.8	34.1	289.9	3099.7	212.8	-69.39	282.15	228.42	3222.39	
11/2012 Average	4.6	19.6	34.5	270.0	3675.0	302.6	-24.66	327.22	273.15	3398.29	
12/2012 Average	4.9	22.7	31.9	245.8	3510.6	278.2	-40.54	318.73	257.27	3122.18	297.81
01/2013 Average	5.1	34.7	32.3	150.8	2603.3	316.4	17.08	299.28	334.84	2563.68	
02/2013 Average	5.0	27.2	29.0	194.6	2392.5	269.2	-34.48	303.72	282.78	2756.14	
03/2013 Average	5.0	23.0	27.5	173.2	2120.9	230.2	-79.16	309.33	238.10	2046.98	
04/2013 Average	4.7	24.9	29.5	200.4	2605.7	271.2	-40.34	311.51	276.92	2634.41	
05/2013 Average	4.8	18.3	35.1	280.3	4773.7	341.3	-36.48	377.81	280.77	3746.92	
06/2013 Average	4.6	23.2	37.4	239.6	3944.5	369.0	19.20	349.83	336.45	3713.77	
07/2013 Average	4.5	15.9	38.6	305.8	5064.8	341.8	-17.45	359.24	299.80	4111.41	
08/2013 Average	4.6	14.6	38.1	334.9	3969.6	304.1	-37.47	341.58	279.78	4310.56	
09/2013 Average	4.7	13.8	38.2	315.9	3797.8	249.3	-48.26	297.56	268.99	3963.34	
10/2013 Average	4.9	14.0	37.0	328.1	3203.6	209.7	-65.80	275.53	251.45	4031.59	
11/2013 Average	5.3	14.3	33.3	316.7	2939.7	170.3	-110.11	280.39	207.14	3477.31	
12/2013 Average	5.3	17.0	34.1	278.7	3426.0	222.6	-78.62	301.25	238.64	3519.33	
01/2014 Average	5.3	22.4	34.6	226.4	3247.4	278.9	-36.76	315.65	253.16	3060.68	317.25
02/2014 Average	5.4	32.3	36.5	176.2	2901.7	322.3	25.46	292.88	319.38	3010.94	
03/2014 Average	5.3	18.1	35.5	256.2	3627.6	268.4	-57.31	325.75	232.61	3128.38	
04/2014 Average	5.1	18.7	36.1	256.2	3231.0	253.1	-39.59	292.74	250.33	3245.20	
05/2014 Average	5.1	15.1	35.0	303.2	3562.7	226.6	-80.16	306.73	209.76	3243.12	
06/2014 Average	5.1	16.6	37.8	300.8	3419.6	228.3	-45.07	273.39	244.86	3727.89	
07/2014 Average	5.1	15.6	36.3	311.1	3460.0	212.1	-67.20	279.34	222.72	3547.95	
08/2014 Average	5.1	13.3	37.7	328.9	3426.0	206.0	-78.64	284.64	211.28	3557.98	
09/2014 Average	5.1	15.3	38.4	307.5	3405.6	221.6	-52.36	274.00	237.56	3696.30	
10/2014 Average	5.1	15.3	37.9	283.5	3482.4	234.8	-55.94	290.77	233.98	3357.95	
11/2014 Average	5.2	18.0	38.6	302.6	3420.3	223.5	-31.35	254.89	258.58	4030.18	
12/2014 Average	5.1	17.9	36.8	265.4	3311.3	247.6	-40.74	288.29	249.19	3339.20	
01/2015 Average	5.1	16.0	35.4	309.4	3325.3	211.2	-68.04	275.23	246.44	3875.44	
02/2015 Average	5.1	13.8	34.1	318.9	3095.2	192.0	-100.39	292.35	214.09	3491.95	
03/2015 Average	5.0	13.4	36.9	354.2	3953.3	216.1	-80.64	296.78	233.84	4173.23	
04/2015 Average	5.1	14.7	38.3	307.9	4416.1	283.7	-99.47	343.13	255.02	4011.46	
05/2015 Average	5.0	17.3	38.3	273.4	3603.8	267.5	-33.19	300.66	281.29	3852.04	
06/2015 Average	5.1	22.8	34.6	244.0	3570.2	289.3	-23.95	313.22	290.53	3593.05	
07/2015 Average	5.1	17.2	34.9	252.5	3016.1	218.8	-63.12	281.89	251.37	3243.39	
08/2015 Average	16.1	36.7	291.3	3290.7			#NUM!		#NUM!	#NUM!	
09/2015 Average	4.5	23.2	37.1	236.9	3537.0	414.8	22.86	391.96	337.35	3582.45	
10/2015 Average	3.7	16.0	37.1	277.8	3402.1	346.0	18.08	327.96	332.56	3412.10	
11/2015 Average	4.3	16.8	38.5	288.1	3910.1	324.5	-2.08	326.60	312.40	3898.24	
12/2015 Average	5.0	19.0	37.1	285.0	4144.3	278.2	-27.27	305.51	287.21	4071.78	
01/2016 Average	3.4	17.7	36.1	219.4	2808.9	368.6	40.96	327.68	388.14	2902.07	
02/2016 Average	4.8	15.9	37.1	268.8	3962.9	299.4	-43.66	343.06	303.52	3915.77	
03/2016 Average	4.1	14.8	37.9	293.6	4580.4	374.2	-9.49	383.70	337.69	4053.51	

Conventional + THP MAD Model Validation

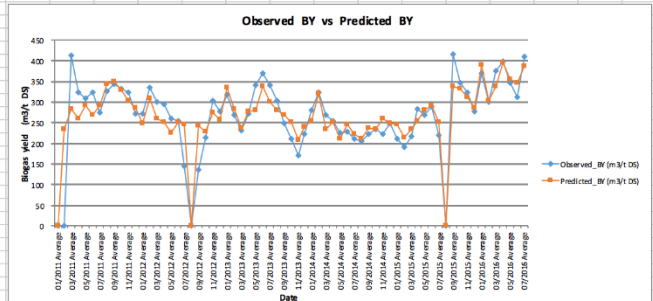
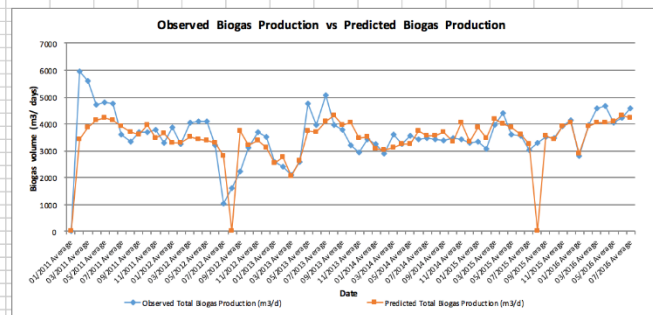
Model validation

The model validation spreadsheet is used to check if the model can accurately capture the changes in digester performance at a specific site.

Input the monthly or weekly average values of DS feed, digester temperature, HRT and observed BY into the respective green cells. Input the digester daily total feed volume and observed total BP to validate the predicted biogas volume.

The model will automatically calibrate based on the entered operational conditions and performance, and is set to recalibrate annually (Note: for weekly data, the calibration frequency needs to be adjusted manually).

The model automatically calculates the predicted BY and generates a plot of the observed BY vs predicted BY and the observed BP vs predicted BP.



Appendix 4-1 Summary of site mean values of the principal parameters collected by all the water companies for managing conventional MAD process control

		BY (m ³ /t DS)		DS Input (%)		HRT (days)		Temperature (°C)	
		Mean	SD	Mean	SD	Mean	SD	Mean	SD
Company 1	Site1	639.1	280.3	4.1	0.7	32.8	5.3	36.3	0.9
	Site2	605.3	190.7	3.0	0.5	22.6	4.3	36.0	1.1
	Site3	589.6	165.4	5.3	1.5	31.3	9.1	35.4	1.5
	Site4	580.1	193.2	3.7	0.6	20.4	6.4	35.9	3.0
	Site5	562.5	187.7	4.3	1.8	13.9	2.5	36.4	1.1
	Site6	540.8	150.0	3.3	0.6	20.6	6.1	36.4	1.0
	Site7	532.6	174.4	3.1	0.5	20.6	4.0	34.1	1.5
	Site8	514.2	174.9	3.0	0.7	21.5	4.3	36.0	1.1
	Site9	498.5	213.5	4.3	1.3	28.1	6.6	36.0	1.1
	Site10	469.9	121.6	4.6	0.7	25.2	6.1	34.5	1.7
	Site11	457.2	172.6	4.5	1.6	25.5	7.0	35.9	1.8
	Site12	456.1	119.1	3.1	0.9	18.2	4.8	34.4	1.2
	Site13	450.8	77.4	4.4	0.8	16.2	3.8	36.4	1.0
	Site14	438.7	132.5	4.1	0.8	27.6	4.6	36.2	1.0
	Site15	435.6	176.8	5.4	0.8	24.3	5.6	36.1	0.8
	Site16	400.5	141.6	4.3	1.1	25.0	7.5	32.9	3.2
	Site17	367.2	105.5	5.3	0.9	26.7	5.6	37.1	0.5
	Site18	355.7	55.3	4.8	0.9	37.6	9.0	35.6	1.6
	Site19	339.9	98.5	4.7	0.6	25.0	3.5	36.0	0.7
	Site20	336.8	55.6	4.5	0.8	19.4	3.5	36.5	0.8
	Site21	326.4	60.4	4.3	1.0	16.0	2.3	35.3	1.8
	Site22	323.3	138.0	4.8	0.9	25.4	6.9	36.5	1.1
	Site23	292.2	160.5	3.8	0.8	19.8	5.7	36.7	2.5
	Site24	267.7	80.9	5.2	1.0	26.1	4.3	35.8	0.6
	Site25	218.6	128.7	4.1	1.0	15.8	6.3	32.5	2.8
	Site26	140.4	174.7	3.5	0.9	27.1	5.5	35.2	1.8
Company 2	Site27	678.1	163.3	4.4	0.9	27.5	5.1	38.8	1.3
	Site28	443.7	106.3	4.5	0.6	20.8	3.7	35.8	2.0
	Site29	407.7	118.3	5.7	1.3	22.9	5.1	38.2	1.9
	Site30	393.0	108.9	4.4	0.9	18.1	3.5	36.8	3.1
	Site31	380.0	50.9	4.2	0.5	22.5	2.5	37.1	1.2
	Site32	378.2	121.7	4.8	0.6	13.9	1.9	37.6	0.8
	Site33	345.8	45.3	5.0	0.3	20.1	2.8	36.7	2.0
	Site34	333.1	41.8	4.7	0.4	14.2	1.8	36.4	1.4
	Site35	330.9	73.7	3.4	0.4	20.7	3.6	36.1	2.0
	Site36	324.2	47.2	4.2	0.3	20.7	3.5	37.8	1.0
	Site37	305.4	59.9	4.8	0.8	22.2	5.2	34.4	2.8
	Site38	294.6	39.3	5.7	0.6	18.4	3.5	40.9	2.2
	Site39	293.0	133.8	4.6	0.7	24.6	6.0	37.1	1.6
	Site40	284.4	59.6	3.4	0.8	21.6	3.4	37.0	1.2
	Site41	283.0	100.0	5.3	1.0	18.0	4.9	35.5	2.3
	Site42	282.5	63.4	4.7	0.5	17.9	4.5	35.9	2.4

Appendix 4-1 continued									
	Site43	219.0	89.8	4.9	0.7	16.5	3.0	36.9	2.0
	Site44	181.2	70.9	6.3	1.0	18.4	4.4	34.9	3.3
Company 3	Site45	568.8	120.7	4.5	0.7	22.3	4.5	35.0	1.6
	Site46	544.4	202.1	3.8	0.9	19.6	4.8	31.2	1.9
	Site47	522.4	160.1	3.8	0.7	29.5	7.5	36.0	1.0
	Site48	302.4	109.5	4.6	1.0	18.3	3.0	36.6	1.0
	Site49	247.7	7.3	3.7	0.4	23.6	3.5	36.4	0.6
	Site50	225.8	106.1	4.4	0.9	19.0	8.5	33.5	2.8
Company 4	Site51	702.9	171.3	2.8	0.6	20.7	5.6	35.6	2.0
	Site52	623.6	172.2	5.7	0.4	18.2	5.2	36.3	0.4
	Site53	550.2	36.9	5.3	0.6	20.4	1.9	36.4	0.1
	Site54	528.8	210.7	3.6	0.6	17.9	8.9	34.3	1.8
	Site55	517.1	124.2	5.0	0.6	25.4	6.6	33.1	2.3
	Site56	514.6	198.4	3.5	0.5	18.0	6.9	35.9	0.5
	Site57	498.7	146.4	5.1	1.3	17.0	5.0	35.6	2.2
	Site58	450.6	268.0	5.4	0.5	15.9	5.2	34.1	2.3
	Site59	419.9	102.1	4.1	0.7	16.4	4.5	34.6	2.0
	Site60	404.4	138.5	5.6	0.7	22.6	5.8	34.9	1.7
	Site61	393.4	106.6	5.0	0.6	16.0	2.7	35.2	1.2
	Site62	370.2	48.3	5.7	0.3	14.5	3.2	37.5	0.4
	Site63	358.8	164.3	6.0	0.4	16.3	2.6	34.6	1.7
	Site64	344.3	136.1	4.9	0.9	25.2	6.3	35.1	0.8
	Site65	335.2	128.9	3.0	0.6	14.7	1.2	36.3	1.9
	Site66	258.6	107.8	5.7	1.0	18.9	6.0	34.5	1.6
Overall	Overall	401.8	178.4	4.5	1.1	21.3	6.9	35.8	2.2
	BY over 500								
	BY 300 - 500								
	BY below 300								

BY- biogas yield; DS feed – dry solids feed; HRT – hydraulic retention time; SD – standard deviation. Note: Company 5 did not operate any conventional MAD sites

Appendix 4-2 Summary of overall site mean values of EY and BY for advanced MAD

	Type	Company	Site	EY (KWh/t DS)		BY (m³/t DS)	
				Mean	SD	Mean	SD
THP	Veolia THP	Company 2	THP site 1	725.1	198.0	299.9	78.7
		Company 4	THP site 2	932.4	175.2	557.9	150.7
	Cambi THP	Company 2	THP site 3	1039.4	350.1	389.2	186.6
			THP site 4	955.9	200.9	414.0	90.8
			THP site 5	815.6	125.0	501.0	172.2
			THP site 6	918.2	95.8	452.1	58.2
		Company 5	THP site 7	857.1	152.3	/	/
		Company 4	THP site 8	739.4	118.2	/	/
	THP site overall			859.7	193.7	447.8	152.9
Enzymic	EEH	Company 5	EEH site 1	741.2	176.7	/	/
			EEH site 2	845.1	103.7	/	/
			EEH site 3	727.2	233.8	/	/
		Company 3	EEH site 4	845.0	265.5	378.1	151.6
			EEH site 5	703.4	291.9	456.7	258.8
	EH	Company 3	EH site 1	878.1	609.7	546.5	106.9
	HpH	Company 5	HpH site 1	1016.3	401.2	/	/
			HpH site 2	1290.3	181.4	/	/
			HpH site 3	1248.1	263.5	/	/
			HpH site 4	1118.6	433.2	/	/
	Enzymic site overall			921.0	339.7	482.4	204.3
	Advanced site overall			888.6	273.9	456.4	167.4

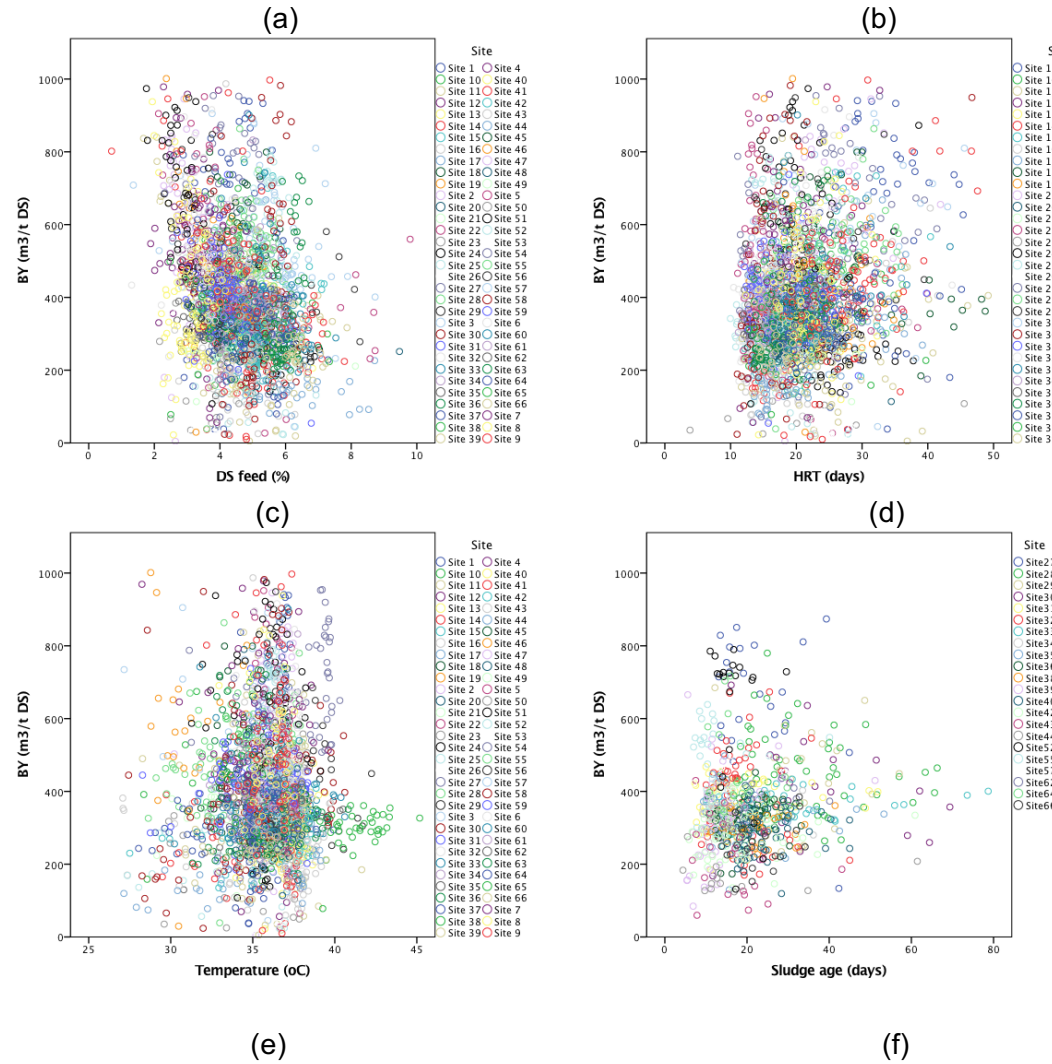
EY - electricity yield; BY - biogas yield; THP - thermal hydrolysis process; EEH - enhanced enzymic hydrolysis; EH - enzymic hydrolysis; HpH - heating pasteurisation hydrolysis; SD - standard deviation

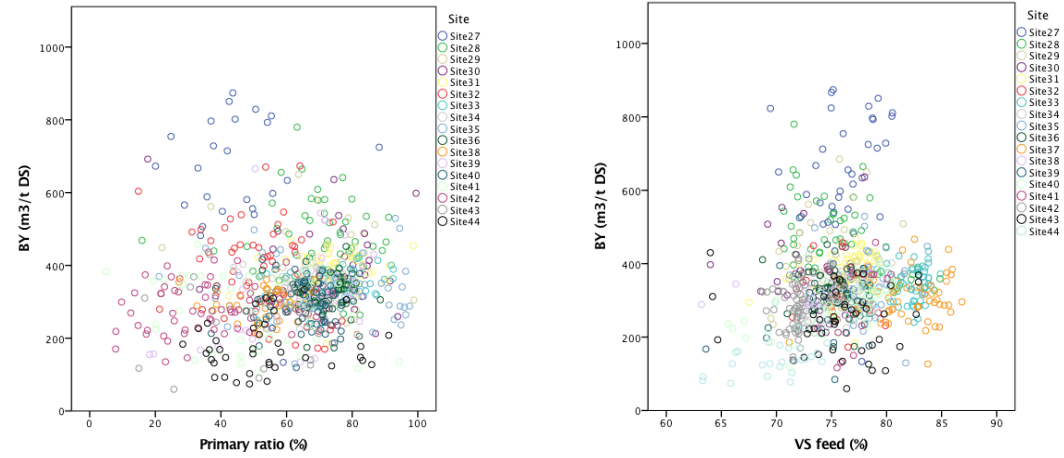
Appendix 4-3 Summary of overall site mean values of principal parameters collected for managing advanced MAD process control

Type	Company	Site	DS feed (%)		VS feed (%)		HRT (days)		Temperature (°C)		pH feed		VFA feed (mg/l)	
			Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Veolia THP	Company 2	THP site 1	6.9	2.1	72.4	2.3	32.4	10.4	37.3	1.7	/	/	/	/
	Company 4	THP site 2	9.6	0.7	/	/	28.5	12.2	36.7	2.5	/	/	/	/
Cambi THP	Company 2	THP site 3	8.7	1.2	70.1	5.6	14.5	4.5	38.2	7.4	/	/	/	/
		THP site 4	9.1	2.0	73.9	2.6	16.8	5.0	40.1	3.8	/	/	/	/
		THP site 5	4.6	0.5	83.1	3.4	14.1	2.1	36.2	2.1	/	/	/	/
		THP site 6	8.7	0.5	84.4	1.0	25.1	4.3	40.1	0.6	/	/	/	/
	Company 5	THP site 7	9.1	0.7	/	/	20.7	4.1	41.5	1.0	/	/	/	/
	Company 4	THP site 8	9.6	0.4	/	/	19.8	5.0	41.4	0.5	/	/	/	/
THP site overall			7.7	2.2	78.9	6.3	21.0	8.9	38.6	3.5	/	/	/	/
EEH	Company 5	EEH site 1	3.9	0.6	/	/	14.3	3.2	36.8	1.1	6.0	0.1	3957.0	613.1
		EEH site 2	6.6	0.4	/	/	14.7	2.5	39.2	0.7	/	/	6874.4	804.0
		EEH site 3	4.4	0.4	/	/	21.6	3.7	39.0	0.5	6.2	0.2	3981.8	329.5
	Company 3	EEH site 4	5.3	0.7	71.9	2.5	20.5	2.0	33.8	1.9	5.5	0.2	/	/
		EEH site 5	2.8	0.1	73.4	4.5	25.3	8.2	36.2	2.1	/	/	/	/
EH	Company 3	EH site 1	4.4	1.0	75.3	2.2	20.9	7.8	33.8	4.7	5.4	0.3	/	/
HpH	Company 5	HpH site 1	6.4	0.8	/	/	23.9	3.3	37.4	1.0	6.4	0.6	4969.5	1449.7
		HpH site 2	5.7	0.5	/	/	22.2	3.9	39.3	1.6	6.4	0.4	8825.5	1038.3
		HpH site 3	6.7	1.0	/	/	25.8	6.6	38.7	0.6	5.8	0.1	7085.5	935.2
		HpH site 4	6.5	0.5	/	/	17.1	7.5	37.8	0.7	6.0	0.2	7910.0	827.0
Enzymic site overall			5.3	1.3	74.0	3.7	23.0	11.2	37.1	2.5	6.0	0.5	5626.8	2125.4
Advanced site overall			6.5	2.2	77.2	6.0	22.0	10.1	37.9	3.2	6.0	0.5	5626.8	2125.4

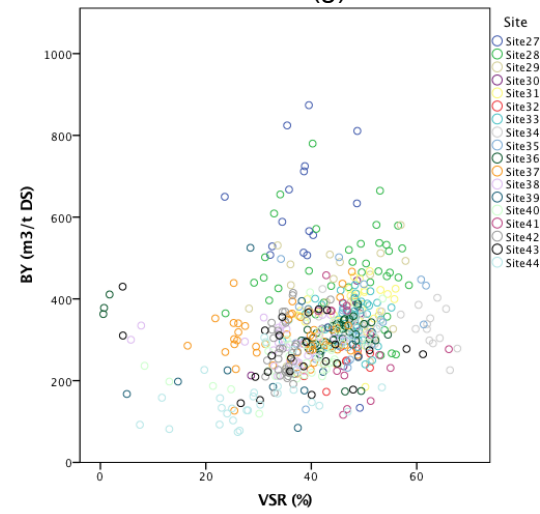
DS - dry solids; VS - volatile solids; HRT - hydraulic retention time; VFA - volatile fatty acids; THP - thermal hydrolysis process; EEH - enhanced enzymic hydrolysis; EH - enzymic hydrolysis; HpH - heating pasteurisation hydrolysis; SD - standard deviation

Appendix 4-4 Scatter plots for conventional MAD BY against: (a) DS feed (%), (b) HRT (days), (c) temperature (°C), (d) sludge age (days), (e) primary SAS ratio (%), (f) VS feed (%), (g) VSR (%)

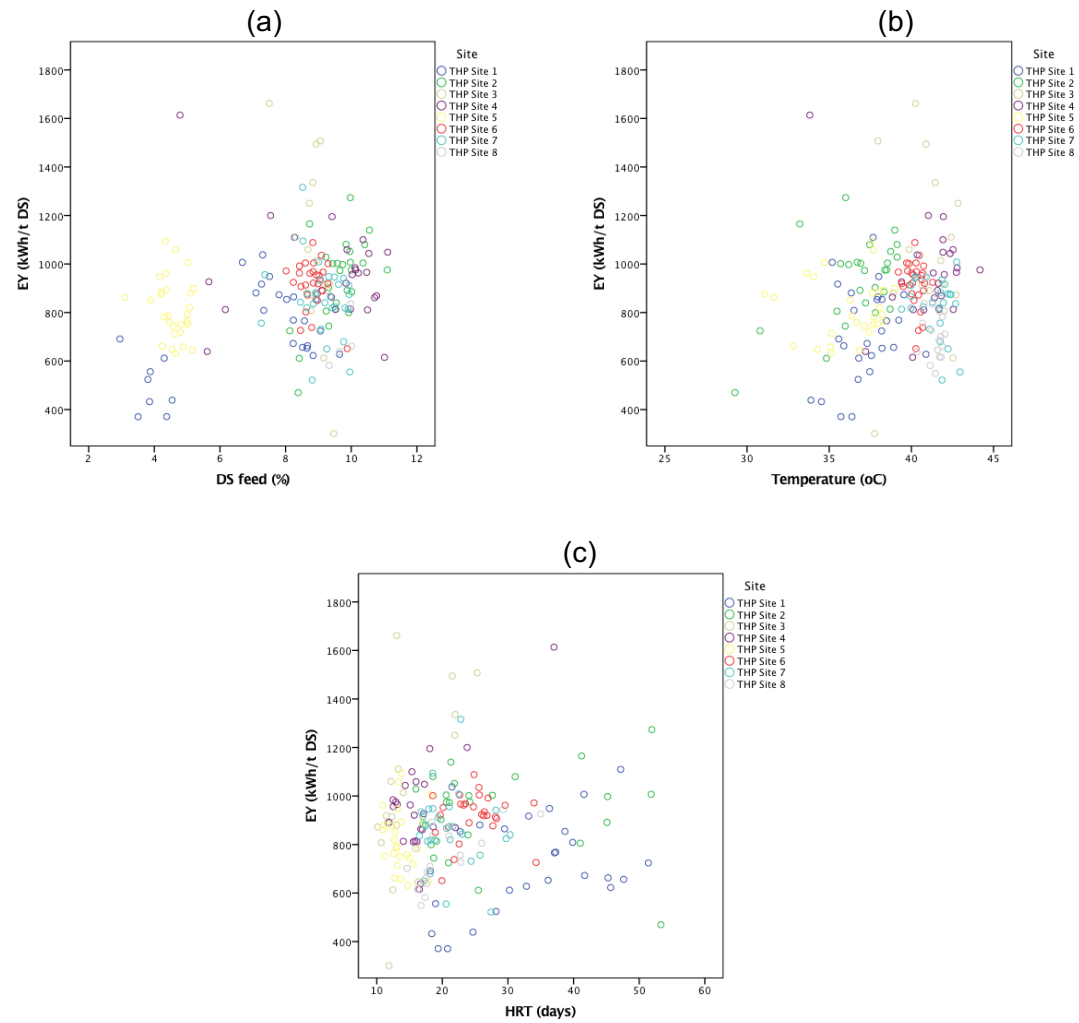




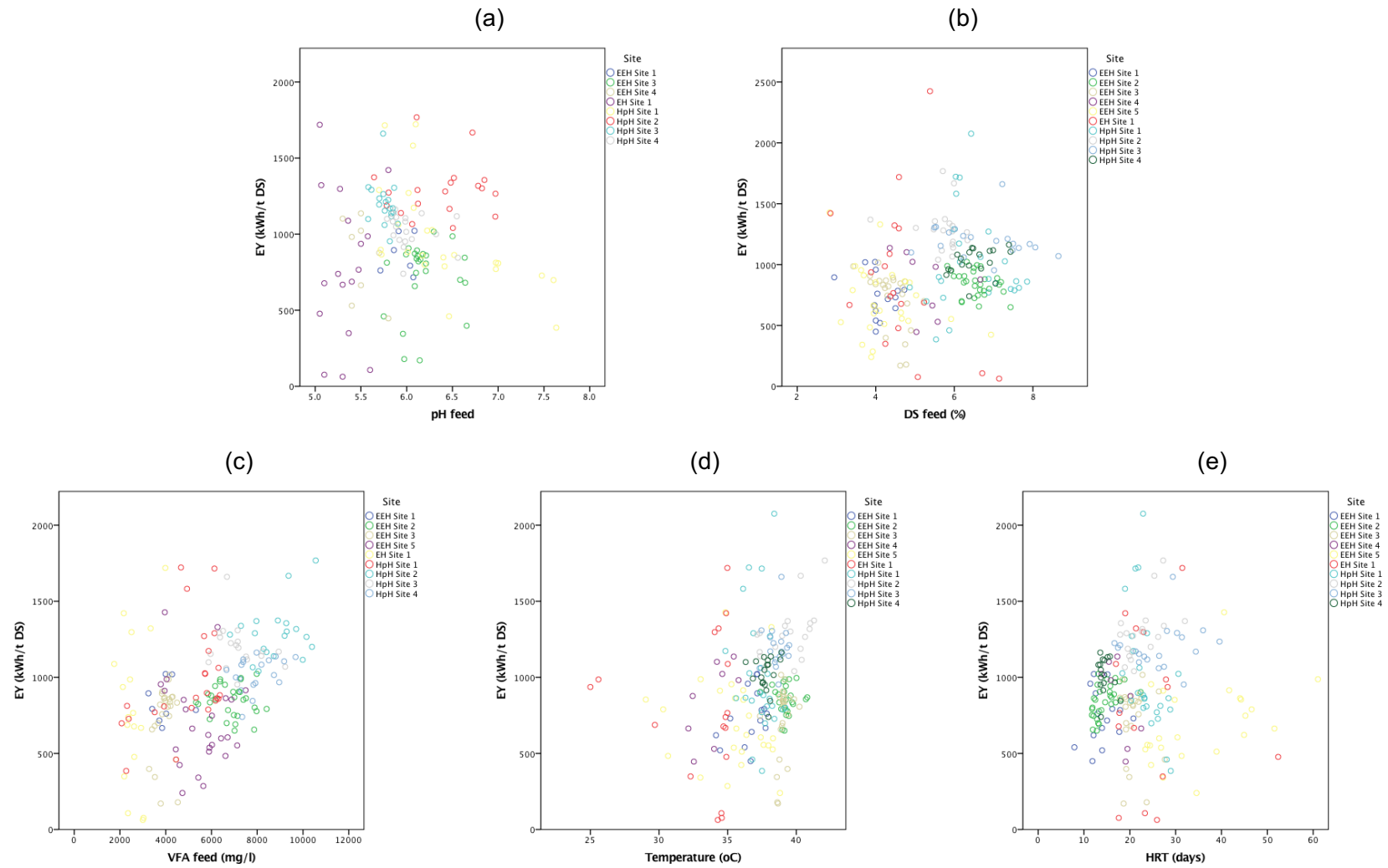
(g)



Appendix 4-5 Scatter plots for THP MAD EY against: (a) DS feed (%), (b) Temperature (°C), (c) HRT (days)

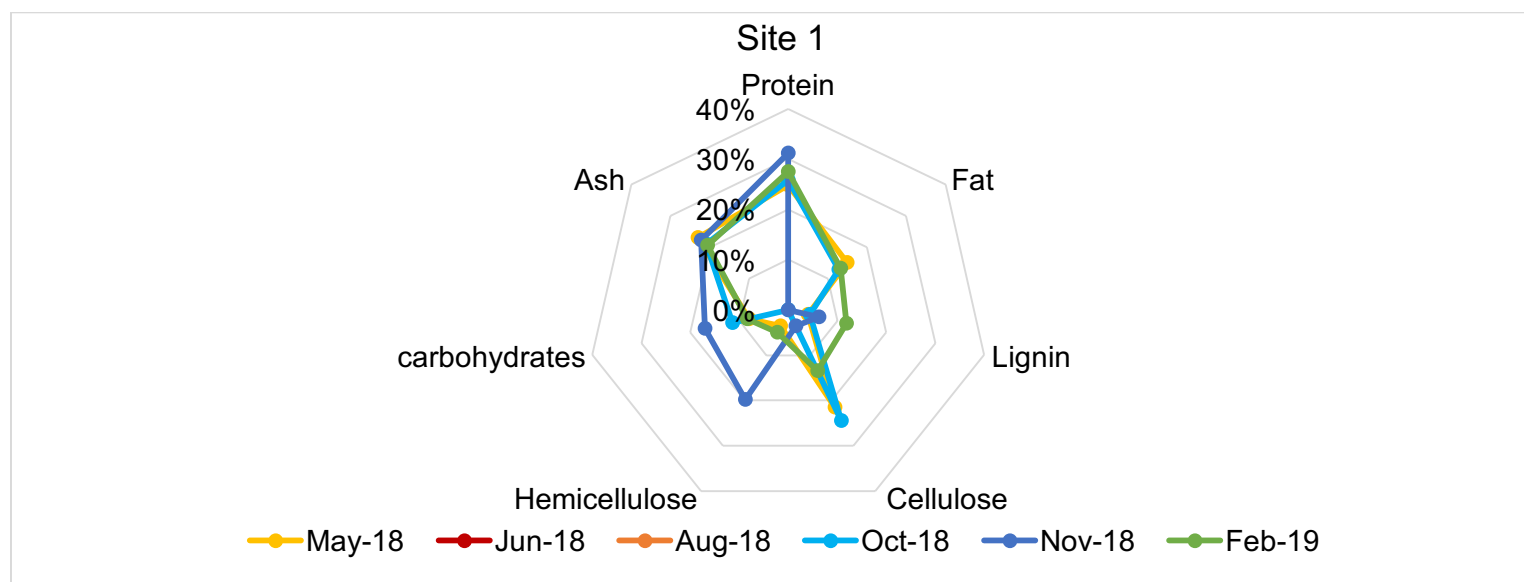


Appendix 4-6 Scatter plots for enzymic MAD EY against: (a) pH, (b) DS feed (%), (c) VFA feed (mg/l), (d) temperature (°C), (e) HRT (days)

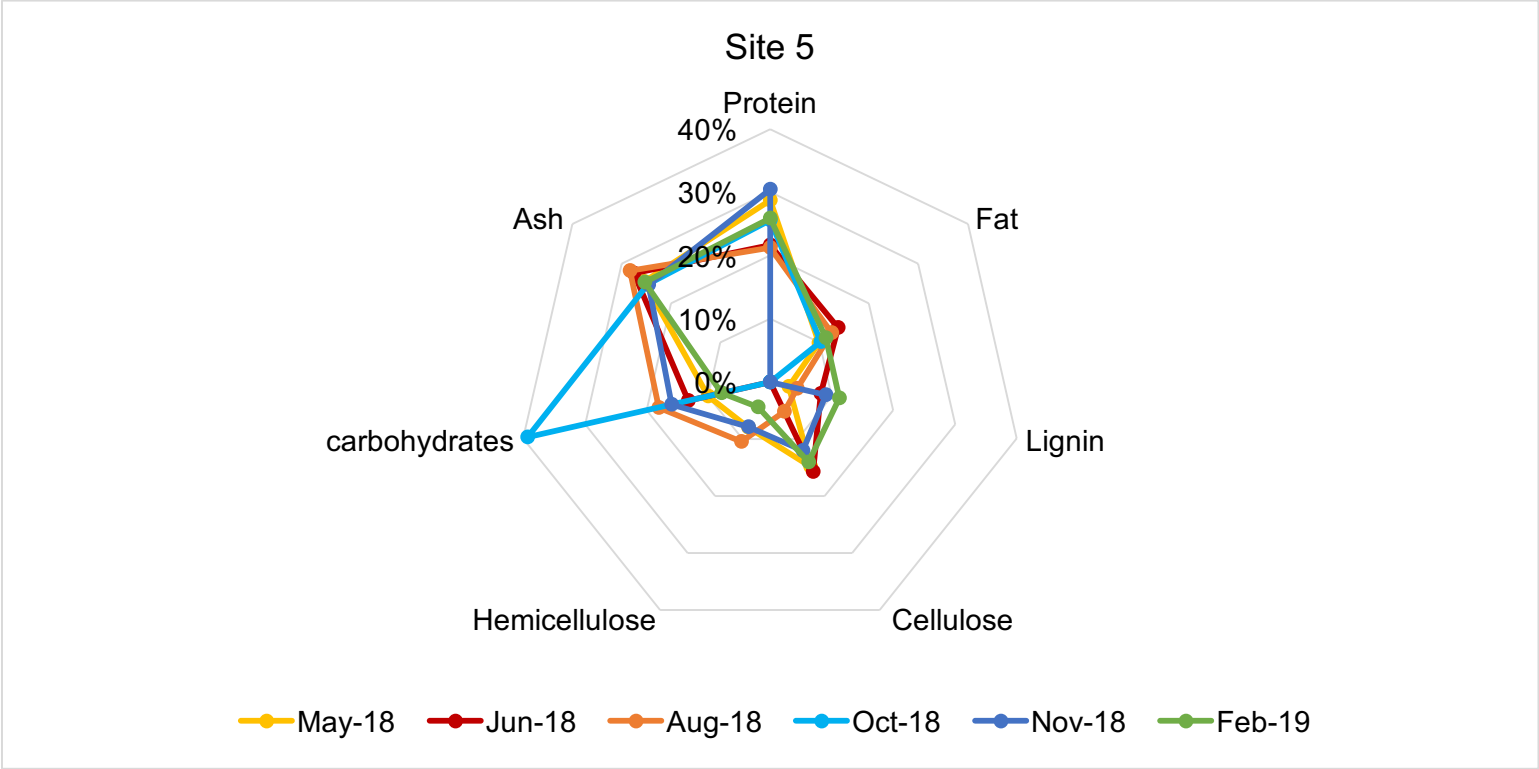


Appendix 7-1 Composition of organic and ash fractions in sludge feed (% DS basis) for the selected sampling sites at different sampling times

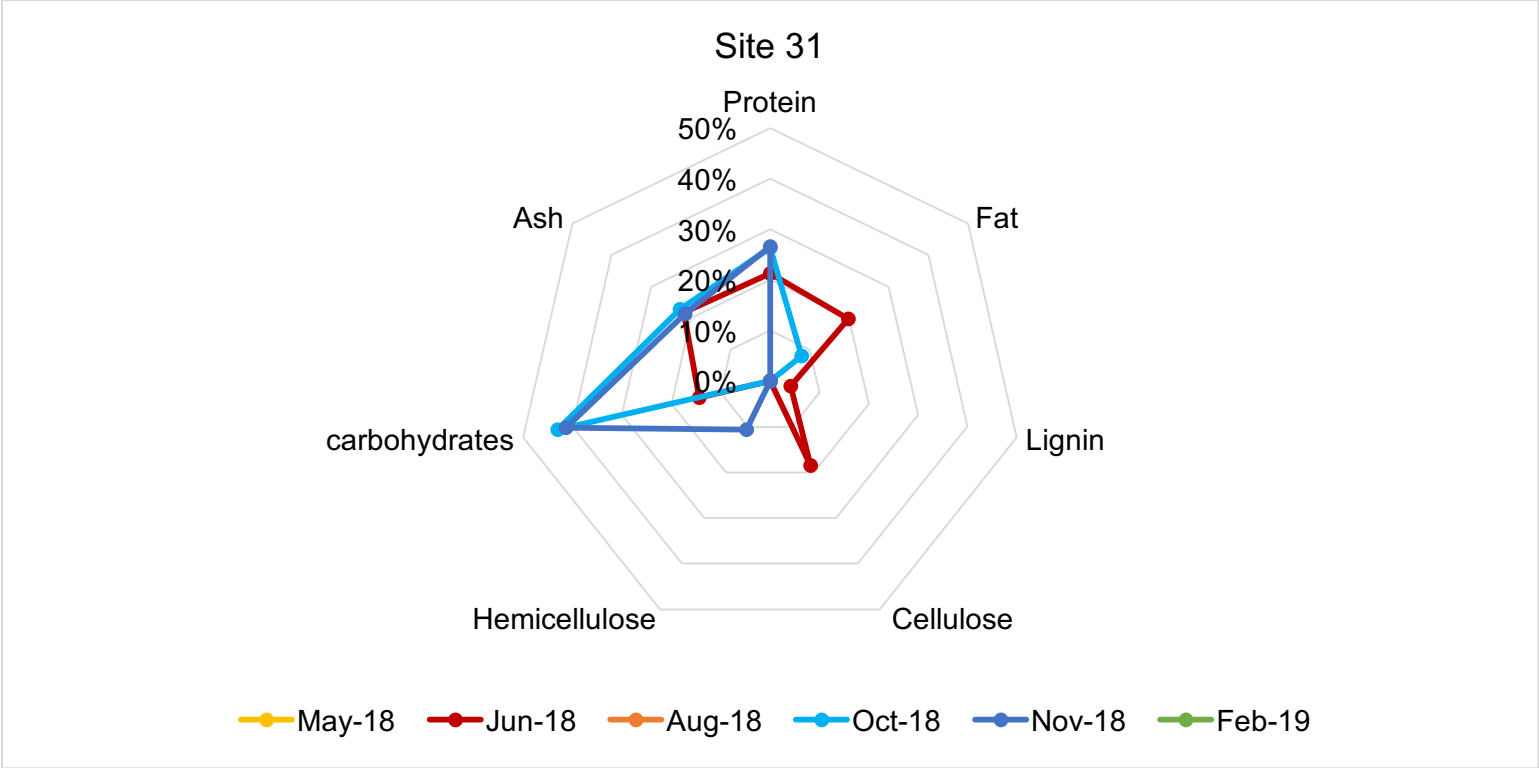
Conventional Site 1	DS	VS	Protein	Fat	Lignin	Cellulose	Hemicellulose	Carbohydrates	Ash
May-18	4.5	77.0	24.8	15.0	4.0	21.5	3.6	8.0	23.0
Jun-18									
Aug-18									
Oct-18	3.6	79.0	25.9	12.9	4.4	24.4	0.0	11.5	21.0
Nov-18	5.4	77.8	31.1	0.0	6.3	3.5	19.8	17.0	22.2
Feb-19	3.6	79.5	27.5	13.3	11.9	13.5	5.0	8.4	20.5



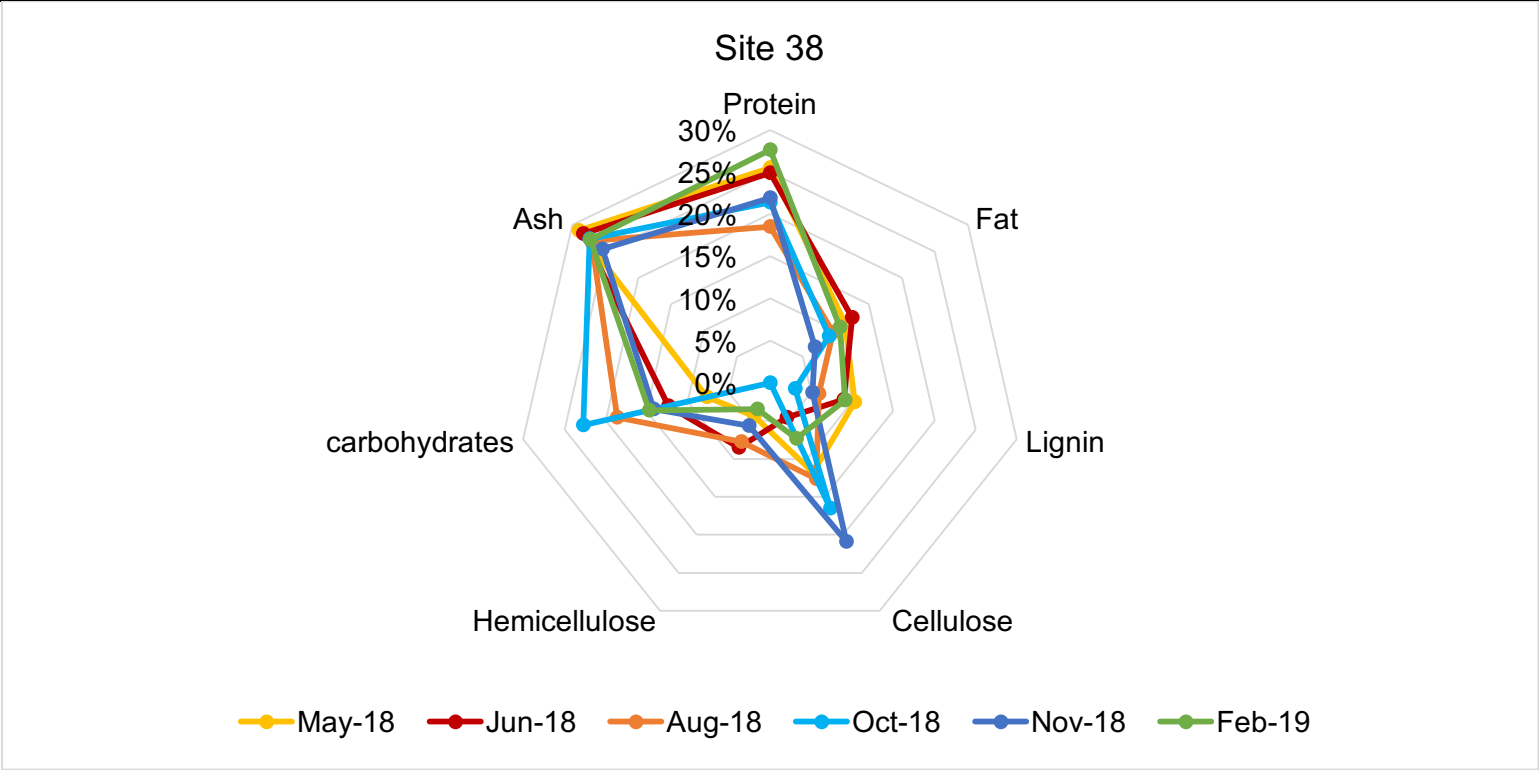
Conventional Site 5	DS	VS	Protein	Fat	Lignin	Cellulose	Hemicellulose	Carbohydrates	Ash
May-18	4.6	74.5	28.7	9.9	3.0	14.9	8.0	10.0	25.5
Jun-18	5.6	72.5	21.6	13.8	8.2	15.7	0.0	13.2	27.5
Aug-18	5.0	71.7	21.2	12.4	4.4	5.2	10.4	18.0	28.3
Oct-18	5.2	75.3	25.6	10.3	0.0	0.0	0.0	39.3	24.7
Nov-18	5.2	75.4	30.4	0.0	9.1	12.0	7.9	16.0	24.6
Feb-19	5.1	74.6	26.0	11.2	11.2	14.0	4.3	7.8	25.4



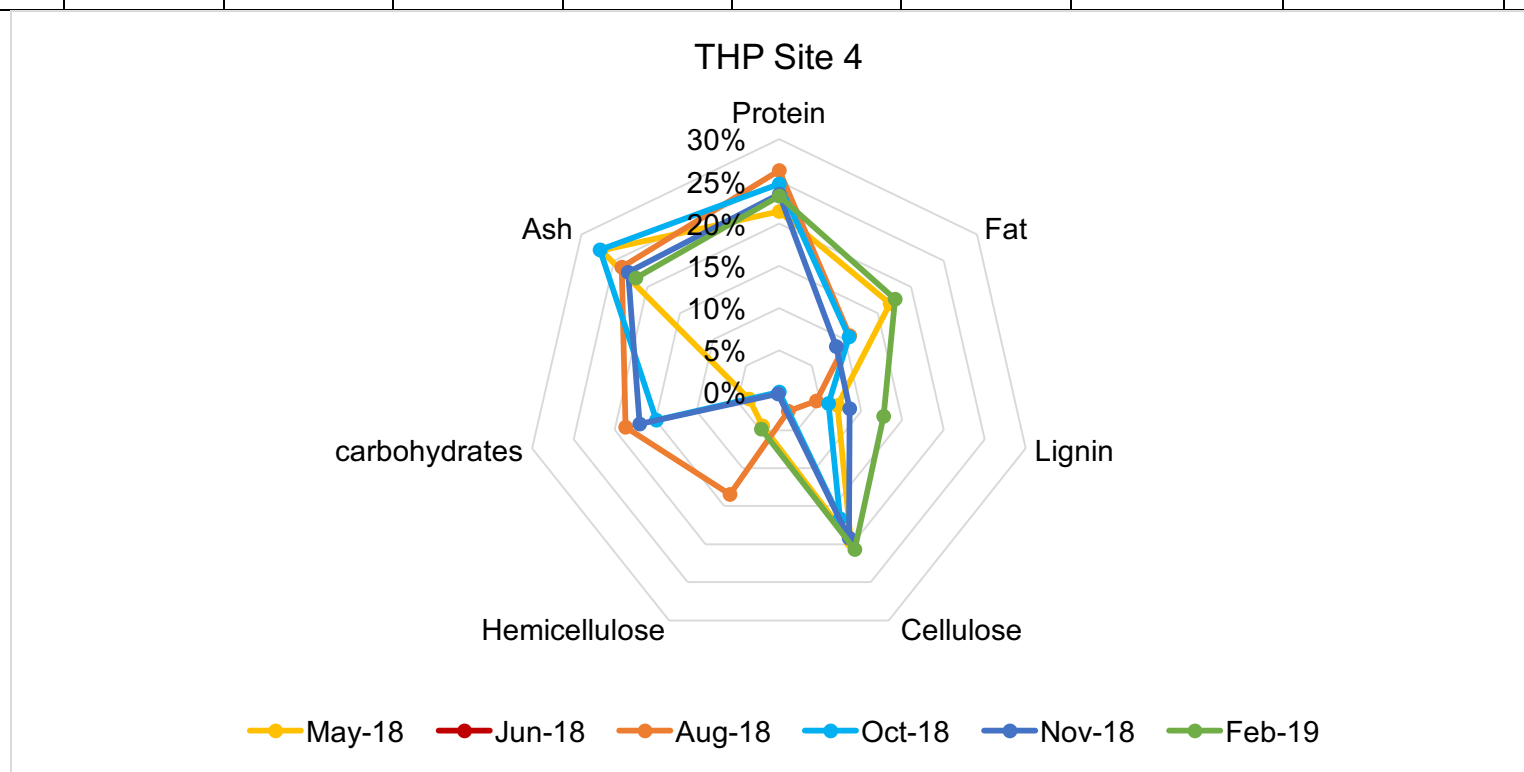
Conventional Site 31	DS	VS	Protein	Fat	Lignin	Cellulose	Hemicellulose	Carbohydrates	Ash
May-18									
Jun-18	3.6	78.1	21.3	19.8	4.2	18.4	0.0	14.4	21.9
Aug-18									
Oct-18	4.0	77.2	26.3	7.9	0.0	0.0	0.0	43.0	22.8
Nov-18	4.2	78.6	26.6	0.0	0.0	0.0	10.6	41.4	21.4
Feb-19									



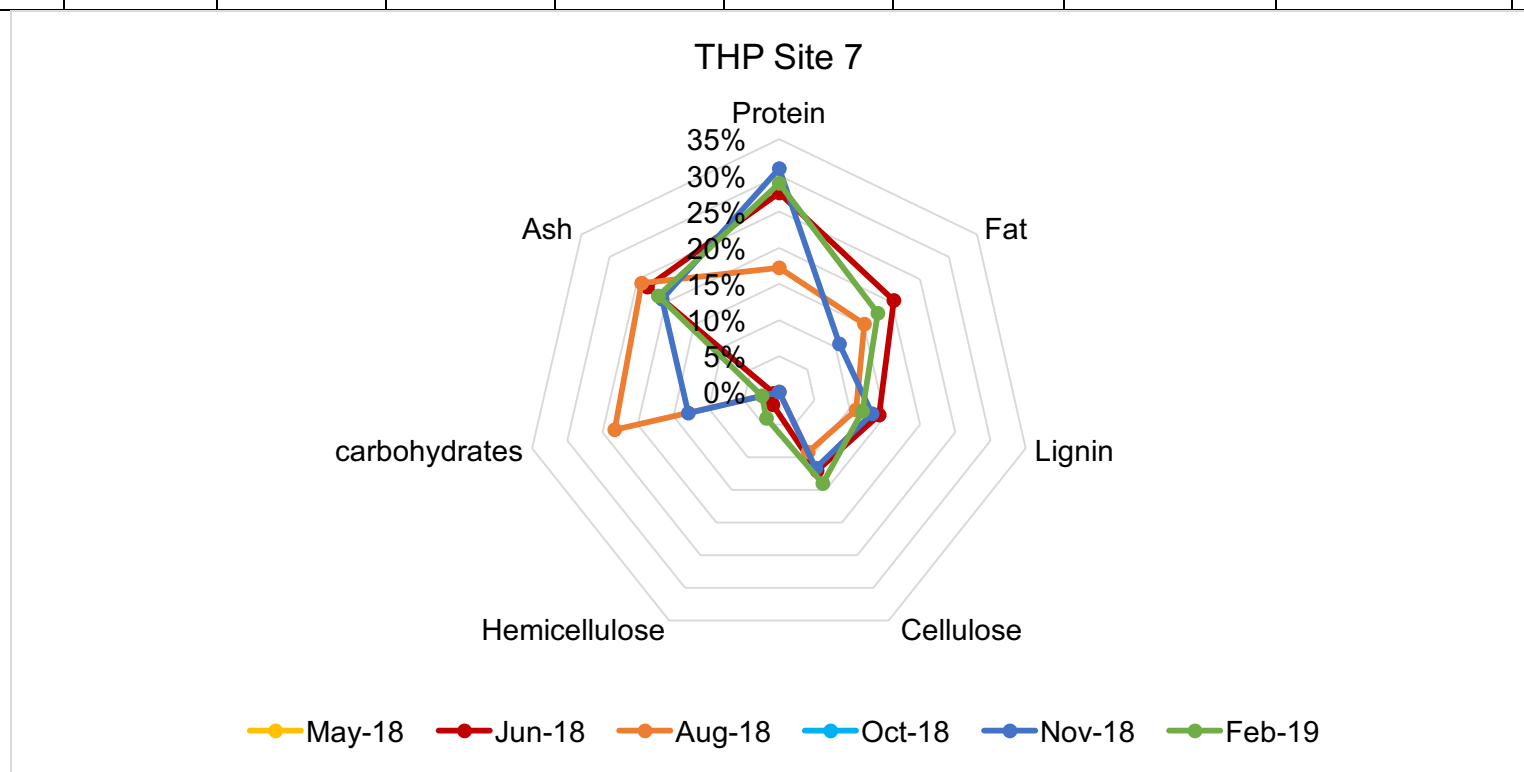
Conventional Site 38	DS	VS	Protein	Fat	Lignin	Cellulose	Hemicellulose	Carbohydrates	Ash
May-18	6.4	71.0	25.5	11.4	10.3	11.8	4.4	7.7	29.0
Jun-18	6.8	71.7	24.9	12.5	8.9	4.5	8.5	12.3	28.3
Aug-18	7.2	72.9	18.5	9.6	6.0	12.6	7.8	18.5	27.1
Oct-18	6.8	72.7	21.4	8.9	3.1	16.6	0.0	22.7	27.3
Nov-18	6.9	74.6	21.9	6.8	5.2	20.9	5.6	14.1	25.4
Feb-19	6.0	72.8	27.6	10.6	9.1	7.3	3.5	14.7	27.2



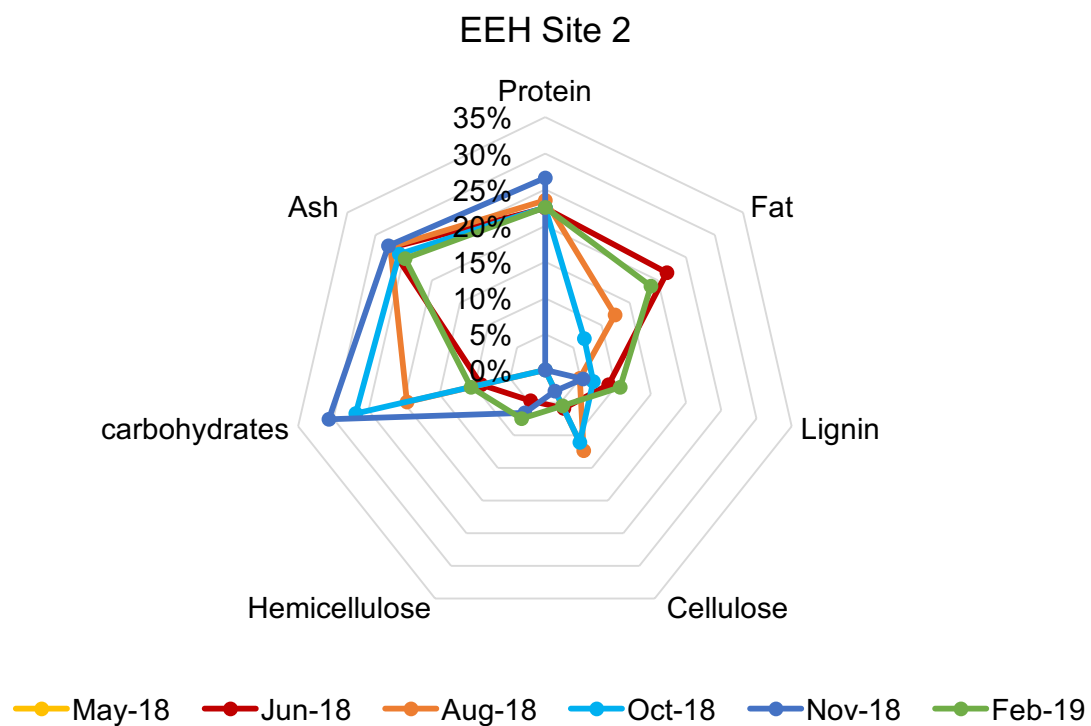
THP Site 4	DS	VS	Protein	Fat	Lignin	Cellulose	Hemicellulose	Carbohydrates	Ash
May-18	10.6	73.1	21.4	16.8	7.1	19.7	4.4	3.6	26.9
Jun-18									
Aug-18	8.8	76.2	26.3	10.7	4.6	2.5	13.4	18.6	23.8
Oct-18	9.7	72.9	24.7	10.6	6.0	16.7	0.0	14.9	27.1
Nov-18	9.6	77.2	23.5	8.7	8.6	19.2	0.2	17.0	22.8
Feb-19	8.9	78.3	23.3	17.6	12.7	20.7	4.8	0.0	21.7



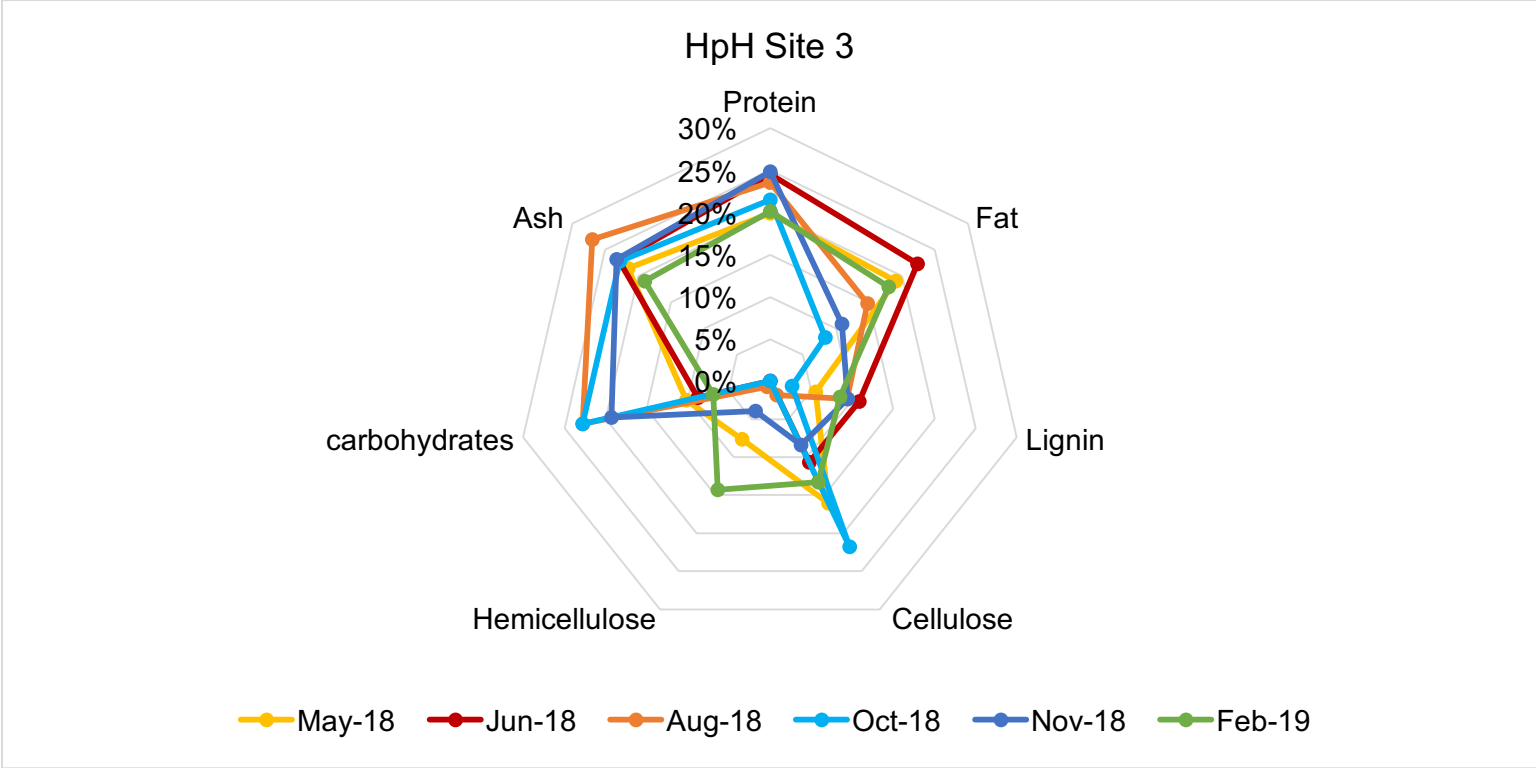
THP Site 7	DS	VS	Protein	Fat	Lignin	Cellulose	Hemicellulose	Carbohydrates	Ash
May-18									
Jun-18	9.9	76.7	27.5	20.3	14.2	12.1	1.9	0.7	23.3
Aug-18	8.6	75.7	17.2	15.1	10.9	9.2	0.0	23.3	24.3
Oct-18									
Nov-18	9.6	79.3	31.0	10.7	13.2	11.6	0.0	12.8	20.7
Feb-19	15.4	78.7	28.9	17.5	11.8	14.0	4.0	2.4	21.3



EEH Site 2	DS	VS	Protein	Fat	Lignin	Cellulose	Hemicellulose	Carbohydrates	Ash
May-18									
Jun-18	6.6	72.7	22.4	21.6	9.1	5.9	4.7	9.1	27.3
Aug-18	6.7	72.6	23.5	12.3	4.9	12.3	0.0	19.6	27.4
Oct-18	6.6	74.2	22.5	7.0	6.8	11.0	0.0	26.9	25.8
Nov-18	6.0	72.3	26.6	0.0	5.3	3.2	6.5	30.7	27.7
Feb-19	6.4	75.3	22.5	18.7	10.7	5.5	7.4	10.5	24.7



HpH Site 3	DS	VS	Protein	Fat	Lignin	Cellulose	Hemicellulose	Carbohydrates	Ash
May-18	7.2	78.5	20.0	19.1	5.6	16.0	7.7	10.2	21.5
Jun-18	6.9	77.1	24.5	22.3	10.8	10.7	0.0	8.8	22.9
Aug-18	6.7	73.1	23.6	14.8	9.4	1.8	0.7	22.8	26.9
Oct-18	8.5	77.2	21.5	8.4	2.7	21.8	0.0	22.8	22.8
Nov-18	6.9	76.8	24.8	10.9	9.4	8.4	3.9	19.3	23.2
Feb-19	6.9	81.0	20.2	18.0	8.5	13.2	14.3	6.9	19.0



Appendix 7-2 Mean dry solids (%), volatile solids and major organic energy constituents in feed sludge (% DS basis) for Conventional Site: 1, 5, 31, 38; THP Site: 4, 7; EEH Site 2 and HpH Site 3 (n = 6)

Site	DS	VS	Protein	Fat	Lignin	Cellulose	Hemicellulose	Carbohydrates
Conventional Site 1	4.3	78.3	27.6	12.6	6.5	14.8	11.0	11.7
Conventional Site 5	5.1	74.0	25.5	9.6	6.1	10.3	5.0	17.5
Conventional Site 31	3.7	78.2	25.3	16.3	8.3	17.8	8.8	20.6
Conventional Site 38	6.7	72.6	23.2	9.9	7.0	12.4	6.0	15.1
Conventional average	5.0	75.8	25.4	12.1	7.0	13.8	7.7	16.2
THP Site 4	9.1	75.4	24.0	14.7	8.0	15.2	5.2	9.2
THP Site 7	9.9	77.2	26.5	15.5	11.5	12.0	4.1	10.3
THP average	9.5	76.3	25.2	15.1	9.7	13.6	4.6	9.8
EEH Site 2	6.5	73.4	23.5	15.1	7.4	7.7	6.0	19.2
HpH Site 3	7.2	77.3	22.4	15.3	7.5	12.4	6.5	15.3
Enzymic average	6.8	75.4	22.9	15.2	7.5	10.1	6.3	17.2

Appendix 7-3 Sludge composition derived CH₄ yield, CH₄ content, BY_c, and observed BY for the selected sampling sites at different sampling times

Site	Date	Sludge composition CH ₄ yield (m ³ /t DS)	Sludge composition CH ₄ content (%)	Sludge composition BY _c (m ³ /t DS)	Observed BY (m ³ /t DS)
Conventional Site 1	05/2018	254.1	60.3	421.4	
	06/2018				
	08/2018				647.0
	10/2018	220.6	60.1	366.9	
	11/2018	210.7	56.6	372.0	
	02/2019	225.8	61.9	364.9	
Site average		227.8	59.7	381.3	647.0
Conventional Site 5	05/2018	211.8	59.7	354.6	422.9
	06/2018	224.7	60.9	368.8	507.6
	08/2018	167.8	49.5	339.2	501.7
	10/2018	188.6	56.7	332.6	413.4
	11/2018	144.4	56.4	256.1	
	02/2019	278.4	60.9	456.9	
Site average		202.6	57.4	351.3	461.4
Conventional Site 31	05/2018				416.5
	06/2018	279.0	61.4	454.7	400.3
	08/2018				436.7
	10/2018	225.7	57.8	390.8	466.2
	11/2018	209.2	53.5	391.3	503.5
	02/2019				537.4
Site average		238.0	57.5	412.3	460.1
Conventional Site 38	05/2018	139.4	60.8	229.1	316.5
	06/2018	198.8	61.8	321.4	378.1
	08/2018	157.0	48.6	322.8	340.4
	10/2018	151.4	50.3	301.2	407.1
	11/2018	186.6	55.3	337.4	430.8

	02/2019	174.8	62.0	281.8	426.2
Appendix 7-2 continued					
Site average		168.0	56.5	299.0	383.2
THP Site 4	05/2018	228.3	59.2	385.4	456.0
	06/2018				506.8
	08/2018	242.1	59.5	407.0	529.1
	10/2018	169.9	58.9	288.6	348.1
	11/2018	194.3	55.2	352.3	496.9
	02/2019	249.9	58.5	426.9	493.8
Site average		216.9	58.3	372.0	471.8
THP Site 7	05/2018				
	06/2018	247.7	62.8	394.6	
	08/2018	189.0	54.3	348.0	
	10/2018				
	11/2018	209.7	61.9	339.0	
	02/2019	310.9	61.2	507.8	
Site average		239.3	60.0	397.3	
EEH Site 2	05/2018				
	06/2018	188.2	63.9	294.7	
	08/2018	174.6	61.0	286.0	
	10/2018	127.3	56.9	223.6	
	11/2018	96.0	52.9	181.7	
	02/2019	238.6	56.3	423.8	
Site average		164.9	58.2	282.0	
HpH Site 3	05/2018	226.0	57.3	394.7	
	06/2018	211.5	60.5	349.4	
	08/2018	144.5	59.4	243.2	
	10/2018	157.8	53.1	297.1	
	11/2018	189.9	57.8	328.4	
	02/2019	218.3	54.8	398.2	
Site average		191.3	57.2	335.2	

Appendix 7-4 Mean inorganic elemental composition

Table A7-1 Inorganic elemental composition of feed sludge for conventional MAD Site 1, 5, 31 and 38 (n = 6)

Element	Site 1 feed	Site 5 feed	Site 31 feed	Site 38 feed	Conventional feed average
Total N (% DS)	5.5	4.8	4.8	5.2	5.1
NH ₄ -N (mg/l)	258.4	553.2	282.3	1065.7	539.9
Fe (% DS)	3.3	5.3	4.2	2.5	3.8
Ca (% DS)	3.3	2.7	4.5	3.4	3.5
P (% DS)	2.1	2.4	2.5	2.4	2.4
S (% DS)	1.1	1.0	1.0	0.8	1.0
K (% DS)	0.4	0.5	0.4	0.4	0.4
Mg (% DS)	0.5	0.5	0.2	0.3	0.4
Na (% DS)	0.3	0.2	0.5	0.1	0.3
Zn (mg/kg DS)	1007.7	556.6	460.4	447.0	617.9
Cu (mg/kg DS)	540.5	172.4	415.8	479.0	401.9
Mn (mg/kg DS)	270.3	250.4	175.2	240.1	234.0
Pb (mg/kg DS)	54.8	68.4	44.0	24.7	48.0
Ni (mg/kg DS)	117.4	39.8	12.5	19.2	47.2
Cr (mg/kg DS)	43.0	31.0	23.2	27.2	31.1
Mo (mg/kg DS)	5.4	10.0	3.7	4.4	5.9
Co (mg/kg DS)	4.4	6.2	4.9	3.4	4.7
Se (mg/kg DS)	3.0	3.0	2.3	3.1	2.8
Cd (mg/kg DS)	0.9	2.1	0.4	0.6	1.0

Table A7-2 Inorganic elemental composition of digested sludge after conventional MAD for Site 1, 5, 31 and 38 (n = 6)

Element	Site 1 digested	Site 5 digested	Site 31 digested	Site 38 digested	Conventional digested average
Total N (% DS)	7.8	7.7	7.3	7.4	7.6
NH ₄ -N (mg/l)	912.2	916.8	711.7	1400.2	985.2
Fe (% DS)	5.2	6.8	5.9	3.7	5.4
Ca (% DS)	3.8	3.7	5.3	4.9	4.4
P (% DS)	3.1	3.2	3.3	3.7	3.3
S (% DS)	1.5	1.4	1.2	1.2	1.3
K (% DS)	0.5	0.8	0.4	0.6	0.6
Mg (% DS)	0.6	0.8	0.3	0.4	0.5
Na (% DS)	0.3	0.4	0.4	0.2	0.3
Zn (mg/kg DS)	706.4	818.1	583.4	641.6	687.4
Cu (mg/kg DS)	406.7	225.3	573.5	704.5	477.5
Mn (mg/kg DS)	298.3	383.9	271.1	338.0	322.8
Pb (mg/kg DS)	48.2	112.1	60.4	35.4	64.0
Ni (mg/kg DS)	32.2	51.1	20.4	26.8	32.6
Cr (mg/kg DS)	46.6	52.8	26.8	39.0	41.3
Mo (mg/kg DS)	7.6	11.7	5.8	6.5	7.9
Co (mg/kg DS)	6.5	9.7	7.2	4.7	7.0
Se (mg/kg DS)	4.0	4.5	3.1	4.6	4.0
Cd (mg/kg DS)	1.2	3.0	0.7	0.8	1.4

Table A7-3 Inorganic elemental composition of feed sludge for THP Site 4, 7; EEH Site 2 and HpH Site 3 (n = 6)

Element	THP Site 4 feed	THP Site 7 feed	THP feed average	EEH Site 2 feed	HpH Site 3 feed	Enzymic feed average
Total N (% DS)	4.4	5.2	4.8	5.8	5.9	5.9
NH ₄ -N (mg/l)	645.8	1006.8	826.3	1393.8	1708.8	1551.3
Fe (% DS)	5.5	4.2	4.8	4.8	2.4	3.6
Ca (% DS)	2.3	2.7	2.5	2.9	2.6	2.8
P (% DS)	2.3	2.5	2.4	2.4	1.9	2.2
S (% DS)	0.9	1.3	1.1	1.5	1.3	1.4
K (% DS)	0.2	0.2	0.2	0.2	0.2	0.2
Mg (% DS)	0.1	0.2	0.2	0.2	0.3	0.2
Na (% DS)	0.1	0.1	0.1	0.2	0.3	0.2
Zn (mg/kg DS)	412.7	481.5	447.1	539.4	573.7	556.5
Cu (mg/kg DS)	172.3	231.7	202.0	215.3	315.1	265.2
Mn (mg/kg DS)	422.7	173.9	298.3	219.5	161.9	190.7
Pb (mg/kg DS)	29.6	24.6	27.1	42.7	41.4	42.1
Ni (mg/kg DS)	17.2	19.0	18.1	25.1	22.8	24.0
Cr (mg/kg DS)	17.3	38.8	28.1	39.6	30.1	34.8
Mo (mg/kg DS)	3.8	6.0	4.9	8.1	6.6	7.4
Co (mg/kg DS)	7.0	2.7	4.8	4.4	3.3	3.8
Se (mg/kg DS)	1.6	2.0	1.8	2.2	3.4	2.8
Cd (mg/kg DS)	0.7	0.6	0.6	0.6	1.0	0.8

Table A7-4 Inorganic elemental composition of digested sludge after THP and enzymic MAD for THP Site 4, 7; EEH Site 2 and HpH Site 3 (n = 6)

Element	THP Site 4 digested	THP Site 7 digested	THP digested average	EEH Site 2 digested	HpH Site 3 digested	Enzymic digested average
Total N (% DS)	7.0	8.5	7.7	7.8	8.6	8.2
NH ₄ -N (mg/l)	2056.0	2357.6	2206.8	1646.4	2094.5	1870.5
Fe (% DS)	8.9	6.7	7.8	6.7	3.5	5.1
Ca (% DS)	4.1	4.3	4.2	4.0	3.8	3.9
P (% DS)	4.0	3.9	4.0	3.3	2.8	3.1
S (% DS)	1.6	2.3	2.0	2.0	1.9	1.9
K (% DS)	0.3	0.3	0.3	0.3	0.4	0.3
Mg (% DS)	0.3	0.3	0.3	0.3	0.4	0.3
Na (% DS)	0.2	0.2	0.2	0.3	0.4	0.4
Zn (mg/kg DS)	701.7	756.5	729.1	716.2	829.8	773.0
Cu (mg/kg DS)	325.7	364.5	345.1	287.8	453.1	370.4
Mn (mg/kg DS)	561.8	272.8	417.3	300.8	234.7	267.7
Pb (mg/kg DS)	51.0	39.6	45.3	62.5	58.9	60.7
Ni (mg/kg DS)	27.9	31.0	29.5	34.5	33.8	34.2
Cr (mg/kg DS)	30.3	66.3	48.3	55.7	45.0	50.3
Mo (mg/kg DS)	6.1	9.7	7.9	11.2	9.6	10.4
Co (mg/kg DS)	10.7	4.2	7.4	5.9	4.8	5.3
Se (mg/kg DS)	2.8	3.4	3.1	2.9	5.0	3.9
Cd (mg/kg DS)	1.1	1.1	1.1	0.9	1.4	1.2

Appendix 7-5 Mean DS, VS and COD in feed and digested sludge at different sampling times

	Feed			Digested			
Date	DS (%)	VS (%)	COD (g/l)	DS(%)	VS(%)	COD (g/l)	COD removal efficiency (%)
Conventional Site 1							
May-18	4.5	77.0		2.8	61.3		
Jun-18						22.0	
Aug-18	0.9	78.5	22.0	2.4	50.9	41.0	
Oct-18	3.6	79.0	48.0	2.5	64.3	34.0	30.0
Nov-18	5.4	77.8	65.0	2.5	63.5	35.0	45.8
Feb-19	3.6	79.5	48.0	2.4	59.2	34.0	29.4
Conventional Site 5							
May-18	4.6	74.5		3.7	55.6		
Jun-18	5.6	72.5	40.0	3.9	56.0	37.0	6.2
Aug-18	5.0	71.7	79.0	3.6	49.8	40.0	49.0
Oct-18	5.2	75.3	80.0	4.1	55.6	48.0	40.8
Nov-18	5.2	75.4	73.0	3.5	59.7	46.0	36.9
Feb-19	5.1	74.6	79.0	1.3	61.7	29.0	63.7
Conventional Site 31							
May-18	3.8	78.2		3.4	57.2		
Jun-18	3.6	78.1	40.0	2.4	63.6	25.0	36.2
Aug-18	1.0	81.7	23.0	2.0	62.0	34.0	
Oct-18	4.0	77.2	55.0	2.2	64.4	31.0	43.3
Nov-18	4.2	78.6	60.0	2.4	63.2	31.0	47.7
Feb-19	3.1	78.7	35.0	2.9	63.7	31.0	11.9
Conventional Site 38							
May-18	6.4	71.0		4.6	63.1		
Jun-18	6.8	71.7	83.0	5.0	60.6	53.0	36.5
Aug-18	7.2	72.9	103.0	4.8	60.2	63.0	38.5
Oct-18	6.8	72.7	91.0	5.2	58.6	57.0	37.7
Nov-18	6.9	74.6	86.0	4.7	59.8	58.0	33.2
Feb-19	6.0	72.8	84.0	4.4	63.5	58.0	31.2

Appendix 7-5 continued							
THP Site 4							
May-18	10.6	73.1		6.6	57.2		
Jun-18	7.3	74.9	50.0	6.5	60.3	76.0	
Aug-18	8.8	76.2	136.0	5.4	62.8	79.0	41.7
Oct-18	9.7	72.9	131.0	6.4	57.2	93.0	29.2
Nov-18	9.6	77.2	126.0	6.6	57.2	81.0	35.5
Feb-19	8.9	78.3	129.0	5.6	55.8	68.0	47.2
THP Site 7							
May-18							
Jun-18	9.9	76.7	128.0	6.1	62.3	67.0	47.7
Aug-18	8.6	75.7	121.0	6.0	58.1	89.0	26.6
Oct-18	6.2	75.7	100.0	5.9	61.1	94.0	
Nov-18	9.6	79.3	145.0	6.4	63.5	98.0	32.6
Feb-19	15.4	78.7	217.0	5.8	59.2	78.0	64.2
EEH Site 2							
May-18							
Jun-18	6.6	72.7	103.0	5.0	61.8	55.0	47.1
Aug-18	6.7	72.6	108.0	5.1	65.7	69.0	35.6
Oct-18	6.6	74.2	107.0	4.9	63.9	57.0	46.6
Nov-18	6.0	72.3	94.0	4.6	64.6	70.0	25.1
Feb-19	6.4	75.3	100.0	4.4	48.5	59.0	41.1
HPH Site 3							
May-18	7.2	78.5		4.7	68.7		
Jun-18	6.9	77.1	109.0	5.4	67.7	65.0	40.1
Aug-18	6.7	73.1	111.0	5.3	66.9	71.0	36.6
Oct-18	8.5	77.2	138.0	5.5	67.4	68.0	51.0
Nov-18	6.9	76.8	106.0	5.3	59.4	69.0	35.4
Feb-19	6.9	81.0	108.0	4.6	69.0	59.0	45.4

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