

# **Soft sensor development and process control of anaerobic digestion**

Submitted by

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## Abstract

This thesis focuses on soft sensor development based on fuzzy logic used for real time online monitoring of anaerobic digestion to improve methane output and for robust fermentation. Important process parameter indicators such as pH, biogas production, daily difference in pH and daily difference in biogas production were used to infer alkalinity, a reliable indicator of process stability. Additionally, a fuzzy logic and a rule-based controller were developed and tested with single stage anaerobic digesters operating with cow slurry and cellulose. Alkalinity predictions from the fuzzy logic algorithm were used by both controllers to regulate the organic loading rate that aimed to optimise the biogas process.

The predictive performance of a software sensor determining alkalinity that was designed using fuzzy logic and subtractive clustering and was validated against multiple linear regression models that were developed (Partner N° 2, Rothamsted Research 2010) for the same purpose. More accurate alkalinity predictions were achieved by utilizing a fuzzy software sensor designed with less amount of data compared to a multiple linear regression model whose design was based on a larger database. Those models were utilised to control the organic loading rate of a two-stage, semi-continuously fed stirred reactor system.

Three 5l reactors without support media and three 5l reactors with different support media (burst cell reticulated polyurethane foam coarse, burst cell reticulated polyurethane foam medium and sponge) were operated with cow slurry for a period of seven weeks and twenty weeks respectively. Reactors with support media were proven to be more stable than the reactors without support media but did not exhibit higher gas productivity. Biomass support media were found to influence digester recovery positively by reducing the recovery period. Optimum process parameter ranges were identified for reactors with and without support media. Increased biogas production was found to occur when the loading rates were 3-3.5g VS/l/d and 4-5g VS/l/d respectively. Optimum pH ranges were identified between 7.1-7.3 and 6.9-7.2 for reactors with and without support media respectively, whereas all reactors became unstable at  $\text{pH} < 6.9$ . Alkalinity levels for system stability appeared to be above 3500 mg/l of  $\text{HCO}_3^-$  for reactors without media and 3480 mg/l of  $\text{HCO}_3^-$  for reactors with support media. Biogas production was maximized when alkalinity was

between 3500-4500 mg/l of  $\text{HCO}_3^-$  for reactors without support media and 3480-4300 mg/l of  $\text{HCO}_3^-$  for reactors with support media. Two fuzzy logic models predicting alkalinity based on the operation of the three 5l reactors with support media were developed (FIS I, FIS II). The FIS II design was based on a larger database than FIS I. FIS II performance when applied to the reactor where sponge was used as the support media was characterized by quite good MAE and bias values of 466.53 mg/l of  $\text{HCO}_3^-$  and an acceptable value for  $R^2= 0.498$ . The NMSE was close to 0 with a value of 0.03 and a slightly higher FB= 0.154 than desired. The fuzzy system robustness was tested by adding  $\text{NaHCO}_3$  to the reactor with the burst cell reticulated polyurethane foam medium and by diluting the reactor where sponge was used as the support media with water. FIS I and FIS II were able to follow the system output closely in the first case, but not in the second.

FIS II functionality as an alkalinity predictor was tested through the application on a 28l cylindrical reactor with sponge as the biomass support media treating cow manure. If data that was recorded when severe temperature fluctuations occurred (that highly impact digester performance), are excluded, FIS II performance can be characterized as good by having  $R^2= 0.54$  and MAE=Bias= 587 mg/l of  $\text{HCO}_3^-$ . Predicted alkalinity values followed observed alkalinity values closely during the days that followed  $\text{NaHCO}_3$  addition and water dilution. In a second experiment a rule-based and a Mamdani fuzzy logic controller were developed to regulate the organic loading rate based on alkalinity predictions from FIS II. They were tested through the operation of five 6.5l reactors with biomass support media treating cellulose. The performance indices of MAE=763.57 mg/l of  $\text{HCO}_3^-$ , Bias= 398.39 mg/l of  $\text{HCO}_3^-$ ,  $R^2= 0.38$  and IA= 0.73 indicate a pretty good correlation between predicted and observed values. However, although both controllers managed to keep alkalinity within the desired levels suggested for stability (>3480 mg/l of  $\text{HCO}_3^-$ ), the reactors did not reach a stable state suggesting that different loading rates should be applied for biogas systems treating cellulose.

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## List of Abbreviations

### Abbreviation    Meaning

AAFEB	Anaerobic Attached- Film Expanded- Bed Reactor
AFB	Anaerobic Fluidised Bed
AD	Anaerobic Digestion
AI	Artificial Intelligence
ANFIS	Adaptive-Network-Based Fuzzy Inference System
ANN	Artificial Neural Network
BA	Bicarbonate Alkalinity
CAD	Computer Aided Design
COD	Chemical Oxygen Demand
CSTR	Continuous Stirred-Tank Reactor
CT	Controller Test
DDA	Daily Difference in Alkalinity
DIC	Direct Inverse Control
EC	Electrical Conductivity
EP	Environmental Permitting
FB	Fractional Bias
FCM	Fuzzy C-means
FIR	Finite Impulse Response
FIS	Fuzzy Inference System
FL	Fuzzy Logic
FLC	Fuzzy Logic Control
FLS	Fuzzy Logic System
FT	Fuzzy Test
GC-FID	Gas Chromatography-Flame Ionization Detection
GMC	Generic Model Control
GPC	Generalised Predictive Control
GPR	Gas Production Rate
HRT	Hydraulic Retention Time
HSGC	Headspace Gas Chromatography

IA	Index of Agreement
IMC	Internal Model Control
IWA	International Water Association
LCFA	Long Chain Fatty Acids
LQPC	Linear Quadratic Predictive Control
MAE	Mean Absolute Error
MIMO	Multiple-Input and Multiple-Output
MISO	Multiple-Input and Single-Output
MLP	Multi-Layer Perceptron
MLR	Multiple Linear Regression
MPB	Methane-Producing Bacteria
MPC	Model Predictive Control
MRAS	Model Reference Adaptive Systems
NMSE	Normalised Mean Square Error
NN	Neural Network
OFMSW	Organic Fraction of Municipal Solid Waste
OLR	Organic Loading Rate
ORP	Organic Redox Potential
P	Proportional
PG	Propylene Glycol
PI	Proportional-Integral
PID	Proportional-Integral-Derivative
PLC	Programmable Logic Controller
PS	Primary Sludge
QP	Quadratic Programming
RBC	Rule-Based Controller
RHONO	Recurrent High Order Neural Observer
RMSE	Root Mean Square Error
RT	Retention Time
SCM	Subtractive Clustering Method
SCOD	Soluble Chemical Oxygen Demand
SGP	Specific Gas Production
SISO	Single-Input Single-Output

SMA	Specific Methanogenic Activity
SRB	Sulphate-Reducing Bacteria
SRT	Solids Retention Time
STR	Self-Tuning Regulators
TPAD	Temperature-Phased Anaerobic Digestion
TPASBR	Temperature-Phased Anaerobic Sequencing Batch Reactors
TS	Total Solids
TSK	Takagi-Sugeno-Kang
UASB	Upflow Anaerobic Sludge Blanket
VA	Volatile Acids
VFA	Volatile Fatty Acids
VS	Volatile Solids
VSR	Volatile Solids Reduction
WWTP	Wastewater Treatment Plant

# Chapter 1 Monitoring and control of anaerobic digestion

## 1.1 Introduction and aim of study

Anaerobic digestion (AD) is a biochemical conversion process that is considered highly attractive for the treatment and recycling of biomass wastes. AD application can lead to the reduction of waste volume, simple and reliable generation of energy-rich gas in the form of methane that can be burnt with limited generation of pollutants, and the production of nutrient-containing final products (Mata-Alvarez et al. 2000)(Evangelisti et al. 2014). Energy generation from biomass and waste is regarded as one of the most promising and consistent future renewable energy sources (unlike solar energy and wind energy) and offers increased benefits over other conversion technologies in terms of energy efficiency such as combustion, pyrolysis and gasification (Appels et al. 2011). Hence, AD technology has been identified as the means to try and produce half of UK's renewable target requirements by 2020 (Mezzullo et al. 2013). Currently there are 106 AD plants in UK, outside of the water industry, processing up to 5.1 millions tonnes of food and farm waste per year with an installed electrical capacity of more than 88 MWe (The Official Information Portal on Anaerobic Digestion 2013).

In AD, a series of reactions are performed by a variety of microorganisms that co-exist in the same environment and the bioconversion of organic compounds to methane is affected by their activity (Gujer & Zehnder 1983)(Lee et al. 2008). The interdependence of different microbial groups can be the cause of system instability (Liu et al. 2004a) since micro-organisms are highly sensitive to disturbances and changes in operating conditions (Steyer et al. 2006). Operating an AD reactor below the theoretical reactor capacity (under-loading) is a way to avoid system instability. However, low loadings result in limited biogas productivity and consequently low turnover. On the other hand, high loading rates offer increased biogas production rates but maximise the risk of reactor overloading that can lead to poor gas production rates and an acidified or sour digester that will require a lot of time to recover (Spanjers & Lier 2006). So, additional costs are required due to the absence of gas productivity and digester restart operations. Therefore, the challenge is to design a monitoring and control system to operate an AD reactor with optimized

biogas production. At the same time, the risk of digester instability, hence, digester failure has to be avoided (Liu et al. 2004a)(Boe 2006).

There are a series of process indicators utilised to characterise process stability and evolution. This is possible with the indirect measurement of activity of the different groups of microorganisms that reflect the current metabolic status of the active organisms in the system (Gujer & Zehnder 1983)(Björnsson et al. 2000). Easily measurable indicators include gas production rate, gas composition, VFAs, pH, alkalinity (Hawkes 1993)(Björnsson, Murto, et al. 2001), ammonia and indirect measurements of organic matter (Pind et al. 2003). In most cases, several process monitoring indicators are utilised in anaerobic digestion applications because they provide complementary information (Hickey et al. 1991). Therefore, a process model (Wang et al. 1996) or an estimation algorithm (Chéruey 1997) can be implemented to estimate the biological state of the AD system. Soft sensor application is considered to be a suitable method of continuous monitoring of easily measured key process variables. Then, the information acquired can be used to make decisions mostly regarding the digester loading that can lead to reduced capital costs and enhanced biogas output (Simeonov et al. 2012). Several soft-sensor applications have been developed in the past to predict the unmeasured on-line variables of acidogenic and methanogenic bacteria, Chemical Oxygen Demand (COD), alkalinity, Volatile Solids (VS), inorganic carbon concentration, volatile fatty acids (VFA) or the state of the AD system (Aubrun et al. 2001)(Bernard et al. 2001)(Alcaraz-González et al. 2002)(Ward et al. 2011)(Gaida et al. 2012)(Montiel-Escobar et al. 2012)(Oppong et al. 2013).

Alkalinity is an indicator of process stability in AD and enables the detection of changes in the buffer capacity of the system (Palacios-Ruiz et al. 2008)(Hawkes 1993). Also, alkalinity is a good indicator of future failure due to reactor acidification (Guwy, Hawkes, Wilcox, et al. 1997). A drop in alkalinity might result in having a 'sour' digester and will take a huge effort to bring the system back to full operation (Sanin et al. 2010). Many control applications utilise the VFA to alkalinity ratio. VFA accumulation can lead to a decrease in pH and cessation of gas production. This justifies why VFAs are widely used to determine the stability of digestion processes. Alkalinity and VFA are two of the most sensitive indicators of process stability (Schoen et al. 2009) which led to a wide application of the VFA/ Alkalinity ratio for the purpose of system monitoring. VFA sensors have been implemented in the past

using analytical instruments. Those include the use of gas chromatography (GC), titrimetry, IR-spectrometry (Spanjers & Lier 2006), spectrophotometry and capillary zone electrophoresis (Zygmunt & Banel 2009). However, on-line sensors have proven to be quite unreliable delivering wrong measurements due to disturbances (e.g. interference of chemical species) (Lardon et al. 2004). Other methods were limited by the fact that the VFA measurement system would only work in a reliable manner if serviced regularly (Boe, Batstone, et al. 2007). In recent years, more accurate VFA sensors have been developed based on headspace gas chromatography (HSGC). A method that applies *ex-situ* VFA stripping with variable headspace volume and gas analysis by gas chromatography-flame ionization detection (GC-FID) has been proposed (Boe, Batstone, et al. 2007).

Alkalinity is a good process indicator of AD process stability. Literature based VFA/Alkalinity ratios are variable and VFA in-line sensors are quite difficult to construct, contain a high level of complexity when it comes to their operation, and can be quite expensive. Therefore, this thesis focuses on the design of a software sensor based on the utilization of cost-effective on-line sensors predicting alkalinity in a more accurate manner than the multiple linear regression models presented in (Partner N° 2, Rothamsted Research 2010) that were developed based on the work presented in (Ward 2009)(Ward et al. 2011).

Takagi-Sugeno-Kang (TSK) Fuzzy Logic (FL) models have been developed to predict alkalinity. TSK FL does not require extensive knowledge of the processes or of the systems under examination. This is useful for AD processes which are highly complex nonlinear microbial processes. However, this technique is capable of providing a good description of those processes (Lauwers et al. 2013). The main advantage of the TSK model over other classes of fuzzy models lies in the fact that it can model a system with great accuracy either locally or globally (Quah & Quek 2006).

The improved performance of fuzzy models over multiple linear regression models in predicting alkalinity is presented in Chapter 3. Two FL models predicting alkalinity are presented in Chapter 4 based on experiments conducted with cow manure. The soft-sensor was validated against process disturbances that included the addition of a buffering agent ( $\text{NaHCO}_3$ ) and water dilution. Additionally, optimum process and stability operating conditions were identified for organic loading rate



(OLR), pH and alkalinity, during the experiments conducted with three different types of biomass support media.

A second fuzzy model developed in Chapter 4 (FIS II) has been utilised to predict alkalinity in a reactor of different configuration and size treating cow manure (Chapter 5). Additionally, it was tested through application in five reactors treating a different substrate than cow manure, cellulose.

Fuzzy logic control (FLC) applications and rule-base controllers designed for AD systems have been successfully developed in the past (Estaben et al. 1997) (Carrasco et al. 2002)(Murnleitner et al. 2002)(Carrasco et al. 2004)(Liu et al. 2004a)(Liu et al. 2004b)(Yordanova 2004)(Scherer et al. 2009) (Ward 2009) (Partner N° 2, Rothamsted Research 2010).The development of two controllers that serve the same purpose (which is to control the OLR of an anaerobic digester): a Mamdani fuzzy logic controller and a rule-based controller, is also presented in Chapter 5. The determination of optimum operating parameter ranges for pH, OLR and alkalinity presented in Chapter 4 forms the basis of the design of the two controllers. These two types of control system are suitable for applications where the user experience can be easily embedded in the controller design. FIS II was set to predict alkalinity, and the alkalinity predictions were then fed into the corresponding controller that would vary the OLR accordingly.

### **1.1.1 Anaerobic digestion definition, Advantages-Disadvantages**

Anaerobic digestion (AD) is the process that involves the production of methane and carbon dioxide through a series of degradation processes that occur as the metabolic outcome of bacterial communities present in organic matter (Skiadas & Lyberatos 1999). Methane and carbon dioxide are the main products of the AD process. Traces of hydrogen and hydrogen sulphide are also present.

AD can be used to process waste organic matter that may only contain less than 5% w/w dry matter. Energy generation and processed organic material are the outputs. Some advantages are listed below:

- Organic wastes treated include municipal sludge, animal manure, industrial sludge, industrial and municipal wastewaters.

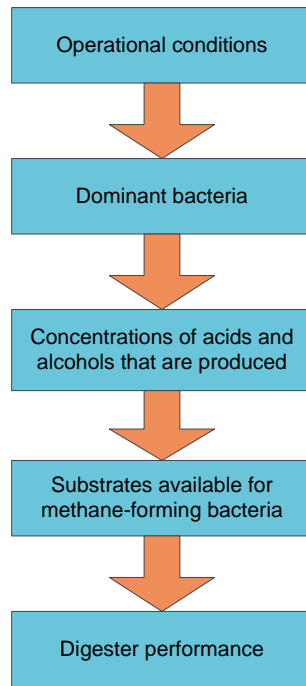
- Energy production can be boosted with the addition of energy crops to organic wastes. This is growing in popularity over the past 20 years. The development of bioenergy is due to increasing oil prices and improved legal framework conditions in Europe (Braun et al. 2009). The methane yields of a number of different feedstock materials can be found in literature (Braun 2007). The use of energy crops is disputed due to the apparent replacement of food growing areas, however energy crops will help maintain higher fossil fuel replacement.
- Biogas can be utilised to heat the reactors, instead of consuming extra energy, and biogas can be stored for future use whereas, aerobic treatment facilities consume but do not generate heat.
- When digesting manure, odours can be reduced and the manure contains less solids and is easier to manage because of a reduced viscosity.
- Greenhouse gas emissions of methane and nitrous oxide are reduced.
- AD has low nutrient requirements especially compared to aerobic treatment processes (Lettinga et al. 1979). Less than 10% of the organic matter removed from an organic waste is transformed into microbial cells using AD, whereas up to 50% of the organic matter removed from the waste can be converted to microbial sludge using aerobic treatment (Wilkie 2005b).
- Digestate is the residual material after the AD of the feedstock and represents (Bermejo & Ellmer 2010) 90-95% of the material fed in the digester. Digestate can be used as a fertiliser with a more immediate crop response because the nutrients nitrogen and phosphorus are more mineralised. For example, different treatments for potato and forage crops in Peru were investigated (Garfí et al. 2011) focusing on the use of guinea pig manure. The results indicated that digestate boosted the potato yield compared to the usage of manure and mixtures of manure and digestate. On the other hand, it was concluded that further investigations have to be made to validate the effectiveness of dry and wet digestate by comparing it with mineral fertilizer applied at several stations characterised by different soil conditions, as the results were mixed (Bermejo & Ellmer 2010).

However, like any process, AD has its limitations. These include:

- Complicated set-up and design of AD units due to legislation. Under the Environmental permitting (EP) scheme, all AD UK plants have to obtain a permit or exception to operate and spread digestate. Further details can be found (Environmental protection England and Wales 2012)
- Significant set-up (planning and legal costs) and operational costs (low values of digestate and heat) (Gebrezgabher et al. 2010) for large scale units.
- Digester processes are unstable (Graef & Andrews 1974) especially during changes in the environment or in the nature of the feedstock (Simeonov et al. 2012).
- Limited knowledge 'surrounding' the digester microbiology and operational data exists.
- The start-up process of a digester at the industrial scale takes several weeks to several months (Lardon et al. 2004). Therefore, it has to be ensured that the system will not collapse.
- High performance and process stability are difficult to be satisfied at the same time since the maximum digester loading rates vary according to the substrate utilised (Staubmann et al. 1997).

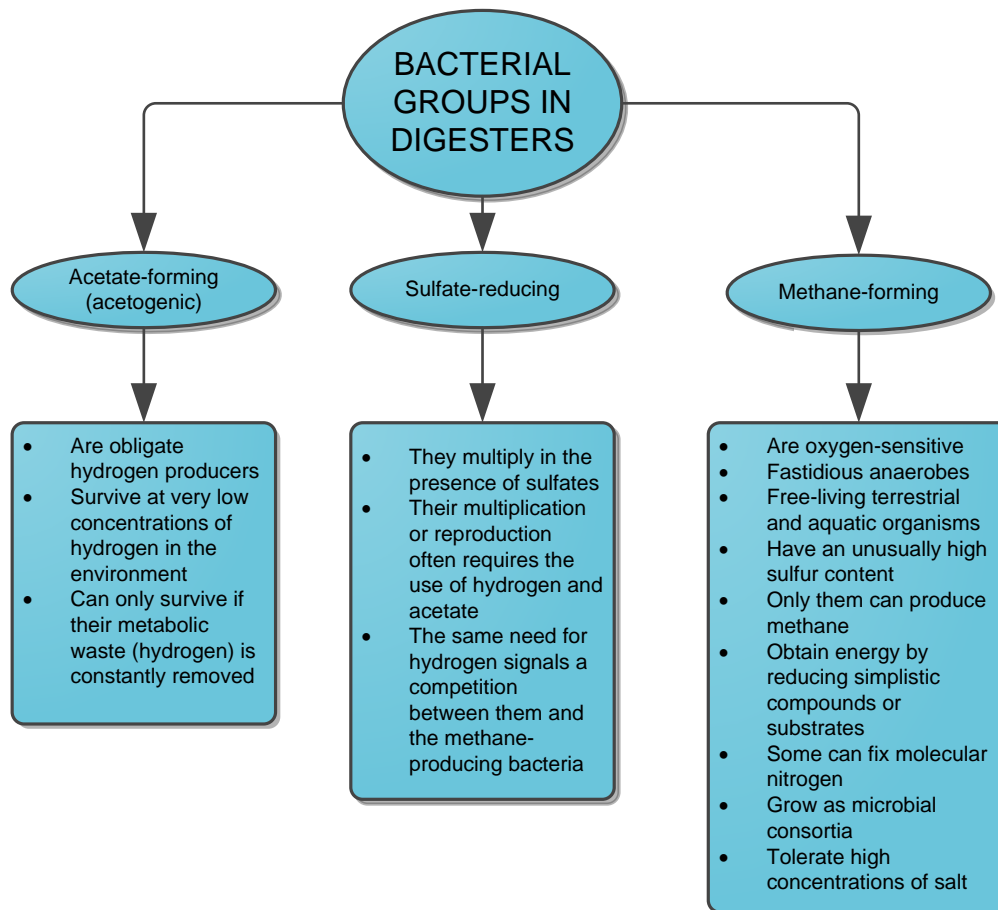
### **1.1.2 Bacteria**

Sufficient monitoring of the AD process requires knowledge of the metabolic states of the process (Ahring et al. 1995) because of a series of different microbial reactions and metabolic transformations (Chartrain & Zeikus 1986). Vital symbiosis of bacteria leads to the production of methane, carbon dioxide and new bacterial cells. Biogas production is affected by a series of events that take place in an anaerobic environment (Figure 1.1).



**Figure 1.1** Bacterial chain of influence (summarised from (Gerardi 2003))

It may be possible to influence the type of bacteria that will enhance biogas production at each stage of the process, or to try and limit the growth of the type of bacteria that inhibits the process (where possible), or to diagnose an AD process by monitoring bacterial community shifts (Lee et al. 2008). Bacteria are divided in three groups according to the substrates utilised by each group: the acetate-forming (acetogenic) bacteria, the sulfate-reducing bacteria and the methane-forming bacteria. The basic properties of these bacterial groups (Gerardi 2003) appear in Figure 1.2.



**Figure 1.2** Bacterial groups in digesters

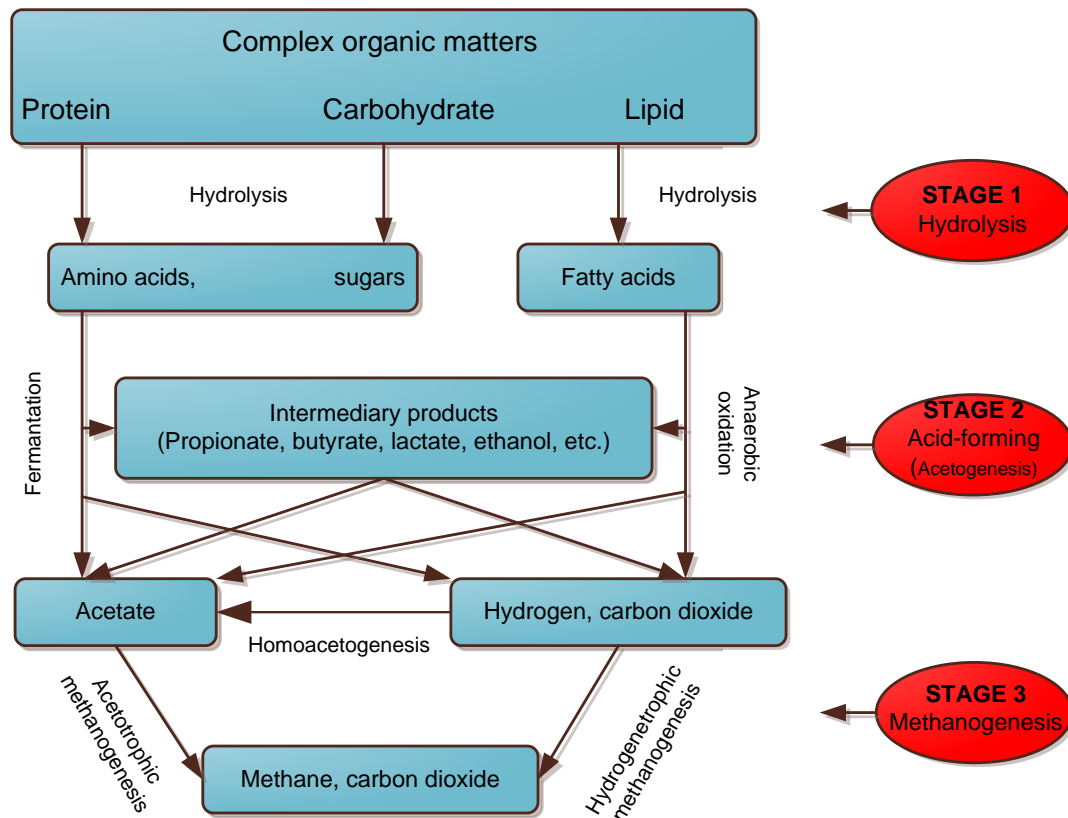
Acetate-forming bacteria are responsible for producing acetate and hydrogen that is consumed by methane-forming bacteria. On the other hand, sulphate-reducing and methane forming bacteria compete for acetate and hydrogen which is vital to their multiplication. Experiments using an anaerobic biofilm have shown (Yoda et al. 1987) that at low acetate concentrations sulphate-reducing bacteria (SRB) dominate over methane-producing bacteria (MPB). Whereas due to the higher maximum growth rates of MPB over SRB, MPB populations are larger at higher acetate concentrations. Finally, high concentrations of sulphate inhibit methane production and ways of limiting SRB activities are suggested in (Hilton & Archer 1988) that include the use of support media and the low level usage of sodium molybdate during start-up and intermittent usage thereafter.

### 1.1.3 Anaerobic Digestion Process Stages

The AD process is a multi-stage process. Three stages are mostly used to describe the sequence of metabolic reactions that take place amongst the microbial communities. These stages include hydrolysis, acid-forming (acetogenesis) and methanogenesis (Gerardi 2003)(Kaspar & Wuhrmann 1978). However, different separations of the process exist. For instance, at (Park et al. 2005) four stages are proposed that include hydrolysis, acidogenesis, acetogenesis and methanogenesis. At (Boe 2006) the stages suggested are hydrolysis, fermentation, acetogenesis and methanogenesis.

It is important to avoid inhibition of each stage of the methanogenesis process. In order to maximise methane production (Vavilin et al. 2008) and achieve a balance between the reaction rates of the steps involved in AD with a series of reactions where some steps are slower than others (Hill 1977). Hydrolysis and methanogenesis are considered to be the slowest steps in AD with suspended or dissolved wastes (Vavilin et al. 1996). If hydrolysis is inhibited the available substrates for the other stages will be reduced which can result in low methane level production. On the other hand, methanogenesis inhibition will also result in low methane production, an organic acid accumulation and an alkalinity and pH drop. In this case, methanogenic bacteria will not survive (Koster & Cramer 1987).

This plethora of bacterial communities varies in terms of (Ghosh & Fredrick G. Pohland 1974) physiology, nutritional requirements, growth kinetic capabilities and sensitivity to environmental stresses. The products of one group are the substrates of another in a sequential degradation process. The three stages of the process along with the chemical reactions appear in Figure 1.3.



**Figure 1.3** Anaerobic Digestion process (Originally taken and modified from (Khanal 2008))

### 1.1.3.1 Hydrolysis

Hydrolysis is defined as (Gerardi 2003) the splitting (lysis) of a compound with water (hydro). In an anaerobic digester complex substrates consisting of (Angelidaki et al. 2009) high molecular weight carbohydrates, fats and/or proteins are being hydrolysed. In this way, large insoluble molecules can be hydrolysed into smaller soluble ones. Hydrolysis is also considered to be a rate-limiting step in processes treating wastes that are high in lipids and/or particulate matter (e.g. sewage sludge, animal manure, food waste) (Khanal 2008).

### 1.1.3.2 Acid-forming Stage

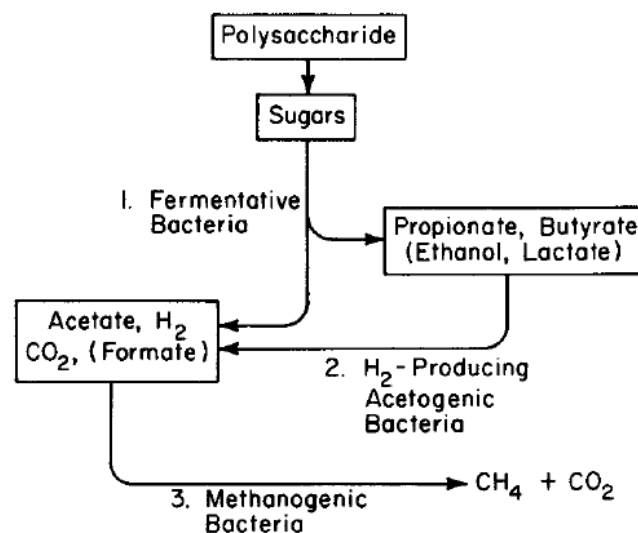
In the second stage of the AD process, the soluble molecules (intermediates) that are either produced in the hydrolysis stage or in the digester are (Eastman & Ferguson 1981) used as substrates for energy and growth leading to the production of fermentation products and cellular material. Those products include (Cohen et al. 1979)(Toerien et al. 1967) hydrogen, carbon dioxide, alcohols, fatty acids and

organic acids. Acetate, formate, methanol and methylamine are used directly by methane-forming bacteria (as a substrate), whereas ethanol, butyrate and propionate are used indirectly (fermentative bacteria degrade them to acetate). The indirect use is also known as the process of acetogenesis. Acetogenesis occurs in the acid-forming stage and is another way to produce acetate (apart from the fermentation of soluble organic compounds). This is achieved by the acetate-forming bacteria (Gerardi 2003).

The most important acid produced in this stage is acetate. The reason is that acetate accounts for a very high percentage of the methane produced that can even reach up to 90% in some cases (Mountfort & Asher 1978). In another experiment (Weber et al. 1984) where ( $UC^{-14}$ ) acetate was used, it was discovered that 65%-96% of the total methane produced came from acetate.

### 1.1.3.3 Methanogenesis

Methanogenesis is the final stage of the AD process. Acetate, carbon dioxide and hydrogen are responsible for the methane formation. However, there are other organic compounds that are responsible for the indirect methane formation such as propionate, butyrate, ethanol and lactate (Figure 1.4).



**Figure 1.4** Relationships of the three general metabolic groups of bacteria or fermentation stages involved in methane fermentation (Bryant 1979)



A rate-limiting step in methane fermentation involves the (Mackie & Bryant 1981) insufficient degradation of fatty acids for digesters that operate under high organic loading rates (OLRs) or short retention times (RT) or both. Therefore, the interspecies hydrogen transfer is also affected due to the inability of methanogenic bacteria to use hydrogen.

Methanogenic bacteria are the most sensitive bacteria involved in the AD process and although ammonia might be a nutrient for bacteria reacting in an anaerobic environment, (Koster & Koomen 1988) increased ammonia concentrations that are found in e.g. livestock waste can inhibit the methanogenesis state. As a consequence, a big drop in pH will limit methane production (Angelidaki & Ahring 1993).

The activity and adaptation of microbes in AD environment can be determined as specific methanogenic activity (SMA) with acetate and hydrogen and culturing techniques can be utilised (Ahring 1995). Research has shown that molecular DNA sequencing techniques are a very effective means of determining the microbial community profile (Delbès et al. 2001).

In conclusion, temperature, OLR, RT, ammonia, pH, alkalinity, stirring rates and feedstock composition affect methanogenic substrate uptake rates and system stability. Therefore, these parameters should be tuned accordingly in order to optimise anaerobic digestion operation.

## **1.2 Operational & process influencing parameters and conditions**

### **1.2.1 Introduction**

The key to anaerobic digestion performance is the effective production of methane by encouraging the growth and metabolism of methane-forming bacteria. So, in order to influence their activity, attention has to be given to the maintenance of optimal operational conditions.

The difficulty in controlling anaerobic digesters derives from the fact that due to the large number of operational conditions that depend on each other (interdependence), it is difficult to maintain the dynamic balance of intermediate AD processes. Changes in parameters that might favour one condition could affect others and vice versa. The cohabitation of many bacterial groups that function

efficiently within different optimum ranges is a challenge since each group has its own optimum operating conditions and is sensitive to several process parameters. Section 1.2 will focus on analyzing these parameters and conditions.

### **1.2.2 Substrate and products**

The consumption of substrates or nutrients is important towards the development of microorganisms. The composition and type of substrate along with the environmental conditions that exist inside a biogas reactor directly influence the biogas process. Therefore it is important to determine the characteristics of the substrate. Substrates are determined by (Angelidaki et al. 2009) the total solids (TS) and volatile solids (VS), chemical oxygen demand (COD), nitrogen and phosphorus.

The composition of particulate substrates plays a vital role in high-solid digestion systems by affecting the process of hydrolysis (Zaher et al. 2009). Hydrolysis rates vary depending on the (Mata-Alvarez et al. 2000) particulate component and the operational conditions when hydrolysis takes place. Substrates that are initially available for degradation include carbohydrates, lipids and proteins (Skiadas & Lyberatos 1999).

The degradation of carbohydrates, lipids and proteins results in the production of intermediates that are further degraded into other intermediates or even methane and carbon dioxide (Pind et al. 2003). Furthermore, monitoring those intermediates can characterise microbial activity. Carbohydrates are (Gerardi 2003) macromolecules or polymers that contain numerous monomers of sugars. They are converted to (Miron et al. 2000) simple sugars before taking the form of volatile fatty acids (VFA). Lipids are hydrolysed to glycerol and long-chain fatty acids (LCFA). LCFA are then used to produce acetate and propionate (Sousa et al. 2007). Proteins are hydrolysed to amino acids and are further degraded through anaerobic oxidation that is linked to hydrogen production or via fermentation according to the Stickland reaction (Kumar et al. 2010) (Miron et al. 2000).

Fatty acids or VFA are not only substrates for methane-producing bacteria, but intermediary products in the AD process as well. However, it has to be ensured that the accumulation of this type of intermediary products will not inhibit the growth of microbial populations that contribute to methane production (Aguilar et al. 1995).

Study cases where volatile acids were utilised as the limiting substrate in the development of kinetic models appear in (Saravanan & Sreekrishnan 2006).

### **1.2.3 Start-up**

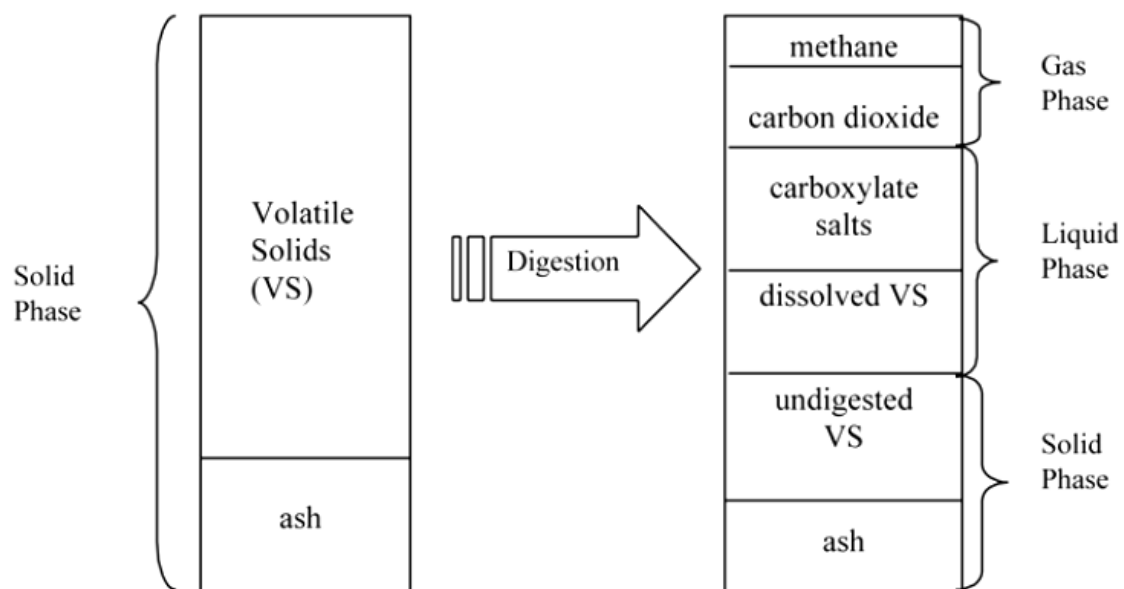
The rate of acidogenesis and methanogenesis is highly affected by the bacterial selection during start-up (Morgan et al. 1991). Substrates encourage the selection of the microbial community during start-up that is constantly influenced by the changes in the dominant species of bacteria (Anderson et al. 1994). A study that identified (mainly methanogenic) bacterial shifts through several stages during start-up in a laboratory-scale continuously mixed anaerobic reactor operating at mesophilic temperatures appears in Leclerc (2001).

Poor start-up can result in having a prolonged period of acclimation and ineffective removal of organic matter (Angelidaki et al. 2006). The amounts of inoculum and the initiation of the feeding should be designed to avoid the accumulation of anaerobic degradation intermediate products such as propionic acid and other VFA and hydrogen, which could inhibit methanogenesis and acetogenesis (Lepistö & Rintala 1995). In this way the optimum loading rate will be reached quicker and the start-up process will be successful.

Start-up operations can be categorised with respect to the loading rate (Biey et al. 2003). Start-up operation can involve low loading rate ensuring that overloading is avoided but results in having a slow and undynamic state of microbial population. On the other hand, a less conservative start-up operation involves a gradual increase of the loading rate leading to a boost in biogas production, but if the reactor overloads process failure will definitely occur. It is pointed out (Liu et al. 2006) that overload can be avoided by process operation below maximum reactor capacity. In this case however, process efficiency, degree of capital investment and operational costs are sacrificed. What is more, modern energy systems demand biogas production and quality to be stable and reliable through economically feasible operations.

## 1.2.4 Volatile solids

Material degradation of particulate and colloidal wastes, sludge feed or OLRs to digesters are expressed in terms of VS (Gerardi 2003). VS destruction is considered to be a stress indicator parameter suitable for detecting gradual changes. However, it is often too slow to detect sudden changes (Ahring et al. 1995). Biomass feed consists of VS and ash (Figure 1.5). VS except for lignin is considered the degradable and methane convertible biomass (Chan & Holtzapple 2003). VS are converted to gas (methane and carbon dioxide), liquid (carboxylate acids, extracellular proteins, energy storage polysaccharides) and solid phases (undigested VS, ash).



**Figure 1.5** Biomass feed digestion and definition (Chan & Holtzapple 2003)

VS are 80-90% of the approximately 8-13% of the total solids content range. One half are converted to the substances that appear in the gas phase and typical solid separation of the influent removes 4% of solids from the influent (Abu Qdais et al. 2010). Solids content varies according to the type of digester used in the AD process.

OLR is normally defined as the amount of VS that are inserted in the digester daily. VS represent the material that can be digested, whereas the remainder of the solids are fixed (Babae & Shayegan 2011). "Fixed" solids and a part of VS are non-biodegradable. OLRs are usually expressed in terms of chemical oxygen demand (COD)/m<sup>3</sup> day or in g VS/l/d. OLR is a very important factor whose manipulation has been the interest of many studies (Pind et al. 2003). Table 1.1 provides recommended COD loading rates with several reactor configurations (Rajeshwari et al. 2000). It can be seen than UASB reactors, anaerobic filter reactors, AAFEB reactors and AFB reactors can operate with increased OLRs of up to 30 kg COD/m<sup>3</sup>/d, 40 kg COD/m<sup>3</sup>/d, 50 kg COD/m<sup>3</sup>/d and 100 kg COD/m<sup>3</sup>/d respectively. Whereas CSTRs and contact reactors operate at much lower loading rates reaching 3 kg COD/m<sup>3</sup>/d and 4 kg COD/m<sup>3</sup>/d respectively.

<b>Anaerobic reactor type</b>	<b>Start-up period (d)</b>	<b>Channeling effect</b>	<b>Effluent recycle</b>	<b>Gas solid separation device</b>	<b>Carner packing</b>	<b>Typical loading rates (kg COD/m<sup>3</sup> day)</b>	<b>HRT (d)</b>
CSTR	-	Not present	Not required	Not required	Not essential	0.25-3	10-60
Contact	-	Non-existent	Not required	Not required	Not essential	0.25-4	12-15
UASB	4-16	Low	Not required	Essential	Not essential	10-30	0.5-7
Anaerobic filter	3-4	High	Not required	Beneficial	Essential	1-40	0.5-12
AAFEB	3-4	Less	Required	Not required	Essential	1-50	0.2-5
AFB	3-4	Non-existent	Required	Beneficial	Essential	1-100	0.2-5

**Table 1.1** Characteristics of different reactor types (Rajeshwari et al. 2000)

Along with the solid content, hydraulic retention times (HRTs) and solids retention times (SRTs) differ as well. Typical amounts of solid content and HRTs for different types of digesters can be found in (Wilkie 2005a). Retention times are important in solids destruction and will be examined in the next section.

### **1.2.5 Retention Times**

Solids retention time or sludge retention time (SRT) and hydraulic retention time (HRT) play an important role in the process control of AD systems. SRT corresponds to the mean residence time of microorganisms (solids) inside the anaerobic digester (Clara et al. 2005). HRT refers (Ekama & Wentzel 2008) to the time the liquid and the dissolved material spends inside the reactor.

Bacterial growth and maintenance is influenced by SRT which ensures that AD fermentation remains functional and stable (Zhang & Noike 1994),(Nges & Liu 2010). A connection between the specific gas production rate (SGP) and SRT exists. More specifically, prolonged SRT results in low biogas production and vice versa (Bolzonella et al. 2005). However, shortening of SRT can cause insufficient destruction of volatile solids and an increase in the quantity of residual sludge for further disposal (Nges & Liu 2010), (Appels et al. 2008). In a study (Moen et al. 2003a) using both lab-scale and pilot-plant digesters it was concluded that the destruction efficiency of VS increased from 53% to 66% as SRT augmented from 6 to 20 days. However, laboratory scale studies utilizing a (semi-)continuous stirred-tank reactor (CSTR) indicated that  $RT < 5$  days are insufficient for stable digestion (Stichting Toegepast Onderzoek Reiniging Afvalwater (STORA) 1985) cited in (Appels et al. 2008). Table 1.2 contains suggested SRT values when SRT is utilised as a design parameter for mixed high-rate digesters. It is also worth mentioning that for low rate digesters SRT is more than 30 days.

Operating Temperature (°C)	Minimum SRT (d)	Minimum design SRT <sub>des</sub> (d)
18	11	28
24	8	20
30	6	14
35	4	10
40	4	10

**Table 1.2** Suggested SRT for the design of completely mixed high-rate digesters (Tchobanoglous et al. 2003)  
(SRT<sub>des</sub>: design value of SRT including a safety margin (d))

Methane generation is highly affected by HRT (Wang et al. 1997). HRT and OLR variation can also influence digester performance. Semi-continuously fed laboratory scale digesters treating poultry slaughterhouse wastes working under mesophilic temperature (31 °C), HRT of 50-100 days and maximum loading of 0.8 kg VS/m<sup>3</sup> d, resulted in a methane yield of 0.52-0.55 m<sup>3</sup>/kg VS<sub>added</sub>. However, increased loading (1.0-2.1 kg VS/m<sup>3</sup> d) and shorter HRT (13-25 days) caused VFA accumulation and drop in methane production that resulted from process inhibition (Salminen & Rintala 2002). On the other hand, a laboratory scale study that focused on the maximisation of acetic acid production (acidogens produce substrates for methanogens) concluded that optimum production took place while operating at HRT close to the washout point (Hwang et al. 2001). However, even at long HRT, low pH values will not boost methane production (LW et al. 2001).

Literature studies contain mixed results whether HRT should be short or long, although long HRT do not destabilise the AD process. There are many parameters that affect HRT and by manipulating those parameters HRT can be minimised without having catastrophic effects on digesters. Those parameters include temperature (thermophilic temperatures shorten HRT) (Espinoza-Escalante et al. 2009), pretreatment techniques (e.g. ultrasonic sludge disintegration (hydrolysis) (Kim & Lee 2012)), type of digester and material characteristics.



## 1.2.6 Temperature

Temperature is the most important parameter in controlling the speed of the metabolic activities of microorganisms in anaerobic environments (Westermann et al. 1989), (Angelidaki & Sanders 2004). Methanogens can be divided into three categories according to the optimum performance and temperature ranges within which they are able to grow and metabolise. These are: psychrophilic, mesophilic and thermophilic. The absence of clear differences between these categories demonstrates a response for (Lettinga et al. 2001) overlapping temperature ranges (Figure 1.6).

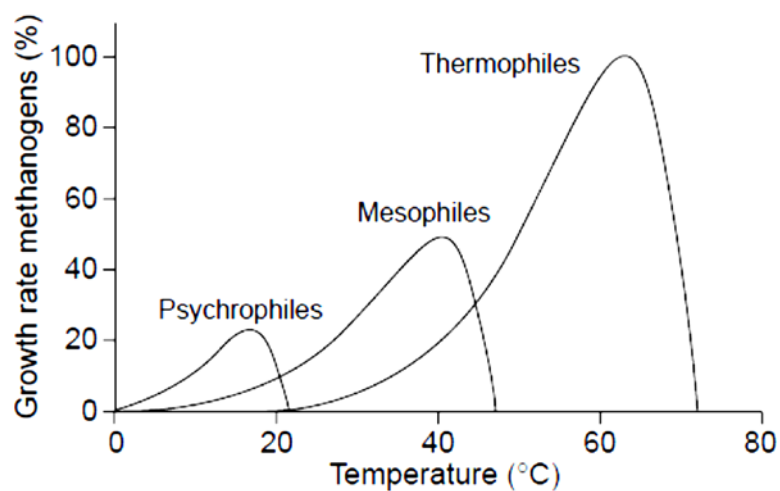


Figure 1.6 Relative growth of methanogens (Wiegel 1990), (Lettinga et al. 2001)

The dependence of optimum methane production on temperature has been investigated by many researchers. Biogas production starts at 0 °C and as the temperature increases so does gas volume. Many studies focus on comparing AD performances under mesophilic and thermophilic temperatures for optimal gas production and a number of studies appear in (Kardos et al. 2011). Digesters operated under thermophilic temperatures (with an optimum temperature of 55 °C) have several advantages over digesters operating under mesophilic temperatures. These include a faster degradation rate, higher metabolic rate, higher gas production rate, improved solid-liquid separation and increased disinfection of pathogenic organisms (El-Mashad et al. 2004), (Yang et al. 2003), (Park et al. 2008). However, thermophilic bacteria frequently experience higher death rates as compared to

mesophilic bacteria (El-Mashad et al. 2004), a lack of diversity resulting in high residual values of volatile acids and are characterised by the inability to treat sludge consistently while operation parameters vary (Gerardi 2003). Also heat is required to maintain the desired operational temperature ranges in a thermophilic digester. Heating requirements can be satisfied by (Sreekrishnan et al. 2007) adopting the auto-heated thermophilic process approach where the digestion process generates the energy needed to keep itself under the thermophilic temperature range.

Another issue regarding temperature is the fact that it should be uniform inside the reactor to avoid accumulation of different microbial populations resulting in partial undesired bacteria performance. Moreover, due to the sensitivity of the thermophilic bacteria population it has to be ensured that temperature does not fluctuate more than 1 °C per day for thermophilic reactors and changes less than 0.5 °C per day are more desirable (Tchobanoglous et al. 2003). A stop in biogas production, a rapid VFA accumulation, a decrease in pH values and the conclusion that when temperature drops inside a thermophilic reactor it should be restored as soon as possible to reach a steady state condition quicker were the findings of a study that simulated heating failure by instant drops of temperature levels from 55 °C to 20 °C (Wu et al. 2006). Low temperature (20 °C) durations were 1, 5, 12 and 24 h respectively and the operating temperature returned to 55 °C within 2 h.

### **1.2.7 pH and buffering capacity**

pH has an impact on the enzymatic activity of microorganisms because each enzyme is active within a specific pH range (Lay et al. 1997). The ideal pH range for the majority of anaerobic bacteria including methanogenic bacteria is 6.8- 7.2 (Sharma, Testa, Cornacchia, et al. 1999), (Ward et al. 2008). However, the optimal pH value for maximum biogas production depends on the substrate and the digestion technique (Liu & Yuan 2008). Rapid acidification that takes place in one-stage reactors results in a decrease in pH, an increase in VFA production and inhibits methane formation compared to two-stage digesters (Bouallagui et al. 2004). This is the reason why separation of stages (hydrolysis, acid-forming and methane-forming stage) is often preferable.

Ideal pH for hydrolysis and acid-forming stages are acidic. This might be due to the weak fermentative ability of acidogenic bacteria while the exoenzyme activity for hydrolysis is still high (Veeken et al. 2000). Optimum pH for acidogenesis of gelatine-rich wastewater is 6.0 (Yu & Fang 2003). In general, optimum pH of hydrolysis and acidogenesis has been reported to be 5.0- 6.0 according to (Ghosh et al. 1975)(Ghosh & Klass 1978)(Droste 1997)(Babel et al. 2004) and 5.5- 6.5 according to (Yu & Fang 2002)(J. Kim et al. 2003)(M. Kim et al. 2003)(Ward et al. 2008). The optimum pH value for methanogenesis is 7.0 (Babel et al. 2004)(Ward et al. 2008). However, in a two-stage anaerobic process treating high concentration methanol wastewater, it was shown that some methanogenic bacteria were able to withstand acidic conditions at pH values 4.9-6.2 (for both reactors) with the aid of granular sludge (Zhou & Nanqi 2007).

Buffer capacity or alkalinity is the equilibrium of carbon dioxide and bicarbonate ion with ammonium ion as a major cation that provides substantial resistance to pH changes (Dohanyos & Zabranska 2001). As bicarbonate is responsible for neutralising VFAs it is considered to be the main buffer (Yang & Anderson 1992). pH is also very dependent on the buffering capacity of the system (Ahring et al. 1995), (Björnsson, Murto, et al. 2001). This is justified in the studies where sugar beet silage was used as a mono-substrate. Sugar beet silage without the leaves is a poor substrate in terms of buffering capacity and the addition of nitrogen and buffering agents was used to maintain pH at stable levels (Demirel & Scherer 2008), (Demirel et al. 2009).

Digester stability is highly influenced by alkalinity. Bicarbonate alkalinity (BA) values above 2500 mg/l enhance digester stability. However, VA accumulation will reduce alkalinity preceding a rapid drop in pH (Fannin 1987). Therefore, monitoring buffering capacity gives an early warning of pH decline than pH alone especially in systems that exhibit a higher buffer capacity (Palacios-Ruiz et al. 2008). A drop in alkalinity that might result in having a 'sour' digester and will take a huge effort to bring the system back to full operation can be prevented by the addition of alkaline chemicals (Sanin et al. 2010). Those chemicals include sodium bicarbonate, potassium bicarbonate, calcium carbonate (lime), calcium hydroxide (quick lime) and sodium nitrate whose addition should be done slowly to avoid disrupting bacterial activity. Finally, due to the fact that methane-forming bacteria require BA, chemicals

that directly release BA are mostly preferred for addition (e.g. sodium bicarbonate and potassium bicarbonate) (Nayono 2009).

### **1.2.8 Toxic/ inhibiting compounds**

A variety of inhibitory compounds that are either present in wastewaters and sludges or are released during the degradation can be responsible for digester upset and/or failure. Inhibition can be detected when the steady-state of methane production and/or VA concentration is decreased, whereas a total cessation of methanogenic activity (Kroeker et al. 1979) and/or decreased substrate removal efficiency (Gruden et al. 2001) is a sign of toxicity. However, detection of inhibition might take either a short or a long time depending on the type and the concentration of the toxic compound. After the exposure of granular or crushed granular sludge to a specific toxicant concentration, it was observed that the derivatives with a higher chlorine content ( $CCL_4, C_2H_2CL_4$ ) did not exhibit inhibition that was present for the derivatives with a lower chlorine content ( $CHCL_3, trans-C_2H_2CL_2$ ) (Rodríguez & Sanz 1998). The main inhibitors of AD process are considered to be ammonia, sulphide, heavy metals, light metal cations, several organic compounds and oxygen.

#### **1.2.8.1 Ammonia**

Nitrogen is a nutrient for anaerobic microorganisms (Mah et al. 1978) and while it should be present in excess of at least  $40-70 \text{ mg N L}^{-1}$  to avoid reduction of biomass activity, high ammonia concentrations inhibit the AD process (Strik et al. 2006). However, significant differences can be found in the literature regarding the inhibiting ammonia concentration (Siles et al. 2010). Methane forming bacteria have the ability to acclimate to ammonia-nitrogen concentrations as high as  $5 \text{ g l}^{-1}$  in digested sewage sludge. On the other hand, considerable time is required to acclimatise concentrations of  $730- 4990 \text{ mg l}^{-1}$  (Van Velsen 1979).

The protein content of food waste especially, is a source of high nitrogen content leading to increased ammonia or ammonium ion concentrations inside the digester and their relative toxicity is pH dependent (Banks et al. 2011), with the more toxic form ( $NH_3$ ) dominating at high pH (Mata-Alvarez 2011). High temperatures also

boost ammonia production beyond a desired point. High hydrolysis rates that are present in thermophilic reactors boost ammonia concentrations and inhibition of the activities of thermophilic methanogens can become significant (Sung & Liu 2003a). The addition of clay minerals to thermophilic reactors and more specifically increased treatment amounts of zeolite (doses 8 and 12 g l<sup>-1</sup>) can reduce the toxic effect of ammonia (Kotsopoulos et al. 2008).

#### **1.2.8.2 Sulphide**

Food processing, pharmaceutical, and pulp and paper industries produce high sulphate concentrations. High amounts of sulphate can cause significant problems in the AD process due to the formation of hydrogen sulphide due to sulphate reduction (Valdes et al. 2006). SRB are a group of anaerobic microorganisms capable of coupling the oxidation of reduced organic or inorganic compounds to the reduction of sulphate for bio-energetic purposes (Colleran et al. 1995) and out-compete MPB for hydrogen and acetate as mentioned in section 1.1.2. The main problem of sulphide production is that it can be toxic for both methanogenesis and sulphate reduction (Wei et al. 2007) and causes reduced biogas (methane) production. Increased H<sub>2</sub>S concentrations in biogas are unwanted as H<sub>2</sub>S is corrosive, toxic and its removal is a costly process (Isa et al. 1986). It also results in producing odour, an increase of liquid effluent COD and reduced quality and quantity of biogas (Lens et al. 2002).

Sulphate can be also beneficial by satisfying the sulphur requirements of various methanogens that are expressed in terms of compounds such as cysteine and glutathione. Therefore, sulphide production by several species may enhance methanogenesis (O' Flaherty & Colleran 2000). Moreover, sulphate benefits wastewater treatment as its production precipitates toxic heavy metals such as Co, Cu, Ni, Pb and Zn (Isa et al. 1986).

#### **1.2.8.3 Light metal anions**

Light metal anions include sodium (Na), potassium (K), magnesium (Mg), calcium (Ca), and aluminium (Al). They can have an impact on AD operation and are found in effluents of anaerobic digesters that originate mainly from industrial

wastewaters or are pH regulator additives (Grady Jr et al. 1999). The light metals are always present but rarely at toxic concentrations (Alkalay et al. 1998) and can only cause problems when present at high concentrations (e.g. heavy pig diet supplementation with bone meal) (Stanogias & Pearce 1987). Moderate concentrations of light metals are needed to stimulate microbial growth (Nayono 2009).

Cation	Concentration in mg l <sup>-1</sup>		
	Stimulatory	Moderately Inhibitory	Strongly Inhibitory
Na	100-200	3500-5500	8000
K	200-400	2500-4500	12000
Ca	100-200	2500-4500	8000
Mg	75-150	1000-1500	3000

**Table 1.3** Stimulatory & inhibitory concentration of light metals sited at (Handajani 2004) modified from (Grady Jr et al. 1999)

#### 1.2.8.4 Heavy Metals

Heavy metals like cobalt (CO), copper (Cu), iron (Fe), nickel (Ni), and zinc (Zn) exist in wastewaters and sludges and eventually end up inside anaerobic digesters (Gerardi 2003). Heavy metal accumulation that is boosted by the efficiency of the digester degradation process might result in increased metal concentrations in the digestate which makes it unsuitable to be utilised as a biofertiliser (Selling et al. 2008). Depending upon their concentration heavy metals can be stimulatory, inhibitory or toxic. While a trace level is sufficient enough to enable enzyme and co-enzyme function or activation, excessive amounts can cause inhibition and toxicity (Kugelman & Mccarty 1965). Heavy metal toxicity is mostly governed by the nature of the physical and chemical environment in which they exist (Mosey et al. 1971)

which explains the variation (from several to several hundred mg/l) in both the reported dosages of heavy metals and their relative toxicity (Chen et al. 2008).

As methanogens are probably the most sensitive members of the bacteria consortium they are highly affected by heavy metal toxicity (Codina et al. 1998). Circumstantial evidence suggests that other trophic groups or organisms within anaerobic digesters other than the methanogenic populations might be affected more by heavy metals (Hickey et al. 1989). Lin discovered that VFA-degrading acetogens were more sensitive than acetic acid-utilising methanogens, some metals can be more toxic to bacteria than others, and that mixtures of heavy metals caused synergistic inhibition on acetic acid degradation (Lin 1992)(Lin 1993)(Lin & Chen 1999). Heavy metal concentrations causing 50% inhibition of methanogenesis were investigated using 100 ml serum vials (Lin 1992)(Lin 1993). Seed sludges originated from a mesophilic sewage sludge digester. The relative sensitivity of acidogenesis and methanogenesis to heavy metals is Cu>Zn>Cr>Cd>Ni>Pb and Cd>Cu>Cr>Zn>Pb>Ni respectively (Lin 1992)(Lin 1993).

In three semicontinuous digesters under thermophilic conditions that were step-fed with cadmium, copper and nickel, a tendency of acclimatisation was observed up to a certain concentration. This acclimatisation was probably due to a variety of processes including enzyme reduction, development of tolerance and changes in metabolism (Ahring & Westermann 1983). The order of decreasing solubility and toxicity was Ni>Cu>Cd. Finally, the addition of Ni, Zn and Cd up to 2.5 ppm considerably enhanced biogas production in a mesophilic digester (37±1°C) treating a mixture of cattle manure and potato waste (Kumar et al. 2006).

#### **1.2.8.5 Organic compounds**

A number of organic compounds can have adverse effects on methane production in anaerobic digesters. Poor solubility and sludge solid absorbance may lead to accumulation of organic chemicals whose accumulation will cause leakage on the cell membrane and eventually in lysis of the cell (Heipieper et al. 1994)(Chen et al. 2008). Organic compounds that are found to be toxic are reported in (Chen et al. 2008). Those include: alkyl benzenes, halogenated benzenes, nitrobenzenes, phenol and alkyl phenols, halogenated phenols, nitrophenols, alkanes, halogenated

aliphatics, alcohols, halogenated alcohols, aldehydes, ethers, ketones, acrylates, carboxylic acids, amines, nitriles, amides, pyridine and its derivatives, some LCFAs, surfactants, and detergents. The parameters that influence toxicity of organic compounds are related to (Yang & Speece 1986): the kind of substance, concentration of toxicant, SRT, biomass concentration, toxicant exposure time, cell age, toxicant administration pattern and temperature.

#### **1.2.8.6 Oxygen**

As AD takes place as the definition implies in the absence of oxygen, oxygen is regarded as a toxic compound. Especially for methanogens that are characterised as strict anaerobes (Kato et al. 1997). Therefore, the presence of oxygen can cause digester instability and poor performance. However, oxygen can be depleted by oxidation of readily available substrate or sulfide (Boe 2006).

#### **1.2.9 Mixing**

Mixing is one of the most important factors governing the operation of anaerobic reactors and its benefits are summarised as follows (Gerardi 2003):

- The bacteria, the substrate and the nutrient distribution favours the digestion process.
- The temperature is maintained stable.
- Acetate-forming and methane-forming bacteria metabolic activities are enhanced due to them being in close range.
- Waste hydrolysis is greatly improved.
- The production of organic acids and alcohols by acid-forming bacteria is enhanced.
- Limits grit settling.
- Restricts scum production.
- Minimises toxicity through quick distribution of toxic content that enters the system.



The main parameters that influence mixing are the intensity and duration of mixing, and the mixing method.

#### **1.2.9.1 Intensity and duration**

Mixing intensity influences the rheological and mechanical characteristics of conditioned sludge (Abuorfi & Dentel 1997). Measurement of rheological properties can be used as a control parameter for the optimisation and dewatering operations at wastewater treatment plants (Ormechi 2007). The intensity of mixing affects digester performance of municipal sludges (e.g. activated sludge). High mixing intensities reduce particle size and diffusion limitation that result in an increase of processing capacity for a digester treating this type of waste (Lanting 2003). And although intense mixing should be avoided during start-up to prevent digester failure, during steady-state conditions intensity variations have minimum effect on digester performance (Hoffmann et al. 2008). Low mixing intensity favours hydrogen utilisation without inhibiting propionate and butyrate degradation. Lower or intermediate mixing intensity was recommended for anaerobic reactors as they exhibit higher methane yield and increased process stability than when under high intensity mixing (Stroot et al. 2001). On the same level, Vavilin and Angelidaki (2005) concluded that at high OLRs intensive mixing inhibits methanogenic activity and growth, suggesting that low mixing intensity favours digester operation. Moreover, lower mixing intensity will reduce operational costs (Luo et al. 2012).

Mixing intensity and duration appear to have different effects as far as different types of anaerobic digester environments are concerned. A series of studies and experiments on mixing duration appear in (Karim et al. 2005)(Kaparaju et al. 2008). The conclusions drawn therein are summarised as follows:

- Adequate mixing improves the distribution of substrates, enzymes and microorganisms in digesters.
- A floating layer of solids due to insufficient mixing can be developed in digesters with low solids. This formation is avoided by increased mixing duration.
- Unmixed digesters might experience floating layers of solids but at the same time produce higher methane yield compared to mixed digesters (Karim et al.

2005). However, different results appeared in (Kaparaju et al. 2008) where higher gas production for continuously mixed digesters compared to unmixed digesters was concluded.

- A shift from continuous to intermittent mixing (2 min of mixing/h) can boost biogas production.
- Intermittent mixing is probably the most optimal for substrate conversion compared to continuous mixing. A biogas yield increase from 2.5% to 14.6% respectively was recorded during the operation of the 800 l reactor thermophilic reactor ( $54\pm 1^\circ\text{C}$ ) treating cow manure (Kaparaju et al. 2008).

### **1.2.9.2 Mixing systems**

Different types of anaerobic digestion mixing techniques exist. Those include:

- Natural mixing that is a result of rising gas bubbles and thermal convection currents that are produced by heating (Schlicht 1999)
- Unconfined gas injection systems that collect gas at the top of the digester, compress it and discharge it through bottom diffusers or through a series of radially-placed top mounted lances (EPA Design information Report 1987).
- Confined gas injection systems that also collect the gas from the top, compress it and discharge it through confined tubes (EPA Design information Report 1987).
- Mechanical stirring systems that might be constituted of large blade impeller-based systems or draft tubes (Cumiskey et al. 2003).
- Jet mixing systems that combine physical modeling and fluid dynamics. The pumped jet recirculation system that is applied in (Harrison et al. 2005) consists of a centrally located at the surface and a base located point where sludge is being drawn from. Then, duty pumps recirculate the digested sludge through six nozzles around the circumference of the digester at three levels.
- Mechanical pumping systems that circulate liquid and are mounted either inside or outside the digester.
- A recent trend in mixing systems involves the utilisation of piston-bubble mixers that not only combine mixing with heating aiming at keeping

temperature stable, but reduce electrical costs and exhibit increased VS destruction (Claro global 2012), (Infilco Degremont 2012).

It is worth mentioning that some of the above mentioned methods (gas injection systems) are based on the principle of gas recirculation which appears to be one of the most efficient ones (Maeng 1995). This is validated by the fact that mechanical mixing consumes a lot of electrical power (Massart et al. 2008) and boosts capital cost (Beddoes et al. 2007). Figure 1.7 contains some mixing system configurations.

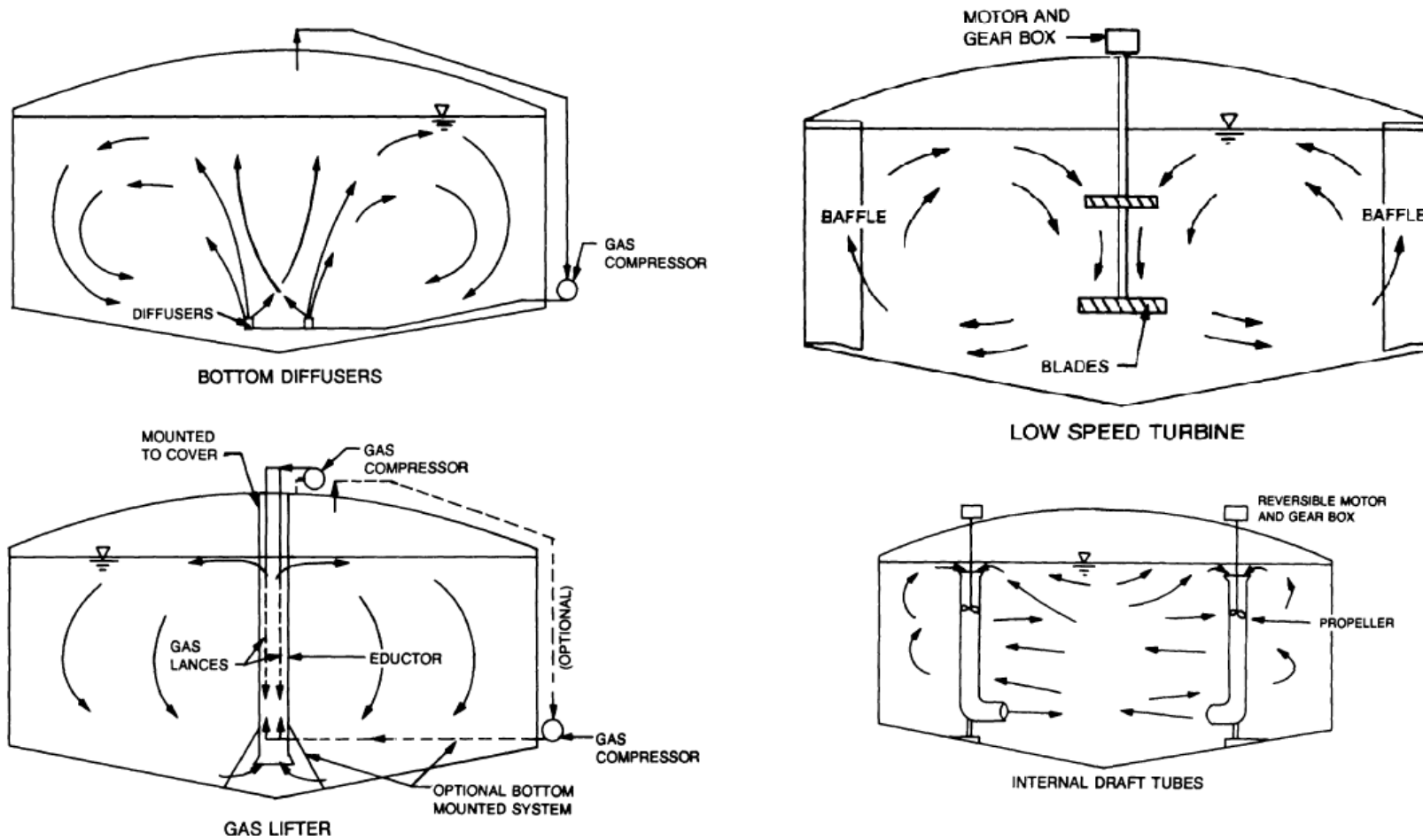


Figure 1.7 Mixing system configurations.

Top left (unconfined gas injection system), Bottom left (confined gas injection system), top right (mechanical stirring system), bottom right (mechanical pumping system).

Modified from (EPA Design information Report 1987).

### **1.2.10 Digester Design and Types**

An efficient reactor set-up must be able produce high amounts of methane and should exhibit high OLR and short HRT. Digester designs vary according to the operational factors that influence the AD process. Based on the critical operating parameters and design, digesters can be characterised as follows (Gerardi 2003),(Li et al. 2011), (Nizami & Murphy 2010):

- Wet or dry depending on the content of solids.
- Plug-flow, complete-mix, fixed-film and covered lagoons based on the design.
- Single- stage (phase) or multi-stage (phase) where separation of the processes usually exists.
- Psychrophilic, mesophilic or thermophilic depending on the operating temperature.
- Batch or continuous where the way of feeding the digester varies.
- High-rate that exhibit high SRTs and high OLRs. Those include anaerobic hybrid reactors, upflow anaerobic sludge blanket and anaerobic membrane reactors.
- Large scale or compact biogas units.

A combination of the above configurations usually exists (e.g. a high-rate, multi-stage thermophilic reactor).

#### ***1.2.10.1 Wet and dry digesters***

The TS content of the feedstock is used to characterise a digester as wet or dry. There is no clear boundary in the literature between dry and wet fermentation. Wet fermentation processes are applied when the TS content does not exceed 15%, whereas in dry or high-solids fermentation the TS content is between 15% and 35% (Weiland 2003)(Weiland 2010)(Li et al. 2011). However, <16% and 22%-40% of TS are the cut-off limits for wet and dry fermentation processes respectively according to (Mata-Alvarez 2002), and 20-40% is considered as the TS content for dry digestion in (Guendouz et al. 2010). Wet processes are operated continuously, whereas dry digestion can be applied either by batch or continuously operated processes. Wet

anaerobic systems are mostly utilised through vertical stirred tank digesters (Weiland 2010). Finally, although high-solids digestion is proving to be more difficult to control, gas production efficiency and OLR are higher (Guendouz et al. 2010).

#### **1.2.10.2 Plug-flow, complete-mix, fixed-film and covered lagoons**

Plug-flow reactors are unmixed reactors that often function horizontally handling a solid content of 10-14% and are mostly operated at mesophilic temperatures. Complete mix digesters or CSTR are systems where material is mixed and ideally should be dispersed evenly in the reactor. However, mixing does not take place constantly, but is discontinuous (Wilkie 2005a). Fixed-film digesters contain supportive media or immobilizing surfaces for bacterial attachment. They mostly operate as flow-through processes where while the material passes through a bed of fixed-film of bacteria growth, soluble organic compounds are absorbed by the bacteria and insoluble organic compounds are attached to their surface (Gerardi 2003). Finally, covered lagoons are impoundments capable of capturing biogas that often operate at ambient temperatures. Common digester types and characteristics are available in Table 1.4.

<b>Characteristic</b>	<b>Covered Storage</b>	<b>Plug Flow Digester</b>	<b>Mixed Plug Flow Digester</b>	<b>Complete Mix Digester</b>	<b>Fixed Film Digester</b>	<b>Induced Blanket Digester</b>	<b>Two-Stage Digester</b>
<b>Digestion Vessel</b>	In-ground clay or synthetically lined storage	Rectangular in-ground tank	Rectangular in-ground tank	Round/square in/above-ground tank	In/above-ground tank	In/above-ground tank	In/above-ground tanks
<b>Level of Technology</b>	Low	Low	Medium	Medium	Medium	High	High
<b>Supplemental Heat</b>	No	Yes	Yes	Yes	Optional	Yes	Yes
<b>Total Solids</b>	3-6%	11-13%	3-13%	3-10%	2-4%	<8%	≈5%
<b>Solids Characteristics</b>	Coarse	Coarse	Medium-Coarse	Coarse	Fine	-	-
<b>Hydraulic Retention Time (days)</b>	60+	15+	15+	15+	<4	3 - 5	10 - 13
<b>Farm Type</b>	Dairy, Swine	Dairy, Swine	Dairy, Swine	Dairy	Dairy, Swine	Dairy, Swine	Dairy, Swine
<b>Optimum Location</b>	All climates	All climates	All climates	All climates	Temperate / warm	All climates	All climates

**Table 1.4** Common digester types and characteristics referenced in (Wisconsin Manure Management 2010) from (Gould & Crook 2009)

### **1.2.10.3 Single- stage and Multi-stage digesters**

AD can occur in single-stage, two-stage or multi-stage configurations. In the single-stage configuration a series of different bacteria populations interact in the same environment. Acid-forming and methane forming bacteria which are the dominant bacteria communities coexist in the same environment and when changes in operational conditions occur (pH, temperature and retention times) their population may be disturbed leading to poor performance and even digester failure. Single stage reactors are characterised by decreased loading rates, long retention time requirements, and are not as stable and flexible in terms of operation compared to two-stage systems. However, the majority of commercial anaerobic digestion units are single stage. They are preferred for their reduced complexity and expense of building despite the fact that two-stage systems can produce higher yields and rates (Nasr et al. 2012). Single phase operation can take place in either parallel reactors or sometimes in reactors in series (multi-stage) (Sharma, Testa & Castelluccio 1999).

Two-stage or multi-stage two-phase digesters are those where the AD stages do not take place on the same reactors. The first phase involves the acid production stage and the second phase involves methane production stage. The benefit of separating these processes has to do with the fact that different stages share different optimum operational conditions. So, by separating these key processes, optimised microbial phases, high loading rates, short retention times, reduced digester costs and enhanced net energy recovery are witnessed (Ghosh et al. 1985). Recent studies have shown that thermal hydrolysis at 70 C° and above is implemented as a separate stage before anaerobic treatment. This leads to an increased hydrolytic activity that can boost VS reduction and biogas yield as appears in (Hartmann & Ahring 2005). However, two-stage systems do not always manage to achieve higher rates and yields and are more expensive to design (Weiland 1993) cited in (Vandevivere et al. 2002). The main advantage of two-stage systems is that they are biologically more reliable for wastes that can cause performance instability in single-stage systems (Vandevivere et al. 2002). Different types of two-stage systems exist. In some, sludge digestion and methane production take place in the same (first) tank and the second tank is used for sludge thickening and storage (Gerardi 2003).



#### **1.2.10.4 Psychrophilic, mesophilic and thermophilic digesters**

Psychrophilic, mesophilic and thermophilic digesters operate at different temperatures. Thermophilic digesters can display better solids destruction and biogas yield compared to mesophilic digesters, but the reactor stability can be easily affected and cause process instability. This was observed in the pilot scale study that was carried out in (Banks et al. 2008).

Mesophilic and thermophilic temperature co-phase digestion has also been investigated (Song et al. 2004) in an attempt to overcome the limitations of mesophilic and thermophilic single-stage digestion. The sludge exchange rate between the digesters operating at different temperature was the performance regulating factor. A 13.6l flow-through mesophilic digester and a 5l retention thermophilic digester were used for the co-phase AD system. Two completely mixed digesters of 12.2l and 5l were used for the single-stage mesophilic and thermophilic digesters treating sewage sludge respectively. The results demonstrated that effluent quality expressed in terms of soluble COD (SCOD) and VFA, specific methane yield and process stability were superior in the co-phase digestion system compared to the single-stage mesophilic reactor. Although pathogen destruction in the co-phase digestion system was almost the same compared to the single-stage thermophilic reactor, higher VS reduction was observed. Temperature-phased systems can outperform two-phase systems treating primary wastewater sludge (PS) and the organic fraction of municipal solid waste (OFMSW) where separation of stages exists (Schmit & Ellis 2001). Temperature-phased anaerobic sequencing batch reactors (TPASBR) can exhibit higher VS removal efficiency and balanced conversion of organics to CH<sub>4</sub> at OLRs of approximately 6.1 g VS/l/d compared to mesophilic two-stage sequencing batch reactors treating a mixture of sewage sludge and food waste (Kim et al. 2011). The advantages of temperature phased anaerobic digestion (TPAD) include high-rate production of biogas and maximised stabilisation efficiency of OFMSW by combining rapid thermophilic and stable mesophilic anaerobic digestion (Kim et al. 2002)(Lv et al. 2010)(Kim et al. 2011).

#### **1.2.10.5 Batch and continuous digesters**

Batch digesters are only loaded once with feedstock and are left to go through all the degradation steps in a sequence. Batch digesters are simple, low-technology, robust when dealing with coarse and heavy contaminants and their low-cost makes them suitable for developing countries. However batch reactors have problems with clogging, produce low biogas yield and operate at low OLRs (Bouallagui et al. 2005)(Vandevivere et al. 2002).

In contrast to batch digesters, continuous digesters are loaded at a regular basis and the feeding rate is variable and suit for the purpose. It is worth mentioning that 90% of the full-scale plants located in Europe focusing on the AD of organic fraction of municipal solid wastes and biowastes rely on continuous one-stage systems (Bouallagui et al. 2005).

#### **1.2.10.6 High-rate digesters**

High-rate reactors are characterised by their ability to produce increased amounts of gas in shorter time than conventional digesters. High-rate systems exhibit higher SRTs over HRTs and although low-solid wastewaters were mainly treated in the past, during recent years anaerobic sequencing batch reactors (ASBR) and baffled reactors have enabled the treatment of high-solids wastes such as animal waste (Angenent et al. 2002). Focus is given on keeping increased amounts of digested material inside the reactor using immobilisation. High sludge concentration can boost conversion rates to 40-60 kg COD m<sup>-3</sup>d<sup>-1</sup> at 30-40° C for soluble wastewaters (Rebac et al. 1995). This is mostly achieved by either single or multi-stage digesters that display higher gas volume production under thermophilic conditions. Staged-reactors are more robust under high loading conditions (Lier 1996), however when operated under thermophilic conditions temperature variations can destabilise the conversion process or even result in reactor failure as mentioned before in this chapter.

### **1.2.10.7 Large scale and compact biogas units**

Large biogas units have the benefit of producing amounts of biogas proportional to their size following an extensive start-up period. However installation, maintenance, occupying space, costs, lack of effective control systems that maximise biogas production and the fact that in order to prevent system failure the unit must not operate at its full potential have started to change the design trend.

Compact biogas units that can handle less material can provide faster results, saving money at the same time. Start-up times are greatly reduced and more efficient control systems can be designed. A very good example, though not from a control point of view, when it comes to working efficiency of small scale biogas systems is the ARTI compact biogas plant that works on waste food. ARTI was developed and distributed to 2000 urban households in India. ARTI is normally 1m<sup>3</sup> and is designed to treat 1-2kg (dry weight) of kitchen waste per day (ARTI 2012). 2kg of starchy or sugary feedstock can produce approximately 500g of methane within 24 hours (Vij 2011). The ARTI design and performance was evaluated in Tanzania and many improvements were suggested in order to maximise its efficiency (Volegeli et al. 2009).

### **1.2.11 Seeding- Immobilisation & Co-digestion**

Seeding, immobilisation and co-digestion represent the means to increase biogas production efficiency. Seeding of anaerobic filters was used to develop a sufficient bacterial population in order to minimise start up time (McCarty 1964), (Young & McCarty 1969). A thorough analysis of immobilisation is performed in (Lettinga 1995) where the importance of developing well-balanced bacterial community is underlined. Different types of media through which immobilisation can be carried out are also listed.

Co-digestion is more beneficial than the digestion of one substrate because it offers (Sosnowski et al. 2003) dilution of potential toxic compounds, increased load of biodegradable organic matter, improved biogas yield, as well as hygienic stabilisation and increased degradation rate under thermophilic conditions. The

development of positive synergism formed in the digestion medium and the existence of the needed nutrients provided by the co-substrate is influential towards biogas production and increased feedstock degradation (Mata-Alvarez et al. 2000). Moreover, it offers a decrease in costs by treating different feedstocks using one plant, better moisture and nutrient content and dilution of inhibiting compounds (e.g. ammonia and degradation products of lipids) (Luostarinen et al. 2009).

### **1.3 Process monitoring indicators**

In order to optimise and control the anaerobic digestion process, important process inputs and outputs have to be monitored during each stage of the process. Ideally, process indicators should be easily acquired and have an impact on process stability and evolution (Pind et al. 2003). This can be achieved by the indirect measurement of the activity of the different groups of microorganisms that reflect the current metabolic status of the active organisms in the system (Gujer & Zehnder 1983)(Björnsson et al. 2000). Common monitoring indicators include gas production rate, gas composition, VFAs, pH, alkalinity (Hawkes 1993)(Björnsson, Murto, et al. 2001), ammonia and indirect measurements of organic matter (Pind et al. 2003). In most cases, several process monitoring indicators are utilised in anaerobic digestion applications because they provide complementary information (Hickey et al. 1991). This section contains a brief overview of some of the most commonly used process monitoring indicators.

#### **1.3.1 Gas production rate**

Biogas is mainly composed of methane gas and carbon dioxide gas and contains traces of hydrogen sulphide, ammonia, nitrogen and other gases. Gas production rates and methane yield in particular can indicate the metabolic status of the digester. A reduction in methane production rates in comparison to the influent rate of organic matter is indicative of soluble acid product accumulation in the liquid phase (Switzenbaum et al. 1990). On the other hand, fairly constant gas production rates are representative of steady state operation (Lin et al. 1997). Gas production is also indicative of a stressed digester. Digester overloading can initially boost biogas production but results in reduced methane yields and inconsistent gas production

rates (Marchaim & Krause 1993). However, in case gas production rate values indicate process imbalance it is often too late to stabilise the process efficiently (Kleyböcker et al. 2012). Therefore, gas production rate is not a reliable early warning indicator (Switzenbaum et al. 1990).

### **1.3.2 Methane and carbon dioxide**

Biogas is a renewable energy source therefore methane yield and methane percentage are of high importance. As mentioned in the previous section, during stable operation gas production rates are constant. The same principle applies to the CH<sub>4</sub> to CO<sub>2</sub> ratio. During stable operation biogas (volume) consists of approximately 60-70% methane and 30-40% carbon dioxide (Mallon & Weersink 2007). This ratio is affected by the primary substrate composition, temperature, the duration of preservation, the bioreactor workload, the homogenous material activation, pressure and pH (Deublein & Steinhauser 2008)(Vilniskis et al. 2011). CO<sub>2</sub> composition depends on pH and alkalinity and has been proven to be an unsuitable parameter for control. Since variations in CO<sub>2</sub> percentage are dependent of the CO<sub>2</sub> 'stored' in the liquid phase as bicarbonates (Guwy, Hawkes, Wilcox, et al. 1997) and changes in pH and alkalinity can affect system performance and consequently gas composition (CH<sub>4</sub> to CO<sub>2</sub> ratio) (Ryhiner et al. 1993). However, methane production is suggested as a better indicator (Liu 2003) as cited in (Boe 2006).

### **1.3.2 Hydrogen**

Hydrogen is an important intermediate and energy carrier in anaerobic digestion that is produced during the degradation of organic matter. The reduction of carbon dioxide by hydrogen is responsible for approximately 30% of methane production generated during anaerobic digestion (Mara & Horan 2003). Hydrogen is produced by fatty acid degradation (especially propionate and butyrate) (Schmidt & Ahring 1993). Therefore, dissolved hydrogen concentration influences the amounts of various end-products of the anaerobic digestion process (Harper & Pohland 1986)(Cord-Ruwisch et al. 1997). High hydrogen concentrations can result in VFA accumulation which justifies the use of hydrogen concentration as an early indicator

for process stability (Archer et al. 1986)(Pauss & Guiot 1993)(Björnsson, Murto, et al. 2001)(Steyer et al. 2002).

Studies have shown that the hydrogen liquid-to-gas mass transfer is limited (Pauss et al. 1990)(Björnsson et al. 2001). This limitation and biological reasons including hydrogen transfer between interspecies in the liquid is the reason that gas-phase hydrogen concentrations are not representative of hydrogen concentrations in the liquid (Whitmore et al. 1987)(Pauss et al. 1990)(Pauss & Guiot 1993)(Guwy et al. 1997)(Björnsson et al. 2001).

### **1.3.3 pH**

As discussed in 1.2.7, pH values inside an anaerobic digester vary depending on the stage of the process (different pH values are optimal during the hydrolysis and acid-forming stage compared to the methanogenesis stage), on the substrate utilised in the process, digester configuration and the pH of the feed. A pH decrease can indicate VFA accumulation and can be a useful indicator in anaerobic digestion systems with a low buffering capacity (Boe et al. 2010). In systems with high buffering capacity, reductions in pH values that are a result of VFA accumulations might take a while to be recorded and will not be able to indicate process imbalance until the process becomes highly unstable (Chapter 5). This phenomenon has also been recorded in the past (Björnsson et al. 2000)(Boe 2006). Therefore, it is suggested that pH should be used as an additional measurement to characterise the state of the digester (Pind et al. 2003).

### **1.3.4 Alkalinity or buffering capacity**

As mentioned in the previous section, sometimes changes in pH are not representative of the anaerobic digestion process. Alkalinity is a more suitable process imbalance indicator since a VFA accumulation will result in reducing the buffering capacity before a noticeable change in pH value occurs (Chapter 5). Reductions in the loading rate, addition of strong bases or carbonate salts and bicarbonate addition (Chapter 4-Chapter 5) can be used to increase a low buffering

capacity in an anaerobic digestion system (Guwy et al. 1997)(Van Lier et al. 2001)(Ward et al. 2008).

### **1.3.5 Volatile fatty acids**

VFAs are the most important intermediates in the anaerobic digestion process. VFA accumulation is representative of the kinetic decoupling between acid producers and consumers and is considered to be typical for stress situations (Ahring et al. 1995). Monitoring of the VFA concentration has been identified as one of the most important parameters for anaerobic digestion (McCarty & McKinney 1961)(Chynoweth & Mah 1971)(Fischer et al. 1984)(Hill & Holmberg 1988)(Hill & Bolte 1989)(Hickey & Switzenbaum 1991)(Anderson & Yang 1992)(Ahring et al. 1995)(Mechichi & Sayadi 2005)(Molina et al. 2009)(Rani et al. 2012). In systems with low buffering capacity pH, alkalinity and VFAs are considered to be efficient in characterizing process activity and stability. However, only VFAs (Murto et al. 2004)(Boe 2006) and VFAs along with alkalinity (Rozzi 1991) (Palacios-Ruiz et al. 2008) are suggested for accurate monitoring of digester stability in system with high buffering capacity.

Methane production was inhibited by more than 50% above 13, 15 and 3.5 g/l of acetate, butyrate and propionate added to granular sludge respectively (Dogan et al. 2005) cited in (Fotidis et al. 2013). Monitoring individual VFA concentrations can provide even more useful information with respect to process stability (Hickey & Switzenbaum 1991)(Ahring et al. 1995)(Pind et al. 1999)(Pind et al. 2003)(Boe et al. 2010). Studies have underlined the importance of using VFA concentration in determining instability in anaerobic digestion systems. However, there are differences regarding which of the individuals VFAs are the most suitable depending on the substrate (Bruni et al. 2013).

Propionate was found to be a good indicator of process imbalance in laboratory scale reactors treating mixed cattle and pig manure when meat and bone meal and lipids were added (Nielsen et al. 2007). Propionate was suggested as a good indicator of process stress under gradual overload (Boe, Steyer, et al. 2007) in a study that concluded that propionate can be used as an overriding alarm parameter for well-buffered anaerobic digestion systems. However, propionic acid accumulation as high as 2750 mg/l might not impact methane production negatively

since it could be a result and not the cause of imbalance (Pullammanappallil et al. 2001). Acetate concentrations exceeding 13 mM were suggested as indicative of process imbalance (Hill et al. 1987) cited in (Ahring et al. 1995). Propionate and acetate were proven to be sensitive to organic overloads in a small scale anaerobic digester treating manure (Boe et al. 2010) and combined with biogas production could indicate both performance and process stability. Propionic acid to acetic acid ratio have also been proposed as indicators of process imbalance (Norstedt & Thomas 1985)(Marchaim & Krause 1993). Ahring (1995) concluded that the propionate/acetate ratio was an insufficient stability indicator and suggested that a parameter reflecting the concentrations of butyrate and isobutyrate could be used for early detection of system instability.

### **1.3.6 Indirect measurements of organic matter**

Measurements of organic matter are important especially in AD systems that focus on stabilizing the organic material before being disposed. This aims to minimise environmental impacts from air and water emissions rather than maximizing biogas productivity. The majority of these AD systems treat industrial and food processing wastewaters prior to discharge (Rapport et al. 2008). As mentioned in section 1.2.4, organic matter destruction is a stress indicator parameter suitable for detecting gradual changes in anaerobic digesters. Therefore, organic matter measurements of the influent and effluent of AD systems can provide useful information regarding process efficiency (Pind et al. 2003). Organic matter removal can be measured in terms of TS, VS (Section 4.3.6), TOC , COD or BOD (Garcia-Calderon et al. 1998)(Steyer et al. 2002)(Amani et al. 2011)(Martin Garcia et al. 2013).

### **1.3.7 Microbial activities**

In order to optimise the performance of an AD system, it is important to know the most effective metabolizing microorganisms that participate in each step of the AD process and to identify their reaction to system disturbances (Delbès et al. 2000). Studies towards the identification of the microbial community structure by using



culture-dependent and culture-independent molecular approaches have been conducted (Sekiguchi et al. 2001)(Sekiguchi 2006). Latest research also focuses on describing the community transitions (population dynamics) during different operational periods (or conditions) (Narihiro & Sekiguchi 2007). Community transitions have been investigated during start-up, successful start-up is important for long-term stability and efficiency, of a batch process (Shin et al. 2010). Identification of dominant bacterial groups by utilizing two-stage AD systems where separation of stages exists can lead to optimisation of specific treatments and loading rates (Rincón et al. 2013). Studies have also proved that different feedstocks impact bacterial communities in different ways. Bacterial communities proved to be quite stable during AD operation using conventional feedstock like maize silage and cattle manure (Ziganshin et al. 2013). However, the same study showed that distinct and less diverse bacterial communities participate in the anaerobic digestion of materials such as chicken manure or *Jatropha* press cake. Microbial and molecular techniques and chemical indicators are utilised to examine microbial activities and further details are available in (Pind et al. 2003)(Sanz & Köchling 2007)(O'Flaherty et al. 2006)(Lauwers et al. 2013). Monitoring of microbial activities is mostly conducted off-line and is used to give an insight of the AD process rather than controlling it.

#### **1.4 Soft sensors**

In an industrial process, such as AD, some process parameters (1.2) and indicators (1.3) cannot be measured directly by a sensor. However, they are important for control and monitoring of the process. In order to solve this problem, a process model (Wang et al. 1996) or an estimation algorithm (Chéruey 1997) can be implemented to estimate the unmeasurable variables. Soft sensor application is considered to be a suitable method where based on the monitoring of easily measured process parameters and/or the utilisation of an AD mathematical model, important process variables can be estimated. Then, the information acquired for the process variables can be used to make decisions mostly regarding the digester loading that can lead to reduced capital costs and enhanced biogas output (Simeonov et al. 2012).

Soft sensors can be split in two categories that include data-based soft sensors and model-based soft sensors (Dewil et al. 2011). Data-based soft sensors are designed as black-boxes in order to predict the unmeasurable variables and some of the most popular techniques are (Dewil et al. 2011): (1) Principle component regression (PCR) (Martens & Næs 1991), (2) Partial least squares (PLS) (Wold et al. 2001), (3) Artificial neural networks (ANN) (1.5.9), (4) Neuro-fuzzy systems (1.5.10), (5) Support Vector Machines (SVM) (Vapnik 1999), (6) Fuzzy systems (1.5.8)(Chapter 3- Chapter 5). Model-based soft sensors include (Dewil et al. 2011): (1) extended Kalman filters (EKF), (2) extended Luenberger observers, (3) adaptive observers (1.5.7), (4) asymptotic observers, (5) internal observers. Different types of observers that have been developed for AD applications are available (Costa et al. 2008).

Several soft sensor applications exist in the literature through which different process parameters and/or process indicators have been predicted. Soft sensors have been used to predict the unmeasured variables of acidogenic and methanogenic bacteria, COD and alkalinity (Alcaraz-González et al. 2002). pH, ORP and EC have been utilised to predict alkalinity (Ward et al. 2011). A soft sensor has been designed to predict VS based on feed flow and gas production rate (Oppong et al. 2013). Inorganic carbon concentration, alkalinity and VFAs have been predicted by software sensors that were designed based on a mass balance model representing the dynamic behaviour of an AD system (Bernard et al. 2001). COD has been estimated based on influent flow rate, methane flow rate and pH of the effluent (Aubrun et al. 2001). Also, models like the widely used Anaerobic digestion Model No. 1 (ADM1) (Batstone et al. 2002), have been proposed to develop state estimators (Gaida et al. 2012)(Montiel-Escobar et al. 2012). ADM1 is a common platform combining 19 biochemical and 2 physicochemical processes describing the way several components within an AD environment evolve (Antonopoulou et al. 2012).

## **1.5 Process control**

### **1.5.1 Introduction**

Despite the fact that anaerobic digesters have many desired characteristics (e.g. reduction of chemical oxygen demand (COD) of the influent leading to the production of methane), AD is known to be an unstable and difficult to control process. This difficulty is due to the fact that there is an inadequate knowledge of the anaerobic digester microbiology, the potential lack of commercial interest, the lack of operator training, the nonexistence of appropriate operational data for installed digesters, the absence of research and academic status, as well as the regrowth required after industrial toxicity episodes (Gerardi 2003).

Anaerobic technology application is strongly linked with the research advances in the field of anaerobic reactors, whose design also influences digester performance (Bouallagui et al. 2005). Efforts regarding system efficiency with respect to control have been made in the direction of optimality and improved performance. Those include the monitoring of microbial populations (Boe 2006) that aims to provide information regarding the changes in the microbial activity throughout the AD process and the way it is related to monitoring and essentially controlling process parameters by exploiting different techniques. It is also pointed out (Van Lier et al. 2001) that the need for successful biological process modelling is essential towards process design, and therefore, process control. Consequently, monitoring and control of the parameters that influence AD processes mostly in the liquid or gaseous phases (not so in the solid phase) is required. On-line monitoring of process parameters (e.g. bicarbonate alkalinity, volatile fatty acids (VFAs), COD) that enables more effective process control has led to more effective controller designs (Alferes et al. 2008). However, one has to take into consideration the sensor and operational costs when those are applied to industrial plants.

Industrial evolution can be strongly linked with control evolution and it would also follow that behind the successful system operation is an effective controller design. Despite the fact that different types of controllers and several control design techniques exist, the concern is whether a controller is suitable for a specific application. The purpose of such a controller especially when it comes to high rate anaerobic digesters is to provide stability, to assure system performance and to

protect the system from collapsing. With the term “collapsing” we refer to the situation where while trying to maximise methane production the system’s robustness deteriorates, indicating a trade-off between robustness and productivity as witnessed in the analysis performed in (Shen et al. 2006).

A control system is usually composed of the manipulated variables, the output variables and the disturbances. The most important manipulated variables include OLR, RT, pH, alkalinity, temperature and waste composition (Pind et al. 2003). Operational and process influencing parameters and conditions were presented in Section 1.2 and the most important process monitoring indicators were presented in Section 1.3. This section presents a basic short introduction to control systems beginning with the simplest closed loop and open loop control systems. Conventional controllers are examined in the next two sections. These include the On-Off controllers, the P (Proportional), PI (Proportional Integral) and PID (Proportional Integral Derivative), as well as Feedforward and Feedback-Feedforward controllers. More complex and/or intelligent control techniques are analysed next. Starting with Cascade controllers, and advancing to Model Predictive and Adaptive Control techniques, concluding with Fuzzy Logic (FL), Neural Networks (NN) and Hybrid Control implementations. Finally, each technique is accompanied by application examples in the field of biotechnological processes focusing on AD.

### **1.5.2 Open-loop and Closed-loop systems**

In open-loop systems the output does not influence or affect the control action of the input signal, which is the reason why these systems are also known as non-feedback control systems. On the other hand, a closed-loop system operates with feedback by comparing the control parameter of interest with the desired system set-point and acting accordingly. In other words, the plant is being driven by the control signal regulations indicated by feedback.

Although open-loop control is rarely utilised in practice as the majority of systems experience some sort of disturbance, it can be applied under certain conditions. Furthermore, open-loop control is an essential tool for engineers and is employed to check and compare the behaviour and response of the system with different control applications (e.g. feedback control). Therefore, open-loop control

can indicate system efficiency that has been achieved with the application of different control techniques. Finally, it is able to identify parameters to control as well as to discover the limitations of controllable behaviour.

## **Applications**

Open-loop control has been used in anaerobic digestion (AD) systems. However, due to the major disadvantage, that the output is input-controlled without taking into consideration any disturbances that might exist, it can be employed only under certain conditions. The majority of control applications in the field of AD are closed-loop. Closed-loop systems can be split into feedback and feedforward systems. However, in this area mostly feedback systems have been utilised.

A hybrid control strategy employed in an anaerobic wastewater treatment process was proposed (Belmonte-Izquierdo et al. 2009) that consists of a neural observer and a fuzzy supervisor utilised in a continuous stirred tank reactor (CSTR). In general, problems that arise from the on-line unavailability of several process measurements can be overcome by utilising a process model along with a limited set of measurements namely state observer (Dochain 2003). The Takagi-Sugeno algorithm-based supervisor, assigned to perform a control action based on the operating conditions, is capable of deciding whether the system should operate in open-loop in case of a small input disturbance (Belmonte-Izquierdo et al. 2009). Therefore, two fuzzy sets are assigned for the “needs” of the supervisor, based on the quantity of organic load that can be treated within a day by a biomass unit. A low-defined fuzzy set is used to limit open-loop control while considering a critical value of the organic load (stability threshold), and a high fuzzy set triggers the proposed closed-loop control action.

A model developed to calculate the risk of foaming in AD systems caused by the microbiological activity was proposed (Dalmau et al. 2010), where a knowledge-based model was utilised to investigate system performance through both open-loop and closed-loop simulation models by making use of the IWA Benchmark Simulation Model No. 2 (Jeppsson et al. 2007). Furthermore, an estimation of the risk of foaming was used as the evaluation criterion, based on the assessment performed on literature parameter findings causing foaming (i.e. filamentous bacteria in the

influent, organic loading rate (OLR), daily OLR variation). The approach followed, investigated different AD system configurations by the comparison of the simulation results in both open-loop and two types of closed-loop systems, providing a clearer idea of the change of parameters. So, in this case, it can be stated that if operational cost was not an issue, the open-loop system and the first closed-loop configuration have the same range and trend regarding OLR and foaming respectively.

Similarly to the previous paper, a nonlinear adaptive control was presented (Mailleret et al. 2004) for bioreactors with unknown kinetics. Once again, the AD system simulation in open-loop proved effective, justifying by comparison the efficiency of the nonlinear controller with an application on an actual wastewater treatment plant.

### 1.5.3 Feedback Control Systems

A standard feedback control system (Fig. 1.8) relies on the appropriate selection of the feedback loop components. The simplest on-off and more complicated proportional –integral and derivative (PID) controllers are preferred in most applications. These two cases will be discussed in this section, along with the equally popular techniques of P and PI control.

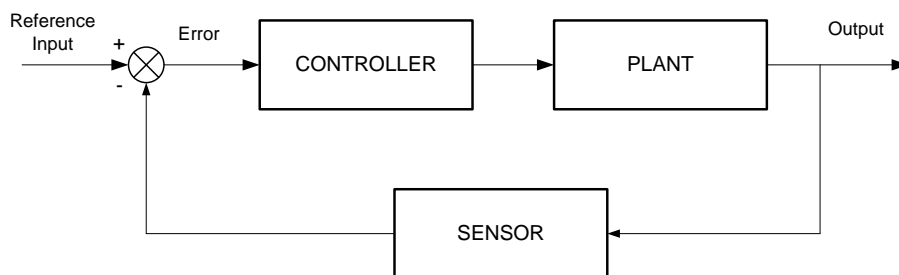


Figure 1.8 Typical Feedback Control System

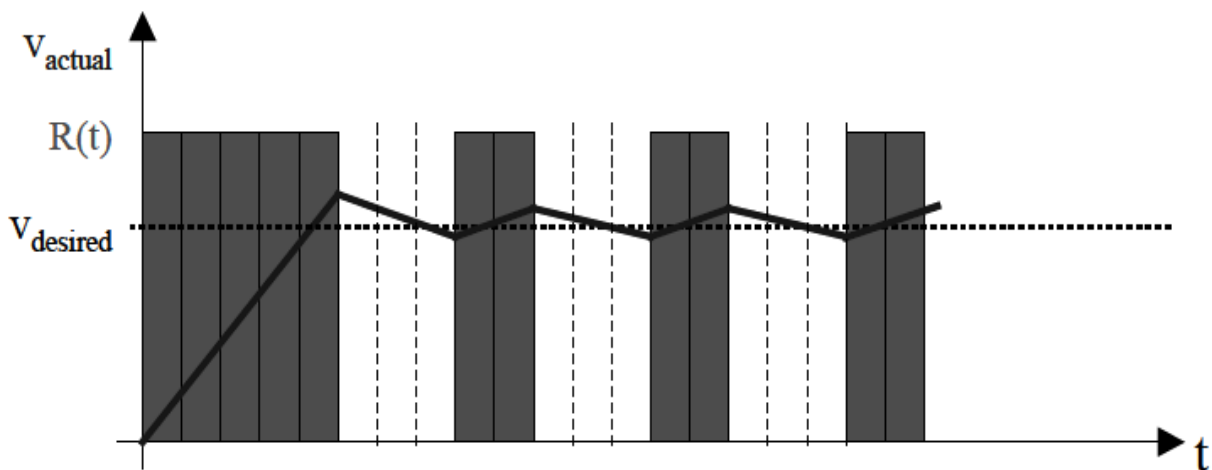
#### 1.5.3.1 On-Off Control

The simplest and cheapest feedback control applications are on-off controllers. The purpose of using such a controller is to maintain a variable within

certain limits or to manipulate it according to a predefined program. Also referred to as bang-bang control (Love 2007) where small, yet finite, errors result in switching the controller output between maximum and minimum values, based on the sign of the error.

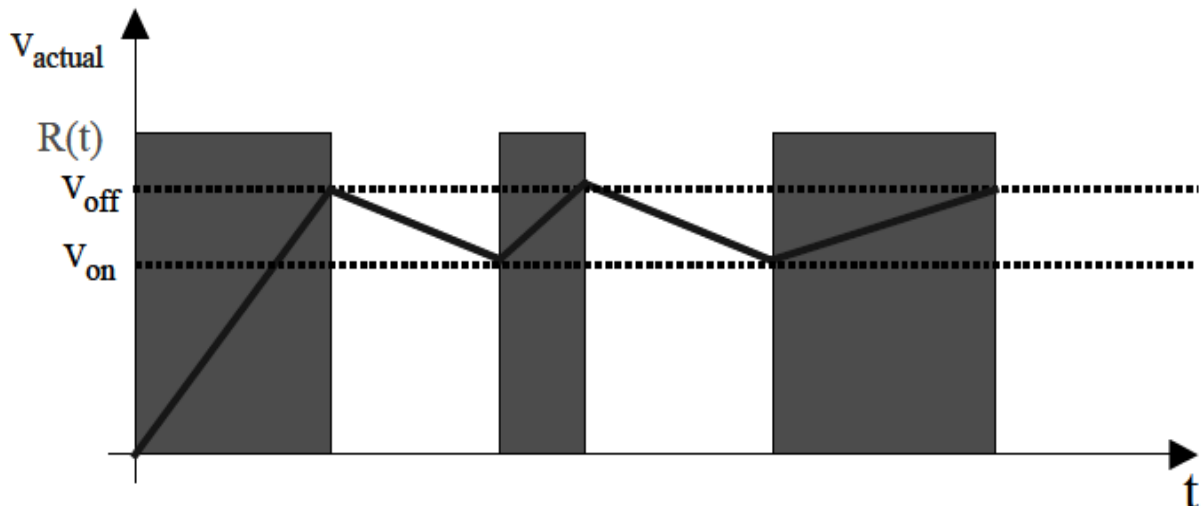
The on-off controller application usually produces variable oscillations around the set-point as shown in the example presented in Figure 1.9.

Several modifications of the on-off controller have been proposed. For example, the introduction of a deadzone (also known as hysteresis) aimed to limit high frequency noise around the desired value and protect the actuating device that might be practically used (Bräunl 2006). However, hysteresis is known to cause some delay (time lag) of the control action. Figure 1.10 shows an example of the hysteresis curve along with the control signal.



**Figure 1.9** Behaviour of an on-off controller (Bräunl 2006).

Process variable (motor speed): solid line, Control variable (constant voltage): shaded area



**Figure 1.10** Behaviour of an on-off controller with hysteresis band (Bräunl 2006)

Process variable (motor speed): solid line, Control variable (constant voltage): shaded area

### **Applications**

Despite the fact that on-off controllers are quite unstable and their application comes with increased overshoot that affects system stability, they have been utilised in several cases to control AD.

A neural network along with an on-off controller had been employed to control the bicarbonate alkalinity (a measure of fermentation stability) in a fluidised-bed anaerobic digester (Guwy, Hawkes, Wilcox, et al. 1997). The designed controller aimed to maintain bicarbonate alkalinity within certain limits, and its operation was tested during organic overload. This on-off controller was capable of maintaining the required bicarbonate concentration, but during organic overloads, a boost in overshoot was observed. Another on-off utilisation was performed (Denac et al. 1990), by tuning the feed rate and by using alkaline consumption as the controlled variable, to control the effluent quality (expressed in total acids concentration). In this case, the controller operation had been proven to be successful. Furthermore, efficient on-off controller application was achieved with pH regulation (through the removal of a weak acid) (Graef et al. 2010). pH regulation was suitable to determine the control action that involved scrubbing carbon dioxide from the gas produced and recirculating the scrubbed gas to the digester. Gas scrubbing and recycle are



presented as a possible solution to problems related to organic overload in anaerobic digesters (e.g. cation toxicity).

### 1.5.3.2 PID Control

PI/PID controllers represent the most common “tool” utilised in process control industry including AD. The controllers are tuned to equip systems with optimal responses to load disturbances (Couper et al. 2009). Essentially, the PID is a controller:

- With an output proportional to the input dictated by a tuning parameter known as controller gain  $K_P$  (P). The P term varies according to the amount of error (e) between measured value and set-point
- That monitors the offset of the set-point and takes action when, and if, such a correction is needed over time (I). I is the integral part whose integral gain is  $K_I$ .
- That enforces the controller to take action based on the rate of the error change producing a derivative (D) term.

So basically, a PID controller represents a summation of the proportional, integral and derivative terms which together calculate the controller output. Figure 1.11 contains a block diagram of a typical PID controller.

The typical mathematical representation of a PID controller is as follows:

In the time domain:

Equation 1.1

$$u(t) = K_P e(t) + K_I \int_0^t e(\tau) d\tau + K_D \frac{de(t)}{dt}$$

or

**Equation 1.2**

$$u(t) = K_P \left[ e(t) + \frac{1}{\tau_I} \int_0^t e(t) dt + \tau_D \frac{de(t)}{dt} \right]$$

In the frequency domain by applying the Laplace transformation on Eq.1.1:

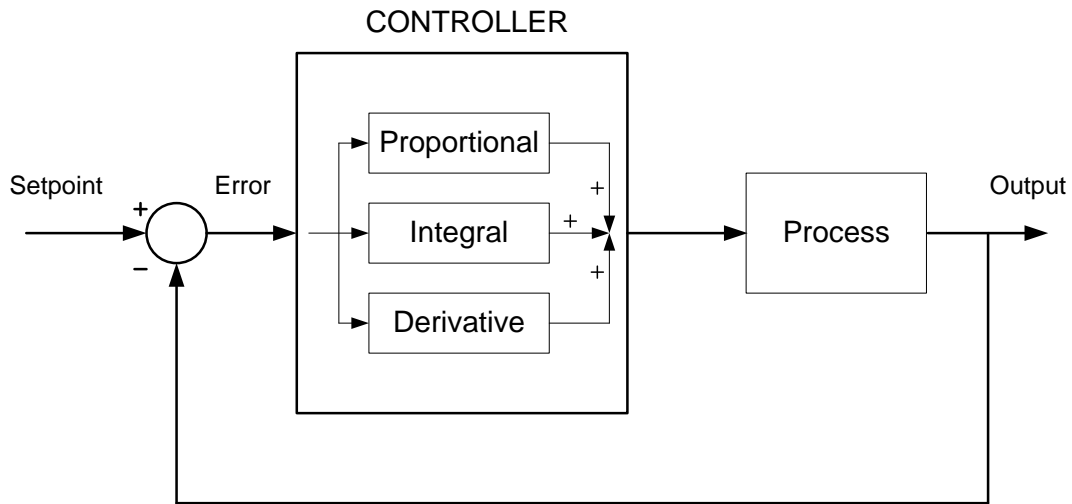
**Equation 1.3**

$$U(s) = \left( K_P + K_I \frac{1}{s} + sK_D \right) e(s)$$

Here:

- $y$  measured process variable
- $u$  control signal
- $e$  control error ( $y_{\text{setpoint}} - y$ )
- $K_P$  proportional gain
- $\tau_I$  integral time
- $\tau_D$  derivative time

In order to utilise a PID controller, the tuning parameters (proportional gain, integral time and derivative time) should be properly defined. This is the reason all PID manufacturers provide reference tuning parameter values. In the literature, several tuning methods are presented (Van der Zalm 2004).



**Figure 1.11** Block diagram of a PID controller

### ***Applications***

Extended use of PID controllers has been made for AD. In a study (Steyer et al. 2002), where an AD fixed-bed process was utilised to treat distillery vinasses, hardware PID implementation was part of the pH measuring and regulating system. Furthermore, a study aimed at developing and testing a nonlinear output feedback control law with applications for biological wastewater treatment processes, such as treating industrial wine distillery wastewater, was shown to be possible (Antonelli et al. 2002). Therein, a local PID was designed to regulate the steam temperature of the heat exchanger. The exchanger treats the fresh substrate with recycled stream, at the optimal temperature of 35° C. And in this case, the PID was programmed to assist in monitoring pH inside the recycling stream. Similarly, a PID has been utilised in (Antonelli et al. 2003).

In a scheme indirectly related to anaerobic digestion, an attempt had been made to maintain the digester temperature within the mesophilic bacterial growth and activity range of 40° C (Alkhamis et al. 2000). The novelty of this effort was that solar energy would provide the digester with the energy required to maintain the temperature level. So, after witnessing a lack of sensitivity in small temperature variations of the designed on-off controller, a PID has been employed for proper temperature control. The reason behind this controller application was mainly that

the PID had increased sensitivity to input value oscillations, operation easiness and capability in the predicting input variations.

### **1.5.3.3 P and PI control**

P and PI controllers are utilised whenever the derivative action is not required for an efficient controlled system application. Thus, they are simply PID controllers where the respective ID (in the P controller case) and D (when PI controllers are concerned) gains are set to 0 ( $k_i, k_d=0$  and  $k_d=0$ ). Despite the fact that in industrial applications whenever a system can be satisfactory controlled by either a P and a PI controller, P control is rarely, if ever, utilised in AD systems. However, its behaviour is very close to the behaviour of on-off controllers that have been examined above. On the other hand, PI controller implementations might not be found regularly, but still they exist even if they are only utilised for comparison purposes in order to investigate and demonstrate the efficiency of another more robust controller.

### **Applications**

Regulation of effluent composition for AD through the utilisation of direct feedback control had been investigated in (Alvarez-Ramirez et al. 2002). The effectiveness of a PI controller applied to regulate the effluent Chemical Oxygen Demand (COD) concentration around a specific set-point was proven. However, as observed time-delays adversely affect controller performance, a cascade controller of an integral feedback and a PI feedback was employed to achieve maximum convergence and disturbance rejection attributes. In another study, a PI controller tuned under the Internal Model Control (IMC) guidelines (a detailed analysis of IMC can be found in (Rivera et al. 1986)) was designed (Neria-Gonzalez & Aguilar-Lopez 2007). The PI controller was utilised for comparison purposes pointing out that the proposed control law, a class of nonlinear proportional control law with adaptive gain, was superior because it could track trajectories in the presence of sustained disturbances. This work was performed while investigating the tracking of sulfate concentration trajectories in a continuous anaerobic bioreactor containing the bacterium *Desulfovibrio Alaskensis*.

### 1.5.4 Feedforward Control

In contrast with feedback control applications, feedforward control strategies enable the system to act as soon as a process disturbance occurs. So, instead of tackling disturbances and system upsets based on error measurement, under circumstances that enable disturbance measurement before it is introduced into the system, a corrective action can be taken (usually in a form of a control signal) to anticipate the incoming disturbance.

A typical feedforward controller scheme is presented in Figure 1.12. The controller takes into consideration the disturbance that affects the system. However, no information is obtained regarding the effects of its action towards the system output. Usually, this application aims to interfere with the system's manipulated variables and so, force the output to stay within the desired limits. As a result, in order to plan an efficient control strategy, experience and very good knowledge of the model describing the behavior of the system is required. The absence of feedback does not induce any instability to the system (Gujer 2008) provided that the above mentioned system prerequisites are satisfied.

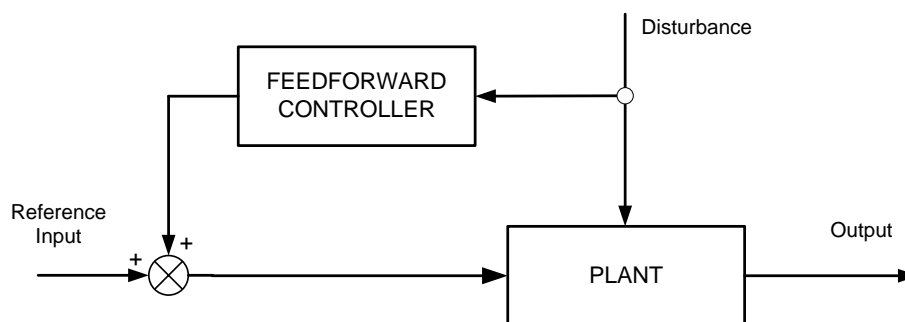


Figure 1.12 A Typical Feedforward Controller

It is also worth mentioning that the basic principles of feedforward control have been applied through another control technique that will be discussed later. Indeed, the foundation in which the neural network control approach has been developed is based on the feedforward concept. Finally, in many cases, hybrid

intelligent control schemes (e.g. neuro-fuzzy controllers) incorporate the same feedforward ideas.

The combination of feedforward and feedback control, namely feedforward-feedback control, can exploit the strengths of both strategies towards the design of a powerful controller. The possibility that feedback control strategies need to incorporate feedforward control in order to analyse the dynamic behaviour of the system (Andrews 1974) can be extended to AD systems

## **Applications**

Numerous applications of feedforward control systems can be found in wastewater treatment processes, as well as activated sludge processes. Most of them involve either predicting the dissolved oxygen concentration (Cordera & Lee 1986), or manipulating sludge recycle rate based on changes in the incoming substrate concentration and flow rate (Vonjeszenszky & Dunn 1976), or even in processes such as the control of phosphate precipitation and pH (Gujer 2008). Furthermore, nitrogen removal (Stare et al. 2007) and comparison of dissolved oxygen control techniques involving feedforward control (Yong et al. 2005) have been examined.

Because of the feedforward philosophy, there was not much use made in AD processes. This is mainly due to the nonlinearities of biochemical processes and the absence of accurate mathematical models. Therefore, a feedforward approach is insufficient on its own to be applied (at least at the moment) to AD processes. Nevertheless, the combination of feedforward and feedback control, namely feedforward-feedback control, can exploit the strengths of both strategies towards the design of a powerful controller. Such a controller was proposed (Mendez-Acosta et al. 2005), where the feedforward signal was utilised as the prescribed reference, with the tracking error being the feedback signal. Furthermore, the possibility that feedback control strategies need to incorporate feedforward control in order to analyse the dynamic behaviour of the system (Andrews 1974) can be extended to AD systems.

## 1.5.5 Cascade Control

Cascade control is a control structure that consists of two or more feedback controllers. To illustrate the idea we will restrict to the simple case of two controllers. The first (primary controller)  $C_2$  is designed to provide the second (secondary controller)  $C_1$  with the system set-point (or reference value).  $C_1$  and the secondary process  $P_1$  form the secondary control loop that is embedded inside the primary loop. The effect of the first disturbance ( $d_1$ ) is limited within this loop, minimising its effect on the primary process. Finally, the outer feedback loop is responsible for coping with the third system input  $d_2$ . A typical cascade control system is presented in Figure 1.13.

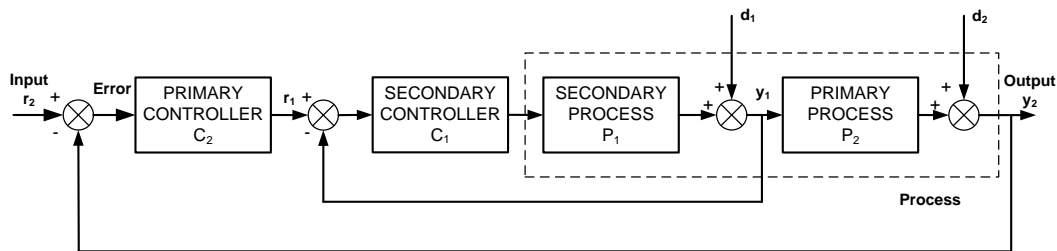


Figure 1.13 A Typical Cascade Control System

The main role of a cascade system is to reduce the effect of external disturbances (Tan et al. 2000) and also to minimise the influence of the actuator or the secondary process gain variations on the control system performance (Cooper et al. 2004). For the controller design, it is common to design the secondary controller (slave) first while keeping the outer loop open. Next, the primary controller (master) should be introduced to the system implementation including the primary process (Morari & Zafiriou 1989). Therefore, in order to obtain a system with maximum effectiveness, the inner loop should demonstrate increased dynamical response compared to the outer one (Tan et al. 2000).

However, despite the fact that practical implementation of such a controller is relatively easy compared to more complex techniques, some difficulty can be witnessed in tuning the two controllers. An example is the case where simple PID's are chosen to perform the control task.

## Applications

The most common industrial cascade implementations involve the application and tuning of the conventional P, PI and PID, with P being proposed as a suitable selection for inner loops due to its design simplicity (Visioli 2006). However, in most AD systems, a fusion is performed between conventional controllers (e.g. PI) and more advanced control techniques (e.g. FLC) to form a cascade controller.

A cascade Fuzzy PI-PI controller (Martínez-Sibaja et al. 2007) was designed to operate an upflow anaerobic sludge blanket (UASB) digester. The control system consists of a conventional PI controller (slave) that feeds the reactor pH back to the influent flow rate, and a Fuzzy PI controller (master) that measures the biogas flow rate and adjusts the pH set-point. Several simulation comparisons of the proposed cascade controller scheme with a conventional PI-PI cascade control implementation demonstrated the superiority of the cascade controller under two operational situations: the start-up process and high load situations where also sufficient disturbance rejection occurred.

The same goals, efficient performance during start-up under high load conditions, rejection of disturbances, as well as solid performance during steady-state running operations characterised the controller proposed in (Liu et al. 2004a) and (Liu et al. 2004c). The controller was applied to an upflow anaerobic fixed reactor. It consisted of a rule-based system that served as a supervisory loop, and two inner feedback loops (cascade system) designed to operate with two P controllers. The secondary controller of the cascade system utilised the reactor pH and manipulated the influent flow rate, whereas the primary controller monitored the biogas flow rate and adjusted the pH set-point value of the slave. However, it is noted that despite the fact that the cascade control consisted of two P controllers an integral action is present in the pH control loop, provided that the feeding rate was continuously tuned. Finally, the reason behind the inclusion of the supervisory system is the maximisation of gas production at all times independently of the offset value off the gas-flow control loop.



### 1.5.6 Model Predictive Control

Model predictive control (MPC) aims to mimic the human decision support system, where we seek to take control actions that will influence a system to produce the best possible predictable output over some limited horizon (Rossiter 2003). Furthermore, this is achieved by making a proper selection of the internal model of the process in question. More specifically, MPC characterises the family of controllers in which there is a direct use of an explicit and separately identifiable model (García et al. 1989). The reader is referred to (Camacho & Bordons 2004), (Rossiter 2003), (Van Den Boom & Stoorvogel 2010) and (Wang 2009) for a thorough analysis of the MPC method.

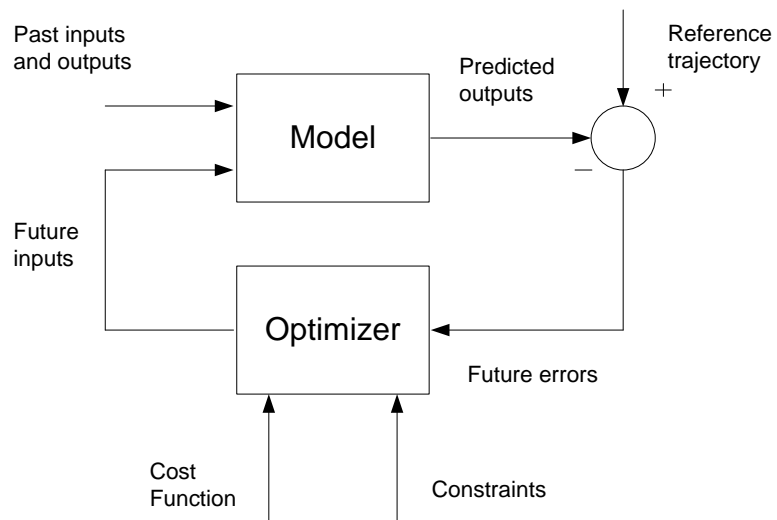
MPC is an optimisation-based control strategy that utilises a plant model to predict the effect of a possible control action on the continuously changing state of the plant. An open-loop optimal control problem is solved at each time step and the new input profile updates the plant while anticipating a new measurement to continue with the input profile updating (Rao & Rawlings 1999). At this updating point, the updated plant is in the middle of a new optimal control problem and the same optimisation process is repeated.

As MPC systems are constructed depending on the process model and process measurements that provide the feedback (and possibly feedforward) element in the MPC structure, a number of possibilities exist according to (Nikolaou 2001):

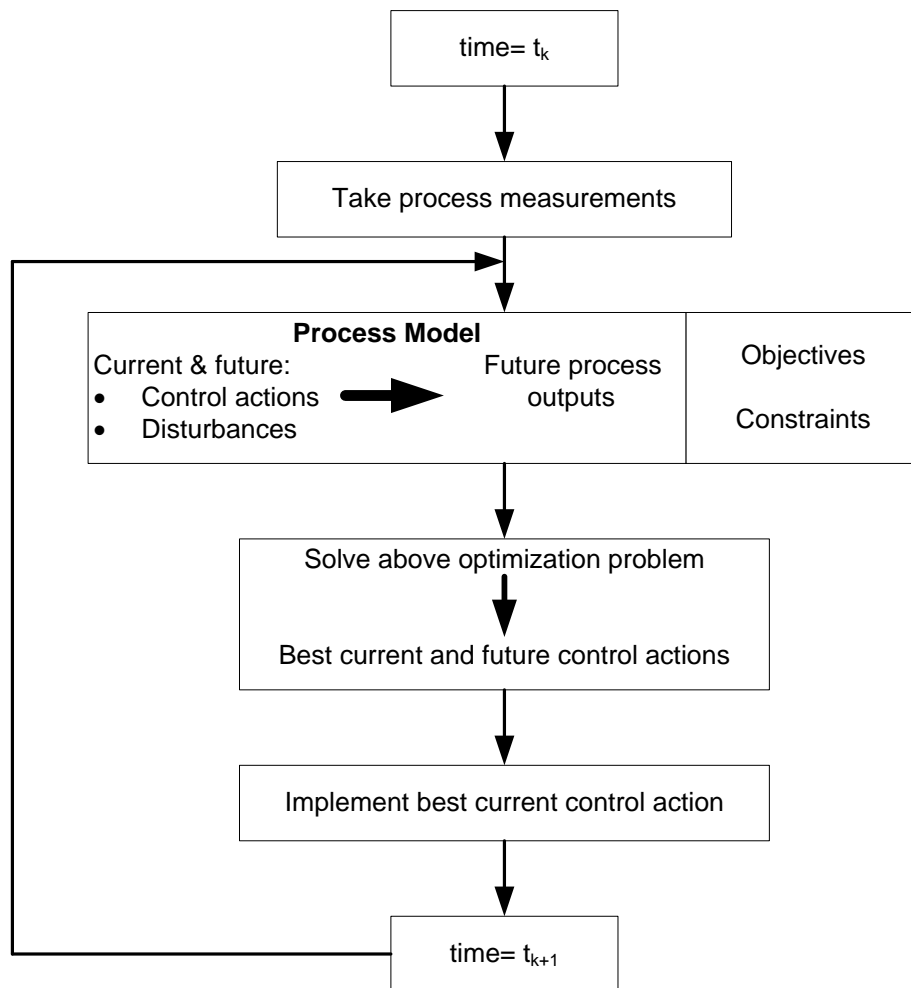
- Input-output model
- Disturbance prediction
- Objective
- Measurement
- Constraints
- Sampling Period (that depicts the frequency of the on-line optimisation solution)

However, as mentioned in (Marlin & Hrymak 1997) and (Skogestad 2000), the appropriate selection of the above should target on-line optimisation, and for this

reason MPC systems can be quite complicated. Figure 1.14 and Figure 1.15 show the typical MPC structure.



**Figure 1.14** Basic structure of Model Predictive Control (Camacho & Bordons 2004)



**Figure 1.15** A Typical Model Predictive Control Scheme in more detail modified from (Nikolaou 2001)

## Applications

The term predictive can be used to describe processes that perform exactly what the word implies following several formats (e.g. prediction is a key in controlling techniques based on monitoring of microbial performance). However, MPC schemes involve the utilisation of the main idea of this method, which is foreseeing the control action that should be taken based on the receding horizon principle aided by an on-line solution of an optimisation problem.

We will not expand on reviewing models that are used in general. So, as focus will be given to applications, the reader is referred to (Rawlings 2000) and the references within for an extended review that involves linear and nonlinear models, neural, fuzzy and local network models. Moreover, an overview of the commercially

available MPC technology is presented in (Qin 2003). Lastly, a framework for multi-parametric programming and control is addressed in (Narciso et al. 2008), where the authors discuss the issue of how balanced truncation and multi-parametric programming techniques can be combined to solve MPC problems posed as quadratic convex problems.

A study focused on determining the transition of ethanol-producing bioreactors from batch to continuous operation and subsequent control subject to constraints and performance considerations (Mhaskar & Aumi 2007). Therein, based on a Lyapunov non-linear model predictive controller and after setting the appropriate stability constraints needed to ensure the feasibility of the optimisation problem, a set of stabilising initial conditions was utilised to determine the time length of the reactor operation in batch mode.

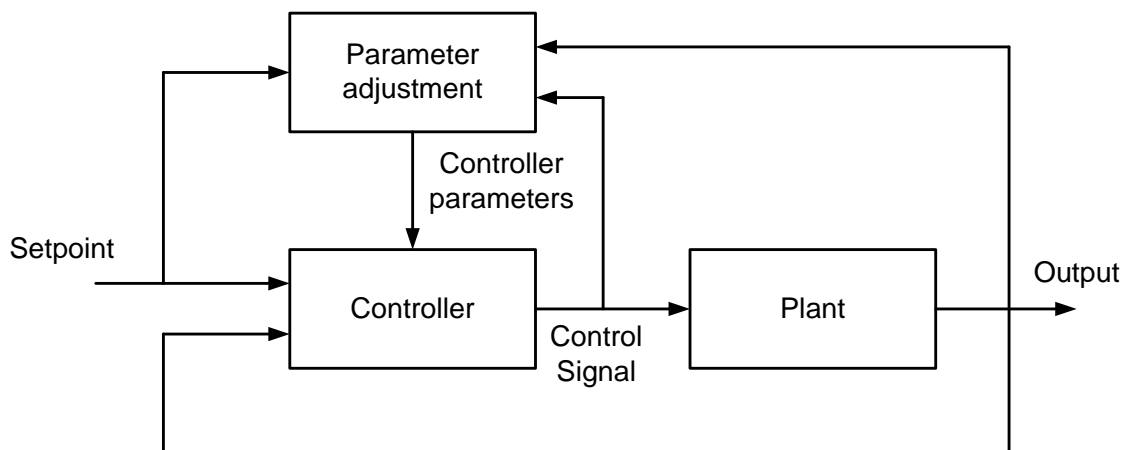
The effect of varying the length of the prediction horizon as well as the input influence on the system behaviour for different initial conditions was examined (Ramaswamy et al. 2005). MPC application in a continuous stirred tank bioreactor was proved to be successful, managing to control it to an unstable operating set-point while examining biomass concentration.

Neural network implementations are going to be analyzed and examined in the Neural Network Section. However, due to the fact that neural network control designs are based on the philosophy of MPC, wide usage has been done by researchers in the field of AD. The suitability of neural network implementation in controlling AD processes is justified in (Azlan Hussain 1999).

Briefly, an identification and control scheme using adaptive on-line trained neural networks applied to an AD process was performed in (Emmanouilides & Petrou 1997). Hydrogen production rates in a sucrose-based bioreactor system (Özkaya et al. 2008) were predicted by employing an artificial neural network. Moreover, by employing MPC strategies through the Matlab Neural Network Toolbox, trace compounds in biogas from AD were predicted (Strik et al. 2005) even under dynamical conditions. Finally, different optimising methods in order to design an external recurrent neural network based Smith predictive controller are analysed (Tan 1996), and the evaluation of the proposed control algorithms was performed with the simulation of an AD process in wastewater treating.

### 1.5.7 Adaptive control

An early definition of an adaptive system is given in (Mishkin & Ludwig 1961), where it is described as “any physical system that has been designed with an adaptive viewpoint”. A pragmatic attitude towards the definition of adaptive controllers is considered (Åström & Wittenmark 1995) where: “an adaptive controller is a controller with adjustable parameters and a mechanism for adjusting the parameters”. Furthermore, an extension of the adaptive control definition to adaptive optimal control appears in (Bitmead et al. 1990) that reflects the adaptive control scheme where the controller is designed based on methods of optimal control theory. A block diagram of an adaptive system is shown in Figure 1.16.



**Figure 1.16** Block diagram of an adaptive system consisting of two loops (Åström & Wittenmark 1995). The first loop consists of the feedback, the process and the controller. The second loop is the one where the parameter adjustment takes place.

Adaptive control can provide automatic process control under uncertainties and fluctuations in system parameters and dynamics. Moreover, it can tackle changes in disturbance characteristics. A variety of adaptive control applications in several industrial areas is listed in (Chalam 1987).

## Applications

First of all, bioprocesses are known to be non-linear and are characterised by complicated dynamics. Second, there is a shortage of cheap and reliable on-line measurement and real-time monitoring equipment (Bastin & Dochain 1990). An adaptive control approach can be considered efficient enough (Petre et al. 2008) to tackle the non-linearity of the processes and the uncertainty of kinetics. For this reason, software sensors (Petre et al. 2008) are utilised to handle the measurement of the state variables. A list of adaptive control applications is available in (Astrom & Wittenmark 1995), underlining their application in the entire industry spectrum.

A model for a single bioreaction occurring in a continuous stirred tank reactor with unknown kinetics was considered (Mailleret et al. 2004). Eventually, after developing a stabilizing non-linear controller around the equilibrium representing the set-point, the control law was extended. In this way, an adaptive version of the controller arose that did not require any prior information of two out of the three parameters considered previously, because they are determined by the value of the targeted set-point for substrate concentration. Based on this work, Dimitrova and Krastanov (Dimitrova & Krastanov 2010) developed a non-linear adaptive feedback law in order to asymptotically stabilise a system that models methane fermentation towards an unknown maximum methane production rate.

Observer-based estimators were implemented and tested while trying to predict the kinetic rates inside a bioreactor (Farza 1998). Furthermore, an adaptive extremum seeking control scheme is presented in (Guay 2004). Moreover, an adaptive observer-based control strategy attempted to tackle with the uncertain models that are present in bioreactor processes (Boskovic 1995). Where, following the stability analysis the simulation results reported satisfactory system response.

A nonparametric statistical approach of process identification (Hilgert 2000) resulted in the design of an adaptive controller capable of handling unpredictable internal changes and process disturbances. A fluidized bed reactor treating industrial wine distillery liquid wastes was used to demonstrate the controller's improved robustness while handling reference set-point changes, as well as ease of implementation and tuning.

The on-line estimation of influent disturbances was the goal in (Theilliol 2003). A novel approach to perform a fusion of non-linear dynamics and control tools was developed (Rincon et al. 2009), where normal form theory and adaptive control were brought together to control an anaerobic digester. Here, based on the system model, a local bifurcation analysis was performed related to the phenomenon of interest. Next, a computation of the corresponding non-linear normal form for this scenario was performed for the controller to be designed.

The pollution level of non-linear bioprocesses with not fully known dynamics has been a subject for control. Adaptive controllers were implemented (Petre et al. 2008), coupled with a state observer and a parameter estimator that served the role of software sensors.

Temperature and pH regulation has been the subject for control in the industry by applying adaptive control (Bastin & Dochain 1990). An adaptive scheme was utilised to control temperature (Brazauskas & Levisauskas 2007), by implementing an adaptive feedback/feed-forward controller in an industrial methane tank operating in municipal sewage plants. A very good example of pH regulation is the controller consisting of a non-linear state feedback law and a gain adaptation that was applied in a biogas tower reactor treating wastewater (Ilchmann & Pahl 1998). However, the controller could only function properly if the input constraints were not too tight.

### **1.5.8 Fuzzy Logic**

The theory surrounding fuzzy sets was originally established by Zadeh in (1965). However, it was Mamdani's work (1974) that led to the acceptance of fuzzy control as a worldwide acceptable control strategy, based on Zadeh's original ideas.

The concept behind the development of fuzzy systems is that a machine is programmed to operate as a human being, by giving the control system the structured reasoning of an experienced operator so that real-time (most of the time) decisions can be performed. The most common way to represent human knowledge is by forming language expressions (linguistic rules) of the type:

"IF (antecedent), THEN (consequent)", which is called the IF- THEN rule-based form. In this way, the complexity of the systems that cannot be successfully modelled mathematically can be overcome by the use of linguistic variables, which can

represent and describe the system uncertainties as fuzzy sets and logical connectives of those sets (Ross 2004). Each fuzzy set consists of membership functions that consist of elements which map to a membership value between 0-1 (Cakmakci 2007).

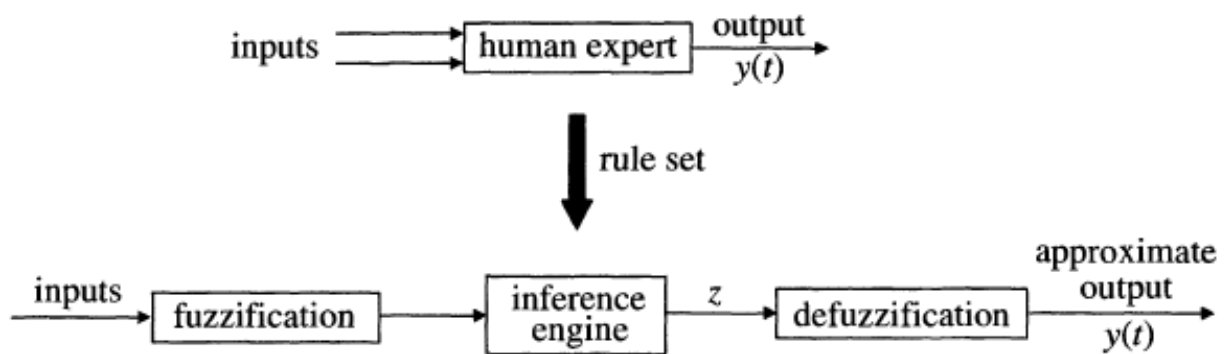


Figure 1.17 A Fuzzy Control System (Jamshidi 2003)

Figure 1.17 shows the relationship between a human expert and a fuzzy system. The fuzzy inference process can be summarised with the following steps (Cakmakci 2007) (Jamshidi 2003), as presented in the above figure:

- Obtain process values (inputs)
- Fuzzy operations are applied to antecedents through a fuzzy rule-based system
- A new set of consequents arises from the above operations
- The rules outputs are aggregated into a new fuzzy set
- The fuzzy set is defuzzified in order to obtain a crisp value

FL is a control method suitable for applications where there is a lack of knowledge regarding the specific model of the system, or if the model is too complex to be controlled by utilizing classic control methods (e.g. PID). FL can be applied when the most optimal solution to the control problem is not required often because it is not possible to achieve one. Furthermore, it enables human experience to be embedded in the controller.

FL is a method like NNs where the controller is considered a “black box” and data acquisition and classification can prove to be difficult tasks. Another important



issue to be addressed is the fact that it is not that easy to investigate optimality and robustness of the designed controller. However, FLC stability studies have been performed (Berenji 1993). Moreover, controller tuning can be complicated because there are no guidelines regarding this process. The most common tuning method is trial and error which can be a time-consuming task as is testing the controller which mainly depends on the rule-size.

## **Applications**

The FL AD controller can be utilised to achieve different goals (Puñal et al. 2003) (Yordanova 2004):

- Keep the required concentration of organic matter at the reactor output
- Reach an optimal methane production level
- Succeed in producing a stable operation in case of systems treating high OLRs affected by input concentration and/or flow rate oscillations.

Several FLC applications can be found in the literature. Yordanova (2004) developed a two-level FLC for the biogas production rate in the anaerobic wastewater treatment plant (WWTP), pointing out the efficiency of the fuzzy approach compared to the application of a conventional PI controller. Another FLC was developed (Scherer et al. 2009) to control biogas reactors using energy crops. The resulting system proved to be successful during start-up and while recovering from failure. The FLC achieved the desired process performance under high OLR and low hydraulic retention time (HRT) without utilizing any special mathematical model or detector or self-learning network. OLR was determined based on pH, specific gas production rate (GPR) and CH<sub>4</sub> content. Specific GPR was chosen instead of volume GPR, as the latter was proven unable to support pH control efficiently. Although redox is widely used as a process parameter, it was not utilised in this case as it was found to be lacking reliability. The number of the FLC rules was selected as 3<sup>x</sup>, where x is the input number. Finally, it is recommended that the FLC process variables should be reconfigured for different substrates.

A FLC based on the utilisation of cheap on-line sensors (Estaben et al. 1997) enables the system to function around a set-point and achieves good chemical

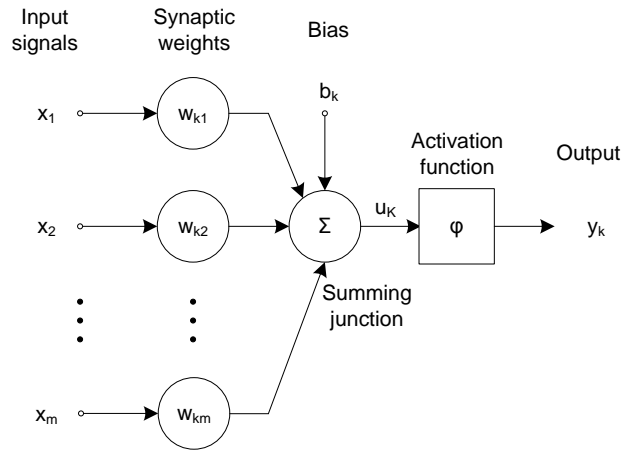
oxygen demand (COD) reduction. Stable operation was possible around a working point with perturbations or overloading conditions.

A two-stage anaerobic wastewater pre-treatment was controlled (Murnleitner et al. 2002) with a FLC system predicting the biological state of the reactors. Control was based on which control actions were taken to maintain process stability and this approach proved to be suitable for applications involving strong volume and concentration variations, or where additional feed can achieve higher biogas production. Finally, the main control issue in the design that appears in (Carrasco et al. 2002) is successful operation recovery in the case of disturbances and, similarly to the previous work, proper state detection of the WWTP.

### **1.5.9 Neural Networks**

Artificial intelligence (AI) is the field of computer science that tries to construct intelligent machines. Several definitions of AI are listed in (Russel & Norvig 2003). NNs or, more specifically, artificial neural networks (ANN)s represent one of the tools that are utilised to solve computer science problems.

A NN is an interconnection consisting of simple processing units (or nodes) that are an abstraction of the behaviour of a human neuron, and is characterised by the ability to learn and respond (Gurney 1997) (Jamshidi 2003). A non-linear model of a neuron is shown in Figure 1.18.



**Figure 1.18** A non-linear model of a neuron modified from (Haykin 1998).

The typical neuron consists of: (1) a set of connecting links named as weights, (2) a summing junction for the input signals, and (3) an activation function. This single-layer neural network is known as perceptron.

The above neuron (k) is characterised by the following equations:

**Equation 1.4**

$$u_k = \sum_{j=1}^m w_{kj} x_j$$

**Equation 1.5**

$$y_k = \varphi(u_k + b_k)$$

where  $x_1, x_2, \dots, x_m$  are the input signals;  $w_{k1}, w_{k2}, \dots, w_{km}$  are the weights of the neuron;  $u_k$  is the linear output of the input signals;  $b_k$  is the bias;  $\varphi$  is the activation function; and  $y_k$  is the output signal.

However, neurons are part of a network and the way they operate is influenced by their (MacKay 2003):

- Architecture

The term architecture involves the network structure, referring to the network variables and their topological relationship. These include single layer

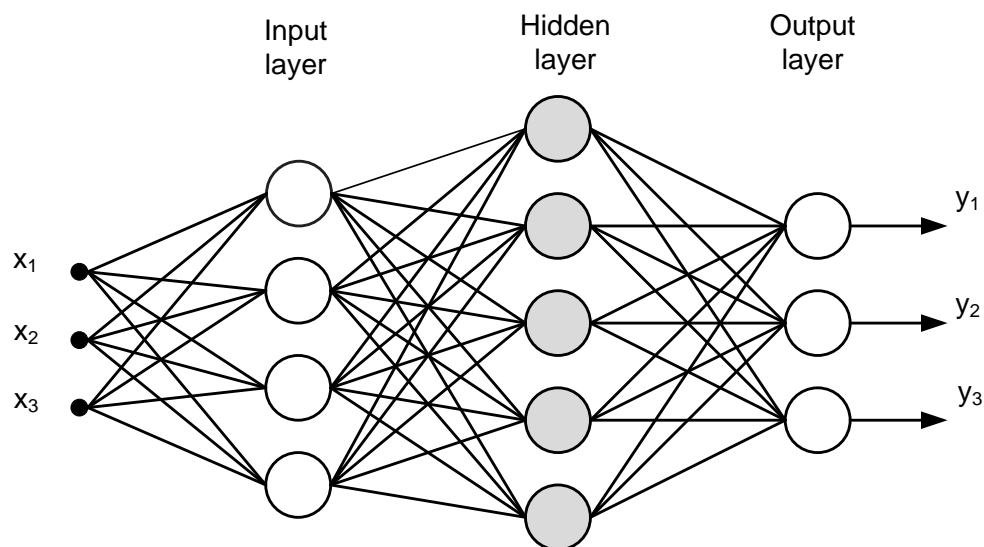
feedforward networks, multi-layer feedforward networks and recurrent networks. A multi-layer NN, a feedforward NN with more than one layer of neurons that is known as a multi-layer perceptron (MLP), is presented in Figure 1.19.

- Activity Rule

Involves the manner in which the neuron activity is modified through interaction between neurons.

- Learning rule

The most important ability of ANN is the ability to learn that can lead to an improved system performance through environmental adaptation. Several methods can be applied with respect to learning. These methods cover a vast area but Haykin (1998) provides a thorough analysis. A popular example is the back-propagation algorithm that is utilised to train an ANN efficiently, being capable of performing the required massive amount of computation required when complicated topologies are involved (Rojas 1996).



**Figure 1.19** A feedforward neural network with one hidden layer

NN are popular because of their powerful properties and abilities (Haykin 1998) that include:

- Nonlinearity

- Input-Output mapping
- Adaptivity
- Evidential response
- Contextual information
- Fault tolerance
- Very large scale integrated (VLSI) implementability
- Uniformity of analysis and design
- Neurobiological Analogy

On the other hand, the reasons behind not adopting this control method include:

- Slow learning when it comes to several applications (e.g. data mining)
- Explicit process or model knowledge is not present due to complex and/or hidden network structure and weights.
- The sample size has to be large enough for the model to be able to generalise (Vogt & Bared 1998).

## **Applications**

As already mentioned in the MPC section, ANN design incorporates predictive capacities. Therefore, a list of ANN applications is available in that section. Several applications that involve different control parameters and objectives will be examined here.

In an extended review of ANN under the scope of MPC (Arumugasamy & Ahmad 2009), the authors point out the efficiency of such control implementations with regard to set-point tracking, as well as disturbance and noise rejection. Furthermore, loading rate regulation during start-up and recovery (Holubar et al. 2003) was achieved with the optimisation of the feeding profile for future time steps, based on gas production and gas composition, through the design of two hierarchical network levels.

The implementation of different MPC strategies involving generic model control (GMC), direct inverse control (DIC) and internal model control (IMC) in a batch reactor using NN techniques has been performed (Mujtaba et al. 2006). NNs were used as a dynamic estimator (for heat release); a predictor (for reactor

temperature) and controller (for jacket temperature) respectively. The above control applications were successful at dynamic set-point temperature tracking. However, the robustness analysis of all three schemes underpinned the superiority of the GMC controller compared to the other two.

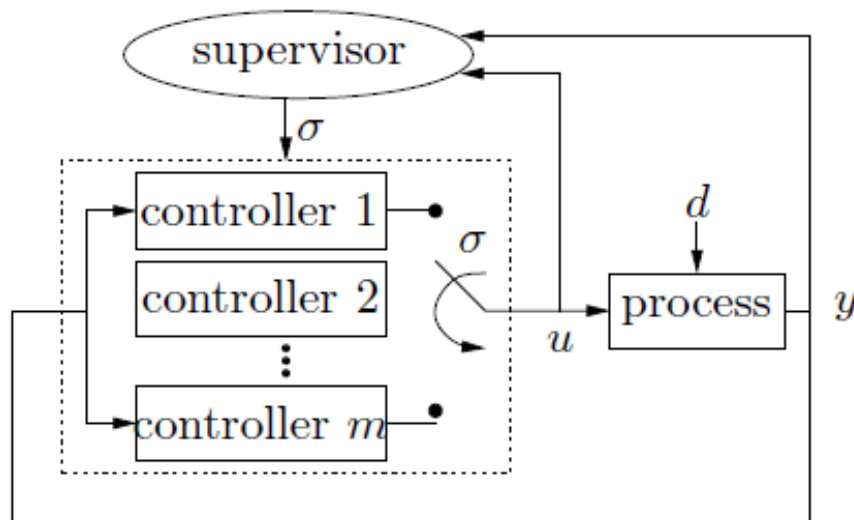
A control application to manage bicarbonate alkalinity (BA) in a fluidised-bed anaerobic digester (Guwy, Hawkes, Wilcox, et al. 1997), consisted of an on-off controller and a NN. The NN controller's goal was to classify the BA data (under steady-state or overload) by using the backpropagation algorithm and successfully kept BA levels under overload conditions without experiencing any overshoot. However, BA control was not sufficient to regulate the entire process, as other undesired parameter level changes (e.g.  $H_2$  and VFA concentrations, % $CO_2$ ) were witnessed.

An example of ANN modelling of non-linear processes is presented (Horiuchi et al. 2001). Instead of describing the system process with differential equations, output data resulting from input changes were used to model the transient system response. More specifically, the response to pH changes in an acid reactor under different retention times was used to model the transient behaviour of the system. However, during the evaluation of this method, some differences were encountered between the simulation and the experimental results. It is stated that those are due to the dissimilar dynamic behaviours encountered for pH up-shift and pH down-shift in the acid reactor. Finally, an ANN capable of utilizing the pH response for on-line measurement of the buffer capacity and alkali consumption during a fermentation process was developed (Hur & Chung 2006) in the absence of expensive sensor equipment to provide biomass estimates of reasonable accuracy.

#### **1.5.10 Hybrid Control**

Hybrid systems are dynamic systems that inter-mix discrete (modelled by means of automata) and continuous-time components (based on differential equations) (Manna & Pnueli 1993) (Chiou & Wang 2008). The need for hybrid control applications lays in the fact that (Lunze 2002) hybrid phenomena can neither be analyzed nor symbolised through techniques that are meant to be applied in either the discrete or the continuous domain.

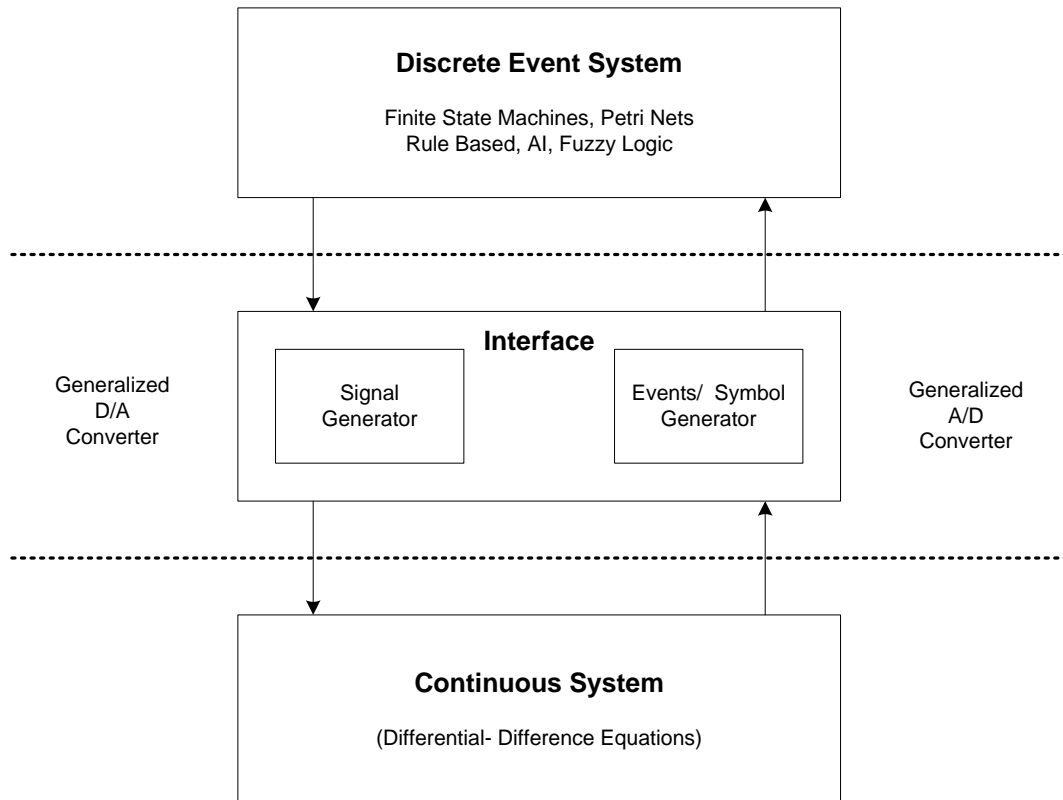
A hybrid controller (Fig. 1.20), consists of (Hespanha 2004) several different (two or more) controllers that are either utilised in the control process or not, depending on the on-line measurements. This switching process aims to (Lemmon et al. 1998) expand the efficient operating scope. Moreover, the element that controls the switching between controllers is called a “supervisor” and is responsible for deciding which controller should participate in the control process at each time.



**Figure 1.20** Hybrid System Architecture I (Hespanha 2004)

A different approach to hybrid control (Fig.1.21) architecture was introduced (Godbole et al. 1995) based on the basic two-level (layer) structure. The lower level contains the plant and conventional controllers, whereas the upper level consists of a more abstract plant description (e.g. FL, Petri nets). The interface is responsible for communication between those levels.

As mentioned above, continuous systems dynamics are usually modelled by differential equations. However, discrete phenomena generally consist of four types: autonomous switching, autonomous jumps, controlled switching and controlled jumps. Discrete and continuous phenomena and a summary of five models of hybrid systems arising from dynamical systems with respect to control are described by (Branicky et al. 1994).



**Figure 1.21** Hybrid System Architecture II (Godbole et al. 1995)

Hybrid controller design is based on a combination of control techniques which mainly utilise fuzzy, NN, MPC and adaptive control ideas. With such control implementations controller efficiency can be maximised by minimising the limitations of an individual controller. Another popular control technique usually considered under the hybrid framework is the ANFIS (adaptive-network-based fuzzy inference system) controller, which is a fuzzy inference system whose design is based on the adaptive network concept (Jang 1993). The controller incorporates the knowledge (e.g. human expertise) and the predictive capabilities of the FIS, as well as the adaptation provided by the NN. So, it is feasible to achieve automated FLC tuning and/or to predict future behaviour more accurately than NN would have predicted. Therefore, the ANFIS system can also learn from the data being modelled, and as the creator of ANFIS states (Jang 1993) it can replace any adaptive or learning control application based on NNs with the same efficiency. The above is also valid in those cases where process understanding in mechanistic sense is poor and NN have to be equipped with other source of knowledge (Zulkeflee & Aziz 2007) that hybrid control is capable of providing.



However, hybrid systems are difficult to be analysed and designed compared to entirely discrete or entirely continuous systems, because (Lygeros 2004) the dynamics may affect system performance with time and system performance with time may affect the dynamics. For example, whereas both discrete and continuous states can be altered with a discrete “jump”, a continuous state can be altered by flowing in continuous time with respect to a differential equation.

## **Applications**

The combination of logical decision-making and continuous control law generation has led to hybrid industrial applications involving (Branicky et al. 1998) programmable logic controllers (PLCs), flight management systems, motors, constrained robotic systems and highway systems. Consequently, hybrid controller implementations have been considered in the field of biotechnological processes.

The lack of sensors for the quantification of control output variables led Seok (Seok 2003) to design a hybrid adaptive control system applying an off-line and an on-line system identification routine in the process. Eventually, the system was tested for the degradation of propylene glycol (PG) in an anaerobic fluidized bed reactor. The overall hybrid adaptive optimal controller performance was successful. However, poor system identification was present during the first hours of operation which could be due to initial model parameter set selection.

Hybrid intelligent control of an anaerobic wastewater treatment process in a CSTR was possible (Belmonte-Izquierdo et al. 2009). A recurrent high order neural observer (RHONO) was designed to estimate biomass and substrate, and an extended Kalman filter was utilised to train the observer. Next, a fuzzy supervisor was applied to control the dilution rate depending on the operating conditions. However, as the resultant controller’s main goal was to avoid washout, it is concluded that although it is capable of doing so, this is not the most ideal solution.

A process for identifying hybrid models in bioprocesses is proposed in (Chen et al. 2000). Whereas a hybrid model based first on the knowledge of the mass balances of the process components, and second on a feedforward network was constructed (Karama et al. 2001). NN implementation within a hybrid approach was able to overcome process difficulties that are encountered due to the lack of

accurate kinetic modelling. Furthermore, it was underlined that as the NN training was based on the overall hybrid model performance and not solely on the NN performance, the direct use of standard training procedures (e.g. backpropagation algorithms) was not an option. Therefore, the standard NN model was trained by the Levenberg Marquardt algorithm, whereas backpropagation combined with a conjugate gradient optimisation method trains the hybrid NN model.

An ANFIS was designed in order to predict the effluent COD reduction from a sugar factory anaerobic WWTP (Perendeci et al. 2009). The model's predictive capabilities were enhanced by the addition of COD data values that exceeded in time those of the overall retention time of the wastewater in the system. In this way, system output estimates showed that despite the data need for appropriate NN training, the model performed satisfactorily.

### **1.5.11 Discussion and Conclusion**

There are a series of control techniques capable of handling non-linear processes including anaerobic fermentation. The lack of accurate modelling resulting from the non-linear nature of the process makes control a difficult task and is the main reason behind the increasing interest in this field, where a diversity of approaches exists.

Control laws are designed to (Steyer et al. 2006) tackle the specific problem they were designed for. Consequently, it is necessary to combine control laws or incorporate them in an advanced scheme, enabling the control system to cope with all kinds of disturbances. Each technique examined in this review can provide adequate control results for specific processes. However the review addresses improvement of process control as no such perfect technique exists. Therefore, control simplicity, expressed in terms of conventional control applications, or control complexity, involving advanced control schemes, is something that should be taken into consideration in the system design. Furthermore, control limitations and/or design parameters that have to be accounted for in each design are also explored.

Open-loop systems are shown to be implemented in order to prove, through comparison, the improved system performance that can be achieved through closed-loop approaches. The application of conventional industrial controllers that include

on/off P, PI and PID approaches can sometimes be inefficient, due to nonlinearities that are inherent in biological systems are not taken into consideration resulting in process instability (Antonelli et al. 2002), or are used in either single input single output (SISO) models or linear cases (Steyer et al. 2006). However, they tend to be utilised for handling vital, yet not very complicated parts of the process (e.g. preventing system failure, controlling one process parameter like pH).

MPC and adaptive control are some of the most effective approaches, but rely on successful process modelling and require advanced mathematical models and calculations. Furthermore, as these methods are linked with AI techniques (e.g. NNs), efforts have been made over the past decade to concentrate on hybrid approaches by overcoming the limitations of each technique (e.g. lack in kinetic modelling,) while exploiting the responsive advantages (e.g. robustness). The ability of intelligent controllers (e.g. FLCs) to handle nonlinear processes, despite design complexity, is most of the time synonymous with enhanced control accuracy. The majority of FLC system designs for AD purposes are mainly based on experience, and extreme care has to be taken to ensure that the system will never encounter situations, or more specifically parameter values, that have not been taken into consideration in the design process. Moreover, with respect to NN implementations, the learning process should be performed effectively, equipping the controller with the knowledge to handle all types of operational situations that might occur. On the other hand, linearised models of the process where stability and convergence properties are local, make it difficult to generalise for the entire operation spectrum (Harmand et al. 2005). Furthermore, insufficient process knowledge can lead to a non-linear model usually lacking in robustness under uncertainties (Belmonte-Izquierdo et al. 2009). In this case, the utilisation of NNs or FL in the control scheme (e.g. a RHONO observer), can help address this issue.

For the future advanced control techniques are growing in popularity and seem to be suitable for non-linear processes. However, optimisation and robustness has to be ensured in order to make such a control system suitable for industrial applications.

## Chapter 2 Fuzzy Logic

This chapter is an introduction to fuzzy logic (FL), off-line data clustering methods and Mamdani FISs with a detailed analysis of the subtractive clustering method and type-1 Sugeno fuzzy logic systems (FLS).

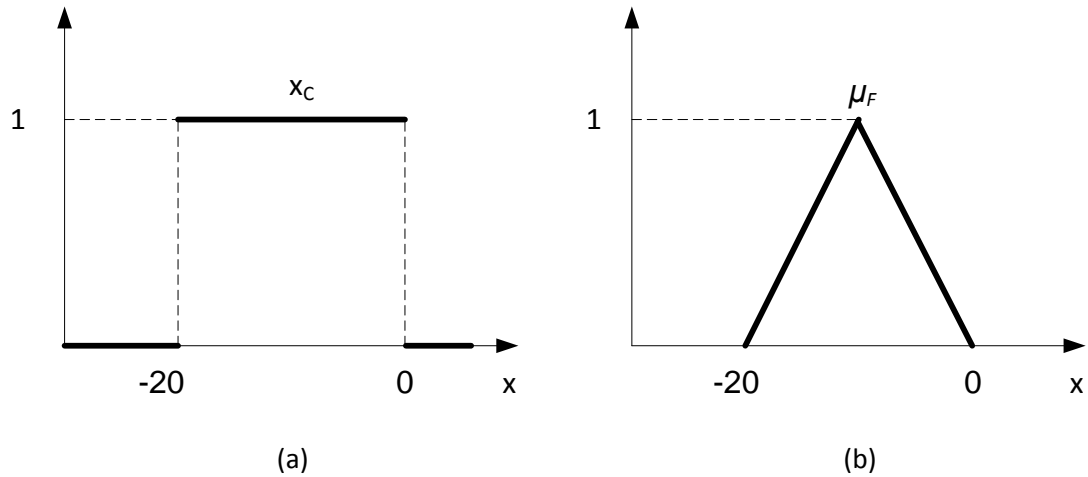
### 2.1 Introduction to Fuzzy Logic

The typical architecture of a Fuzzy Inference Systems (FIS) (Figure 2.2) consists of four components: the fuzzifier, the rule-base, the inference engine and the defuzzifier. There are three types of fuzzy inference that have been employed in a series of applications: the Mamdani fuzzy inference (Mamdani & Assilian 1974), the Takagi-Sugeno-Kang (TSK) fuzzy inference also known as Sugeno fuzzy inference (Takagi & Sugeno 1985) and the Tsukamoto fuzzy inference (Tsukamoto 1979) with the first two being the most popular. The main differences between these fuzzy models are the consequents of the fuzzy rule and the aggregations and defuzzification procedures. This chapter will mostly focus on examining the Sugeno FIS.

#### 2.1.1 Fuzzy Sets

Crisp set theory is used to define the membership or the non-membership of individuals in a given universe of discourse. However, fuzzy set theory, established originally by Zadeh in the 1960's (Zadeh 1965), states that a grade of membership can be assigned to each individual belonging to a certain class. The membership of an element (individual) in a fuzzy set (class) is given by number between 0 and 1.

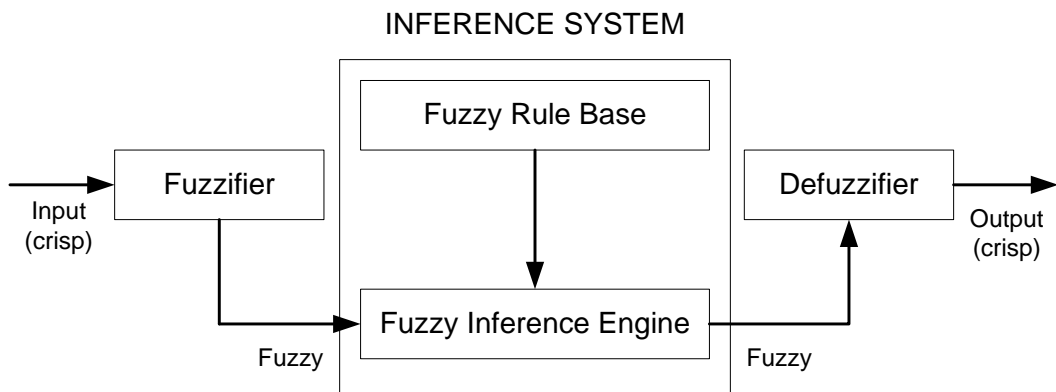
Let  $U$  be a collection of points, where  $U$  is the universe of discourse. For a given element  $x \in U$ , a set  $C$  is characterised as crisp only if  $x \in C$  or  $x \notin C$ . However, a fuzzy set  $F$  in the same universe of discourse is characterised by a membership function  $\mu_F: U \rightarrow [0,1]$ . An example of crisp versus fuzzy set is shown in Figure 2.1 by illustrating the possible definition of cold temperature ( $^{\circ}\text{C}$ ).



**Figure 2.1** Cold temperature membership function for (a) crisp set C and (b) fuzzy set F

### 2.1.2 Fuzzification

Fuzzification is the first key component of a FL system. As described in the previous section, the fuzzification operation is the process of mapping from a crisp point to a fuzzy set and involves the decomposition of system inputs and outputs into one or more fuzzy sets. Fuzzification is important because many quantities that are crisp and deterministic are not deterministic at all. Imprecision, ambiguity or vagueness might lead to uncertainty so that fuzzy representation through a membership function is more suitable (Ross 2004).



**Figure 2.2** Fuzzy Logic System configuration

Two types of fuzzifiers exist: singleton and non-singleton. The most common fuzzyfier is the singleton fuzzifier. In this case, the crisp point  $x \in U$  is mapped into a fuzzy set  $F$  with support  $x'$  where  $\mu_F(x') = 1$  for  $x' = x \in U$  and  $\mu_F(x') = 0$  for  $x' \neq x \in U$ . Non-singleton fuzzyfiers are those in which the support is more than one point. In these fuzzifiers,  $\mu_F$  achieves a maximum value at  $x' = x$  ( $x'$  might be more than one point) and decreases as it moves away from that point. Singleton FL systems can be found in (Lee 1990) and non-singleton FL systems are available (Mouzouris & Mendel 1997) and the references within. The most common non-singleton fuzzyfiers are the Gaussian, the triangular and the trapezoidal fuzzyfiers.

### 2.1.3 Rule base

The rule-base is comprised of a set of rules that are developed based on system knowledge and aim to approximate the relationship between input and output (Kim et al. 2006). If-then rule statements are used to formulate these conditional statements and are usually of the following form:

$$IF X \text{ is } X_1 \text{ AND } Y \text{ is } Y_1 \text{ OR } Z \text{ is } Z_1, THEN U \text{ is } U_1,$$

where  $X, Y, Z$  are fuzzy input variables,  $U$  is a fuzzy output variable,  $X_1, Y_1, Z_1, U_1$  are fuzzy linguistic values defined by fuzzy sets on the range of  $X, Y, Z, U$  respectively (Saemi & Ahmadi 2007)(Jamshidi 2003), 'AND', 'OR', 'NOT', are connectives of the rule. The IF-part of the rule statement ' $X \text{ is } X_1 \text{ AND } Y \text{ is } Y_1 \text{ OR } Z \text{ is } Z_1$ ' are called the antecedents or premises, and the THEN-part of the rule ' $U \text{ is } U_1$ ' is the consequent. Depending on the type of FIS that we are dealing with the consequent can take the form of a fuzzy set or a function.

### 2.1.4 Inference Engine

The inference engine considers all the fuzzy rules in the fuzzy rule-base and transforms an input (or a set of inputs) to the corresponding output(s) based on the fuzzy inference method applied. The Sugeno and the Mamdani inference engines vary in the way that the output is calculated. The Sugeno output membership

functions are either linear or constant and are expressed as an equation or an analytical expression, whereas the Mamdani membership functions are linguistic.

### **2.1.5 Defuzzification**

Defuzzification is the last step in the fuzzy inference process and involves the conversion of the fuzzy output from the inference engine to a crisp number. Several defuzzification methods exist: the weighted average, maximum membership, average maximum membership, centre of gravity etc. The most common is the centre of gravity due to its simplicity and accuracy.

## **2.2 Clustering**

Data clustering is a method that identifies similarities in data and aims to group the data based on those similarities. Data plays an important role in the construction of data-driven models, especially in cases where data contains noise, conflicts (same input(s) result in having different output(s)) or are inconsistent. However, if data is treated carefully by utilising a suitable clustering technique to identify data patterns, deal efficiently with conflicting data and remove any outliers, then an accurate model based on those groupings can be constructed. Therefore, clustering techniques are widely used for fuzzy modelling.

Several clustering techniques exist. The most representative off-line techniques include: K-means (or Hard C-means) clustering, Fuzzy C-means clustering, Mountain clustering and Subtractive clustering.

### **2.2.1 K-means clustering**

The K-means clustering (MacQueen 1967)(Hartigan & Wong 1979) is an algorithm that aims to locate data clusters in a dataset by partitioning M points in N dimensions into K clusters so that a cost function (or objective function) is minimised. The algorithm proceeds by selecting the initial position of the cluster centres and then updates them until there is no further improvement of the cost

function. The main disadvantage of this method is that the algorithm is sensitive to the initial cluster locations. Therefore, several runs with different initial clusters centres have to be performed to obtain the optimal solution to the problem (Ray & Turi 1999) (Likas et al. 2003).

### **2.2.2 Fuzzy C-means clustering**

The Fuzzy C-means clustering method (FCM) was presented (Dunn 1973) and further developed (Bezdek 1981). The basic idea behind FCM is that (Bezdek et al. 1984) each data point has a membership in each cluster that is specified by a degree that varies between 0 and 1. FCM is an unsupervised method that also minimises a cost function and always converges. However, FCM requires (Guillaume 2001) some parameters to be set in advance (e.g. the number of the cluster centres), is sensitive to noise and requires a long computational time (Hung & Yang 2001).

### **2.2.3 Mountain clustering**

Mountain clustering (Yager & Filev 1994) is based on the construction of a function related to the density of data points, a so called the mountain function. After forming the grid of the data space, the construction of a mountain function from the data at every grid point is followed by the destruction of the mountains to obtain the cluster centres. The performance of this method is affected by the rise in the computational time that increases exponentially with the dimension of input data (Hammouda 2000).

### **2.2.4 Subtractive clustering**

The Subtractive Clustering Method (SCM) was introduced by Chiu (Chiu 1994)(Chiu 1997). This method is a modification of the mountain clustering method. The main differences between these two methods are mainly the potential value estimation method, the way a neighbouring data point is influenced, and how a new



cluster centre is acquired (Doan et al. 2005). SCM is a fast method with reduced computational time compared to mountain clustering. The computational time and the data dimension are analogous as the computational time does not increase exponentially with the dimension of input data.

Each data point is considered as a potential cluster centre and the higher the density around a specific data point the higher are the chances that this data point will become a cluster centre.

A group of  $n$  data points  $\{x_1, x_2, \dots, x_n\}$  in an  $M$ - dimensional normalised space where all the data points are bounded by a hypercube is considered. The potential  $P_i$  for each data point to become a cluster centre is calculated as follows:

**Equation 2.1**

$$P_i = \sum_{j=1}^n e^{-a||x_i-x_j||^2}$$

where

**Equation 2.2**

$$a = \frac{4}{r_a^2}$$

$r_a$  is a positive constant of the radius defining a neighbourhood and data points outside this radius have little influence on the potential and  $||.||$  denotes the Euclidean distance.

After the computation of the potential of every data point has been completed, the data point  $x_1^*$  with the highest potential  $P_1^*$  is selected as the first cluster centre. Then, the potential of the remaining data points is revised according to

**Equation 2.3**

$$P_i \Leftarrow P_i - P_1^* e^{-\beta ||x_i-x_1^*||^2}$$

where

$$\beta = \frac{4}{r_b^2} \text{ and } r_b = \eta * r_a$$

$r_b$  is the radius that defines the neighbourhood which will have measurable reductions in potential and  $\eta$  is called the squash factor. Since closely spaced cluster centres are not desired,  $r_b$  is typically chosen to be greater than  $r_a$ .

After the potential of all data points have been revised by using Equation 2.3, the data point with the highest remaining potential is chosen as the second cluster centre. Then further reduction of the potential of each data point according to their distance from the second cluster centre is performed. After the  $k$ th cluster centre has been obtained, the potential of each point is revised by the formula

**Equation 2.4**

$$P_i \Leftarrow P_i - P_k^* e^{-\beta \|x_i - x_k^*\|^2}$$

where  $x_k^*$  is the location of the  $k$ th cluster centre and  $P_k^*$  is its potential value.

The process of acquiring a new cluster centre and revising potential is repeated until the remaining potential of all data points is below some fraction of the potential of the first cluster centre  $P_1^*$ . Additional criteria are used for accepting or rejecting cluster centres to avoid marginal cluster centres:

if  $P_k^* > \bar{\epsilon} P_1^*$

Accept  $x_k^*$  as a cluster centre and continue

else if  $P_k^* > \underline{\epsilon} P_1^*$

Reject  $x_k^*$  and end the clustering process

else

Let  $d_{min}$  = shortest of the distances between  $x_k^*$  and all previously found cluster centres

if  $\frac{d_{min}}{r_a} + \frac{P_k^*}{P_1^*} \geq 1$

Accept  $x_k^*$  as a cluster centre and continue

Else

Reject  $x_k^*$  and set the potential at  $x_k^*$  to 0. Select the data point with the next highest potential as the new  $x_k^*$  and test again.

$\bar{\varepsilon}$  (accept ratio) specifies a threshold for the potential above which we will definitely accept the data point as a cluster centre and  $\underline{\varepsilon}$  (reject ratio) specifies a threshold below which we will definitely reject the data point.

Subtractive clustering has four parameters that directly influence the number of rules and the error performance measures: the cluster radius  $r_a$ , the squash factor  $\eta$ , the accept ratio  $\bar{\varepsilon}$  and the reject ratio  $\underline{\varepsilon}$ . For example a small value of  $\eta$  will generate fewer clusters that might not be a precise representation of the system (model) investigated. On the other hand, a very large value of  $\eta$  will generate a large number of rules leading to an over defined system. As a consequence, a parameter search has to be performed to find the optimal values for a given dataset. Recommended values for those parameters were introduced by Chiu. Also a parametric search on various clustering parameters to identify the best model by proposing an extended subtractive clustering method by identifying the ranges that provide the best models was performed (Demirli et al. 2003) (Table 2.1).

Symbol	Chiu	Demirli
Cluster radius	[0.25, 0.50]	[0.15, 1]
Squash factor	1.25	[0.05, 2]
Reject ratio	0.15	[0, 0.9]
Accept ratio	0.5	[0, 1]

Table 2.1 Recommended values for parameters in subtractive clustering (Ren et al. 2006)

### 2.3 Mamdani Fuzzy logic

As mentioned in 2.1, the major difference between the Mamdani and the TSK FISs lies in the way the consequent of the fuzzy rules are represented. This means that the aggregation and the way the defuzzification process is performed are different (Al-Jarrah & Abu-Qdais 2006).

The first two parts of the Mamdani fuzzy inference process involve the fuzzification of the inputs and the application of the fuzzy operators (Puñal et al. 2003). Then fuzzy operations are applied to the antecedents through the rule-base

that can be developed based on human expert knowledge (Murnleitner et al. 2002). The ability to include expert knowledge in the form of linguistic rules and combine them with rules that can be automatically generated from datasets, representing the behavior of the system, makes Mamdani FLSs attractive (Casillas et al. 2000). The consequents of the rules have the form of fuzzy sets and the next step involves the aggregation of the rule outputs into a new fuzzy set. The defuzzification process is then performed in order to obtain a crisp output value. The selected defuzzification method for the fuzzy controller developed in Chapter 5 will be the centroid approach which is one of the most commonly used techniques (Turkdogan-Aydinol & Yetilmezsoy 2010).

Let us consider a fuzzy system with two noninteractive inputs (Ross 2004)  $x_1$  and  $x_2$  (antecedents) and a single output  $y$  (consequent) that is described by a collection of  $r$  linguistic IF-THEN propositions of the form:

**Equation 2.5**

$$IF\ x_1\ is\ A_1^k\ and\ x_2\ is\ A_2^k\ THEN\ y^k\ is\ B^k\ for\ k = 1, 2, \dots, r$$

where  $A_1^k$  and  $A_2^k$  are the fuzzy sets representing the  $k^{th}$  antecedent pairs, and  $B^k$  is the fuzzy set representing the  $k^{th}$  consequent. Also, let us consider  $x_1$  and  $x_2$  to be the crisp value inputs of the fuzzy system whose membership functions are described by

**Equation 2.6**

$$\mu(x_1) = \delta(x_1 - input(i)) = \begin{cases} 1, & x_1 = input(i) \\ 0, & otherwise \end{cases}$$

**Equation 2.7**

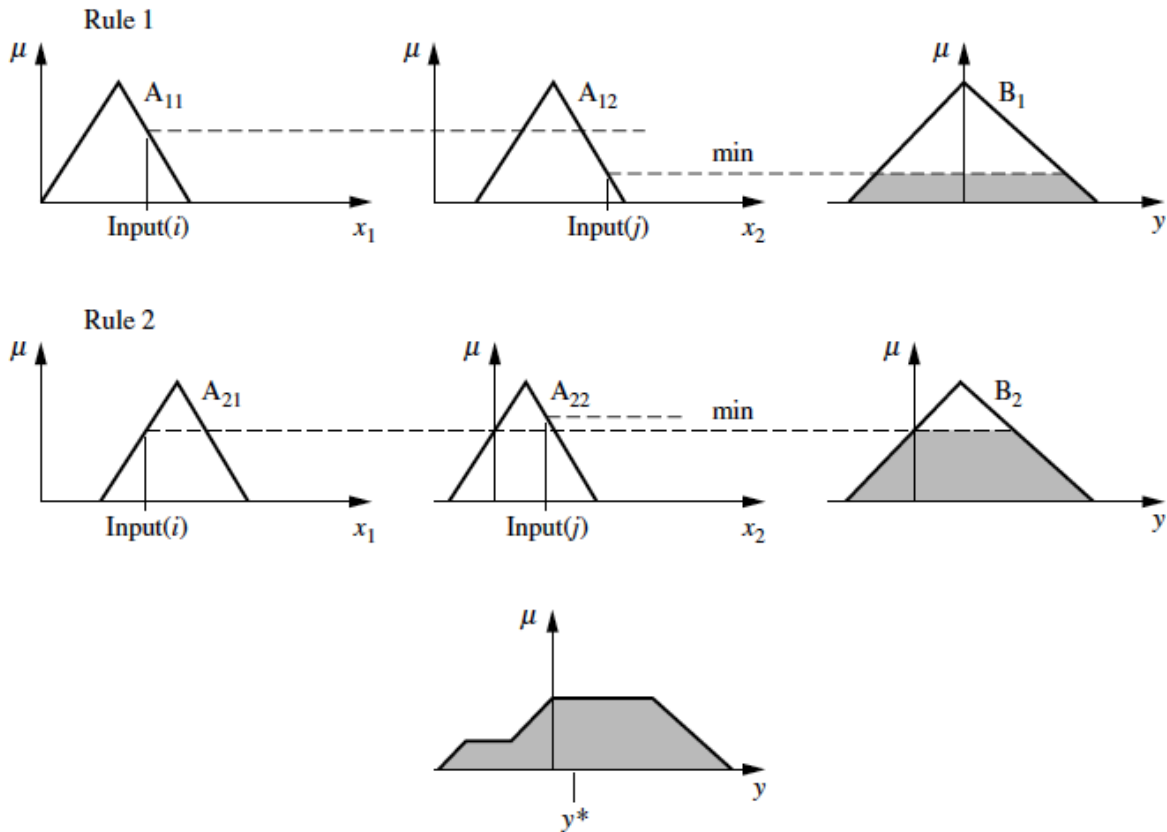
$$\mu(x_2) = \delta(x_2 - input(j)) = \begin{cases} 1, & x_2 = input(j) \\ 0, & otherwise \end{cases}$$

By choosing to use a max-min Mamdani implication method of inference and for a set of disjunctive rules, the aggregated output for the  $r$  rules is:

Equation 2.8

$$\mu_{B^k}(y) = \max_k \left[ \min \left[ \mu_{A_1^k}(\text{input}(i)), \mu_{A_2^k}(\text{input}(j)) \right] \right]$$

for  $k = 1, 2, \dots, r$



**Figure 2.3** Graphical Mamdani (max-min) inference method with crisp inputs (Ross 2004).

The graphical analysis of the two rules is depicted in Figure 2.3.  $A_{11}$  and  $A_{12}$  correspond to the first and second antecedents of the first rule respectively and  $B_1$  is the fuzzy consequent of the first rule. Similarly,  $A_{21}$  and  $A_{22}$  correspond to the first and second antecedents of the second rule respectively and  $B_2$  is the fuzzy consequent of the second rule. The minimum function (Equation 2.8) is illustrated in Figure 2.3 and results from the logical 'and' connection of the antecedents that is present in the rule structure (Equation 2.5). The minimum membership value results in shaping the membership function for the consequent of each rule (grey shaded area corresponding to the consequent membership function of each rule in Figure 2.3). The resultant consequent membership functions for the two rules are then aggregated (aggregation operation 'max', Equation 2.8). Therefore, the output is represented with an aggregated membership function comprising of the outer

envelope of the individual membership forms of each rule. Finally, a crisp value ( $y^*$ ) can be obtained from the output membership function using the desired defuzzification method (2.1.5).

## 2.4 TSK Fuzzy logic

TSK FL was introduced (Takagi & Sugeno 1985)(Sugeno & Kang 1988) in order to approximate and/or identify a variety of systems and functions by utilising input-output data. System approximation is achieved with the generation of fuzzy rules. The fuzzy sets in the consequent are substituted by a linear equation of the input variables. Linear models are used to locally represent the dynamics of the state-space regions, and an interpolation of those represents the overall system model (Figure 2.4). TSK FL does not require extensive knowledge of the processes or of the systems under examination, especially for AD processes which are highly complex nonlinear microbial processes. However, it is capable of providing a good description of those (Lauwers et al. 2013). The main advantage of the TSK model over other classes of fuzzy models lies in the fact that it can model a system with great accuracy either locally or globally (Quah & Quek 2006).

For a multiple-input and single-output (MISO) system with  $n$  rules, similar to the ones that will be presented in this thesis, the  $n^{\text{th}}$  rule of in a TSK FLS is of the following form:

**Equation 2.9**

$$R^n: \text{if } x_1 \text{ is } A_1^n, \dots, \text{ and } x_k \text{ is } A_k^n$$

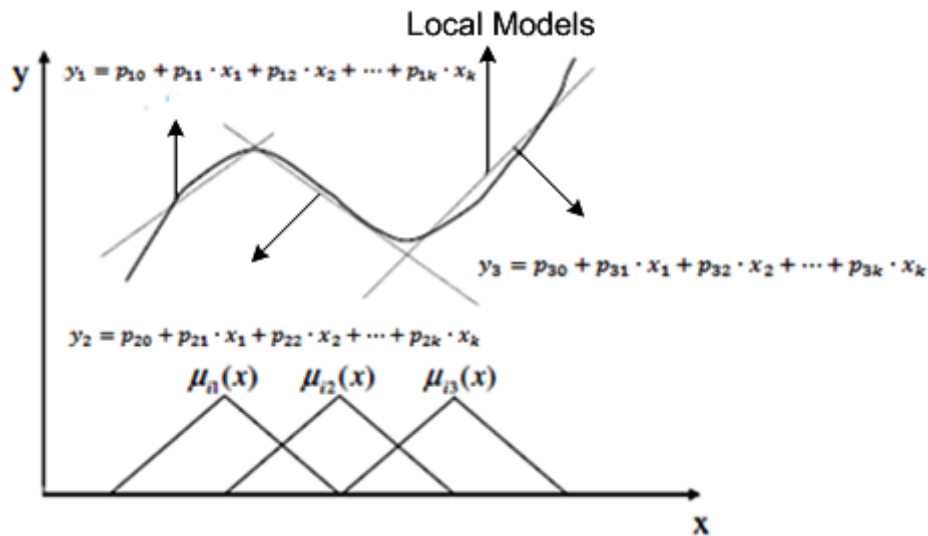
$$\text{then } y^n = p_0^n + p_1^n \cdot x_1 + \dots + p_k^n \cdot x_k$$

where  $x_1, \dots, x_k$  and  $y$  are respectively the linguistic input and output variables,  $A_1, \dots, A_k$  are the fuzzy sets representing a fuzzy subspace, and  $p_0^n, \dots, p_k^n$  are the consequent parameters. The final output  $z$  of the fuzzy model is computed as follows (weighted average aggregation):

Equation 2.10

$$z = \left[ \sum_{i=1}^n \mu_i y_i \right] \div \left[ \sum_{i=1}^n \mu_i \right]$$

where  $\mu_i$  represents the degree to which the  $n^{\text{th}}$  rule is fired (firing strength) and is derived using a T-norm operator (the product operator  $*$  or the minimum operator  $\wedge$ ).



**Figure 2.4** TSK model as a smooth piece-wise linear approximation of a non-linear function. Modified from (Lohani et al. 2006).

There is an absence of a systematic way to derive rules and parameters of the membership functions of the antecedent part of the fuzzy model. However, data clustering (or fuzzy clustering) techniques are utilised to solve this problem. System identification is achieved by “embedding” the cluster centres into rules.

## 2.5 TSK based on subtractive clustering

By implementing the subtractive clustering algorithm the number of fuzzy rules, that is equal to the number of clusters, and the rule premises (cluster centres) are established (Chiu 1994)(Chiu 1997).

Consider a set of  $m$  cluster centres  $\{x_1^*, x_2^*, \dots, x_m^*\}$  found in an  $M$  dimensional space where the first  $N$  dimensions correspond to the input variables and the last

$M - N$  dimensions correspond to the output variables. Each vector  $x_i^*$  is decomposed into two component vectors  $y_i^*$  and  $z_i^*$ . The vector  $y_i^*$  contains the first  $N$  elements of  $x_i^*$  corresponding to the cluster centre coordinates in the input space and the vector  $z_i^*$  contains the  $M - N$  elements corresponding to the cluster centre coordinates in the output space.

Each cluster centre  $x_i^*$  is considered to be a fuzzy rule of the following form that is representative of the system's behaviour:

*Rule i: if {input is near  $y_i^*$ } then the output is near  $z_i^*$ .*

Given an input vector  $y$ , the degree of fulfilment of rule  $i$  is defined as:

**Equation 2.11**

$$\mu_i = e^{-a\|y-y_i^*\|^2}$$

where  $a$  is defined in Equation 2.2. Then the output vector  $z$  is given by:

**Equation 2.12**

$$z = \left[ \sum_{i=1}^m \mu_i z_i^* \right] \div \left[ \sum_{i=1}^m \mu_i \right]$$

The above computational model can be viewed in terms of a FIS employing fuzzy if-then rules of the following form:

**Equation 2.13**

$$\text{if } Y_1 \text{ is } A_{i1} \ \& \ Y_2 \text{ is } A_{i2} \ \& \ \dots \ \text{then } Z_1 \text{ is } B_{i1} \ \& \ Z_2 \text{ is } B_{i2} \ \dots$$

where  $Y_j$  is the  $j^{\text{th}}$  input variable and  $Z_j$  is the  $j^{\text{th}}$  output variable,  $A_{ij}$  is an exponential membership function in the  $i^{\text{th}}$  rule associated with the  $j^{\text{th}}$  input and  $B_{ij}$  is a membership function in the  $i^{\text{th}}$  rule associated with the  $j^{\text{th}}$  output.

The membership function of the  $i^{\text{th}}$  rule that is represented by cluster centre  $x_i^*$  is:



Equation 2.14

$$A_{ij}(Y_j) = \exp \left\{ -\frac{1}{2} \left( \frac{Y_j - y_{ij}^*}{\sigma_{ij}} \right)^2 \right\}$$

$B_{ij}$  is a symmetric function centred around  $z_{ij}^*$ ,  $y_{ij}^*$  and  $z_{ij}^*$  are the  $j^{\text{th}}$  elements of  $y_i^*$  and  $z_i^*$  respectively and  $\sigma_{ij} = 1/\sqrt{2a}$ .

Chiu (Chiu 1994)(Chiu 1997) showed that the accuracy of the TSK model can be improved by implementing a Type-1 FLS by setting the consequent parameter  $z_i^*$  in Equation 2.8 to be a linear function of the input variables (the consequent parameters in a zero order TSK FLS are constants):

Equation 2.15

$$z_i^* = G_i y + h_i$$

Then, the process of obtaining the optimal consequent parameters (matrice  $G_i$ , constant  $h_i$ ) involves substituting Equation 2.15 in Equation 2.12 and solving a linear least squares estimation problem (Takagi & Sugeno 1985).

### 2.5.1 TSK example based on subtractive clustering (function approximation)

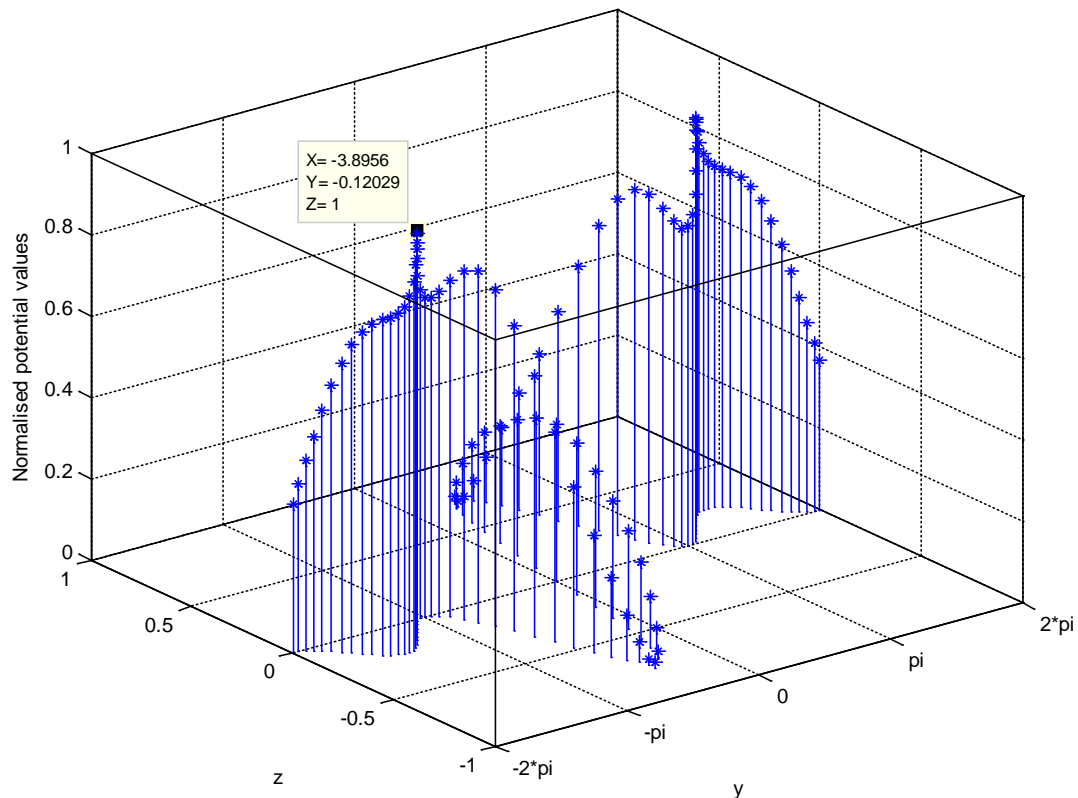
Let us consider the problem of modeling the nonlinear function in Equation 2.16.

Equation 2.16

$$z = \frac{\sin(y)^2}{y}$$

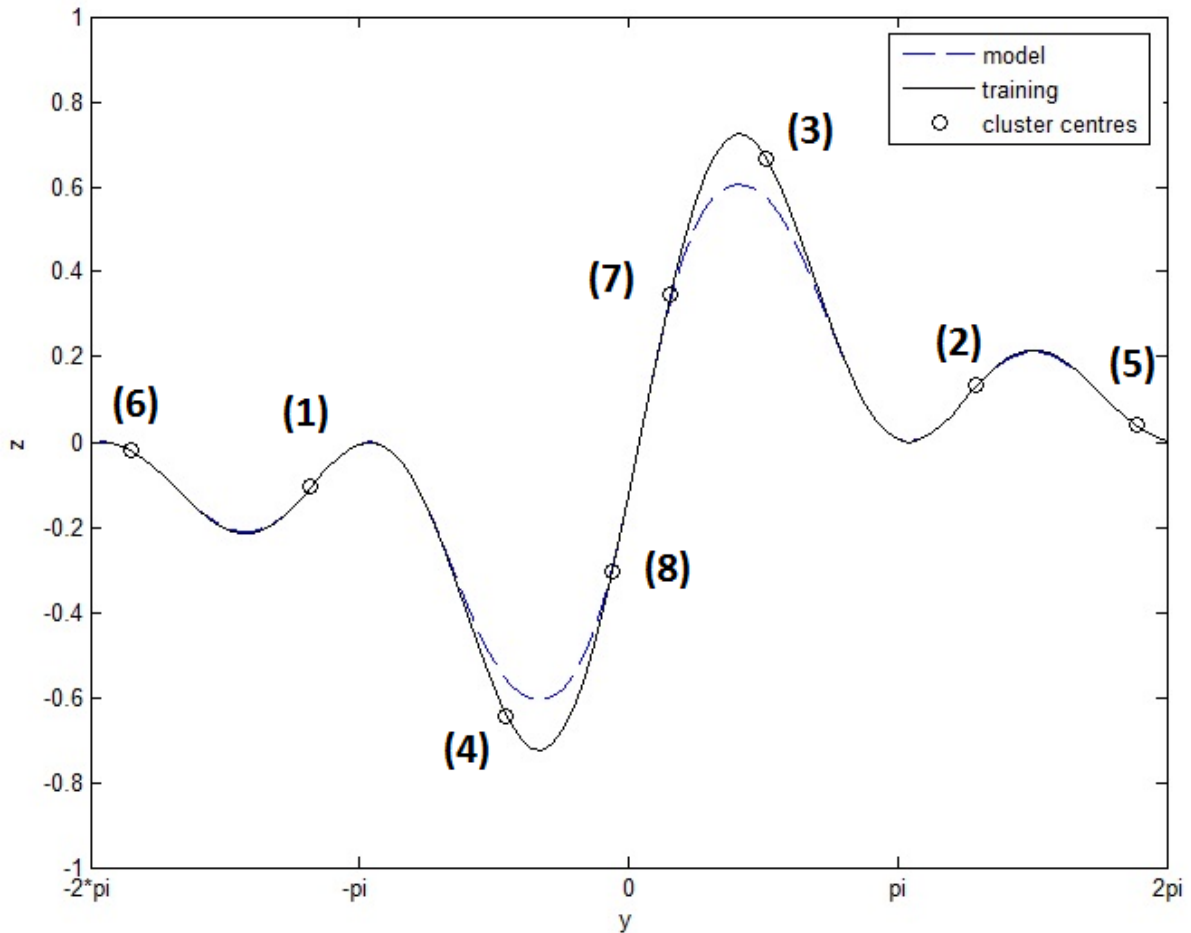
Equally spaced  $y$  values were used to generate 100 training data points for the range  $[-2\pi, 2\pi]$ . Then, all training data were normalised so that they were bounded by a unit hypercube. The recommended parameter values for subtractive clustering were chosen as suggested by Chiu (Table 2.1). Therefore, the squash factor was set to 1.25, accept ratio= 0.5, reject ratio= 0.15 and cluster radius was set to 0.25. By applying the process described in 2.2.4, eight cluster centres were found.

After the potential for each data point to become a cluster centre was calculated using Equation 2.1 (Figure 2.5), the data point with the highest potential was selected as the first cluster centre as depicted in Figure 2.5 and in Figure 2.6 (1).



**Figure 2.5** Potential values using Subtractive Clustering

Next, the potential of the remaining data points was revised (Equation 2.3). The data point with the highest remaining potential was selected as the second cluster centre ((2) in Figure 2.6). Then, further reduction of the potential of each data point according to the distance from the second cluster centre was performed. Equation 2.4 was used to calculate the revised potential for each point after obtaining the cluster centres. Additional criteria are being used for accepting and rejecting cluster centres and revising potential (2.2.4), which resulted in having eight cluster centres that were acquired in the order depicted in Figure 2.6.



**Figure 2.6** Comparison of training data with the first order TSK model  
 ((1)-(8): position of the eight cluster centres)

The clusters determined from the data identified eight regions in the input space that map into the associated class. Therefore, each cluster centre can be translated into a fuzzy rule for identifying the class.

As soon as the clustering process was completed, the fuzzy system in Equation 2.12-Equation 2.14 was designed. There, the consequent parameter  $z_i^*$  (Equation 2.12) was set to be a linear function of the input variables. The 'if-then' rules then became the TSK type of the form of Equation 2.9 (Table 2.2). The degree of fulfillment of each rule is depicted in Figure 2.7 and the membership function parameters are shown in Table 2.3. This is a single input-single output problem. Therefore, in Equation 2.13 and Equation 2.14,  $j=1$  and is omitted for simplicity of representation (e.g.  $A_1$  instead of  $A_{11}$ ). The process of obtaining the optimal consequent parameters involves solving a least-squares estimation problem (2.4). Figure 2.5 shows the output of the fuzzy model that produced an output close to the desired system output.

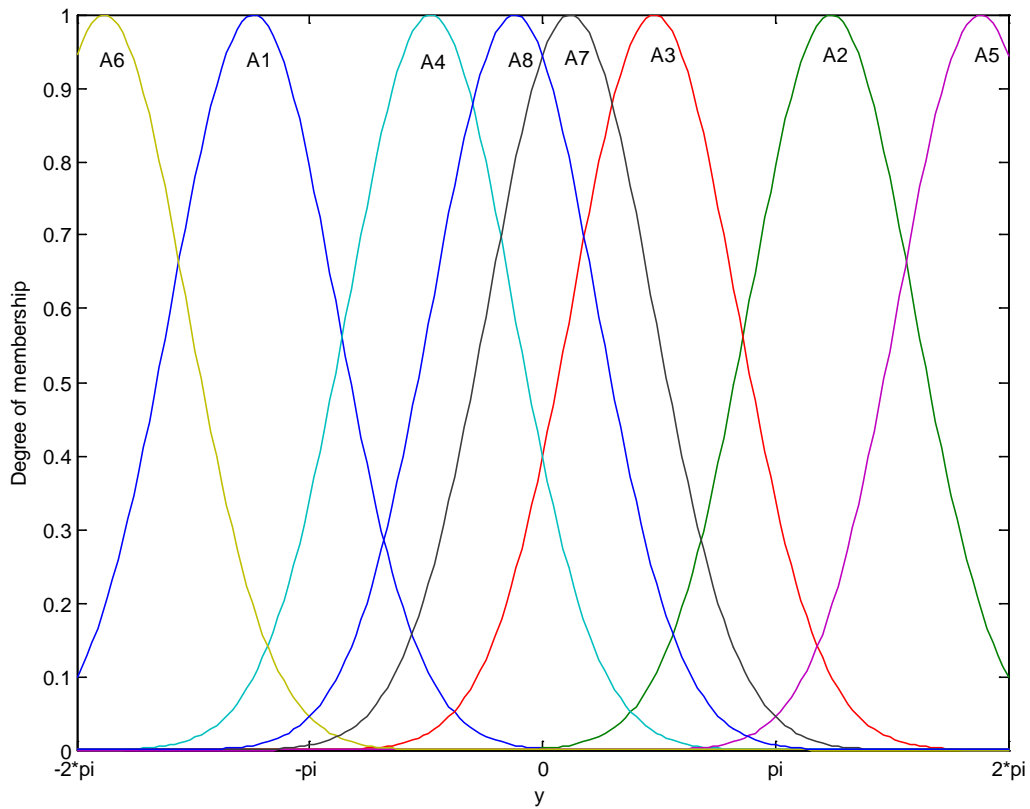


Figure 2.7 Degree of fulfillment of each rule

Rule	Rule description of the form If ... Then $z = p_1 y_1 + p_0$
1	If $y$ is $A_1$ Then $z_1 = 0.8988 \cdot y + 3.5258$
2	If $y$ is $A_2$ Then $z_2 = 0.8988 \cdot y - 3.5258$
3	If $y$ is $A_3$ Then $z_3 = 6.106 \cdot y - 25.87$
4	If $y$ is $A_4$ Then $z_4 = 6.106 \cdot y + 25.87$
5	If $y$ is $A_5$ Then $z_5 = 0.2568 \cdot y - 1.8393$
6	If $y$ is $A_6$ Then $z_6 = 0.2568 \cdot y + 1.8393$
7	If $y$ is $A_7$ Then $z_7 = 36.288 \cdot y - 78.814$
8	If $y$ is $A_8$ Then $z_8 = 36.288 \cdot y + 78.814$

Table 2.2 TSK fuzzy model rules

<b>Cluster</b>	<b>Membership functions</b>	<b>Centres (y)</b>	<b>Width (y)</b>
1	$A_1$	-3.89	1.11
2	$A_2$	3.89	1.11
3	$A_3$	1.508	1.11
4	$A_4$	-1.508	1.11
5	$A_5$	5.906	1.11
6	$A_6$	-5.906	1.11
7	$A_7$	0.377	1.11
8	$A_8$	-0.377	1.11

**Table 2.3** Membership function parameters obtained using subtractive clustering

## **Chapter 3 Development of a software sensor for Alkalinity monitoring based on fuzzy logic**

In this chapter a software sensor predicting alkalinity was developed using fuzzy logic and subtractive clustering. It is compared with the software sensors based on multiple linear regression models that were developed (Partner N° 2, Rothamsted Research 2010) to predict alkalinity. Those models were utilised to control the OLR of a two-stage, semi-continuously fed stirred reactor system.

### **3.1 Introduction- Alkalinity as a monitoring parameter**

The AD process is difficult to control and can become unstable especially when changes in fermentation are induced by rapid OLR changes or variations in the feedstock composition (Spanjers & Lier 2006). High loading and in most cases overloading result in poor gas production rates and an acidified or sour digester that will require a lot of time to recover. The recovery period varies according to the substrate added, the size of digester and the nature of the controller that regulates the OLR in automated processes. Gentle recovery strategies are suggested, an example of which can be found in Scherer et al. (2009) and Demirel & Scherer (2008). To avoid a sour digester that will require between two days and two months to recover, introducing a control system that will prevent overloading has to be designed.

Gas production of AD systems is optimised for increased OLRs and in certain cases control systems focus on preventing system imbalance rather than driving the system towards maximum gas production (Pullammanappallil et al. 1998). However, efficient operation at high load rates is vital to get the most out of AD systems ensuring that the system will be protected against disturbances and overloading situations (Liu et al. 2004c).

Alkalinity is an indicator of process stability in AD and enables the detection of changes in the buffer capacity of the system (Palacios-Ruiz et al. 2008)(Hawkes 1993). Moreover, alkalinity is a good indicator of future failure due to reactor acidification (Guwy, Hawkes, Wilcox, et al. 1997).

Many control applications utilise the VFA to alkalinity ratio. VFA accumulation may lead to a decrease in pH and cessation of gas production. This justifies why VFAs are widely used to determine the stability of digestion processes. Alkalinity and VFA are two of the most sensitive indicators of process stability (Schoen et al. 2009) which led to a wide application of the VFA/ Alkalinity ratio for the purpose of system monitoring. Alkalinity is influenced by the VFA concentration which is the reason why it should be monitored together with alkalinity to provide as accurate and complete information as possible of the digester stability (Ahring & Angelidaki 1997). Values between 0.1 and 0.4 are considered to be indicative of stable process operation avoiding any risks of acidification bounds (Switzenbaum 1990)(Zickefoose & Hayes 1976). Tighter bounds have also been proposed. A ratio between 0.1-0.25 is considered favourable, a rise above 0.3-0.4 dictates that corrective measures should be taken and values that exceed 0.8 result in severe digester failure (Khanal 2008). A ratio of at least 1.4:1 of bicarbonate/VFA should be maintained for a well-buffered and stable digestion process despite the fact that the stability of the ratio, and not its level, is of prime importance (Appels et al. 2008).

VFA sensors have been implemented in the past using analytical instruments. Those include the use of gas chromatography (GC), titrimetry, IR-spectrometry (Spanjers & Lier 2006), spectrophotometry and capillary zone electrophoresis (Zygmunt & Banel 2009). However, on-line sensors have proven to be quite unreliable by delivering wrong measurements due to disturbances (e.g. interference of chemical species) (Lardon et al. 2004). Other methods were limited by the fact that the VFA measurement system would work in a reliable manner if serviced regularly (Boe, Batstone, et al. 2007). In most recent years, more accurate VFA sensors have been developed based on headspace gas chromatography (HSGC). A method that applies *ex-situ* VFA stripping with variable headspace volume and gas analysis by gas chromatography-flame ionization detection (GC-FID) has been proposed (Boe, Batstone, et al. 2007). The sample analysis might be time consuming (sampling duration is 25-40 minutes) but the individual VFA component analysis is in good agreement with off-line analysis.

Due to the fact that alkalinity is a good process indicator of AD process stability, that literature based VFA/Alkalinity ratios are variable and that VFA in-line sensors are quite difficult to construct, contain a high level of complexity when it

comes to their operation and can be quite expensive, it was decided to proceed with the design of a software sensor that predicts alkalinity.

## **3.2 Materials and methods**

### **3.2.1 Input selection**

pH, electrical conductivity (EC), organic redox potential (ORP) and temperature (in one of the designs) were selected as the inputs of the FIS that would predict alkalinity. These parameters were chosen to form the input set because they could be obtained by cheap and easy to maintain sensors performing the measurements with the use of simple electrodes (Ward et al. 2008). Moreover, they have been used in the past to predict alkalinity and assess stability of AD plants (Partner N° 2, Rothamsted Research 2010)(Ward et al. 2008)(Ward et al. 2011). Finally, by utilizing the same inputs and the same data used to predict alkalinity a comparison of the proposed fuzzy logic design could be made with the regression models.

### **3.2.2 Description of the AD plant setup**

#### ***3.2.2.1 Digester configuration***

The pilot scale anaerobic digester consisted of a two stage, semi-continuously fed stirred reactor system treating manure. The first tank was used for the hydrolysis process and the second tank for the methanogenesis process. The two tanks were based at North Wyke Research and were designed to be 1m<sup>3</sup> and 1.5m<sup>3</sup> respectively. Both tanks had several ports on the top to allow for a number of probes to be installed. The digester was operated with cattle slurry at mesophilic temperatures (37° C).



### **3.2.2.2 Sensors- Data acquisition**

Partech Waterwatch 2610 flow cells (St Austell, Cornwall UK) provided pH, ORP, EC and temperature measurements. Sensors were installed in each tank and data was downloaded into files with the utilisation of LabView™ software.

### **3.2.2.3 Process control configuration**

LabView software (version 8.2, National Instruments Ltd.) was used to record process parameter data, regulate pumping and mixing events and control the AD process by adjusting the OLR according to the magnitude of alkalinity.

When the OLR was controlled through a rule-based system configuration, it would be increased or decreased following the predicted alkalinity evolution. The OLR adjustment was defined by the rule-based system based on the alkalinity deviation from an established midpoint.

If the predicted alkalinity was higher than its midpoint, then the OLR would increase according to the distance from the midpoint. Consequently, if the predicted alkalinity was below the midpoint, then the feeding rate would decrease according to the distance from the midpoint. In addition, the current alkalinity prediction value was compared to the previous alkalinity prediction value and their difference also influenced the following feeding rate.

Derivative weighting was also utilised to control the rate of OLR change due to alkalinity adjustments. Therefore, a high derivative weighting value corresponded to a high rate change in accordance with the alkalinity value.

The system operation was also governed by a maximum OLR value that would prevent system failures due to alkalinity predictions and/or sensor malfunctions

### **3.2.3 Order of experiments**

The experiments that were performed so as to evaluate the proposed control methodology were as follows:

1. 07/05/2008- 26/07/2008

During the first experiment (M-A(1)) the OLR was adjusted manually and focused on developing and evaluating the proposed alkalinity prediction algorithm.

2. 28/07/2008- 02/10/2008

The second experiment (RB- A(2)) tested the developed algorithm and the OLR was determined by the rule-based system based on the soft-sensor predictions.

3. 02/10/2008- 20/10/2008

An improved alkalinity prediction algorithm was designed based on data collected from the previous experiments. Experiment (M-B(3)) aimed to test the new adjusted soft-sensor. OLR was controlled manually in order to test the system response to higher OLRs.

4. 20/10/2008- 07/12/2008

During the experiment (RB-B(4)), a new algorithm was implemented and a rule-based system was responsible for varying the OLR accordingly.

### **3.2.4. Chemical analysis (Alkalinity)**

Alkalinity was measured using off-line titration (Metrohm 716 DMS Titrino, Metrohm House, Unit 2, Top Angel, Buckingham Industrial Park, Buckingham MK18 1TH). Alkalinity concentration was measured in the form of  $\text{CaCO}_3$  and was converted to  $\text{HCO}_3^-$  by multiplication of the result by a factor of 1.22 as suggested in the operating procedure.

### **3.2.5. Multiple linear regression alkalinity prediction models**

An algorithm was developed to predict alkalinity values based on sensor data (Partner N° 2, Rothamsted Research 2010). Although the algorithm remained the same throughout the duration of all four experiments, the factors of the equation differed. More specifically, as data accumulated during the experiments the algorithm was improved by incorporating newly available data. The factors for the alkalinity soft

sensor equation were determined using multiple linear regression based on the pH, ORP and EC values provided by the sensors:

#### Algorithm 1 (MLR1)

The first algorithm (MLR1) was developed during the first experiment (M-A(1)) and utilised during the first and the second experiment (RB- A(2)). Equation 3.1 corresponds to the predicted alkalinity value:

##### Equation 3.1

$$\text{Alkalinity} = -8906 + (1678 * pH) + (1.998 * ORP) + (384.2 * EC)$$

#### Algorithm 2 (MLR2)

The second algorithm (MLR2) was an improved version of MLR1 since its constituting parameters were based on more data. MLR2 was used during the third (M-B(3)) and the fourth (RB-B(4)) experiment. Equation 3.2 corresponds to the predicted alkalinity value from the soft-sensor:

##### Equation 3.2

$$\text{Alkalinity} = 4876 + (22 * pH) + (0.16 * ORP) + (-223 * EC)$$

#### Algorithm 3 (MLR3)

A third algorithm that incorporated temperature as well was proposed in future co-digestion (manure and grass were used as substrates) experiments that were conducted between 02/02/2009- 23/07/2009 (Partner N° 2, Rothamsted Research 2010). The reason why temperature was incorporated is because it provided a better regression. Sufficient data were not collected (four alkalinity samples were analyzed off-line for alkalinity) and therefore this algorithm cannot be part of the comparison that will be conducted in the thesis. However, for the sake of completeness the proposed algorithm (Equation 3.3) is provided:

### Equation 3.3

$$\text{Alkalinity} = k + (k * pH) + (k * ORP) + (k * EC)$$

where the first k is a general constant and each remaining k is a unique constant associated with each process parameter.

### 3.2.6. FIS Design to predict alkalinity

#### 3.2.6.1 Introduction

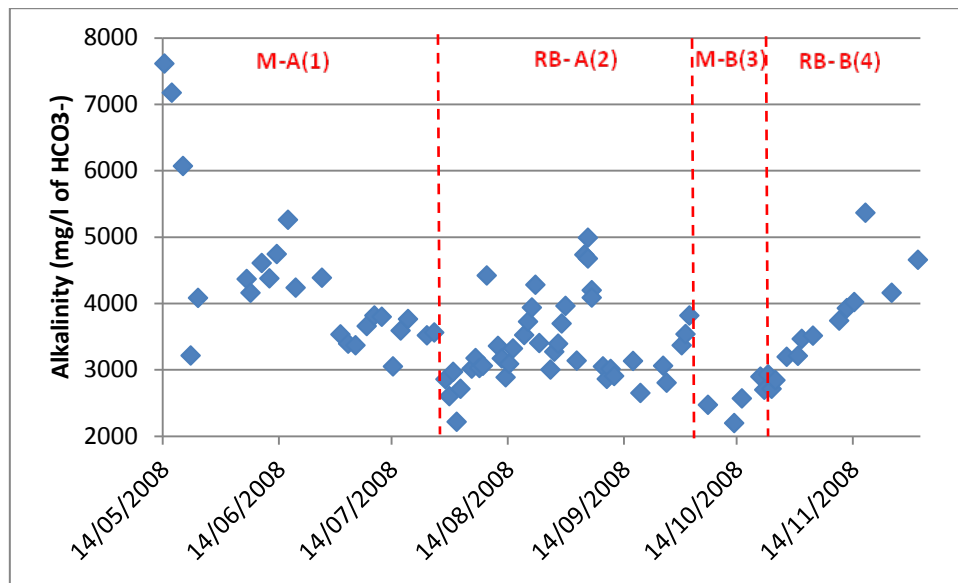
Two different FISs were implemented in order to demonstrate the improved efficiency of FL systems in variable prediction compared to multiple linear regression models. The FISs were designed using the same inputs that the multiple linear regression models used: pH, ORP and EC. First order Takagi-Sugeno FISs based on subtractive clustering were constructed to predict alkalinity.

The first FIS (FIS1) was designed and tested from data that were collected during (M-A(1)) and (RB- A(2)). FIS1 will be compared with MLR1 which is the soft-sensor that predicted alkalinity for the duration of the first two experiments. FIS1 will then be compared with MLR2 which is an improved version of MLR1 and was applied in the last two experiments. This evaluation will demonstrate that the performance of an improved multiple linear regression model is inferior to the FL system proposed.

The second FIS (FIS2) was designed based on data from the first three experiments. Because the comparison results between the first two regression models and FIS1 favoured FIS1, the second fuzzy model implementation aimed to demonstrate that as the database increases, so does the accuracy of the model. Moreover, systematic data acquisition also under extreme operational conditions enhances the model performance and robustness.

The training, checking and validation datasets for the design of FIS1 and FIS2 were selected in a random way but followed certain rules. Since the off-line alkalinity measurements were recorded in a sequential way, the training, checking and validation sets follow the same pattern.

The maximum and minimum values of each input/output variable are part of the training set. Since all the samples were collected in a sequential way it was ensured that the training, checking and validation sets followed the same pattern. The most representative way to divide the database into the three aforementioned sets is to select 10% of the samples for the validation set, 65% of the samples for the training set and the remaining 25% for the checking set. Observed alkalinity values from all experiments are depicted in Figure 3.1.



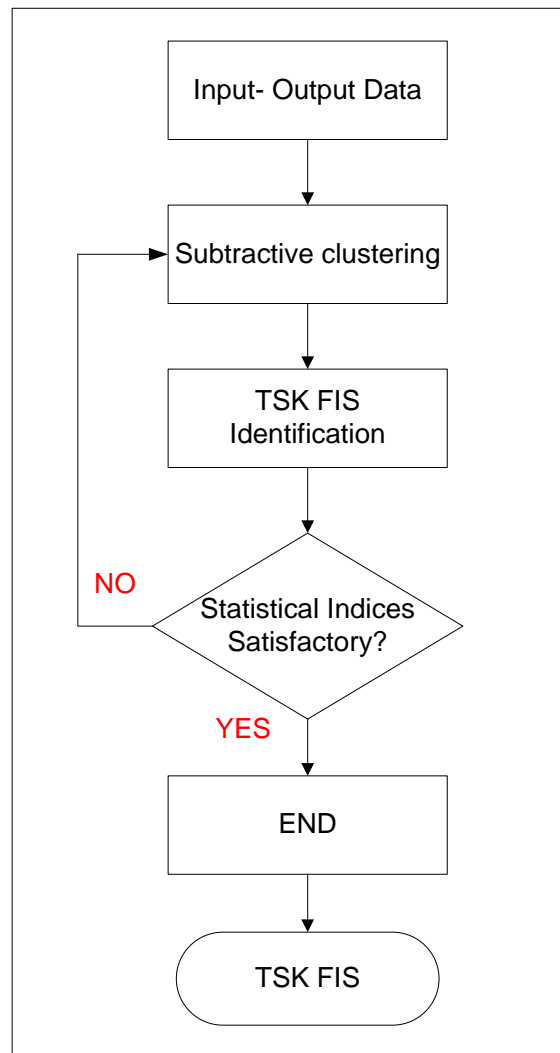
**Figure 3.1** Samples analysed for alkalinity for experiments M-A(1), RB-A(2), M-B(3) and RB-B(4).

### 3.2.6.2 Fuzzy modelling algorithm

The flow diagram of the fuzzy modelling algorithm is presented in Figure 3.2. The process followed is quite similar to the one utilised (Ren et al. 2011). Subtractive clustering (Chiu 1994) was used to locate the position and number of cluster centres and a first order Takagi-Sugeno FIS (Takagi & Sugeno 1985)(Sugeno & Kang 1988) was implemented based on the clustering performed.

Based on the investigation performed by varying the cluster radius, the squash factor, the reject ratio and the accept ratio, the cluster radius appeared to have the most important influence in the cluster estimation. Even after identifying a FIS that exhibited the best performance based on the statistical indices (3.2.7) with a specific cluster radius, the variation of all the other three parameters had very little, if

any, positive influence to our fuzzy system. Therefore, based on the parameter identification presented (Chiu Table 2.1) it was decided to vary only the cluster radius from 0.15 to 1.0 with a step of 0.01 and not to vary any of the other parameters. It is also worth mentioning that any FIS with a cluster radius greater than 0.5 exhibited similar behaviour. The Fuzzy Logic Toolbox within the framework of Matlab 7.10 was used to implement the FISs.



**Figure 3.2** Sugeno FIS based on subtractive clustering design

### **3.2.7 Performance Indicators**

There are two ways to evaluate how good model estimations are: using numerical methods and visual methods. Visual methods include the use of several plots (e.g. plotting of predicted against observed values) and can give a general idea

about the model performance. Numerical methods can accurately characterise the model performance and in some cases suggest how to improve the model.

The multiple linear regression models and the FISs performance were evaluated with the use of several statistical measures. The performance indices that follow were used because each index provides information about a different aspect of the model and some indices provide a better overall performance description if they are used in conjunction. Also, considering the application under examination some of them influence the model design more than others. In this case for example bias is considered quite important whereas in other applications the coefficient of determination ( $R^2$ ) might be the crucial indicator. We denote by  $O_i$  an observed value and by  $P_i$  a predicted value.  $O_m$  and  $P_m$  represent the average value of the observed and predicted values respectively.

The coefficient of determination ( $R^2$ ) also known as the multiple correlation coefficient provides information about the proportion of variability taken into consideration by the model.  $R^2$  is frequently used in classical regression analysis as a measure of successful prediction (Nagelkerke 1991) and takes values between 0 (poor performance) and 1 (best performance).

**Equation 3.4**

$$R^2 = \frac{(\sum_{i=1}^n (O_i - O_m)(P_i - P_m))^2}{\sum_{i=1}^n (O_i - O_m)^2 \sum_{i=1}^n (P_i - P_m)^2}$$

The mean absolute error (MAE) measures the distance of the estimates from the observed values (Equation 3.5). MAE gives information about the error in its original magnitude and scale and is less sensitive to extreme values than squared errors. The reason why MAE is preferred over root mean square error (RMSE) is that although it is used in the evaluation of many FL designs, RMSE is only indicative of the model ability to predict a value away from the mean (Nayak et al. 2005). RMSE gives a relatively higher weight to larger errors and is mostly useful when large errors are particularly undesirable (Shahi 2009). Finally, it is concluded (Willmott & Matsuura 2005) that MAE is an unambiguous measure of average error magnitude and should be used over RMSE which tends to be higher or equal to MAE at all times.

**Equation 3.5**

$$\text{Mean absolute error (MAE)} = \frac{1}{n} \sum_{i=1}^n |P_i - O_i|$$

Bias is a performance index similar to MAE but it does not use the absolute value in the difference between the predicted and observed values. Bias provides information about how much the model under predicts or over predicts. Bias values should ideally be 0.

**Equation 3.6**

$$\text{Bias} = \frac{1}{n} \sum_{i=1}^n (P_i - O_i)$$

The index of agreement (IA) was introduced by Willmott (1981) to characterise the degree of the model prediction error and varies between 0 (no agreement) and 1 (perfect agreement). IA can help to identify additive and proportional differences in the observed and predicted means and variances (Moriasi et al. 2007).

**Equation 3.7**

$$IA = 1 - \frac{\sum_{i=1}^n (P_i - O_i)^2}{\sum_{i=1}^n (|P_i - O_m| + |O_i - O_m|)^2}$$

Normalised mean square error (NMSE) is mostly used in conjunction with MAE and measures the deviation between predicted and observed values and ideally should have a value of 0 indicating a perfect agreement. However, high NMSE values do not imply that the model under examination is completely unreliable.

**Equation 3.8**

$$NMSE = \frac{\sum_{i=1}^n (P_i - O_i)^2}{\sum_{i=1}^n (P_i)^2}$$



Fractional Bias (FB) is a measure of agreement between mean observed and predicted values (Cakmakci 2007) and a value of 0 signifies a perfect agreement.

**Equation 3.9**

$$\text{Fractional Bias (FB)} = \frac{P_m - O_m}{0.5(P_m + O_m)}$$

### **3.3 Results and discussion**

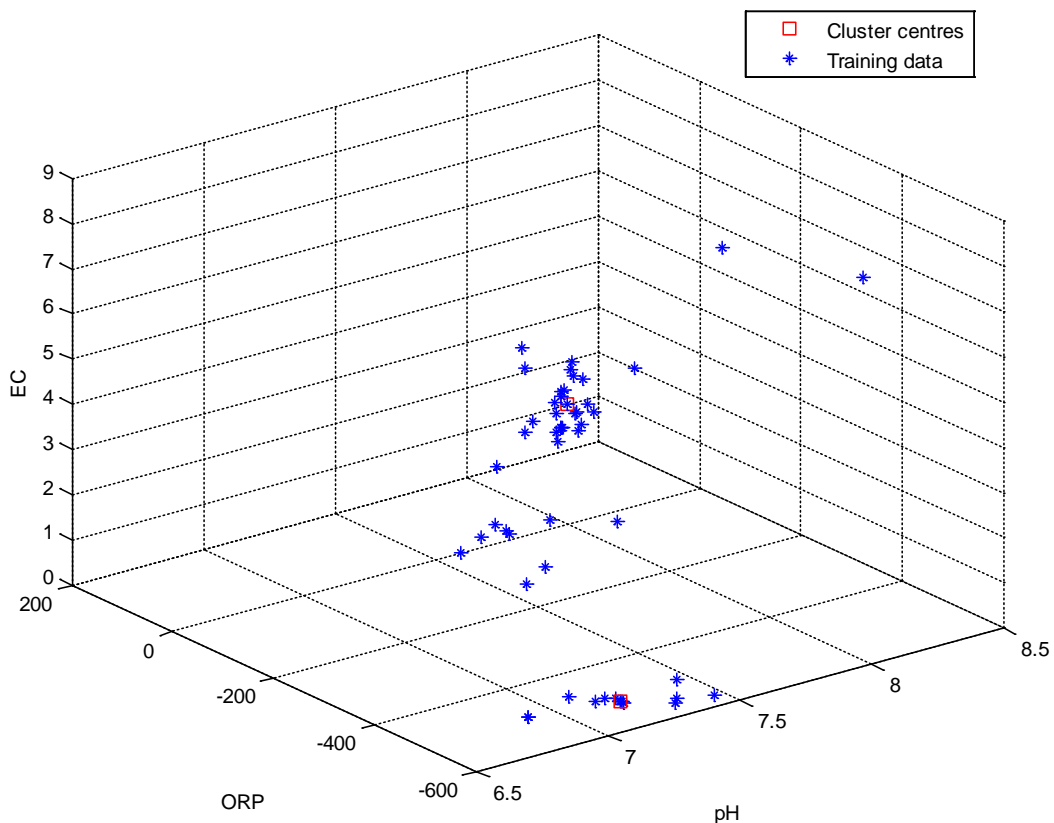
#### **3.3.1 FIS1 design and comparison with MLR1**

The maximum and the minimum input/output values were selected to be part of the training set of the FIS. 78% of the data was part of the training set and 22% of the data constituted the checking set. Since the data were collected in a sequential manner the training and checking set values followed the same pattern (one checking set data point every four training data points). This selection came as a result of previous attempts where both datasets were selected in random, ensuring though that the maximum and minimum values were always part of the training set, but the results were not satisfactory and in many cases although the resulting FIS performance appeared to be really good the evaluation using newly available data proved to be poor mainly due to the poor spread of the training dataset values.

The FIS design was based on the process described in Figure 3.2. Least-square estimation was used to identify the consequent parameters of the TSK FIS (Chiu 1994). The consequent functions of the model are linear. The premise structure and parameters as well as the consequent structure and parameters were set and tuned in a recursive manner.

During the variation of the cluster radius, the squash factor, the reject ratio and the accept ratio, the cluster radius appeared to have the most influence in the cluster estimation. After identifying the best data fitted FIS with a specific cluster radius, the variation of all the other three parameters had very little, if any, positive influence to our fuzzy system. Therefore, it was decided to vary only the cluster radius from 0.15 to 1.0 with a step of 0.01 and not to vary any of the other parameters.

The parameters based on which the best FIS was designed were: cluster radius 0.5, squash factor 1.25, reject ratio 0.15 and accept ratio 0.5. Two cluster centres were identified using subtractive clustering (Table 3.1) based on which the Gaussian membership functions for each input were defined. Each input had two fuzzy sets based on the clustering performed (Figure 3.4) and two rules governed the fuzzy system's function (fuzzy model) (Table 3.2). For EC there is a uniform distribution of the fuzzy sets which is depicted by the distance in the location of the cluster centres. For pH there is a higher degree of overlap. This is probably due to the fact that the range of pH values is limited between 5.9 and 8.9 and the majority of them have values close to 7. This explains why the two cluster centers are both located quite close to this value. All the ORP values apart from a very few exceptions are centered around -500. This is the reason why there is a high amount of overlap between the two membership functions.



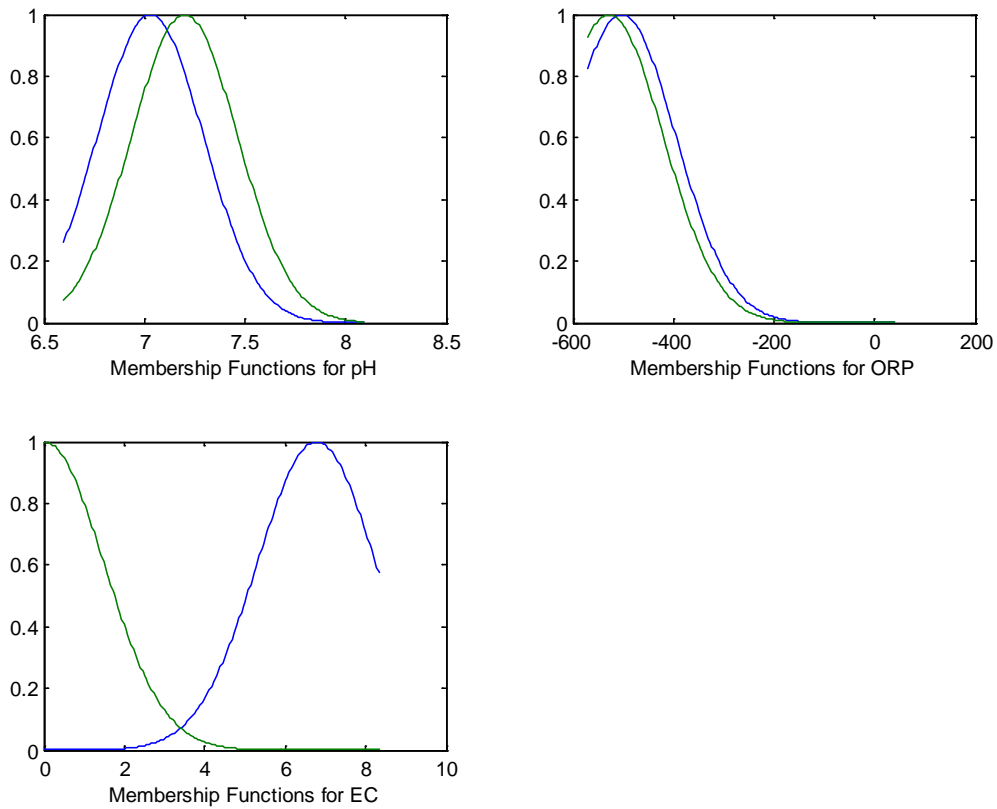
**Figure 3.3** 3D representation of subtractive clustering for FIS1

Cluster	Membership Functions	Centres			Width (spread)		
		pH	ORP	EC	pH	ORP	EC
1	MF1	7.03	-503	6.78	0.263	107.66	1.473
2	MF2	7.2	-521	0.02	0.263	107.66	1.473

**Table 3.1** Membership function parameters obtained using subtractive clustering for FIS1

Rule	Rule description of the form If ... Then $y = p_1x_1 + p_2x_2 + p_3x_3 + p_0$
1	<i>If <math>pH_{in}</math> is <math>pH_{in}MF1</math> and <math>ORP_{in}</math> is <math>ORP_{in}MF1</math> and <math>EC_{in}</math> is <math>EC_{in}MF1</math> Then Alkalinity = <math>-43.545 \cdot pH + 1.186 \cdot ORP - 33.244 \cdot EC + 4485.959</math></i>
2	<i>If <math>pH_{in}</math> is <math>pH_{in}MF2</math> and <math>ORP_{in}</math> is <math>ORP_{in}MF2</math> and <math>EC_{in}</math> is <math>EC_{in}MF2</math> Then Alkalinity = <math>4411.727 \cdot pH - 3.8 \cdot ORP + 534.276 \cdot EC - 29215.7</math></i>

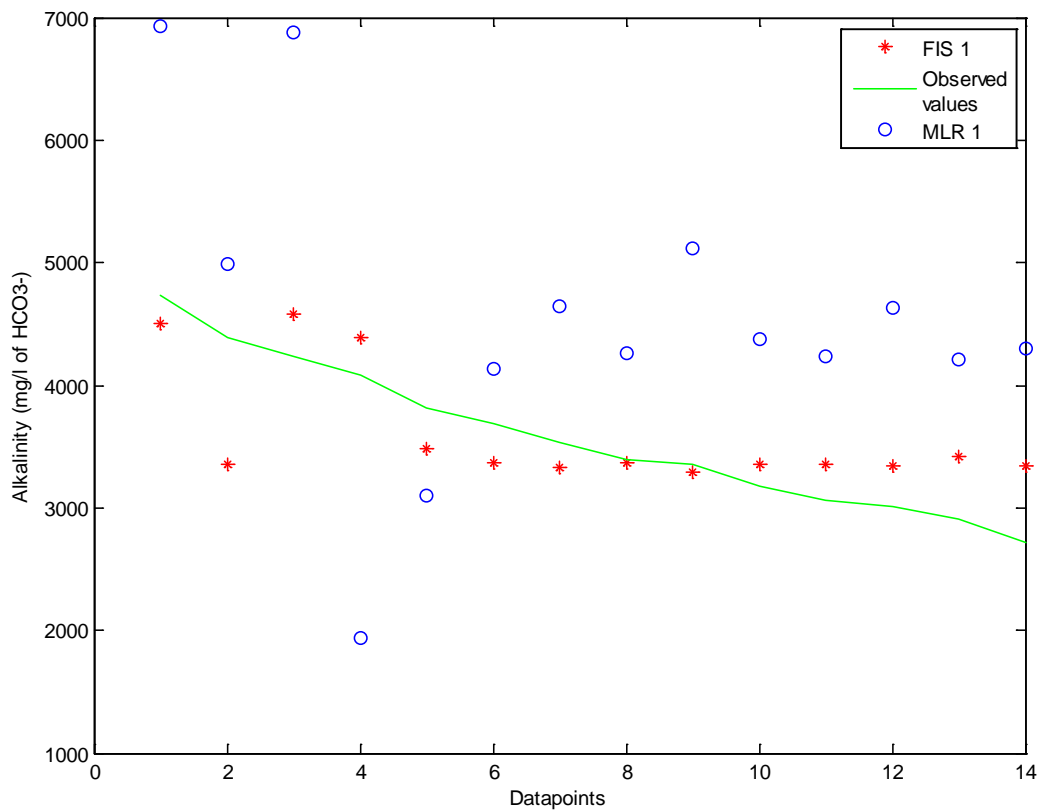
**Table 3.2** TSK fuzzy model rules and consequent parameters for FIS1



**Figure 3.4** Fuzzy sets for FIS1

FIS1 was compared with MLR1 in order to evaluate its performance (Figure 3.5). Since both MLR1 and FIS1 were designed based on data obtained during the experiment M-A(1), the performance indices provided in Table 3.3 do not correspond to the evaluation of these models with newly available data. They demonstrate however, whether these techniques (multiple linear regression and fuzzy logic) are suitable to predict the system's output (alkalinity) in an accurate manner.

The data points used to train both models were few (52 data points for FIS1 and 68 for MLR1) and in a nonlinear process like anaerobic digestion data-based approaches require a much larger amount of data for the designed model to be quite accurate. This is one of the reasons why the performance indices for MLR1 appeared to be very poor.



**Figure 3.5** Observed and predicted alkalinity values using FIS1 and MLR1

Performance indices	MLR1	FIS1
$R^2$	0.122	0.494
MAE	1383.3	344.23
Bias	976.3	27.219
IA	0.475	0.824
NMSE	0.103	0.013
FB	0.24	0.008

**Table 3.3** Alkalinity performance for MLR1 and FIS1

MLR1 was poor in predicting alkalinity values (Table 3.3). Taking into consideration that alkalinity ranged between 1600-6700, a MAE of 1383 and a bias value of 976.3 indicate that the regression model is unsuitable to predict the process

parameter under investigation. The rest of the performance indices also indicate an insufficient input-output mapping.

FIS1 demonstrated an improved efficiency in predicting alkalinity. Although the coefficient of determination was 0.494, the low MAE value, the even lower value for bias and the almost zero NMSE and FB values indicate that the fuzzy logic model could be possibly control the OLR based on alkalinity predictions. However, since some of the observed alkalinity values were outside the training range of FIS1, the fuzzy model is not considered to be reliable for future applications.

### 3.3.2 Comparison between FIS1 and MLR2

MLR2 was developed based on data from the first two experiments M-A(1)) and RB- A(2) and was applied to predict alkalinity during the third M-B(3) and the fourth RB-B(4) experiment. MLR2 was essentially an improvement of MLR1 since its design was based on more data that described the anaerobic digestion evolution process in a larger range of operation.

MLR2 was compared with FIS1 against newly available data from M-B(3) and RB-B(4) to demonstrate that an improved multiple linear regression model could not predict alkalinity more accurately than a fuzzy model that was designed with less amount of data. Additionally, certain input values laid outside the FIS1 training range since changes in the anaerobic digestion environment over that period resulted in having new extreme input values. The response of the FIS to these extreme values was positive as indicated by the performance indices in Table 3.4.

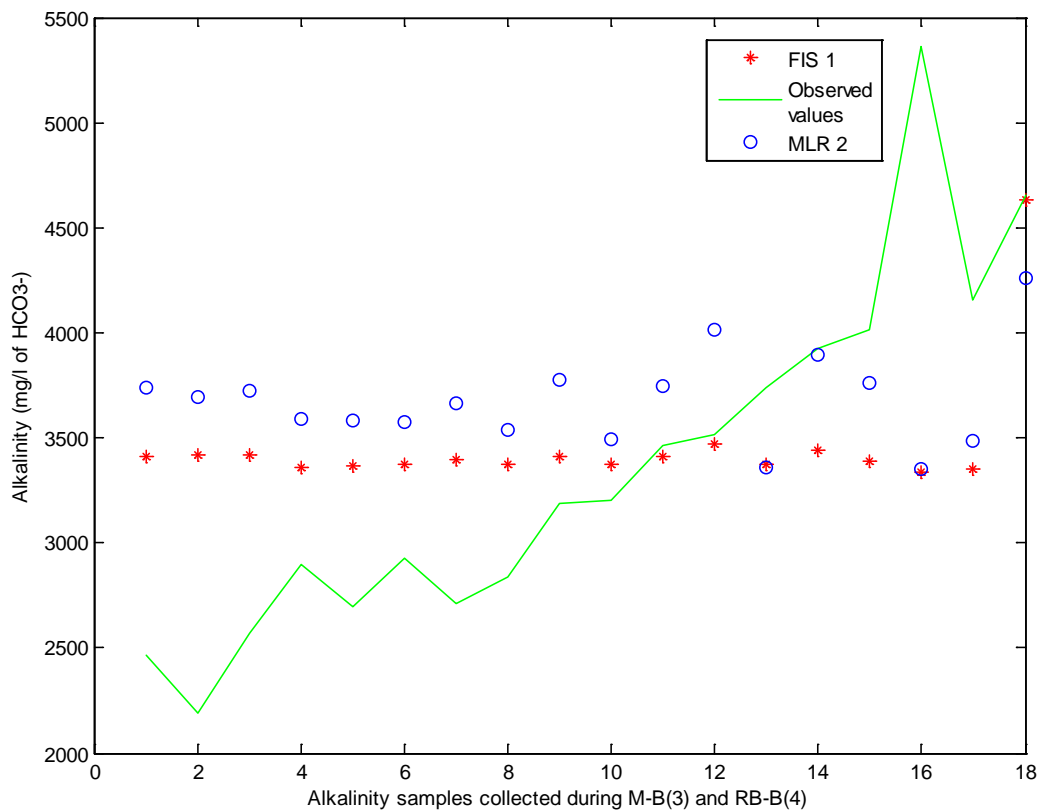
<b>Performance indices</b>	<b>MLR2</b>	<b>FIS1</b>
R <sup>2</sup>	0.004	0.127
MAE	733.86	591.28
Bias	319.74	99.494
IA	0.361	0.4
NMSE	0.057	0.048
FB	0.09	0.03

**Table 3.4** Alkalinity performance for MLR2 and FIS1

It appears that the performance of both models was quite inadequate for the FIS1 and inadequate for MLR2 (Figure 3.6). However, by taking into consideration that the system did not operate during extreme conditions (e.g. really high loading rates) during the first experiment, FIS1 cannot be considered as a reliable alkalinity predictor. Therefore, it needs to be ensured that input/output values that were outside its design range are embedded in the design of the fuzzy model.

The MLR2 performance indices indicate that the regression model is quite unsuitable for this application. A coefficient of determination with a value of zero is indicative of the model performance and only the MAE value is quite satisfactory. Still, a model that was designed based on data over a five month period should provide more accurate results especially compared with an undertrained model such as FIS1.

Although the  $R^2$  value is extremely low for FIS1, the MAE and the bias indicate that the predicted values do not deviate by a huge amount from the actual values. However, an IA value of 0.4 is really low and the NMSE and FB values are slightly high. Figure 3.6 shows that the FIS1 predictions do not deviate that much and that the fuzzy model is not very sensitive to alkalinity changes based on pH, ORP and EC fluctuations.



**Figure 3.6** Observed and predicted alkalinity values using FIS1 and MLR2

### 3.3.3 FIS2 design

A new FIS was designed in order to demonstrate that by increasing the training data base a more accurate model can be designed. However, systematic data acquisition is required (preferably on a daily basis) to sufficiently monitor the evolution of the input/output parameters. During all four experiments samples were not analyzed for alkalinity on such a frequent basis which resulted in a not so accurate process representation.

MLR3 was superior to the previous multiple linear regression models presented above because temperature was also part of the system inputs. By including temperature a more precise alkalinity value could be forecasted considering the fact that temperature fluctuations severely impact the anaerobic digestion process. Moreover, by including temperature as an input a better regression was achieved (Partner N° 2, Rothamsted Research 2010). However,



FIS2 did not include temperature as an input parameter because the fuzzy models did not respond positively to this addition.

Data from the first three experiments M-A(1), RB- A(2)), and M-B(3)) were used in the fuzzy logic implementation that was validated with data from the fourth experiment(RB-B(4)). It was ensured that the fuzzy logic model would accommodate maximum and minimum input values. The selection process of the training and checking sets for the model implementation was performed in a similar manner to the process followed for the implementation of FIS1.

FIS2 implementation was based on the process depicted in Figure 3.2. The consequent parameters were determined by utilizing the least-square estimation method (Chiu 1994). The consequent functions of the model are linear. The premise structure and parameters as well as the consequent structure and parameters were set and tuned in a recursive manner.

The cluster radius was varied from 0.15 to 1 with a step of 0.01 during the best model identification process. The values of the squash factor, the reject ratio and the accept ratio were set to 1.25, 0.15 and 0.5 respectively.

The parameters based for the best FIS designed were: cluster radius 0.27, squash factor 1.25, reject ratio 0.15 and accept ratio 0.5. Three cluster centres were identified using subtractive clustering (Table 3.5) based on which the Gaussian membership functions for each input were defined. Each input had three fuzzy sets based on the clustering performed and three rules governed the fuzzy system's function (fuzzy model) (Table 3.6).

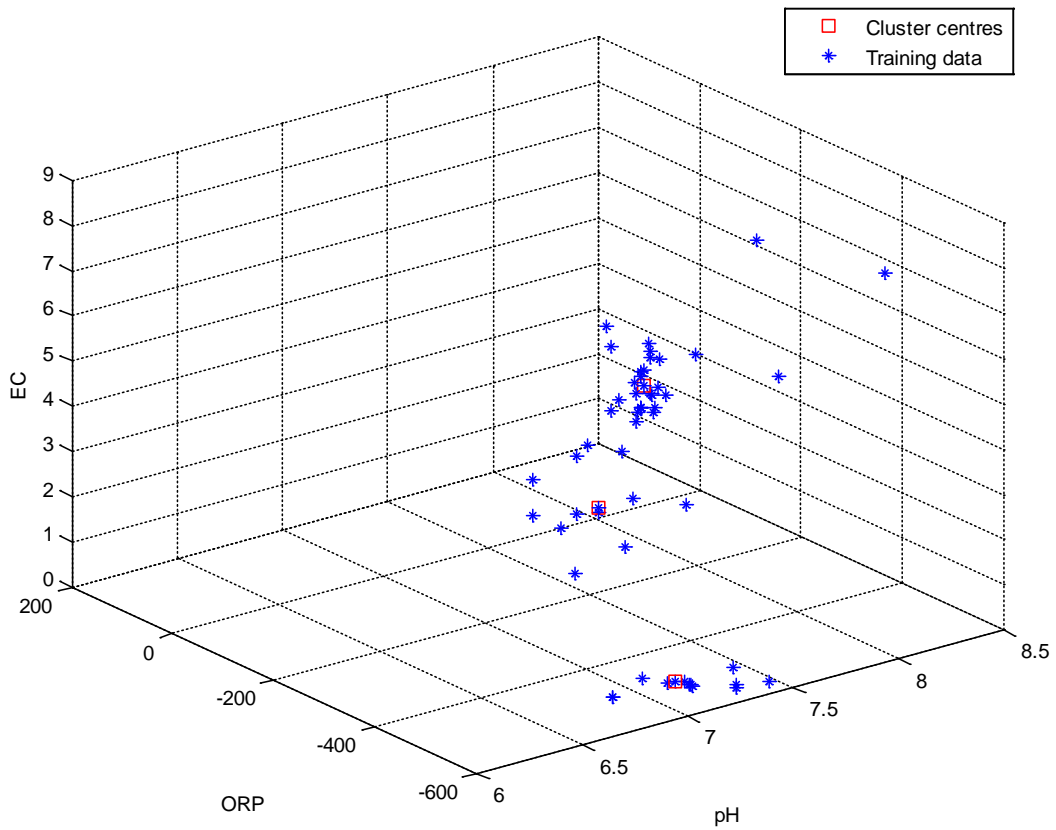


Figure 3.7 3D representation of subtractive clustering for FIS2

Cluster	Membership Functions	Centres			Width (spread)		
		pH	ORP	EC	pH	ORP	EC
1	MF1	7.03	-503	6.78	0.142	58.135	0.795
2	MF2	7.18	-501	0.02	0.142	58.135	0.795
3	MF3	6.76	-525	4.51	0.142	58.135	0.795

Table 3.5 Membership function parameters obtained using subtractive clustering for FIS2

Rule	<p style="text-align: center;"><b>Rule description of the form</b></p> <p style="text-align: center;"><b>If ... Then <math>y = p_1x_1 + p_2x_2 + p_3x_3 + p_0</math></b></p>
1	<p><i>If <math>pH_{in}</math> is <math>pH_{in}MF1</math> and <math>ORP_{in}</math> is <math>ORP_{in}MF1</math> and <math>EC_{in}</math> is <math>EC_{in}MF1</math> Then</i></p> <p style="text-align: center;"><i>Alkalinity = <math>-29.329 \cdot pH + 1.171 \cdot ORP - 28.395 \cdot EC + 4349.795</math></i></p>
2	<p><i>If <math>pH_{in}</math> is <math>pH_{in}MF2</math> and <math>ORP_{in}</math> is <math>ORP_{in}MF2</math> and <math>EC_{in}</math> is <math>EC_{in}MF2</math> Then</i></p> <p style="text-align: center;"><i>Alkalinity = <math>5716.502 \cdot pH - 0.081 \cdot ORP - 36581.335 \cdot EC - 36068.709</math></i></p>
3	<p><i>If <math>pH_{in}</math> is <math>pH_{in}MF2</math> and <math>ORP_{in}</math> is <math>ORP_{in}MF2</math> and <math>EC_{in}</math> is <math>EC_{in}MF2</math> Then</i></p> <p style="text-align: center;"><i>Alkalinity = <math>350.181 \cdot pH + 22.5 \cdot ORP - 484.338 \cdot EC + 14956.646</math></i></p>

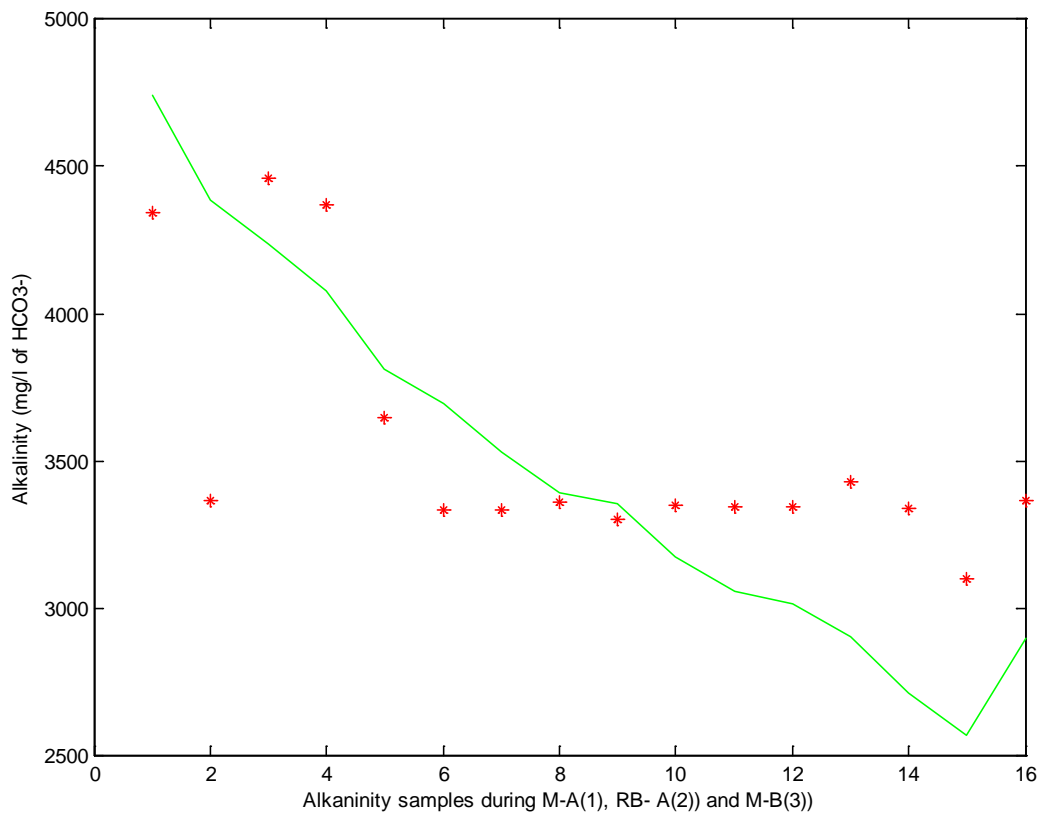
**Table 3.6** TSK fuzzy model rules and consequent parameters for FIS2

FIS2 proved to be much better than FIS1 in predicting alkalinity values by examining the statistical performance indices (Table 3.7). FIS2 predicted values were much closer to the actual values during the validation process than the training and checking process. This indicates that the model is capable to predict alkalinity values more accurately than FIS1, MLR1 and MLR2.

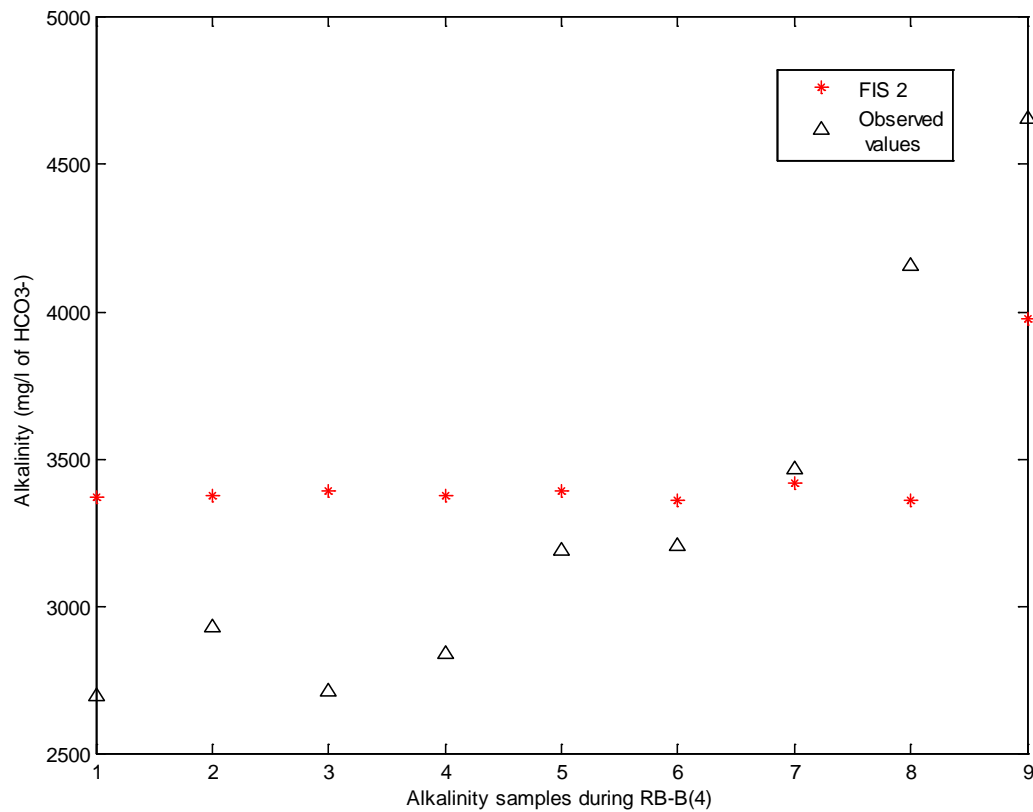
A 0.534 value for  $R^2$  and a similar value of the IA indicate that the observed values are quite close to the actual values. The MAE and the bias point out that the deviation from the actual values is not high and that the predictions follow the measurements relatively closely. Finally, quite low values for NMSE and FB signify that the fuzzy model values are in quite good agreement with the actual output parameter values. Training and checking alkalinity values are depicted in Figure 3.8, and observed and predicted alkalinity values for the RB-B(4) experiment are depicted in Figure 3.9.

Performance indices	FIS2	
	Training	Validation
R <sup>2</sup>	0.496	0.534
MAE	334.66	468.72
Bias	16.233	130.97
IA	0.814	0.594
NMSE	0.013	0.024
FB	0.005	0.039

**Table 3.7** Training and validation performance for FIS2



**Figure 3.8** Observed and predicted alkalinity values for FIS2 during M-A(1), RB- A(2) and M-B(3))



**Figure 3.9** Observed and predicted using FIS2 alkalinity values during RB-B(4)

### 3.4. Conclusions

This chapter presented a fuzzy logic technique to predict an important process parameter (alkalinity) using subtractive clustering. Two first order Takagi-Sugeno (TS) FISs based on subtractive clustering were developed and tested against already applied multiple linear regression models.

The FISs (FIS1 and FIS2) demonstrated an improved ability to predict alkalinity values from known input parameters such as pH, ORP and EC compared to MLR1 and MLR2. However, even the fuzzy model performance was only adequate indicating a need to either identify different input parameters or even better record data systematically in order to improve the model response.

Multiple performance indices were used to evaluate how each model performed. Since alkalinity off-line measurements can also vary while analyzing the

same sample for alkalinity, MAE values of up to 500-600 can be considered as acceptable. This implies that  $R^2$  and IA values will not be as high as in other AD applications where different input/output parameters are utilised.

Although (Partner N° 2, Rothamsted Research 2010) a newly developed model that included temperature as an input was able to successfully control the OLR that dictated the operation of the system, approaches that included this input parameter and applied fuzzy logic principles were unsuccessful. This means that the three input parameters used in the fuzzy designs were suitable for the purpose and that the addition of temperature would not improve the model. Moreover, sufficient validation data for MLR3 could not support the argument that its predictions were superior to the other regression models as only four samples were analyzed for alkalinity during the five month application of MLR3.

The modelling approach proposed is suitable for AD applications provided that the training database consisted of continuous samples over a long period of time, based on which the loading rate of an AD system is controlled. Daily sample analysis for alkalinity would result in having a more precise representation of alkalinity evolution since OLR was varied on the same basis.

## **Chapter 4 Monitoring of anaerobic digestion, identification of key process parameters and process investigation of anaerobic digesters with and without support media**

The development and evaluation of two fuzzy logic models predicting alkalinity based on the operation of small scale anaerobic digesters is presented in this chapter. The suitability of first order TSK FIS using the design process proposed in Chapter 3 is evaluated through application on three 5l reactors with different support media. However, different inputs were utilised in the design of the FISs. Those were pH, daily difference in pH, gas production volume and daily difference in gas production volume for the first FIS and pH, daily difference in pH, gas production volume/reactor volume and daily difference in gas production volume/reactor volume for the second FIS. The performance of the reactors with support media is compared with the performance of three 5l reactors without support media and optimum performance and stability OLR, pH and alkalinity process parameters are identified for all six reactors.

### **4.1 Introduction**

Anaerobic digestion research is a time consuming task due to the large time periods required for start-up, especially for high capacity biogas units, for digester stabilisation and for observing the influence that a variation in the process parameters has on system performance.

As mentioned in Chapter 1 (1.2.5), hydraulic retention times vary. The experimental work presented in this chapter focuses on the operation under thermophilic conditions that generally require lower retention times than digesters operating at mesophilic temperatures. Indicatively, at thermophilic temperatures solids retention times can vary from a couple of days (4 days) to 20 days (Table 1.2), (Moen et al. 2003b). In order to maximise methane productivity, SRT has to be

decreased and loading rates have to be increased up to a point to avoid overloading that might produce a sour digester (such an investigation is presented (Ferrer et al. 2010)). Also, in order to draw safe conclusions regarding the operation of an anaerobic reactor under a specific OLR, the system should be allowed to complete at least three retention times.

An anaerobic digester that consisted of three 120l tanks with support surfaces was constructed to investigate the stages of start-up, stable operation, failure and recovery. Also, a fuzzy model predicting alkalinity would be developed based on the manual digester operation. In this way, a controller that would vary the OLR based on alkalinity predictions would be implemented aiming to stabilise the AD process and maximise biogas productivity. However, although operational failures did not allow for this system to work, important lessons were learnt concerning the design of anaerobic reactors (Appendix I).

Instead, three 5l reactors without support media were operated for a period of about three months (18/07/2012- 01/10/2012) and three 5l reactors with different support media were operated for a five month period (18/07/2012-21/12/2012). Data from the first four months were examined since temperature problems resulted in having corrupted measurements during the last month of operation.

The aim of the experiments was that all the reactors would undergo the processes of start-up, failure, recovery and stable operation with different retention times. In this way, results could be drawn with respect to:

- Performance differences between different types of reactors (reactors with and without biomass support media)
- Performance differences between reactors with different support media.
- Optimum operational conditions for performance and stability (pH, alkalinity, OLR)
- Stabilisation and maximisation of biogas production
- Alkalinity evolution

Data from these experiments were used in the design and evaluation of two first order TSK fuzzy logic systems to predict alkalinity. TSK FISs were implemented to predict alkalinity levels in all reactors with support surfaces. So, based on the



alkalinity predictions a controller that would vary the OLR would be developed (Chapter 5) and its function would be tested in further experiments.

## 4.2 Small scale experiments Materials and Methods

### 4.2.1 Construction of single-stage reactors

Six 5l vessels were used to construct the single stage reactors (Figure 4.1). Each reactor was filled with 4l of substrate to allow for head space. A mechanical stirrer was utilised to mix the contents through a stirring rod that existed in the middle of the vessel sealed with silicone to avoid gas leakages.

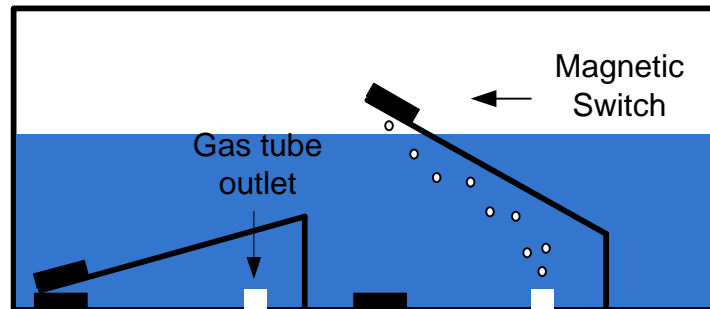


**Figure 4.1** Single-stage digester without support media

Different biomass support media were lining the inside of three of the tanks after being formed into a cylinder. Stainless steel wire was utilised to keep it in its place to prevent the support media from collapsing after extensive period of usage (section 4.2.2 provides detailed analysis of the support media).

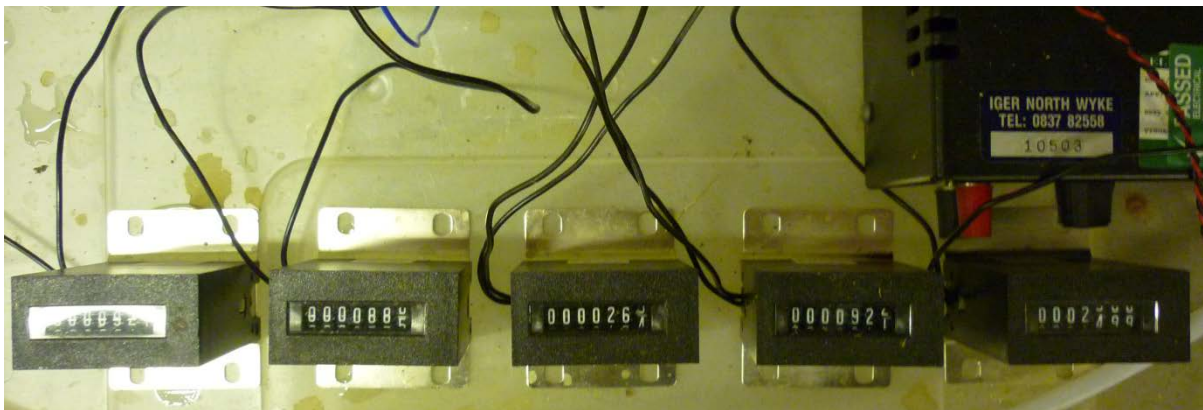
The gas outlet located at the top of the vessel was connected to a liquid displacement system made out of cells of known volume capacity. The cell system

was designed to work based on the principle of buoyancy. Magnetic reed switches were connected to each cell to providing information about the switching behaviour of each cell. Side profile diagram of a cell is depicted in (Figure 4.2).



**Figure 4.2** Gas outlet tubes are connected through the bottom of the water bath directing gas into each cell. Magnetic reed switches record gas production volumes.

The magnetic reed switches were connected to a system of mechanical counters to enable off-line recording of gas volume production (Figure 4.3).

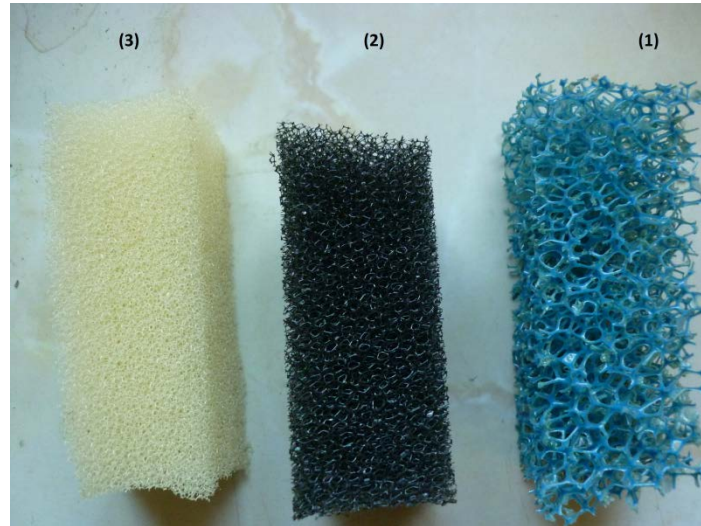


**Figure 4.3** Mechanical counters connected to the magnetic reed switches

#### 4.2.2 Biomass support media

Three of the reactors were equipped with support media (Figure 4.4). Biomass support media were attached to the reactors to enhance stability during system operation under different OLRs, high OLRs and short HRTs (Show & Tay 1999)(Demirer & Chen 2005). The chosen support material for the first reactor was burst cell reticulated polyurethane foam coarse with a ca. 2.5 mm pore diameter.

Burst cell reticulated polyurethane foam medium with a ca. 1 mm pore diameter was inserted to the second reactor, and sponge with a ca. 1mm pore diameter was selected as the biomass support surface for the third reactor.



**Figure 4.4** Biomass support media. (1) Burst cell foam coarse, (2) burst cell foam medium, (3) sponge

The size of the support material in each reactor was approximately 35 cm×20 cm×3 cm. Experiments with both water and cow slurry were conducted under room temperature to measure the density of each material (Equation 4.1). The total solids (TS) percentage was 4.55% and the volatile solids (VS) percentage was 4.13% of the cow slurry.

**Equation 4.1**

$$\text{Density} = \frac{\text{Weight of sample (kg)}}{\text{Length} \times \text{Width} \times \text{Height (m}^3\text{)}}$$

The porosity of each material was also determined with the use of water by Equation 4.2:

**Equation 4.2**

$$\text{Porosity} = \frac{\text{Void volume}}{\text{Total volume of the sample}} \times 100$$

The support media were also examined with respect to how much volume of slurry they can absorb using water and cow slurry with the same characteristics as above. The absorptive capacity measurement would indicate the amount of working

volume that exists inside each reactor by determining how much volume each material could absorb. This experiment was conducted under room temperature. Absorbency (%) was calculated as:

**Equation 4.3**

$$Absorbency\% = \frac{(WA - WB)}{WB} \times 100$$

where WA= support media weight after immersion (g) and WB= weight of dry support surface before immersion (g). The process followed was similar to the one presented (Das et al. 2009) with the difference that in the experiments conducted with slurry the materials under investigation were left in the liquid for 24 hours.

Two samples of each support media were used to determine density, porosity and absorbency with water. Each material was tested for a second time after being left to dry out and an average value was recorded. Three samples of each support media (nine samples in total) were used to determine density and absorbency with the use of slurry. Then an average value was calculated for each material. Table 4.1 contains the experimental results.

	<b>Foam coarse</b>	<b>Foam medium</b>	<b>Sponge</b>
Dry density	0.030	0.023	0.024
Wet density	0.369	0.510	0.605
Porosity	60.00	57.37	47.53
Absorbency	1136.65	2032.43	2399.03
Slurry density	0.35	0.44	0.37
Slurry absorbency	1390.00	1933.33	1731.58

**Table 4.1** Biomass support media characteristics at room temperature

Maintenance reactor operations that took place during the operation period (4.2.4) (the tanks were operated under thermophilic conditions 55°C) provided different results regarding slurry absorbency. A higher temperature value (55°C), a larger surface area or the fact that the total solids and the volatile solids percentage were lower than the original experiments influenced all three biomass surfaces to absorb higher amounts of slurry. Each support material volume was measured once

and the recorded absorbed volume was validated by weighting the remainder volume inside each vessel. All three support surfaces were able to hold similar amounts of slurry varying between 1.470 kg- 1.490 kg for TS%= 2.8-3.1 and VS%= 2.35-2.7. Therefore, slurry absorbency percentage was much higher compared to the original experiments by holding an average value of 5710 which is approximately 2.5 times greater than the values in Table 4.1.

#### **4.2.3 Feedstock**

Cow manure was used for the operation of the six 5l reactors. Cow manure originating from the beef cattle located at North Wyke Research was collected every seven days. Cow manure was diluted with water and screened through a mesh with a 10 mm× 10mm hole size to remove most of the hay present in the manure. Cow Slurry was then stored in a deep freezer at +4° C during this period to delay the anaerobic digestion process. The feed material was inserted on a daily feeding basis in a water bath that was kept at 55°C for 5-6 hours. This period corresponds to the time that cow slurry takes to reach this temperature from room temperature. This process would ensure that temperature inside each reactor would remain stable at all times.

#### **4.2.4 Reactor operation**

The experimental work initiated in 18/07/2012 using cow slurry that contained 4% total solids and 2.6% of volatile solids and had an alkalinity value of 3350 mg/l HCO<sub>3</sub><sup>-</sup>. The total solid content and volatile solid content of the feed material was slightly different every week due to the collection of fresh cow manure. Each reactor was fed on average once a day five days a week due to working rules. The feeding process was carried out manually by disconnecting the gas tube from the top of each reactor and by withdrawing and inserting equal amounts of volume.

The operation of the six reactors, three without support media (1, 2, 3) and three with support media (4, 5, 6) lasted approximately seven weeks until 06/09/2012. Burst cell foam coarse was attached to reactor 4, burst cell foam medium was attached to reactor 5 and sponge was the selected support media for

reactor 6. All reactors were filled with 4l of fresh cow slurry with the above characteristics and no other inoculum was used.

#### **4.2.5 Mixing**

Experiments regarding the impact of mixing were not conducted. An adequate mixing regime was selected. The contents of each reactor were mixed with a stirring rod inserted through the top of each vessel. The mixing speed was approximately 120 rpm. A 24 hour mechanical time switch provided power for 15 minutes every hour.

#### **4.2.6 Off-line monitoring**

The six reactors were monitored once a day for pH and CH<sub>4</sub>%. Gas volume measurements were recorded once a day between 19/07/2012- 10/09/2012 and twice a day between 10/09/2012- 20/11/2012 to enable the identification of possible leakages in the gas collection system or the reactors. Influent and effluent total solids and volatile solids content were determined at regular intervals. Samples were analyzed for alkalinity on an average of four days a week.

pH was measured off-line after feeding using a HANNA INSTRUMENTS HI9025 microprocessor-based pH meter. The pH meter was calibrated on a weekly basis using pH 4 and pH 7 buffer solutions.

##### **4.2.6.1 Methane**

CH<sub>4</sub>% was measured before feeding using a Crowcon Triple + plus IR gas monitor. The device utilises an infrared sensor to determine methane levels between 5- 100% volume scale as well as 0-100% LEL (lower explosive limit). Therefore, since there is no numerical display for % volume until the level of gas exceeds 100% LEL, 100% LEL corresponds to CH<sub>4</sub>= 5 %.

#### **4.2.6.2 Biogas volume**

Daily gas volume production was recorded by multiplying the number of times that each switch was activated that was displayed on the mechanical counters (Figure 4.3) with the volume that each cell could hold.

#### **4.2.6.3 Total solids**

Samples for total solids percentage and volatile solids percentage were taken at regular intervals from both the feedstock (after heating) and the effluent originating from each tank. Total solids are determined as the residue that is left following the evaporation of liquid from a sample that resulted from drying the sample in an oven operating between 103° C to 105° C to a constant weight or mass. Samples were inserted in aluminium foil trays and were weighted before entering the oven. The samples remained inside the oven for 24 hours to ensure that the sample was dry and then weighted again.

#### **4.2.6.4 Volatile solids**

Volatile solids are calculated by measuring the sample weight loss after ignition. Volatile solids determination was performed by using the dried samples above, placing them in ceramic trays and inserting them in a muffle furnace at 550° C for 4 hours to achieve a constant mass. Then, the samples were weighed again to determine the volatile solids percentage. TS and VS percentages were calculated using Equation 4.4 and Equation 4.5 respectively.

**Equation 4.4**

$$\% \text{ Total Solids} = \frac{\text{Dry solids weight}}{\text{Liquid weight}} \times 100$$

**Equation 4.5**

$$\% \text{ Volatile Solids} = 1 - \frac{\text{sample after ashing}}{\text{dry sample}}$$

Volatile solids reduction (VSR) which is indicative of the anaerobic digestion process performance and is expressed as a percentage was also calculated using Equation 4.6:

**Equation 4.6**

$$VSR = \frac{VS_{in} - VS_{out}}{VS_{in} - (VS_{in} \times VS_{out})} \times 100$$

where  $VS_{in}$  = volatile solids that enter the digester and  $VS_{out}$  = volatile solids that are removed from the digester.

#### **4.2.6.5 Alkalinity**

Samples that were collected after the feeding process were examined for total alkalinity. Samples were first centrifuged for five minutes at 3000 rpm. Then a mixture that contained 0.25 ml of the sample and 80 ml of deionised water was analyzed for total alkalinity with the use of an auto-titrator (Metrohm 716 DMS Titrino). The auto-titrator calculated total alkalinity in terms of  $\text{CaCO}_3$  concentration. Next, alkalinity was converted from  $\text{CaCO}_3$  to  $\text{HCO}_3^-$  because alkalinity in terms of  $\text{HCO}_3^-$  provides a more accurate measure of the buffering capacity in anaerobic digestion environments (Hattingh et al. 1967)(Lahav & Morgan 2004). Also, because digester pH is controlled by the  $\text{CO}_2$  concentration in the gas phase and by the  $\text{HCO}_3^-$  alkalinity in the liquid phase (Appels et al. 2008). The alkalinity conversion from mg/l as  $\text{CaCO}_3$  to mg/l as  $\text{HCO}_3^-$  was conducted as follows: Consider the reaction



and the fact that the molecular weight of  $\text{CaCO}_3$  is 100 g/mol and the molecular weight of  $\text{HCO}_3^-$  is 61 g/mol. So, one mol of  $\text{Ca}(\text{HCO}_3)_2$  is equivalent to one mol of  $\text{CaCO}_3$  and holds  $2 \times 61 = 122$  g of  $\text{HCO}_3^-$ . Therefore, alkalinity is expressed (California department of public health 2013) in terms of  $\text{HCO}_3^-$  (mg/l) =  $1.22 \times$  alkalinity as  $\text{CaCO}_3$  (mg/l).



## 4.3 Results and discussion

### 4.3.1 Start-up

Identical loading rates for all of the reactors without support and identical loading rates for the three reactors with support were applied. The difference in performance under similar OLRs would provide data for the performance evaluation of all reactors.

For the duration of the start-up period, which is defined as the period that is required for the system to start producing biogas, all reactors were fed manually at variable organic loading rates that did not always influence the system operation positively. This was a result of inexperience but was also due to the intention to record process parameter data during variable operating conditions. In this way the database that would be utilised to form the alkalinity inference system would be as complete as possible by incorporating extreme operating condition data. This involved operation at really low loading rates (<1.5 g VS/l/d), at high loading rates (>3.5 g VS/l/d) and rapid changes in the feeding regime aiming to calibrate the fuzzy soft sensor as accurately as possible.

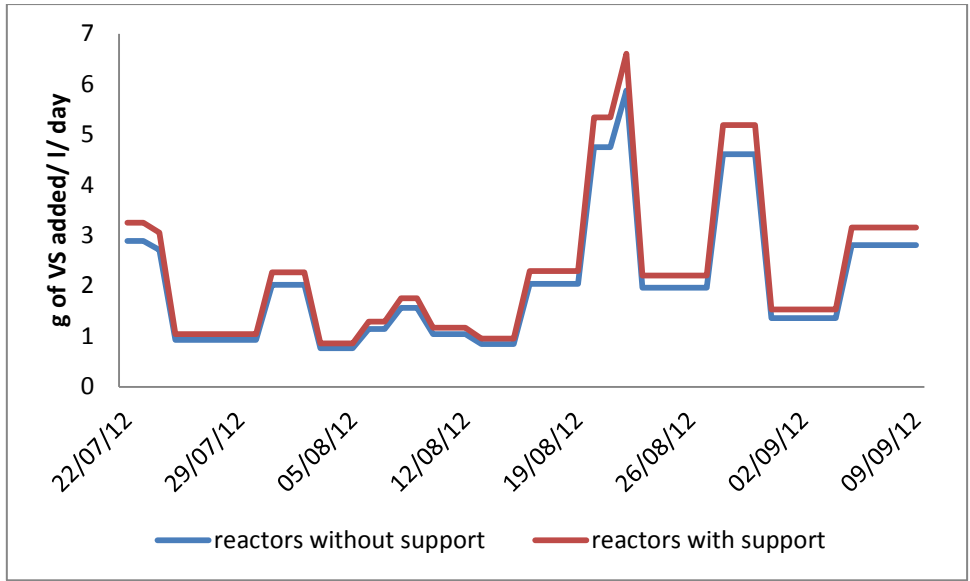
During start-up, AD systems should be operated under low loading rates so as to form a proper microbial community. Failure could result in (Angelidaki et al. 2006) having extended acclimatisation periods (Wu et al. 2001) and poor organic matter removal (Griffin et al. 1998). High OLRs during the first two weeks of operation hindered the stabilisation of the reactors and resulted in reducing the feeding rate to an average of 3 days a week. Despite the fact that OLRs were more than 3 g VS/l/d and the reactors were expected to become quite acidic, the pH remained at quite high levels 7.39- 8.14 in all reactors. However, gas production volumes and gas composition values were very low.

OLRs were reduced during the following three weeks to boost system stabilisation and microbial growth and were set to higher levels the week after 20/08/2012 (week six) in an effort to increase biogas production. However, drops in pH values resulted in adjusting the OLR from 5.8 g VS/l/d to 1.96 g VS/l/d for reactors without support media and from 6.6 g VS/l/d to 2.2 g VS/l/d for reactors with support media to avoid acidification. Further increases in loading rates to 4.6 g VS/l/d and 5.18 g VS/l/d for reactors with, and without, support media respectively, drove

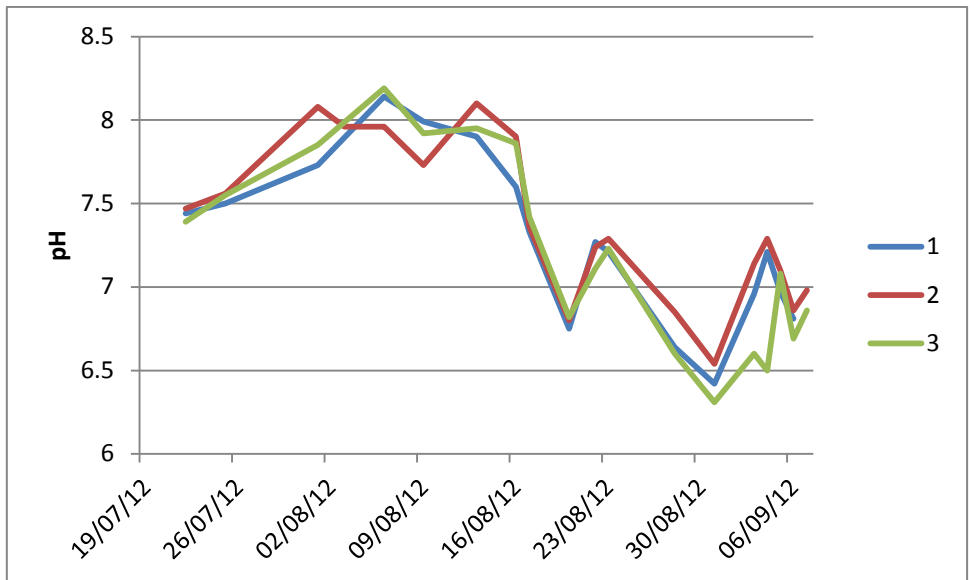
the system towards acidification and OLRs were reduced on 31/08/2012 (week 8) to force pH values to go higher than 6.9. Figure 4.5 depicts the average loading rates for each reactor during the start-up period.

Extreme OLR variations resulted in having massive changes in pH. The OLR was doubled on 16/08/2012 (week five) in all reactors to 2 g VS/l/d and pH dropped by 0.5 within four days. OLR was further increased to 5.87 g VS/l/d for reactors without support and to 6.6 g VS/l/d for reactors with support media on the 22/08/2012 (week six) to examine the systems' reaction to even higher OLR that would help identify the maximum loading rate that the reactors could tolerate. As a result, pH went down by approximately 0.5 in all reactors. The pH trend indicated that OLR should be decreased dramatically to enable all reactors to recover. However, OLRs were further increased during week seven to 4.6 g VS/l/d and 5.18 g VS/l/d for vessels 1, 2 and 3 and vessels 4, 5 and 6 respectively. This loading boost aimed to drive the reactors to acidification and see how they recover followed a week of low feeding rates of approximately 1.5 g VS/l/d. Figure 4.6 and Figure 4.7 depict the pH evolution of reactors without and with support media.

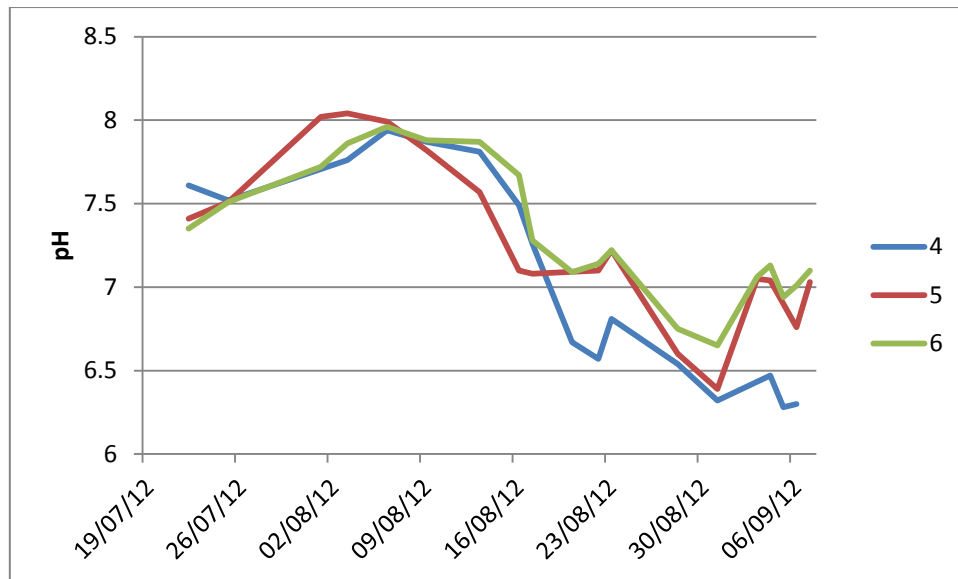
The evaluation of the data collected from the operation of the six reactors, especially during the periods that followed overloading incidents, proved very useful especially when it comes to future variations of the organic loading rate. It was observed that reactors without support media were becoming unstable when the loading rates exceeded 3-3.5 g VS/l/d. Reactors 4,5 and 6 were able to cope with higher feeding rates due to the biomass support media installation ranging between 4- 5.5 g VS/l/d.



**Figure 4.5** Average OLR (g VS added/ l/ day) for reactors with and without biomass support media



**Figure 4.6** Start-up pH values for reactors without biomass support media (Reactors 1-3).



**Figure 4.7** Start-up pH values for reactors with biomass support media (Reactors 4-6).

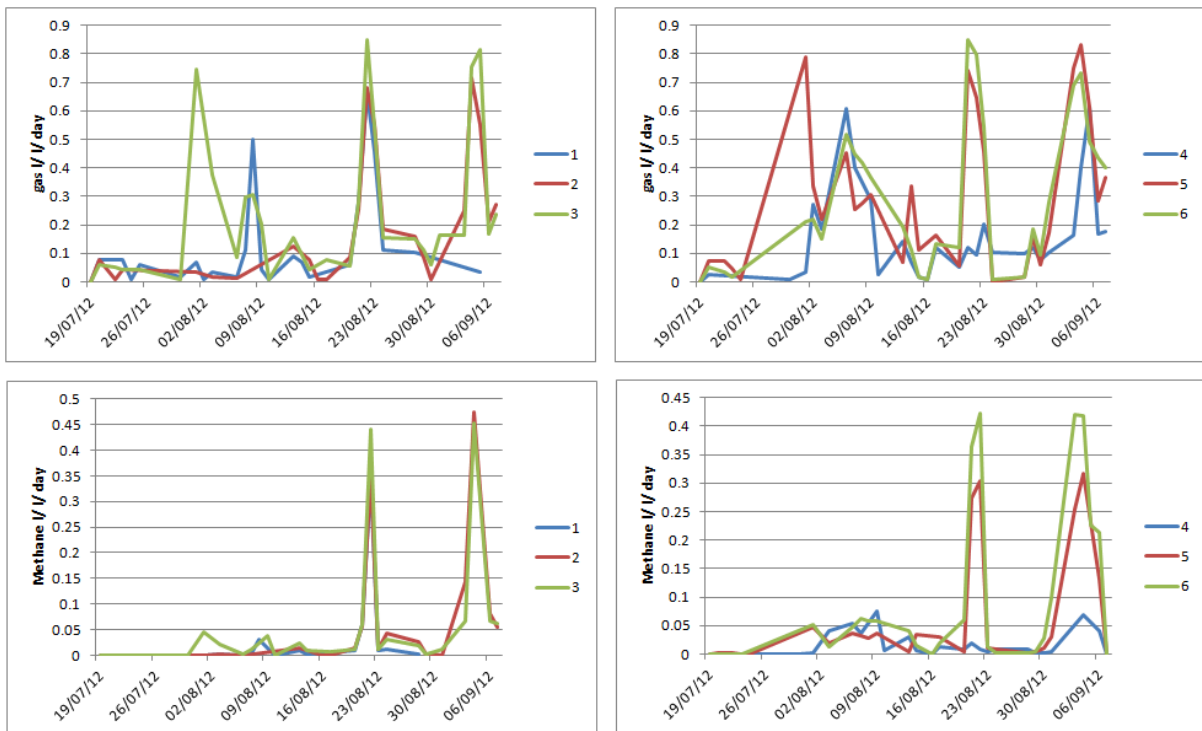
Since the reactors were initially operated under relatively high loading rates, two weeks were approximately required for each vessel to start producing gas. Gas and methane production levels were relatively low and varied according to the loading rate. However, since the reactors were still in the start-up phase, changes in the OLR heavily influenced pH and consequently alkalinity values. So, although biogas production reached 0.8 l/d for reactor 3, 5 and 6 on certain occasions (Figure 4.8), OLR decreases aiming to avoid system destabilisation resulted in reducing the amount of biogas produced to 0.2 l/d.

Reactor 1 did not perform well in terms of biogas production compared to reactors 2 and 3 although these reactors were supposed to behave in a similar way. This was probably due to the fact the vessel had to be serviced at regular intervals for gas leakages which is validated by the fact that pH and alkalinity values between tanks 1-3 were quite similar.

Although reactors 4, 5 and 6 were operated under the same OLRs, pH and gas production values of reactor 4 were lower than the values that characterised the operation of reactors 5 and 6 especially between 21/08/2012 and 07/09/2012 (weeks six and seven). Vessels 5 and 6 produced the most amount of gas during the start-up phase followed by reactor 3. The average biogas and methane production values during start-up are presented at Table 4.2.

	Reactor 1	Reactor 2	Reactor 3	Reactor 4	Reactor 5	Reactor 6
Average Biogas Production (l/l/d)	0.12	0.18	0.23	0.16	0.29	0.28
Average CH <sub>4</sub> Production (l/l/d)	0.024	0.055	0.049	0.019	0.063	0.101

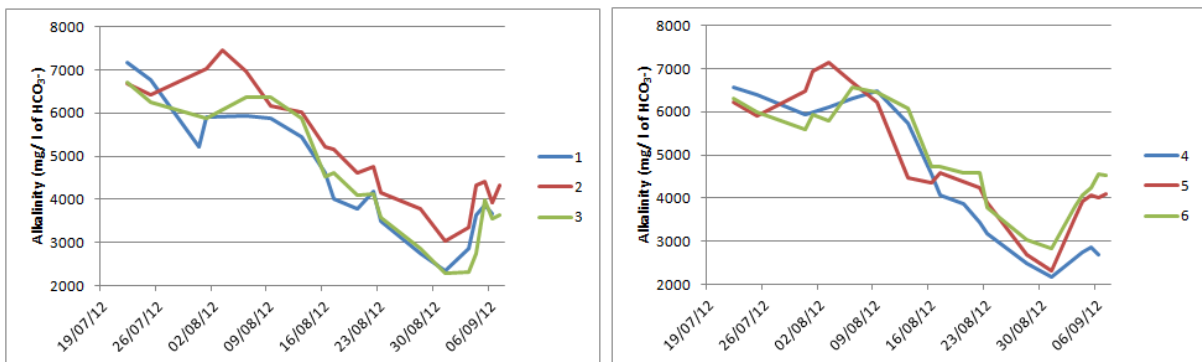
**Table 4.2** Average biogas and methane production during start-up for reactors with (Reactors 4-6) and without biomass support media (Reactors 1-3).



**Figure 4.8** Gas and methane production during start-up. Top left (gas production for reactors without biomass support media), top right (gas production for reactors with biomass support media), bottom left (CH<sub>4</sub> production for reactors without biomass support media), bottom right (CH<sub>4</sub> production for reactors with biomass support media)

Alkalinity results are depicted in Figure 4.9. Since pH and alkalinity values of reactor 1 are similar to reactors 2 and 3 it can be concluded that the low gas production values of reactor 1 were due to gas leakages. Alkalinity evolution followed

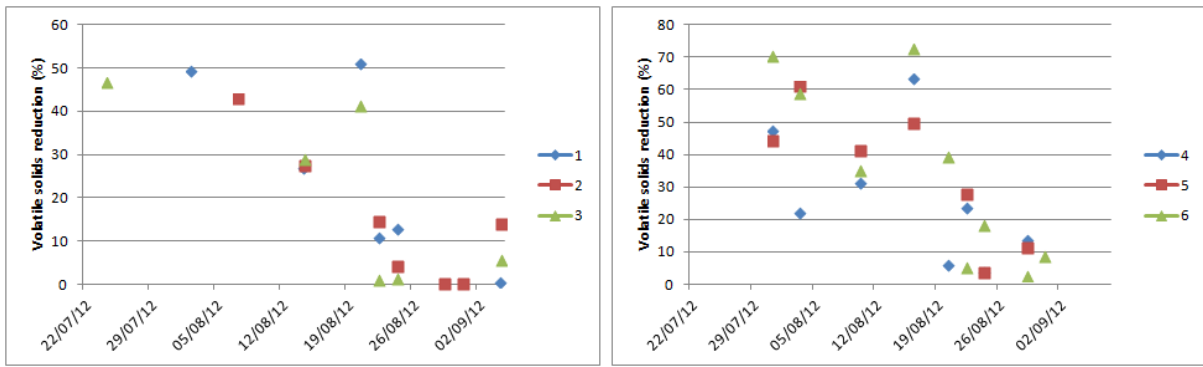
the same trend for all reactors and was due to the loading regime. Overloading resulted in reducing the buffering capacity of the reactors by driving the alkalinity to values close to 2500 mg/l of  $\text{HCO}_3^-$ . On the other hand, corrective operation actions that involved drastic drops in OLR enhanced alkalinity. An alkalinity value between 3490-4000 mg/l of  $\text{HCO}_3^-$  appeared to be, based on the data collected so far, the threshold for maintaining a pH value above 6.9 in reactors 1, 2 and 3 and between 2700-3890 mg/l of  $\text{HCO}_3^-$  for reactors 4, 5 and 6. This suggests that threshold alkalinity concentrations are probably lower for digesters containing support surfaces. Alkalinity concentrations <2700 mg/l of  $\text{HCO}_3^-$  result in having reduced gas production and consistent high loading rate application will lead to gas cessation.



**Figure 4.9** Start-up alkalinity values for reactors with (Reactors 4-6) and without biomass support media (Reactors 1-3).

Volatile solids reduction rates (VSR) for all six reactors are depicted in Figure 4.10. The maximum VSR in reactors 1-3 was around 50% and declined during the periods that followed the overloading of the reactors (15/08/2012 and 28/08/2012). Such low VSR percentages were expected as all the reactors became acidic. Also, VSR calculated shortly after recovery incidents were significantly higher (e.g. on 05/09/2012 for reactors 1, 2 and 3) since anaerobic digestion systems become more stable.

VSR rates for reactors 4-6 were much higher compared to the reactors without support surfaces by reaching 70% and exhibiting higher rates even when the anaerobic environment was becoming unstable. However, low reduction rates for all reactors approximately ten days before the end of the start-up period corresponded to low alkalinity and pH that signalled a process decline.



**Figure 4.10** Volatile solids reduction (VSR) during start-up for reactors with (Reactors 4-6) and without biomass support media (Reactors 1-3).

Reduction rates were expected to stabilise during steady state operation especially for the reactors with support media unlike start-up where overloading incidents ceased gas production and volatile solid destruction.

### 4.3.2 Stable operation

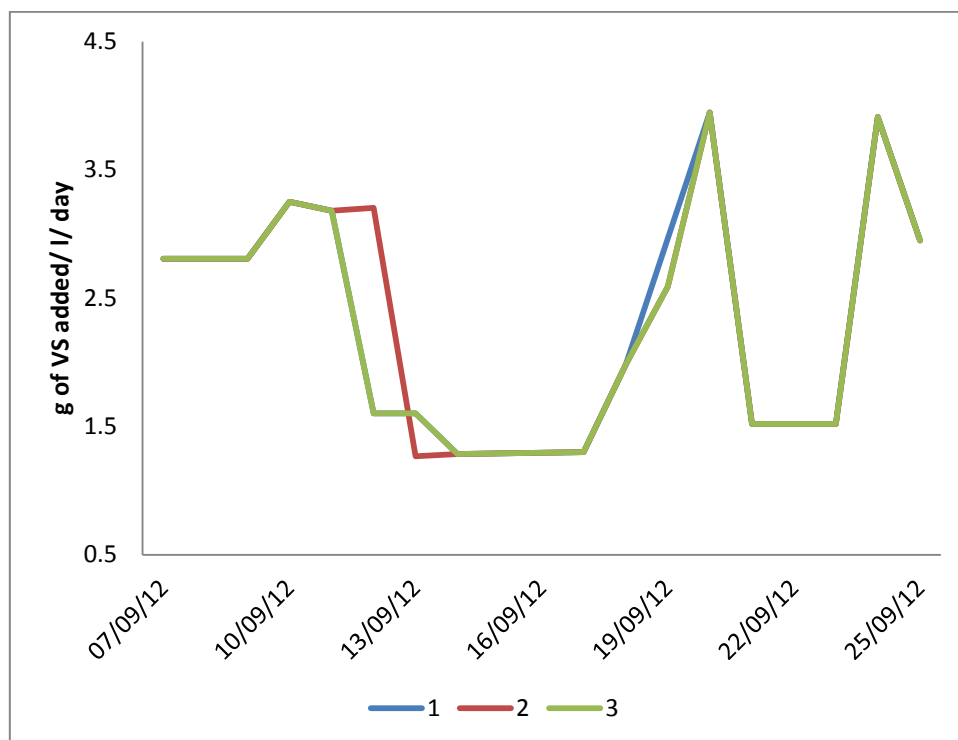
After the process stabilisation in all reactors following the last overloading incident that took place on 30/08/2012, all reactors reached a steady state around 07/09/2012 (week eight). Stabilisation was determined by the fact that pH was above 6.9, biogas production was consistent, methane composition was around 40% and alkalinity values were above the values suggested as the threshold during start-up. After that point the aim was to maximise biogas production by increasing the OLR, yet ensuring that each system was stable and did not become acidic. Unsupported Reactors 1, 2 and 3 were operated for a short period of time (four weeks) since focus was given on the performance of reactors 4, 5 and 6.

#### 4.3.2.1 Reactors 1, 2, 3

Based on the response of the reactors to OLR variations during the start-up phase each reactor was driven by a similar loading rate trying to raise biogas productivity. Vessels 1, 2 and 3 were operated from week 8-18 (06/09/2012-26/09/2012) at loading rates varying from 1-5 g VS/l/d exceeding the limits that were previously suggested (3-3.5 g VS/l/d). This was done to validate the OLR thresholds

and to examine the changes in operating conditions not only between digesters with similar set-up (without support media) but also between digesters with different set-up (with biomass support media).

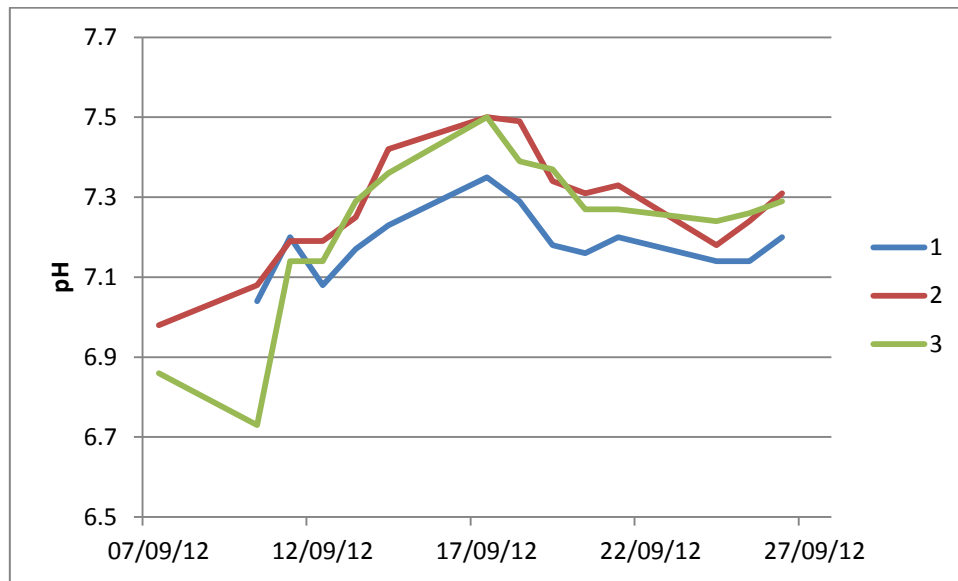
When the reactors recovered, the loading rate was reduced to 1.2 g VS/l/d and was gradually increased to 4.55 g VS/l/d before getting reduced again on the ninth week (21/09/2012) to avoid process destabilisation (Figure 4.11) and record alkalinity fluctuations.



**Figure 4.11** Stable operation OLR for reactors without biomass support media (Reactors 1-3).

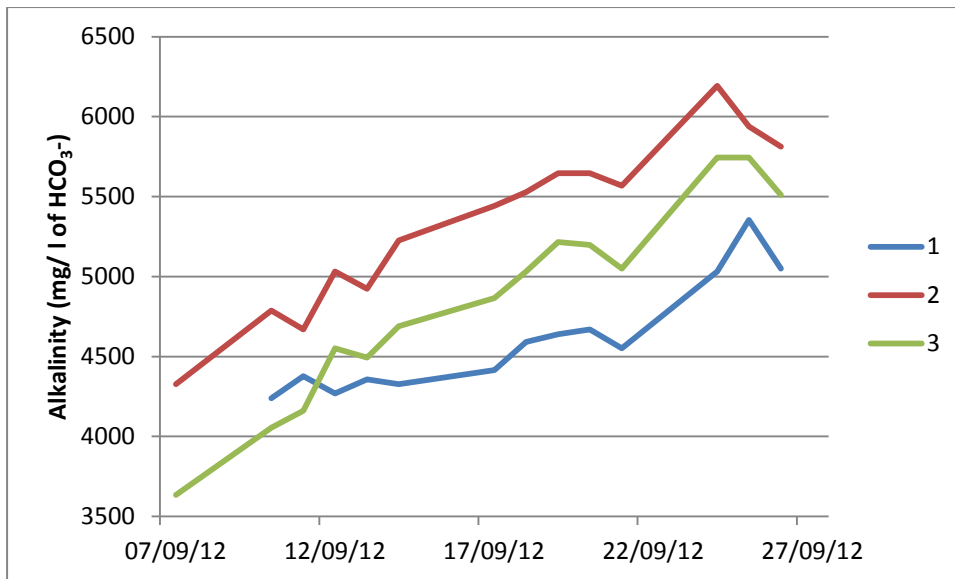
As soon as pH reached the value of 7, OLR was decreased to boost pH values after 13/09/2012 (week eight). OLR was steadily increased to achieve higher biogas generation levels while aiming to maintain pH at stable levels and above 6.9 (Figure 4.12). However, in order to maintain a steady pH inside the three tanks a decrease in OLR was needed after the 21/09/2012 otherwise both alkalinity and pH would be below the thresholds suggested during start-up. This is validated by the fact that both pH and alkalinity started to rise after the decrease in OLR, boosting the buffering capacity of the reactors.





**Figure 4.12** Stable operation pH for reactors without biomass support media (Reactors 1-3).

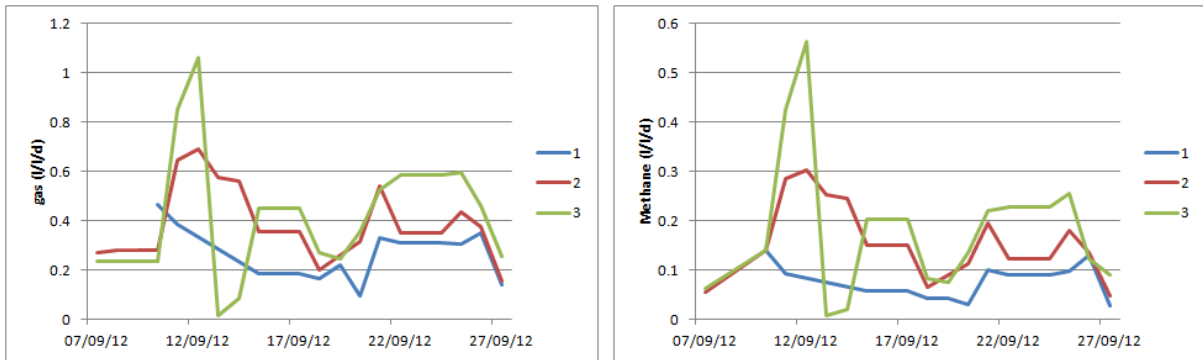
Alkalinity remained within acceptable levels at all times (Figure 4.13). However, when trying to boost biogas production by operating close to the limit of destabilizing the biogas process, alkalinity will always be very close to the detected threshold. Therefore, OLR variations should aim at keeping its value above 3490 which was achieved throughout this three week period. Alkalinity continued to rise through the duration of the stable operation. The alkalinity value dropped on 21/09/2012 (week ten) as a result of increased OLR and was positively influenced again by dropping the loading rate.



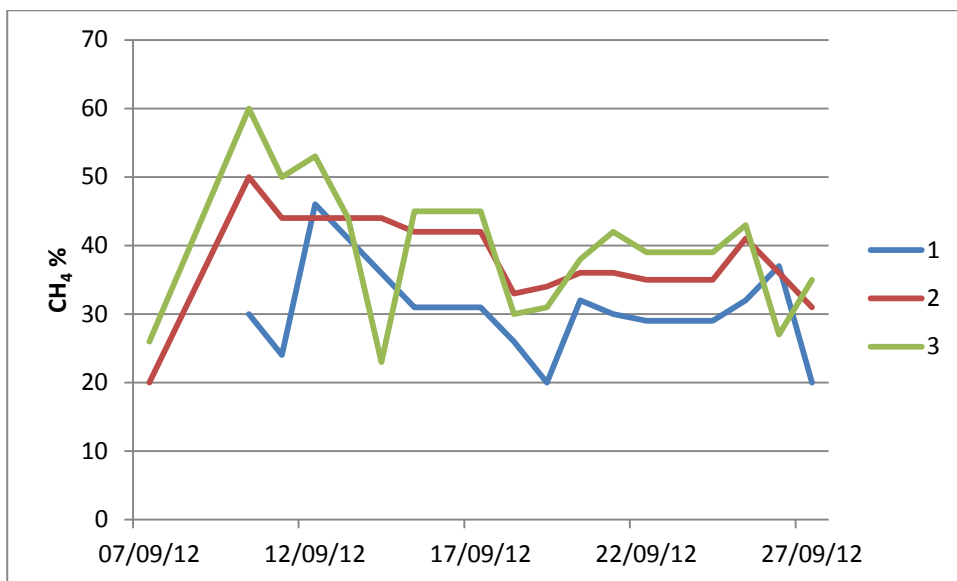
**Figure 4.13** Stable operation alkalinity for reactors without biomass support media (Reactors 1-3).

Gas production and methane production were quite erratic for all reactors (Figure 4.14) during the first ten days. Both of them peaked between 11/09/2012 and 12/09/2012 which resulted from system recovery during start-up. A drop was witnessed during the next day but that was caused by temperature fluctuations due to problems experienced with the heating of the water bath (temperature was around 20°C). This event signifies the importance of maintaining stable temperature as temperature fluctuations lead to a cessation in biogas production. Overheating of the reactors has similar results as witnessed on 18/09/2012 when the temperature reached 64°C and gas production dropped in all reactors.

Tank 1 did not produce much gas although system conditions were similar to the other tanks which might be due to gas leakages. Tank 3 exhibited the best behaviour by having an average daily gas production of 0.447 l/l/d, followed closely by tank 2 with 0.38 l/l/d and tank 1 with 0.26 l/l/d (without taking data from 13/09/2012 and 18/09/2012 into consideration). Also, methane percentages were not very high (especially for tank 1) but remained stable during this period with average values of 30% CH<sub>4</sub>, 40% CH<sub>4</sub> and 40% CH<sub>4</sub> for reactors 1, 2 and 3 respectively (Figure 4.15).



**Figure 4.14** Stable operation average daily gas and methane production for reactors without biomass support media (Reactors 1-3).



**Figure 4.15** Stable operation methane percentage for reactors without biomass support media (Reactors 1-3).

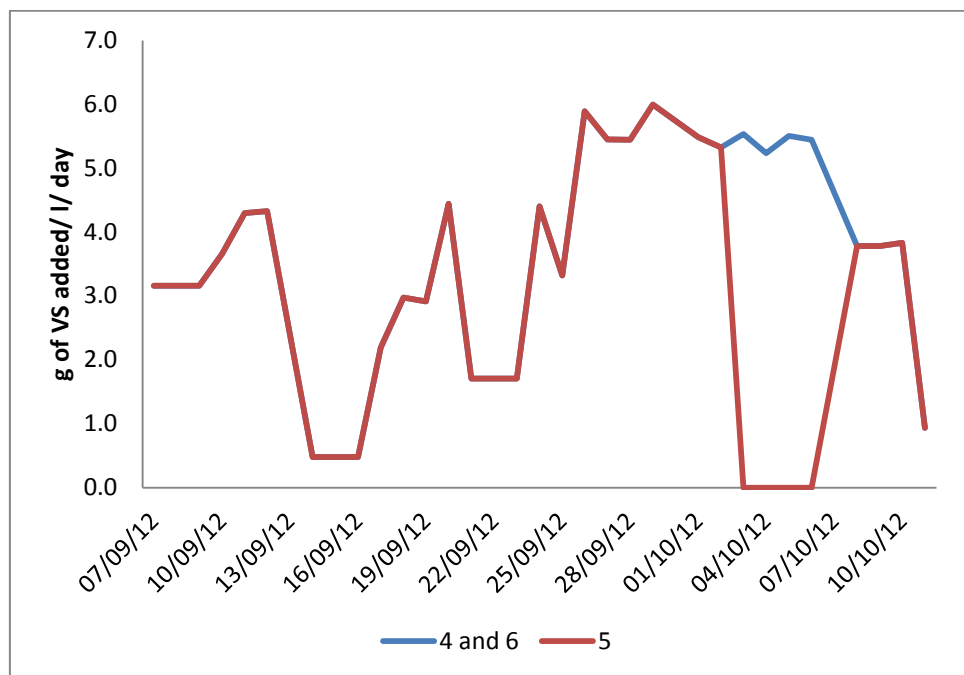
Very few samples were analyzed for %TS and %VS for reactors 1, 2 and 3 during this period (four in total) and three of them were collected during the days that temperature fluctuations affected the reactors. This resulted in having VSR between 3-5 % which might not be representative of the reactors' operation, but certainly justify the cessation of gas production due to temperature fluctuations.

#### **4.3.2.2 Supported Reactors 4, 5, 6**

Following start-up, all reactors reached a stable-state between 07/09/2012-10/09/2012 (week eight) after which severe pH fluctuations were not witnessed.

Process parameter data during start-up suggested that reactors with biomass support media could cope with loading rates up to 4-5.5 g VS/l/d. All vessels were operated with loading rates up to 6 g VS/l/d. High OLRs aimed to push all reactors to function close to the limit where maximum biogas production occurs yet an overloading incident could easily result in system failure.

Stable operation took place between 07/09/2012- 11/10/2012. Identical loading rates were applied to all digesters during this period (Figure 4.16) to assess the differences and similarities involving the utilisation of different support media in small scale digesters. On 03/10/2012 (week eleven) glass reactor 5 broke and although comparable performance conclusions to reactors 4 and 6 could not be drawn until 08/10/09 when the loading process was reinitiated for reactor 5, rapid digester recovery due to the existence of biomass support media provided valuable data and underlined the supplementary role that support media could play in AD systems.

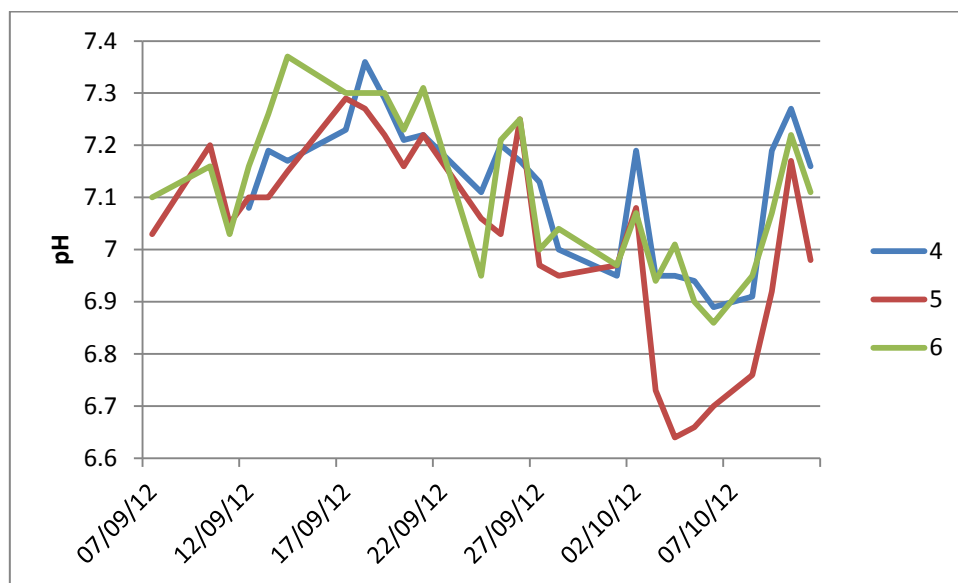


**Figure 4.16** Stable operation OLR for reactors with biomass support media (Reactors 4-6).

After the reactors reached a stable state OLR was increased on 11/09/2012 to 4.3 g VS/l/d trying to force the system to increase biogas production. However, a slight decline in pH values suggested a decrease in loading rates (0.5 g VS/l/d) that were applied over the next four days. Experimentation with the loading rates that

also aimed to capture process parameter data under various OLR ended on 20/09/2012 (week ten). High OLRs started being applied for three weeks trying to examine the systems' reaction under high loading rates (with the exception of reactor 5).

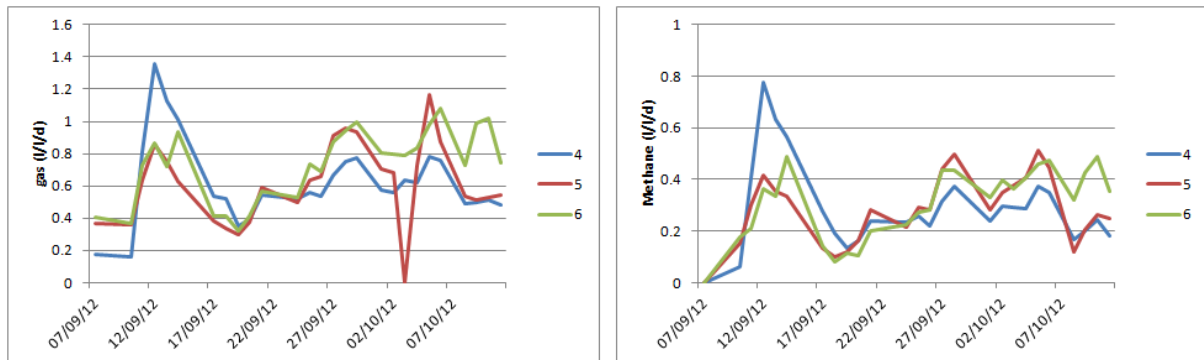
The impact of OLR on the pH in all three vessels was positive (Figure 4.17). pH remained stable through the entire seven week period having values above 6.9. Data dictated that 6.9 was the critical pH value for process stability (values below 6.9 correspond to reactor 5 during the days following the breakage incident). pH values also indicate that reactors with support media can maintain a stable pH. pH levels were lower compared to the values acquired from reactors 1, 2 and 3, however, under higher loading rates than reactors with support media. This implies that the support surfaces provide stability to the reactors and result in having a less sensitive to changes environment more suitable for microbial activities.



**Figure 4.17** Stable operation pH for reactors with biomass support media (Reactors 4-6).

Gas and methane production were much higher compared to reactors 1-3 (Figure 4.18). Their levels remained stable, especially after 24/09/2012 when reactors were operated under higher loading rates, ranging between 0.5-1.1 l/l/d for gas production and 0.2-0.5 l/l/d for methane production. Reactor 6 exhibited the best performance with respect to gas and methane production by delivering an average of 0.73 l/l/d of gas followed closely by reactors 4 and 5 that produced 0.62 l/l/d and 0.61

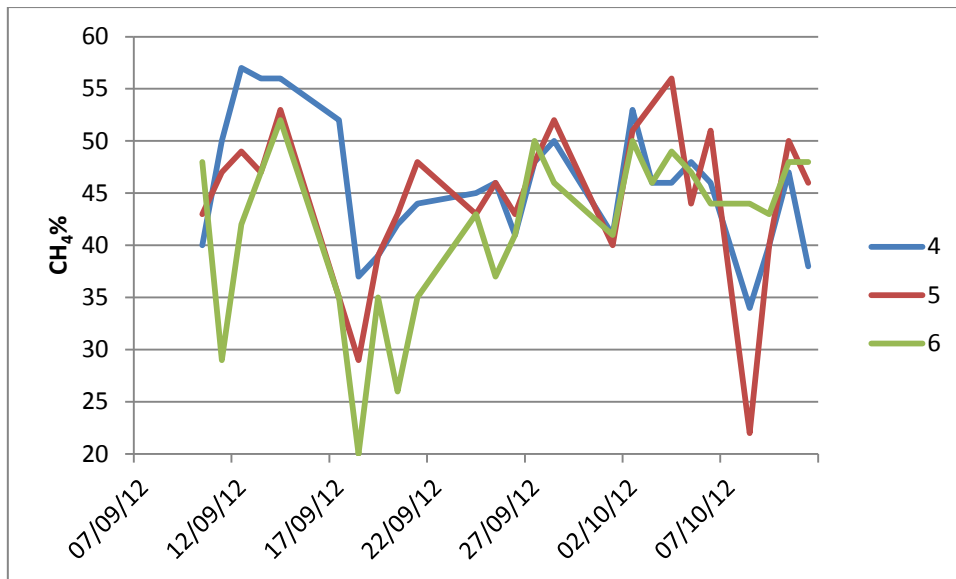
l/d. Methane production was similar with reactor 6 generating an average of 0.3 l/d, reactor 4 generating an average of 0.29 l/d and reactor 5 an average of 0.28 l/d.



**Figure 4.18** Stable operation gas and methane production for reactors with biomass support media (Reactors 4-6).

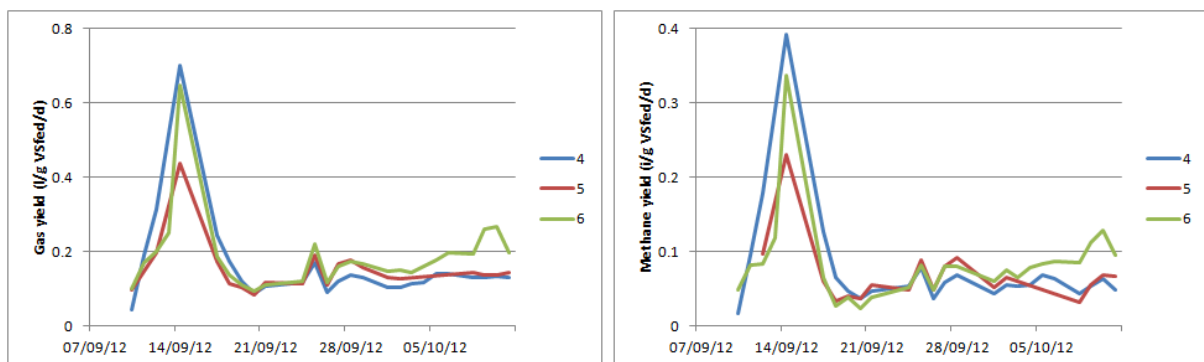
CH<sub>4</sub>% remained stable and at high levels (Figure 4.19) considering that some oxygen intake took place during loading and unloading the reactors. Average methane composition levels were 46%, 44% and 43% for reactors 4, 5 and 6 respectively. Fixed methane composition values around 45% validated the fact that all reactors remained stable throughout this period. The exception was methane percentages with values varying between 20%- 37% that were recorded around the 18/09/2012. Those were a result of the decrease in OLR aiming to maintain pH values above the threshold of 6.9.

Reactor 5 operation was re-initiated on 03/10/2012 (week eleven) and the biomass support media massively assisted in the reactor recovery. The anaerobic digester did not stop producing gas despite the fact that the working volume not attached to the support media was lost. This suggests that biomass media could help anaerobic digesters by minimizing recovery periods. In the case of reactor 5, although a week was needed for pH to climb beyond 6.9, gas production was quite consistent despite the fact the system was still recovering.



**Figure 4.19** Stable operation methane percentage for reactors with biomass support media (Reactors 4-6).

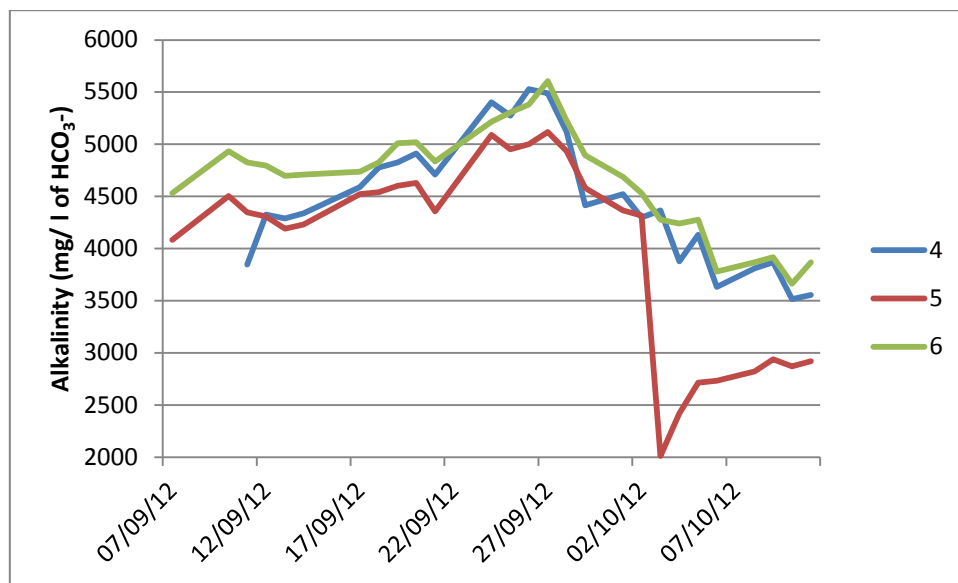
Gas and methane yield remained stable from week nine to week eighteen following the pattern of gas and methane production despite the minor variations applied in the loading rate (Figure 4.20).



**Figure 4.20** Stable operation gas and methane yield for reactors with biomass support media (Reactors 4-6).

Alkalinity values remained between 3500 mg/l of  $\text{HCO}_3^-$  and 5600 mg/l of  $\text{HCO}_3^-$  during the stable operation period (Figure 4.21). Values below the aforementioned threshold that characterised the material inside reactor 5 are due to process re-start and the positive trend of alkalinity values after 03/10/2012 signalled the beginning of a recovery period. OLR declines influenced the buffering capacity of the system (13/09/2012) and system operation at increased loading rates drove

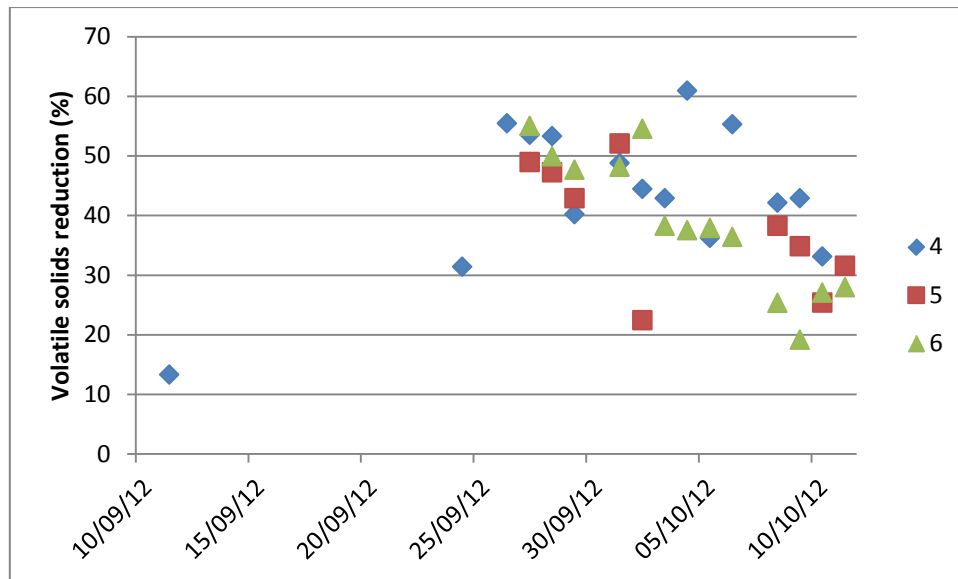
alkalinity close to 3500 mg/l of  $\text{HCO}_3^-$ . Massive alkalinity drops were recorded after 24/09/2012 (week 10) which was when loading rates peaked at 6 g VS/l/d indicating that the system would fail if loading rates were not reduced. Alkalinity was stabilised slightly above the minimum measured values for reactors 4 and 6 at loading rates of 3.8 g VS/l/d. This suggests that OLR should definitely not exceed a value of 5.5 g VS/l/d and should ideally be close to 4 g VS/l/d to achieve maximisation of biogas production and maintain a stable system at the same time.



**Figure 4.21** Stable operation alkalinity for reactors with biomass support media (Reactors 4-6).

Volatile solids reduction (Figure 4.22) ranged between 20%-60 % after 25/09/2012 (week ten). Higher VSR reduction percentages were recorded when OLRs were around 5.5 g VS/l/d and at the same time biogas production was maximised and remained stable. Especially between 26/09/2012 and 02/10/2012 VSR in all reactors was between 40%-56%. However, to avoid process instability OLRs were lowered and as a result reduction rates declined. Although a reduction of above 40% continued to exist in reactor 4, the percentages in reactor 6 dropped below 30% while the rates in reactor 5 that was still recovering were similar.





**Figure 4.22** Stable operation volatile solids reduction percentage for reactors with biomass support media (Reactors 4-6).

### 4.3.3 Under loading, failure and FIS evaluation

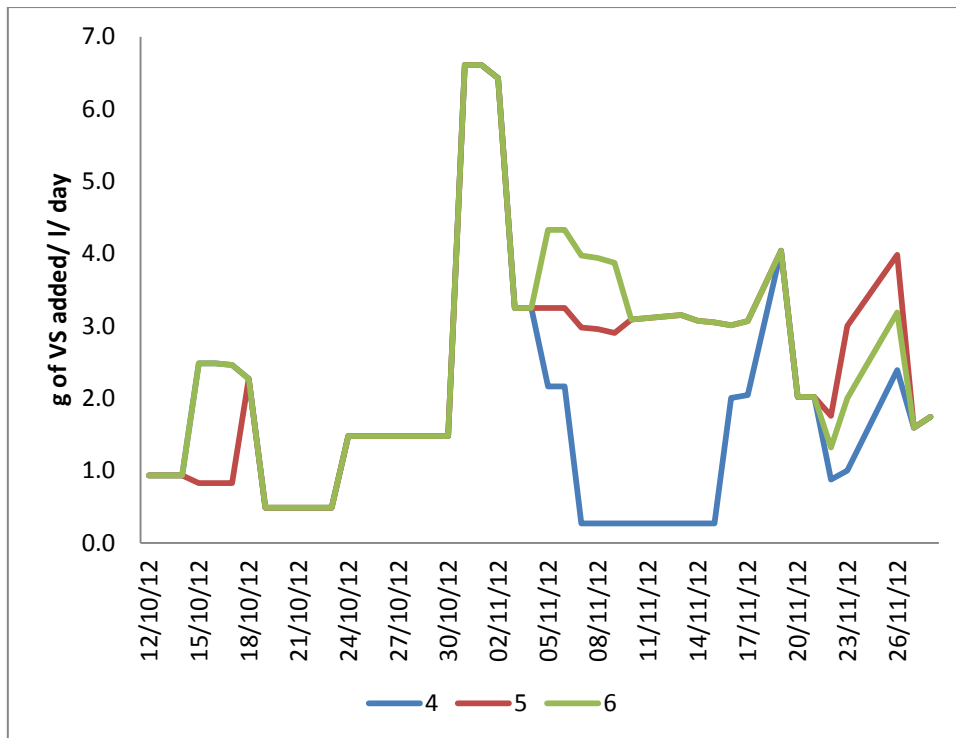
Following the stable operation period each reactor was run at different loading rates in an effort to capture each system's behaviour to severe OLR fluctuations that included not feeding the reactors on a daily basis. This resulted in having low pH values (<6.9), alkalinity values below the suggested threshold of 3500 mg/l of  $\text{HCO}_3^-$  and reduced gas and methane production. Recording process parameter data after periods of keeping OLRs to a minimum and then suddenly boosting loading rates would provide useful data regarding process parameter evolution.

FISs that predicted alkalinity (to be presented in 4.4.5) that were designed with data from the start-up and stable operation period would be validated against these newly available data. Previous experiments showed that maximum gas production occurs when the reactors operated close to the limit of becoming unstable. The predicted alkalinity values of the fuzzy models proposed should follow real alkalinity values closely especially between the range of 2500 mg/l of  $\text{HCO}_3^-$  and 4000 mg/l of  $\text{HCO}_3^-$ . Therefore, it was necessary to conduct experiments where reactors 4, 5 and 6 would also exhibit a poor behaviour by keeping pH, alkalinity and gas production at low levels.

Experiments with severe loading rate fluctuations initiated on 11/10/2012 and lasted until 28/11/2012 (weeks nineteen to twenty one). Part of those experiments included studies of how  $\text{NaHCO}_3$  addition, that aims to boost pH and alkalinity values, and water dilution influence gas production. Also, apart from investigating the effects of buffer addition to the system, it was intended to examine whether the proposed fuzzy inference mechanism would be able to predict alkalinity values accurately by introducing some form of disturbance into the system.

The loading rate varied from 0-6.6 g VS/l/d for all reactors. OLRs that did not exceed 2.5 g VS/l/d were applied on average until 30/10/12. Then a shock load of 6.6 g VS/l/d was deliberately applied in all reactors (31/10/2012- 02/11/2012) causing system failure. Reactor 4 broke shortly after (07/11/2012) and was replaced. This explains the absence of loading until 16/11/2012.

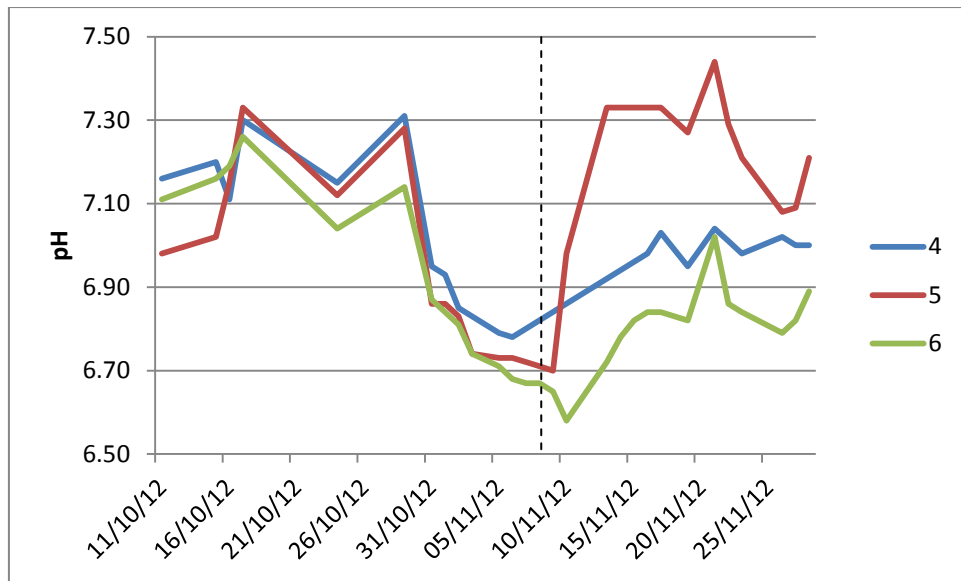
3 g/l of buffer ( $\text{NaHCO}_3$ ) were added to reactor 5 (09/11/2012 and 10/11/2012) and 1.6 l of substrate was replaced with fresh water in reactor 4 (09/11/2012). Buffer addition was chosen because it increases the biodegradability of the organic fraction of solid waste, biogas productivity and VSR (Abdulkarim & Evuti 2010). Water dilution combined with addition of fresh cow slurry was selected for tank 6 for practical reasons. Because although water dilution is not the best strategy to help in digester recovery, fresh manure could not be inserted inside the reactors due to mixing restrictions and the small diameter of the inlet hole. The results presented (Palatsi et al. 2009) where seven recovery strategies were tested against long chain fatty acid inhibition in manure thermophilic digestion indicated that feeding cessation was proved to be the most poor approach whereas reactor dilution with inoculum was proven to be the most process influential approach. Water dilution accompanied by the addition of reactor effluent and manure also influences the recovery speed of ammonia-inhibited thermophilic anaerobic digesters (Nielsen & Angelidaki 2008). However, ammonia inhibition usually takes place at higher pH values (Sung & Liu 2003b) than the ones existing inside tanks 4, 5 and 6 at that point. Average loading rates and pH values are depicted in Figure 4.30 and Figure 4.31 respectively.



**Figure 4.23** Under loading, failure and FIS evaluation OLR for reactors with biomass support media (Reactors 4-6).

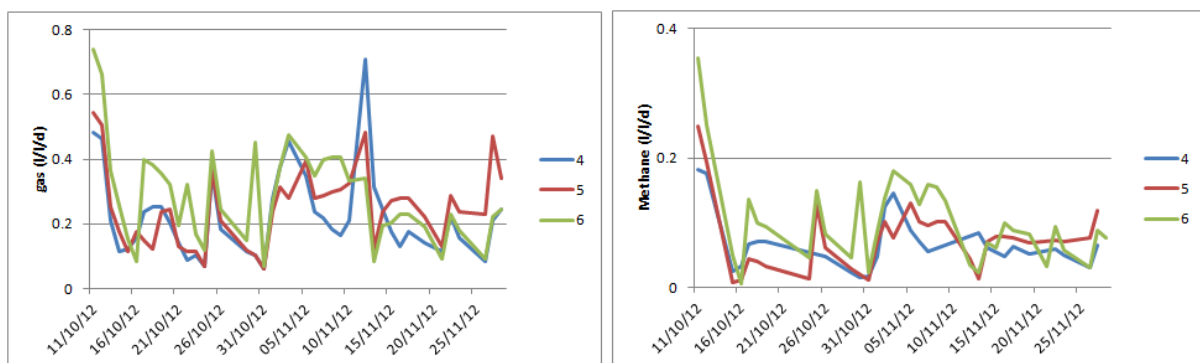
pH values ranged, as intended, from 6.6-7.45, reaching the highest value following the buffer addition. The maximum loading rate applied forced pH to drop around 6.7 on 08/11/2012 before going up again while all reactors were recovering. pH variations covering a big range around the desired operating point where maximum biogas production occurred during steady state conditions (6.9-7.1) would provide a good validation set.

Water dilution did not have an obvious impact on reactor 6 pH since lowering the loading rate would result in lifting pH values which is believed to be the reason for the pH trend after 09/11/2012. On the other hand,  $\text{NaHCO}_3$  addition rocketed pH values in reactor 5 from 6.7 to 7.3 in three days as expected. pH remained at higher levels and slightly oscillated according to the OLRs applied.

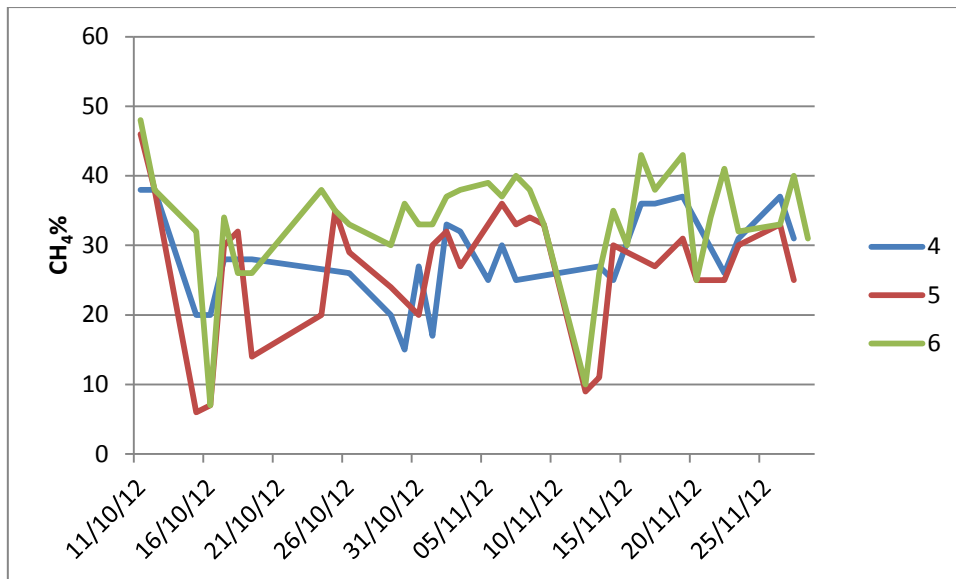


**Figure 4.24** Under loading, failure and FIS evaluation pH for reactors with biomass support media (Reactors 4-6).

Gas and methane production were low compared to the volumes that the reactors were able to produce during stable operation. Their levels dropped to half (Figure 4.32) indicating process imbalance. The addition of  $\text{NaHCO}_3$  in reactor 5 and the water dilution accompanied by slurry feeding in reactor 6 were not enough to boost gas production even three weeks after their application. Reactor performance is also reflected by the average methane percentages of 28%, 27% and 34% for reactors 4-6 respectively. Methane percentages are available in Figure 4.33.

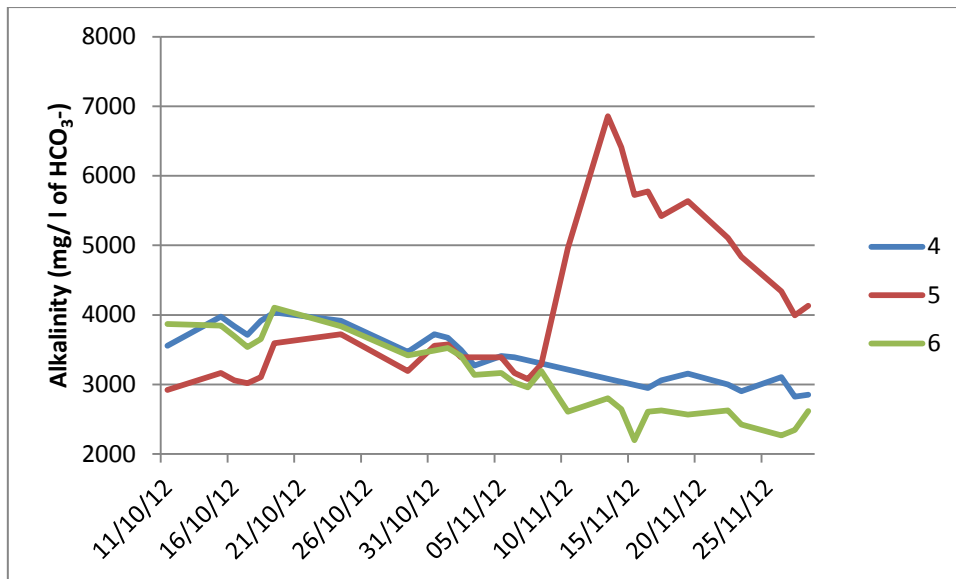


**Figure 4.25** Under loading, failure and FIS evaluation gas and methane production for reactors with biomass support media (Reactors 4-6).



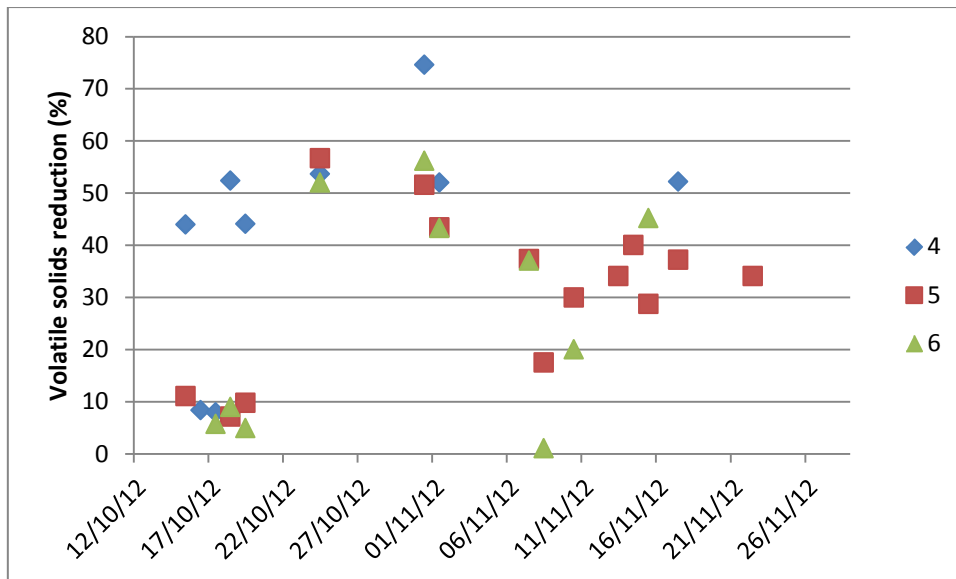
**Figure 4.26** Under loading, failure and FIS evaluation methane percentage for reactors with biomass support media (Reactors 4-6).

Alkalinity for reactors 4 and 6 was close to the border of indicating process imbalance and reactor 5 values were below 3500 mg/l of HCO<sub>3</sub><sup>-</sup> since it was still recovering from the accident that took place a week before 11/10/2012 (Figure 4.34). The sudden boost in the feeding regime drove alkalinity to levels around 3000 mg/l of HCO<sub>3</sub><sup>-</sup> signifying system failure. Water dilution with fresh slurry did not force alkalinity to reach higher levels but on the contrary, alkalinity remained around 2600 mg/l of HCO<sub>3</sub><sup>-</sup> demonstrating that dilution on its own is not suitable to reinstate digester stability at loading rates between 1.5-3 g VS/l/d. On the other hand, buffering addition rocketed alkalinity similarly to pH from 3300 mg/l of HCO<sub>3</sub><sup>-</sup> to 6860 mg/l of HCO<sub>3</sub><sup>-</sup>.



**Figure 4.27** Under loading, failure and FIS evaluation alkalinity for reactors with biomass support media (Reactors 4-6).

Volatile solids reduction rates were quite high for reactor 4 ranging between 40%- 75% with the exception of the rates for 16/10/2012-17/10/2012 where the low percentages are probably due to the loading rate adjustment from 0.27 g VS/l/d to 2 g VS/l/d that eventually resulted in boosting VSR rates even further. Reactor 5 destruction rates were slightly lower with a median value of 34% which is characteristic of the percentages existing after the addition of NaHCO<sub>3</sub> to the system. Reactor 6 rates had the same trend with those of reactor 5 resulting from the similarities in the feeding regime.



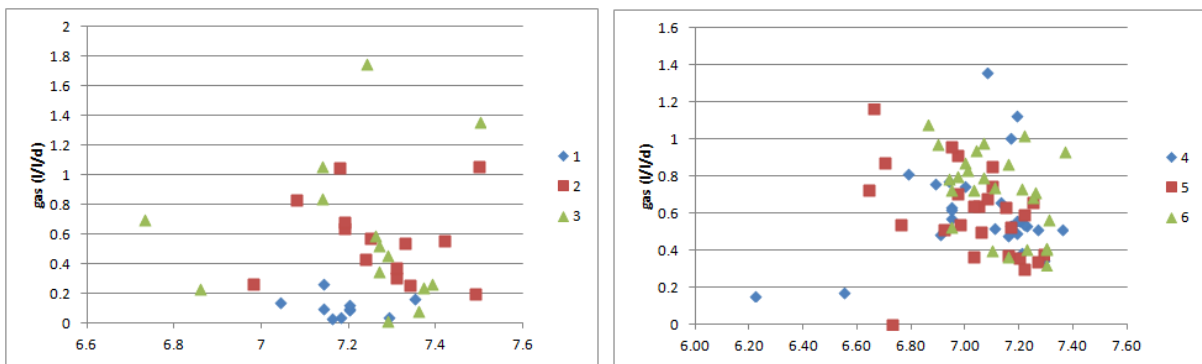
**Figure 4.28** Under loading, failure and FIS evaluation volatile solids reduction percentage for reactors with biomass support media (Reactors 4-6).

#### 4.3.4 Performance comparison of reactors 1-6 and identification of optimum process parameter values.

Reactors 1, 2 and 3 were able to withstand maximum loading rates between 3- 3.5 g VS//d and higher OLRs caused system imbalance. Vessels with biomass support surfaces were operated at higher loading rates and could produce higher amounts of gas when the loading rate varied between 4- 5 g VS//d. During the aforementioned experiments a vast range of feeding regimes was applied and tested in all reactors and although systems could operate after being supplied with higher amounts of cow slurry their stability declined. Also, despite the fact that reactors 4, 5 and 6 worked with different types of support surfaces the upper OLR limit was similar for all of them.

pH fluctuated in all vessels during extreme loading rate alterations especially when the aim was either to avoid reactor destabilisation or deliberately enforcing the digesters to fail. However, during start-up operations when the reactors were overloaded, pH declined fast in reactors 1, 2 and 3 whereas a gradual reduction was recorded in vessels 4, 5 and 6. This means that systems with support surfaces are more stable to OLR fluctuations and pH variations. pH variations are indicative of process stability and can help in applying corrective loading rate actions before AD systems reach a point where recovery will require a longer time to be completed.

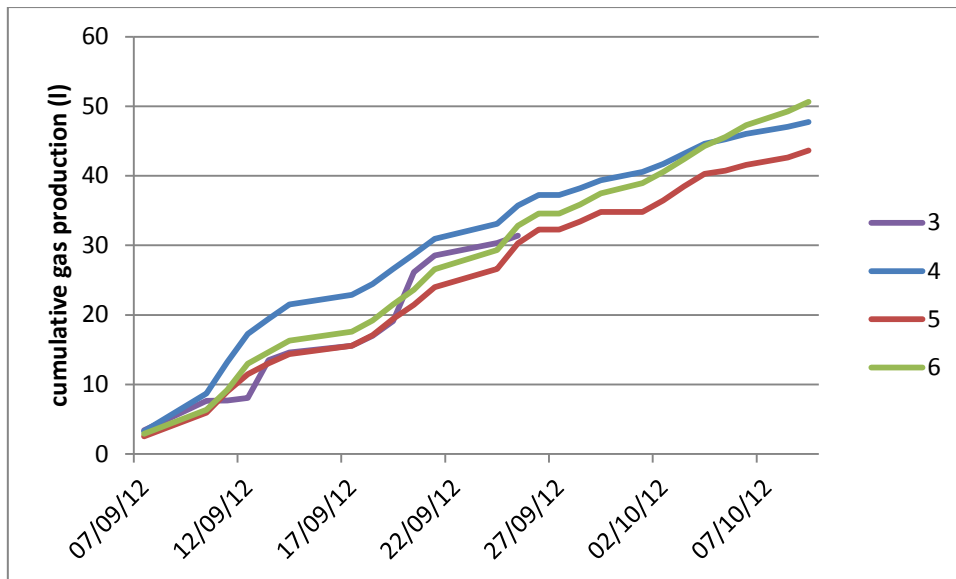
During stable operation pH was kept at steady levels for all reactors by fluctuating between 7-7.5 and 6.9-7.35 for reactors 1, 2, 3 and 4, 5, 6 respectively. Consequently, pH levels where maximisation of biogas production occurred were different for reactors with and without support surfaces. As depicted in Figure 4.29, the optimum pH ranges were 7.1-7.3 and 6.9-7.2 for reactors 1-3 and 4-6 respectively and were identified from data collected during stable state (Reactor 1 values appear to be low due to gas leakages). pH <6.9 indicated process imbalance in all vessels and suggested the application of a reduced loading rate aiming to increase the buffering capacity of the digester.



**Figure 4.29** Optimum pH for all reactors

Gas production was not much higher for reactors with support media throughout the duration of all experiments. From the reactors without support surfaces reactor 3 exhibited the best performance followed by reactors 1 and 2 that had slightly lower cumulative production values. Sponge appeared to be able to provide a more suitable environment for the growth of methanogens compared to the two types of reticulated foam inserted in the other two reactors. Reactor 6 was the most productive of the systems with support surfaces. This leads to the conclusion that biomass media do not have a huge impact in enhancing biogas production. Stability is what they mostly offer to AD systems. Cumulative gas production data during stable operation are depicted in Figure 4.30.





**Figure 4.30** Stable operation cumulative gas production for reactors 3, 4, 5 and 6

Stability was guaranteed in all reactors for alkalinity values above 3500 mg/l of  $\text{HCO}_3^-$ . Biogas productivity was maximised when alkalinity was close to the threshold that signified system failure. This value was different for reactors 1-3 and 4-6. Reactors without support media had increased biogas yields when alkalinity was approximately above 3500 mg/l of  $\text{HCO}_3^-$  and below 4500 mg/l of  $\text{HCO}_3^-$ . However, alkalinity levels that boosted methane yield for reactors 4-6 were slightly lower varying between 3480- 4300 mg/l of  $\text{HCO}_3^-$ . These rates suggested that loading rates should focus on keeping alkalinity close to the limit where the system is prone to become unstable for maximisation of biogas production. On the other hand, maintaining higher levels would limit reactor productivity but provide a stable environment.

#### 4.3.5 Software sensor development based on Fuzzy Logic

##### 4.3.5.1 Introduction

As mentioned in 1.3, common process monitoring indicators include pH and gas production rate (Hawkes 1993)(Ahring & Angelidaki 1997)(Boe et al. 2010). A survey of 400 full-scale AD plants worldwide, installed by Biomethane, concluded

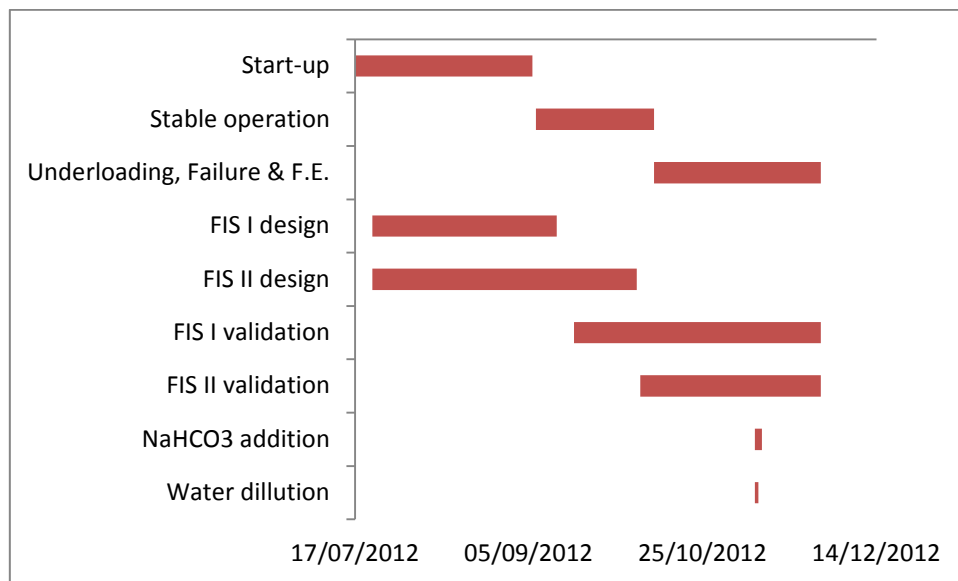
that in 95% of the plants, pH and biogas flow in-line meters were installed (Spanjers & Lier 2006). Biogas production rate has been proposed as a means to measure stability in methanogenic treatment plants (Steyer et al. 2006), and along with pH and the difference between the actual biogas flow and its set point have been identified as the most realistic variables for monitoring AD processes efficiently at high load (Liu et al. 2004b). pH, gas production rate and hydrogen content have been used to calculate the variation of the feed rate (Ehlinger et al. 1994)(Moletta et al. 1994) as cited in (Liu et al. 2004b). Similarly, the feed rate was controlled only by gas flow rate and pH (Estaben et al. 1997)(Steyer et al. 1999) since these sensors could be easily used in industrial applications and were characterised as cost-effective. Additionally, it was proven that the aforementioned sensors were sufficient for control. Therefore, methane composition was not part of the control algorithm.

The selection of the input parameter set was made based on the ability of pH and gas production rate to effectively determine the stability status of AD systems. Alkalinity, which is a stability indicator, is closely related to pH (1.2.7)(1.3.4)(3.1) and consequently the gas flow rate (as mentioned above). Additionally, pH and gas flow can be measured by low maintenance sensors suitable for the development of a cost effective software tool for process optimization through stability.

Data collected during the experiments conducted with reactors 4, 5 and 6 were used to design at first a fuzzy inference system that would predict alkalinity levels based on pH, daily gas volume, daily difference in pH and daily difference in gas volume. Next, a second FL system was designed that had pH, daily gas volume/reactor volume, daily difference in pH and daily difference in gas volume/reactor volume. The change in input selection was performed to make the software sensor applicable in different reactor setups (e.g. different shape reactors, higher capacity). The two fuzzy inference systems that were developed were applied in the reactors containing biomass support media.

The first fuzzy system was designed during the early stages of the experimentation process aiming to prove that data-based models can be improved with the addition of more data. More specifically, the first FIS (FIS I) was designed based on data collected during the start-up phase and during the first ten days of the stable operation period (22/07/2012- 13/09/2012). The second FL system (FIS II) was constructed on 06/10/2012 towards the end of the stable operation period. FIS II implementation aimed not only to prove that a model trained with more data would

exhibit a better performance, but also to check the model suitability through validation with newly available data. Validation data included those that were collected throughout the further experimentation period where loading rates were deliberately varied, buffering agent ( $\text{NaHCO}_3$ ) was added inside reactor 5, and water dilution was performed in reactor 6. In this way, fuzzy model performance could be evaluated against disturbances. Data utilised for each design are depicted in Figure 4.31.



**Figure 4.31** Data used in fuzzy logic design and validation (F.E. corresponds to FIS evaluation)

First order fuzzy inference systems based on subtractive clustering were developed based on the method presented in Chapter 3 (Figure 3.2). Approximately 75% of each database constituted the training set and 25% of the data formed each checking set. Since random categorisation of training and checking sets resulted in having overdefined fuzzy models, the selection of each set was conducted similarly to Chapter 3 by following a time sequential pattern in order to get clear data representation of all input values.

#### 4.3.5.2 FIS I design

FIS I was developed based on data from reactors 4 and 5 and was initially validated from data originating from reactor 6 until 13/09/2012. This selection was made because the proposed model was intended to be able to determine alkalinity in all reactors. It was ensured that minimum and maximum input values were part of the training set. Least squares estimation was used to determine the consequent functions of the TSK FIS. Premise parameters and structure, consequent parameters and structure were set and tuned in a recursive manner and the consequent functions were linear.

Cluster radius was varied from 0.15 to 1 with a step of 0.01. The squash factor was set to 1.25, the reject ratio to 0.15 and the accept ratio to 0.5. The cluster radius of the FIS that provided the best statistical indices during the evaluation process was 0.15. Twenty-nine cluster centers were identified using subtractive clustering (Table 4.3) and resulted in having the same amount of membership functions for every input (Figure 4.32) and the same number of rules that regulated system output. The third rule is characteristic of all the rules and is of the following form:

If  $pH_{in}$  is  $pH_{in}MF3$  and Gas volume<sub>in</sub> is Gas volume<sub>in</sub>MF3 and Daily pH difference<sub>in</sub> is Daily pH difference<sub>in</sub>MF3 and Daily gas volume difference<sub>in</sub> is Daily gas volume difference<sub>in</sub>MF3

Then Alkalinity= $699.9 \cdot pH - 149.8 \cdot \text{Gas volume} + 0 \cdot \text{Daily difference in pH} - 9.568 \cdot \text{Daily difference in gas volume} + 0$

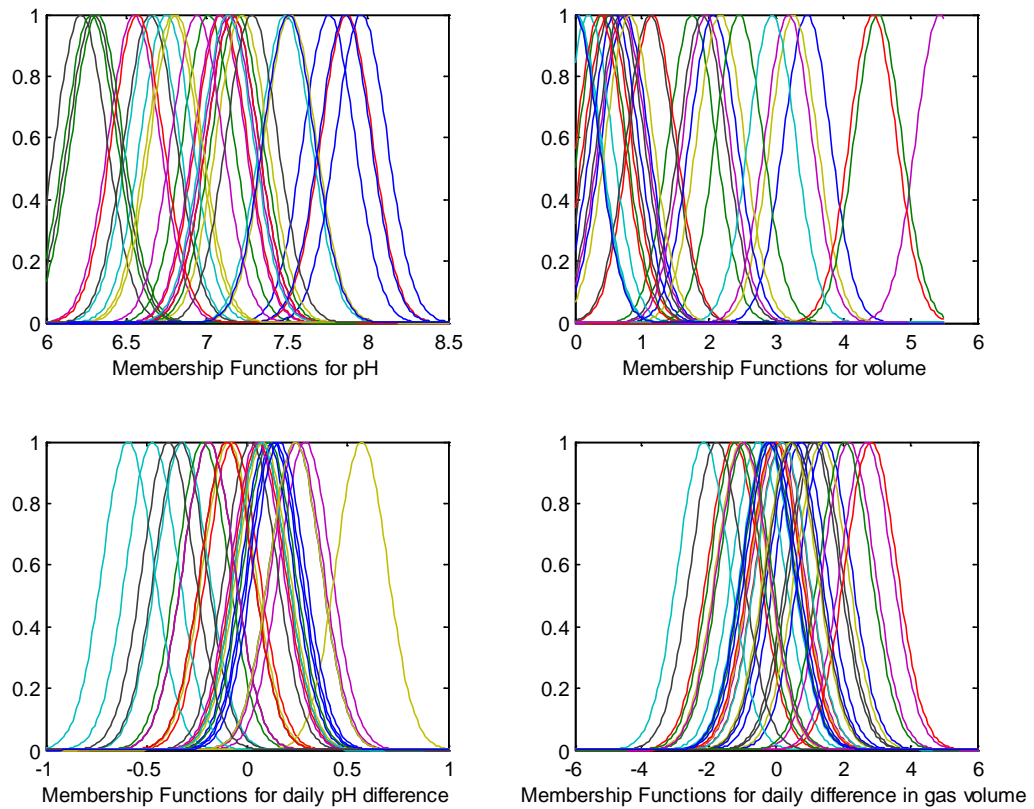


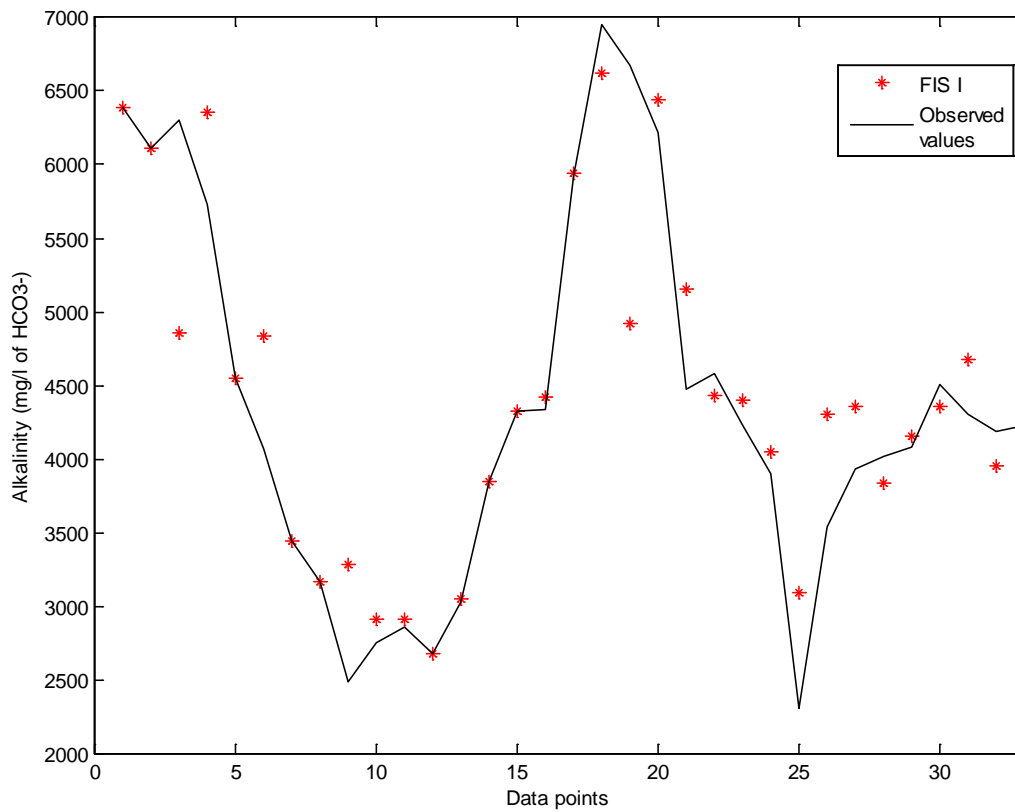
Figure 4.32 Fuzzy sets for FIS I

Cluster	centres				width (spread)			
	Input 1	Input 2	Input 3	Input 4	Input 1	Input 2	Input 3	Input 4
1	7.76	0.748	0.24	0.748	0.159	0.35	0.127	0.763
2	7.01	1.75	0.07	-0.21	0.159	0.35	0.127	0.763
3	7.87	1.156	-0.07	-1269	0.159	0.35	0.127	0.763
4	7.49	0.034	-0.32	-0.533	0.159	0.35	0.127	0.763
5	6.55	0.714	0.25	0.034	0.159	0.35	0.127	0.763
6	7.52	0	-0.09	0	0.159	0.35	0.127	0.763
7	6.22	1.904	-0.33	1.19	0.159	0.35	0.127	0.763
8	7.16	3.465	0.13	0.56	0.159	0.35	0.127	0.763
9	6.32	0.408	-0.22	-1.19	0.159	0.35	0.127	0.763
10	7.09	0.49	-0.19	-0.035	0.159	0.35	0.127	0.763

11	6.67	0.204	-0.59	-0.272	0.159	0.35	0.127	0.763
12	7.08	5.44	0.29	2.176	0.159	0.35	0.127	0.763
13	6.79	3.264	0.57	1.36	0.159	0.35	0.127	0.763
14	6.65	1.12	-0.1	1.103	0.159	0.35	0.127	0.763
15	7.96	2.065	0.1	1.47	0.159	0.35	0.127	0.763
16	7.19	4.522	0.11	-0.918	0.159	0.35	0.127	0.763
17	7.16	4.445	0.06	2.835	0.159	0.35	0.127	0.763
18	6.75	0.018	-0.47	-2.152	0.159	0.35	0.127	0.763
19	7.14	3.185	0.05	2.695	0.159	0.35	0.127	0.763
20	7.22	2.17	0.08	-1.015	0.159	0.35	0.127	0.763
21	6.3	0.68	0.02	-1.768	0.159	0.35	0.127	0.763
22	7.86	0.595	0.14	-0.245	0.159	0.35	0.127	0.763
23	6.28	2.448	-0.19	2.049	0.159	0.35	0.127	0.763
24	6.57	0.374	-0.1	0.17	0.159	0.35	0.127	0.763
25	7.13	2.94	0.07	0.1867	0.159	0.35	0.127	0.763
26	6.94	1.96	-0.19	-0.98	0.159	0.35	0.127	0.763
27	6.81	0.816	0.24	0.442	0.159	0.35	0.127	0.763
28	7.28	0.525	-0.39	0.49	0.159	0.35	0.127	0.763
29	7.51	0	0.16	-0.14	0.159	0.35	0.127	0.763

**Table 4.3** Membership function parameters obtained using subtractive clustering for FIS I

FIS was evaluated with data from all reactors following the design period. Despite the fact that the TSK system was developed with data based from reactors 4 and 5 alkalinity predictions were more accurate when the model was applied to reactor 6 (Table 4.4).  $R^2$  values were quite low for all reactors and almost 0 for reactor 4. MAE was satisfactory averaging around 700 for all reactors but the Bias values were encouraging since they were kept at minimum levels considering the application especially for reactor 5 where bias= 76.344. IA was quite good for reactors 5, 6 and at acceptable levels for reactor 4 by being just under 0.44. NMSE was not kept very close to 0 in all reactors but FB was particularly good for reactor 5 by having a value of 0.018.



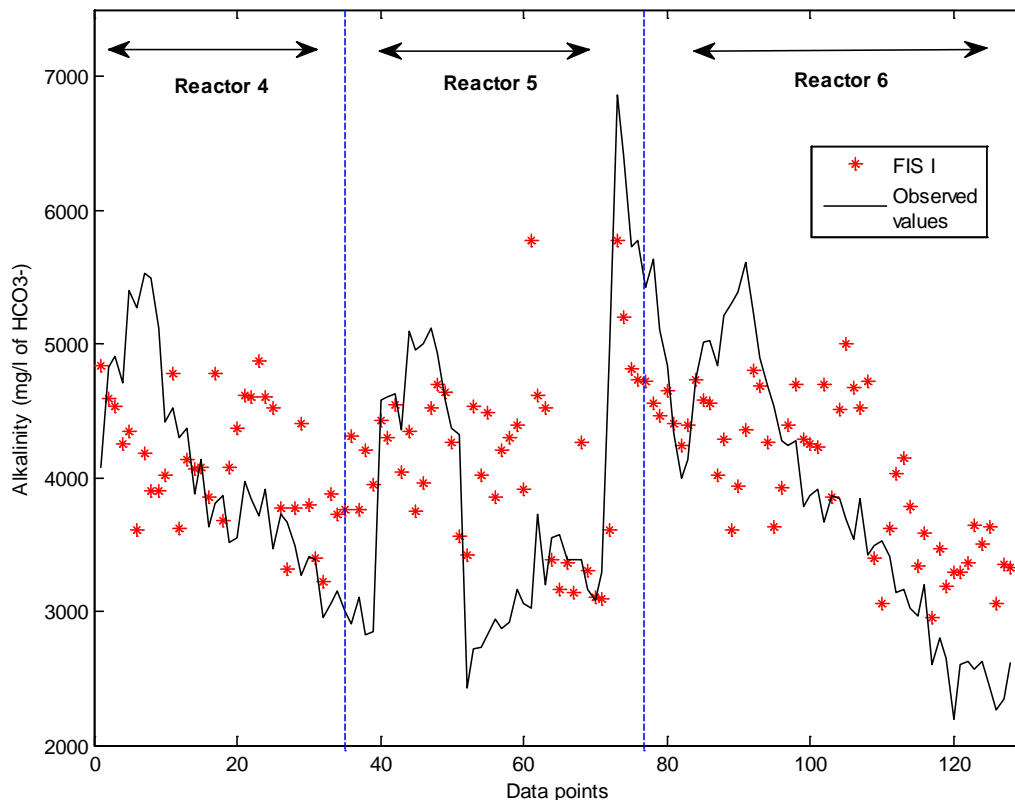
**Figure 4.33** Observed and predicted alkalinity values for FIS during training

Performance indices	FIS I			
	Reactor 4	Reactor 5	Reactor 6	Training
R <sup>2</sup>	0.042	0.242	0.398	0.823
MAE	686.18	747.28	692.23	328.78
Bias	181.6	76.344	251.34	46.405
IA	0.439	0.666	0.702	0.947
NMSE	0.04	0.05	0.04	0.014
FB	0.045	0.018	0.066	0.01

**Table 4.4** Alkalinity performance for FIS I during training and based on newly available data

Alkalinity predictions for all three reactors are depicted in Figure 4.34. Although the bias values are kept at quite low levels a visual representation of the results shows that the fuzzy model overpredicts most of the alkalinity values that are

below 3500 mg/l of  $\text{HCO}_3^-$  in the second reactor and under predicts the majority of the values in all reactors that are over 5000 mg/l of  $\text{HCO}_3^-$ . This is probably due to the fact that alkalinity values over 5000 mg/l of  $\text{HCO}_3^-$  were mostly recorded during the first few days of reactor operation since loading rate increases managed to reduce alkalinity to levels below 4500 mg/l of  $\text{HCO}_3^-$ .



**Figure 4.34** Observed and predicted alkalinity values using FIS I

Around the seventy-fifth data point a boost in alkalinity values is present in reactor 5. The FIS output seems to be following the real alkalinity values quite closely during that period which is when pH started to reach higher levels due to the addition of  $\text{NaHCO}_3$ . However, the last twelve predicted values in Figure 4.34 that correspond to the period following the water dilution that took place in reactor 6 indicate a huge deviation from the actual values.

By taking into consideration that:

- The FIS was designed with the inclusion of minimum amount of data from the stable operation period



- Only data from two out of the three reactors were utilised in each design
- The fuzzy model responded positively to one out of the two disturbances that were inserted to the systems

Embedding reactor 6 data to increase the training set data base and adding data from the majority of the stable operation period would increase the functionality of the fuzzy predictor.

#### **4.3.5.3 FIS II design**

FIS II was constructed with data from all three reactors (4, 5 and 6). Data until 06/10/2012 were used to train and check the model and alkalinity measurements that were taken until the end of the further experimentation period constituted the validation set. FIS consequent functions were set following least squares estimation. The premise and consequent parameters and structure were determined and tuned recursively and FIS consequent functions were linear.

Cluster radius was varied from 0.15 to 1 with a step of 0.01. The squash factor was set to 1.25, the reject ratio to 0.15 and the accept ratio to 0.5. The cluster radius of the FIS that was selected to determine alkalinity was 0.41. Five cluster centres were determined using subtractive clustering (Table 4.5) which correspond to an equal amount of membership functions and rules that regulated system output. Fuzzy rules are depicted in Table 4.6.

FIS II had fewer cluster centres compared to FIS I (the number was reduced from twenty to five). This was a result of the higher value that was assigned to the cluster radius (0.41) which probably resulted in a more accurate system representation. It is possible that the smaller  $r_a$  value (0.15) that was selected during the design of FIS I, might have led to an over defined system characterized by an excessive number of rules (Mollaiy Berneti 2011).

Cluster	centres				width (spread)			
	Input 1	Input 2	Input 3	Input 4	Input 1	Input 2	Input 3	Input 4
1	7.08	0.683	0.11	-0.026	0.167	0.197	0.186	0.176
2	7.3	0.411	0	-0.003	0.167	0.197	0.186	0.176
3	7.13	0.663	-0.04	0.128	0.167	0.197	0.186	0.176
4	6.9	0.978	-0.11	0.145	0.167	0.197	0.186	0.176
5	7.1	0.645	0.02	0.48	0.167	0.197	0.186	0.176

**Table 4.5** Membership function parameters obtained using subtractive clustering for FIS II

Rule	Rule description of the form If ... Then $y = p_1x_1 + p_2x_2 + p_3x_3 + p_0$
1	<p><i>If <math>pH_{in}</math> is <math>pH_{in}MF1</math> and <math>GV/RV_{in}</math> is <math>GV/RV_{in}MF1</math> and <math>pH\ dif_{in}</math> is <math>pH\ dif_{in}MF1</math> and <math>GV/RV\ dif_{in}</math> is <math>GV/RV\ dif_{in}MF1</math> Then</i></p> $\text{Alkalinity} = -1082.98 \cdot pH + 65.827 \cdot GV/RV\ dif + 4147.465 \cdot pH\ dif - 4033.91 \cdot GV/RV\ dif + 8873.479$
2	<p><i>If <math>pH_{in}</math> is <math>pH_{in}MF2</math> and <math>GV/RV_{in}</math> is <math>GV/RV_{in}MF2</math> and <math>pH\ dif_{in}</math> is <math>pH\ dif_{in}MF2</math> and <math>GV/RV\ dif_{in}</math> is <math>GV/RV\ dif_{in}MF2</math> Then</i></p> $\text{Alkalinity} = 5103.667 \cdot pH - 3814.17 \cdot GV/RV\ dif + 1193.006 \cdot pH\ dif - 1035.44 \cdot GV/RV\ dif - 32529.7$
3	<p><i>If <math>pH_{in}</math> is <math>pH_{in}MF3</math> and <math>GV/RV_{in}</math> is <math>GV/RV_{in}MF3</math> and <math>pH\ dif_{in}</math> is <math>pH\ dif_{in}MF3</math> and <math>GV/RV\ dif_{in}</math> is <math>GV/RV\ dif_{in}MF3</math> Then</i></p> $\text{Alkalinity} = 9275.63 \cdot pH - 4479.68 \cdot GV/RV\ dif + 2642.83 \cdot pH\ dif - 11848.7 \cdot GV/RV\ dif - 55558.3$
4	<p><i>If <math>pH_{in}</math> is <math>pH_{in}MF4</math> and <math>GV/RV_{in}</math> is <math>GV/RV_{in}MF4</math> and <math>pH\ dif_{in}</math> is <math>pH\ dif_{in}MF4</math> and <math>GV/RV\ dif_{in}</math> is <math>GV/RV\ dif_{in}MF4</math> Then</i></p> $\text{Alkalinity} = 5083.47 \cdot pH - 2472.1 \cdot GV/RV\ dif - 2345.85 \cdot pH\ dif + 1686.734 \cdot GV/RV\ dif - 28906.5$

5	<p><i>If <math>pH_{in}</math> is <math>pH_{in} MF5</math> and <math>GV/RV_{in}</math> is <math>GV/RV_{in} MF5</math> and <math>pH dif_{in}</math> is <math>pH dif_{in} MF5</math> and <math>GV/RV dif_{in}</math> is <math>GV/RV dif_{in} MF5</math> Then</i></p> $Alkalinity = 189.851 \cdot pH + 145.331 \cdot GV/RV dif - 1155.1 \cdot pH dif + 1135.225 \cdot GV/RV dif + 1672.771$
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**Table 4.6** TSK fuzzy model rules and consequent parameters for FIS II (GV/RV is gas volume/reactor volume, and dif stands for difference)

During the evaluation of FIS II it was observed that the supposedly more accurate model did not have many differences with FIS I. However, the most important finding was that during the design process there was always a tradeoff between  $R^2$  and MAE and Bias. Throughout the design process of FIS II, models that had high  $R^2$  values ranging up to 0.7 had MAE and Bias values that were higher by at least 25% of the MAE and Bias values that characterise FIS II. Since the latter statistical indices are considered to be more important in this design, FIS II coefficient of determination values are kept at low levels (<0.5), for all three reactors. MAE values averaged at 629 and Bias values averaged at 568 which are sufficient enough since the alkalinity stability and optimum operation range is between 3500 mg/l of  $HCO_3^-$  and 4500 mg/l. Predicted and observed alkalinity values are depicted in Figure 4.35 demonstrating that bias is kept at desired levels.

Index of agreement had an average value of 0.52 for all reactors, which shows an acceptable agreement between predicted and observed values, and fractional bias had an average value of 0.17 which is not as close to 0 as desired. FIS II was able to characterise alkalinity levels in reactor 6 with more accuracy than reactor 4 and reactor 5 (Table 4.7)

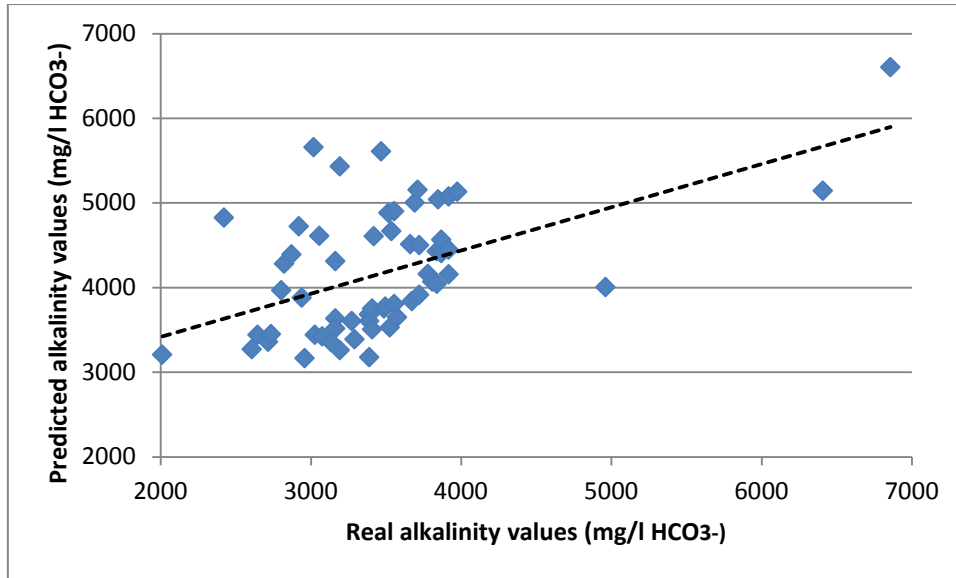
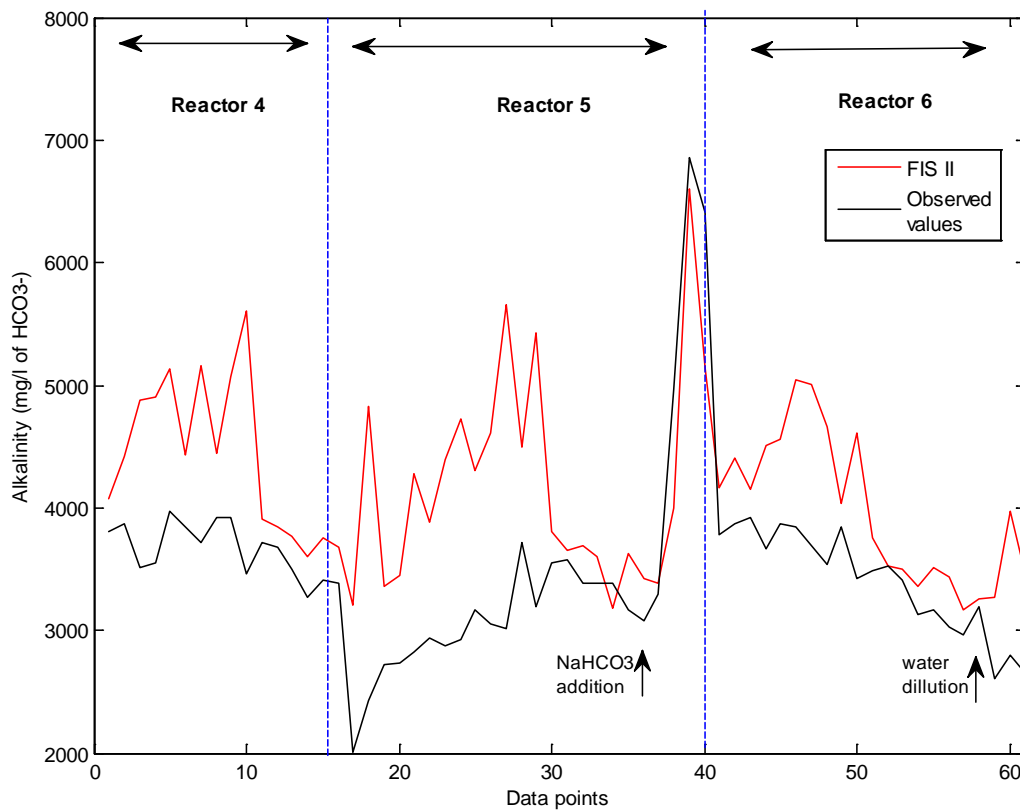


Figure 4.35 Observed and predicted alkalinity values using FIS II

Similarly to FIS I, FIS II was able to detect boosted alkalinity levels due to addition of  $\text{NaHCO}_3$  in an accurate manner, however when water dilution was applied to reactor 6 the predicted alkalinity levels started rising compared to the measured values and declined by 1300 mg/l of  $\text{HCO}_3^-$  on average. So, it is concluded that in order to enable the reactors to detect values after dilution this data should be embedded in the fuzzy design (Figure 4.36).

Performance indices	FIS II		
	Reactor 4	Reactor 5	Reactor 6
$R^2$	0.143	0.281	0.499
MAE	621.77	800.81	466.53
Bias	621.77	616.93	466.53
IA	0.3	0.66	0.603
NMSE	0.045	0.082	0.031
FB	0.188	0.197	0.155

Table 4.7 Alkalinity performance for FIS II based on newly available data



**Figure 4.36** Observed and predicted alkalinity values for all reactors using FIS II

## 4.4 Conclusions

Evaluation of the performance of six 5l reactors was conducted. Three reactors did not contain any support surfaces and three reactors had burst cell foam coarse, burst cell foam medium, and sponge respectively attached as biomass support media. All reactors were operated under similar loading regimes to observe and determine differences in the way they function.

Reactors with biomass support media proved to be more stable compared to the reactors without support media, but did not exhibit significantly higher gas productivity. Biomass support media appeared to play a vital role in digester recovery which was also witnessed after reactor breakages, where the system had to be restarted, by speeding up the recovery period. Sponge had a higher positive influence on gas production followed by burst cell foam coarse and burst cell foam

medium. From reactors without support surfaces (reactors 1-3) only reactor 3 managed to reach gas production levels similar to reactors with support media during its limited time of operation.

Maximum sustainable loading rates varied between different types of reactor set-ups and increased biogas production was found to exist between 3-3.5 g VS/l/d for reactors without support media and 4-5 g VS/l/d for reactors with support media. Optimum pH ranges were identified for reactors 1, 2 and 3 between pH 7.1- 7.3 and were slightly lower for reactors 4-6 (pH 6.9-7.2). All reactors became unstable when pH was <6.9. Also, alkalinity levels for system stability appeared to be above 3500 mg/l of  $\text{HCO}_3^-$  for reactors with no media and 3480 mg/l of  $\text{HCO}_3^-$  for digesters with support media. Biogas production was boosted when alkalinity was between 3500-4500 mg/l  $\text{HCO}_3^-$  for vessels 1-3 and between 3480-4300 mg/l of  $\text{HCO}_3^-$  for vessels 4-6.

A reformulation of the inputs that would be part of the fuzzy logic system designed to predict alkalinity was performed. A new selection of inputs based on low cost reliable sensors that included gas volume production which is indicative of the digester performance was conducted. Therefore, instead of having pH, ORP and EC as system inputs pH, gas volume/reactor volume, daily pH difference and daily gas volume/reactor volume difference were selected as the new inputs.

Two first order TSK fuzzy systems were developed during different periods throughout the experiments trying to capture alkalinity behaviour. These software predictors intended to form the basis of a controller that would regulate the loading rate based on alkalinity. FIS I appeared to be better than FIS II, however due to the limited input range during the evaluation process some values were outside the specified range making it more unstable for future utilisation. FIS II was supposed to perform better than FIS I since a larger database was utilised during the design process. FIS II behaviour was slightly inferior to FIS I, however a higher degree of completeness in the training data set would enhance the accuracy of this software predictor in future applications. FIS II was characterised by quite good MAE and bias values of 466.53 mg/l of  $\text{HCO}_3^-$  for reactor 6 and an acceptable value for  $R^2 = 0.498$ . NMSE was close to 0 with a value of 0.03 and a slightly higher FB= 0.154 than desired.

Data collected throughout the duration of the experiment contained some uncertainties mainly due to practical problems encountered during reactor operation.

Those included gas leakages, oxygen intake during reactor loading and unloading, reactor breakages and the fact that water from the water bath containing the cells would flow into the reactors through the gas collection tubes during high loading periods taking place at start-up. However, these uncertainties make the fuzzy system more robust since they are contained in the inputs and the outputs that were used to determine the system structure itself (Ross 2004). System robustness was tested by adding  $\text{NaHCO}_3$  to reactor 5 and by diluting reactor 6 with water. FIS I and FIS II were able to follow the system output closely in the first case, but not in the second. This means that process data recorded during the days that followed water dilution should be embedded in the system design to enable it to detect similar changes in the future.

## **Chapter 5 Alkalinity software sensor application and development of rule-based and fuzzy logic organic loading rate control systems for anaerobic digesters**

FIS II (developed in Chapter 4) is utilised as an alkalinity predictor. FIS II functionality is tested through the application on a 28.34l cylindrical reactor with biomass support media treating cow manure. In a second experiment a rule-based and a Mamdani fuzzy logic controller are developed to regulate the organic loading rate based on alkalinity predictions from FIS II. They are tested through the application in five 6.46l reactors with biomass support media treating cellulose.

### **5.1 Introduction**

Process control plays an important role in AD systems to stabilise the fermentation as well as optimise the biogas output primarily by regulating the amount of volatile solids that enter the digester on a routine basis. High OLR can require continuous digester feeding (hourly) whereas when lower loading rates are applied the digester needs to be fed once a day (Mattocks 1984). OLR is adapted to the biological conversion capacity of AD systems (Verma 2002) by process control and is directly associated with the retention time via the active biomass reactor concentration (Schoen 2009). Also, biogas production depends on the organic matter that is biodegraded by anaerobic microorganisms. OLRs depend on the substrate utilised which is the reason why optimum OLR values range according to the substrate and operating conditions (e.g. differences exist between mesophilic and thermophilic AD plants operating with identical substrates). Reactor set-up is an additional factor that influences OLR. Van Lier (1996) provides a table of optimal OLR values for different substrates operated under thermophilic temperatures.

A fuzzy logic AD controller can be utilised to achieve different goals (Puñal et al. 2003) (Yordanova 2004):

- Keep the required concentration of organic matter at the reactor output
- Reach an optimal methane production level



- Succeed in producing a stable operation in case of systems treating high OLRs affected by input concentration and/or flow rate oscillations.

Several FLC applications can be found in the literature. Yordanova (2004) developed a two-level FLC for the biogas production rate in the anaerobic wastewater treatment plant (WWTP), pointing out the efficiency of the fuzzy approach compared to the application of a conventional PI controller. Another FLC was developed (Scherer et al. 2009) to control biogas reactors using energy crops. The resulting system proved to be successful during start-up and while recovering from failure. The FLC achieved the desired process performance under high OLR and low hydraulic retention time (HRT) without utilizing any special mathematical model or detector or self-learning network. OLR was determined based on pH, specific gas production rate (GPR) and CH<sub>4</sub> content. Specific GPR was chosen instead of volume GPR, as the latter was proven unable to support pH control efficiently. Although redox is widely used as a process parameter, it was not utilised in this case as it was found to be lacking reliability. The number of FLC rules was selected as 3<sup>x</sup>, where x is the input number. It is recommended that the FLC process variables should be reconfigured for different substrates. A FLC based on the utilisation of cheap on-line sensors (Estaben et al. 1997) enables the system to function around a set-point and achieves acceptable reduction of chemical oxygen demand (COD). Stable operation was possible around a working point with perturbations or overloading conditions. A two-stage anaerobic wastewater pre-treatment was controlled (Murnleitner et al. 2002) with a FLC system predicting the biological state of the reactors. Control was based on which control actions were taken to maintain process stability and this approach proved to be suitable for applications involving strong volume and concentration variations, or where additional feed can achieve higher biogas production. Finally, the main control issue in the design that appears in (Carrasco et al. 2002) is successful operation recovery in the case of disturbances and, similarly to the previous work, proper state detection of the WWTP.

Knowledge based rule structures have also been applied to control AD systems. Some of the applications were based on fuzzy logic and were implemented as a rule-base (Carrasco et al. 2002) (Carrasco et al. 2004). Other designs include the utilisation of rule-based supervisory systems to control the influent flow rate (Liu

et al. 2004a)(Liu et al. 2004b)and the OLR (Ward 2009) (Partner N° 2, Rothamsted Research 2010).

This chapter focuses on the development of two controllers that serve the same purpose which is to control the OLR of an anaerobic digester: a fuzzy logic controller and a rule-based controller. The determination of optimum operating parameter ranges for pH, OLR and alkalinity was presented in Chapter 4 and formed the basis of the design of the two controllers. FIS II (Chapter 4) was set to predict alkalinity which is indicative of digester stability. Alkalinity predictions were then fed into the controller that would vary the OLR accordingly.

Both approaches were very similar since they were designed based on the alkalinity and OLR optimum parameter ranges identified in Chapter 4. The first controller is a Mamdani fuzzy logic controller that regulates the OLR based on the daily alkalinity value and the daily difference in alkalinity. The second controller is a rule based system that varies the OLR using the FIS II alkalinity predictions and the daily difference in alkalinity.

## **5.2 Materials and methods**

### **5.2.1 Order of experiments**

The experiments that were performed are as follows:

1. Evaluating the alkalinity predictor FIS II using a different reactor set-up (02/10/2012- 03/12/2012)

During the first experiment, fuzzy test 1 (FT1), the OLR was adjusted manually and focused on evaluating FIS II using a 28.34l reactor

2. Evaluation of a rule-based system to control OLR (09/01/2013- 08/03/2013)

The second experiment, controller test 1 (CT1), tested FIS II. OLR was determined by a rule-based controller (07/02/2013- 08/03/2013) using control strategy II and was applied on two 6.46l reactors treating cellulose.

3. Evaluation of a fuzzy logic system to control OLR (09/01/2013-08/03/2013)

The third experiment, controller test 2 (CT2), tested FIS II. OLR was varied using a Mamdani fuzzy logic controller (07/02/2013- 08/03/2013) using control strategy I and was applied on three 6.46l reactors treating cellulose.

The order of experiments is depicted in Figure 5.1 where FLC corresponds to the fuzzy logic controller and RBC to the rule-based controller.

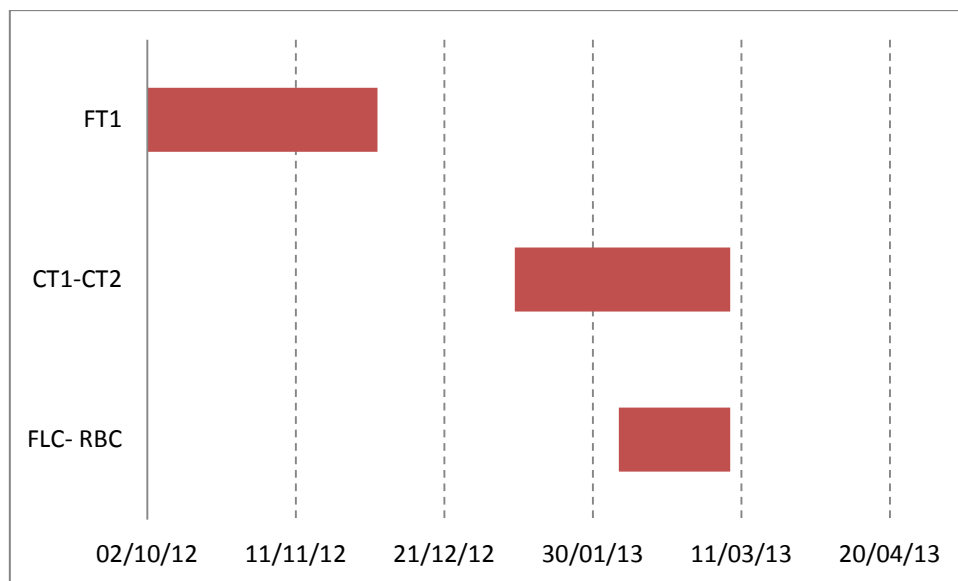
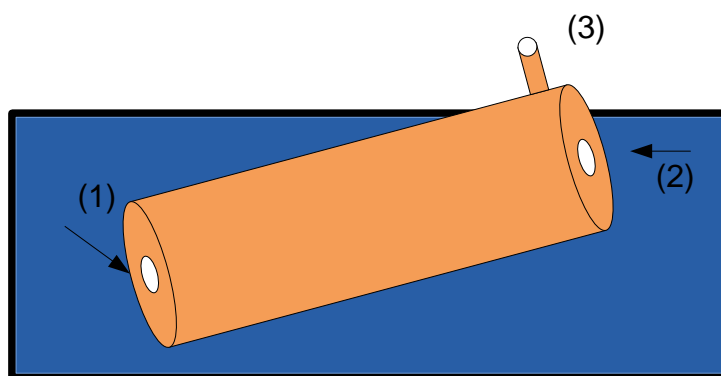


Figure 5.1 Order of experiments and controller application

## 5.2.2 Reactor construction

### 5.2.2.1 Reactor used in experiment FT1

The anaerobic digester used in FT1 consisted of a 28.34l single stage cylindrical reactor with a working volume of 25l to provide a headspace (Figure 5.2). Biomass support media lined the inside of the tank after being formed into a cylinder and stainless steel wire was utilised to keep the support media in place.



**Figure 5.2** Schematic of larger digester. Mixing was implemented by pumping digestate from inlet 1 to inlet 2. (3) is the gas outlet.

The gas outlet located at the top of the reactor was connected to a liquid displacement system made of a cell of known capacity that operated based on the principle of buoyancy. A magnetic reed switch provided information with respect to the number of times each cell was moved (Figure 4.2). The switch was connected to a mechanical counter that recorded gas production off-line (Figure 4.3). The reactor was placed at a c.a. 30° angle inside a water bath to prevent the gas outlet from getting blocked.

### **5.2.2.2 Reactors used to evaluate fuzzy logic and rule-based controllers**

6.46l cylindrical reactors with a working volume of 5l were used in CT1-CT2. These reactors were placed vertically inside a water bath. The reactor-set up was similar to the one used in FT1 (inlet-outlet for mixing, gas outlet) and off-line gas volume production was recorded in the same way.

### **5.2.3 Biomass support media**

All reactors were equipped with biomass support media attached around the inside of each reactor. Based on the reactor performance presented in Chapter 4 (where it was concluded that the reactor with the sponge produced the highest amount of biogas), sponge with a c.a. 1 mm pore diameter was selected as the biomass support surface. The size of the support material in the 28.34l reactor was 80 cm× 38 cm× 3cm and 56 cm× 20 cm× 3 cm in the 6.46l reactors.

## **5.2.4 Feedstock**

Cow slurry was used for the reactor operated during the first experiment (FT1). Manure was collected from the beef cattle located at North Wyke Research every seven days. Cow manure was diluted with water and then screened through a 10 mm diameter mesh to reduce the amount of straw bedding that would enter the digester. Cow slurry was then stored in deep fridge below +4° C to reduce degradation processes. Cow slurry was heated to 55°C for 5-6 hours on a daily basis prior to feeding in order to minimise temperature imbalance inside the reactor. Cellulose was used as a feedstock for experiments CT1- CT2.

## **5.2.5 Reactor operation start up procedures**

### ***5.2.5.1 Reactor used in experiment FT1***

The reactor used in experiment FT1 was initially loaded with 25l. Of the 25l, 14l was digestate originating from reactors 1-3 (Chapter 4) that used cow slurry containing 4.35% total solids and 3.07% volatile solids holding a total alkalinity value of 5400 mg/l  $\text{HCO}_3^-$ . The remaining 11l were fresh cow slurry with 3.9% total solids and 3.27% volatile solids with a total alkalinity value of 1826 mg/l  $\text{HCO}_3^-$ .

### ***5.2.5.2 Reactors used in experiments CT1-CT2***

The five 6.46l reactors that were used in CT1 and CT2 were originally filled with 5l of cow slurry of 3.35% total solids and 2.93% of volatile solids. For the duration of all experiments each reactor was fed once a day on an average of five days during the working week. The feeding process was conducted manually during FT1 by connecting the peristaltic pump to point (1) for substrate withdrawal and point (2) for substrate insertion (Figure 5.2). During CT1-CT2 each reactor was fed manually by removing the top of each 6.46l reactor.

### **5.2.6 Mixing**

The pump that was used in all experiments was a Watson-Marlow 323 U/D peristaltic pump (Watson-Marlow Bredel pumps, Falmouth, Cornwall TR11 4RU, UK). Mixing was performed manually for 10 min before every feeding incident and for 10 min after every feeding during FT1 because the pump blocked when operated automatically. The mixing time corresponds to the amount of time it took for the pump to mix the contents of the reactor twice.

Although the substrate viscosity and composition should enable the peristaltic pump to function automatically during experiments CT1-CT2, however the pump would block most of the time. Therefore, gentle stirring with a rod manually took place for 1 min before sample collection and 1 min after daily feeding.

### **5.2.7 Off-line monitoring**

Throughout the duration of all experiments each reactor was monitored once a day for pH and CH<sub>4</sub> content in the biogas. Gas volume was recorded twice a day to ensure that no gas leakages existed in the gas collection system or the reactors. Total solids and volatile solids analysis was conducted at frequent intervals during FT1. Samples were analyzed for alkalinity on an average of five times a week.

pH was measured off-line using a HANNA INSTRUMENTS HI9025 microprocessor-based pH meter during FT1, CT1-CT2. The pH meter was calibrated weekly using pH 4 and pH 7 buffer solutions. During FT1 pH was recorded during the feeding incident. During experiments CT1-CT2 pH was measured five hours before the feeding incident.

Methane content, biogas volume, total solids, volatile solids and alkalinity were measured using the same methods described in section 4.2.6.

### **5.2.8 Fuzzy Inference System predicting alkalinity**

First order fuzzy logic systems FIS I and FIS II were developed and tested in Chapter 4. FIS II was developed with a bigger database than FIS I and although it

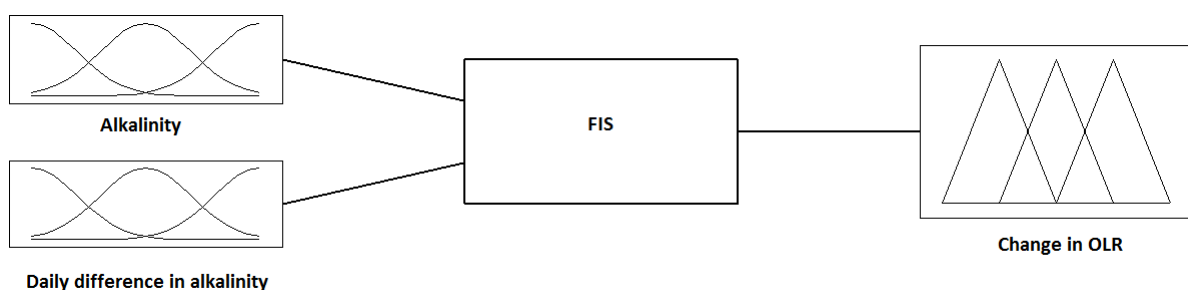
proved to be slightly inferior to FIS I, data size would make it more robust than FIS I in future applications.

FIS I and FIS II were tested against disturbances that included bicarbonate addition and water dilution. Both systems reacted positively to bicarbonate addition but their predictions were not as accurate as expected regarding alkalinity values that existed in the reactors during the days that followed water dilution. Therefore, previous data were included in a design of a new FIS as suggested in the previous chapter. This inclusion produced a less accurate model for normal operation. So, since the aim of the design of a software sensor for an AD process is also to maintain a stable system, it should also aim at avoiding correcting actions that might include diluting reactor contents with water. For these reasons FIS II was selected as the alkalinity predictor for all experiments.

### 5.2.9 Control strategy I (Fuzzy Logic)

A fuzzy logic system (Mamdani) was developed to determine the OLR variation to control the fermentation during experiments CT2 and CT4. The controller was based on Mamdani's fuzzy inference method (Mamdani & Assilian 1975).

Fuzzy Logic Toolbox within the framework of Matlab 7.10 was used to create the FIS. Input variables representing alkalinity and daily difference in alkalinity and the output variable representing the change in OLR expressed in g/l/d were developed using the FIS editor (Figure 5.3).



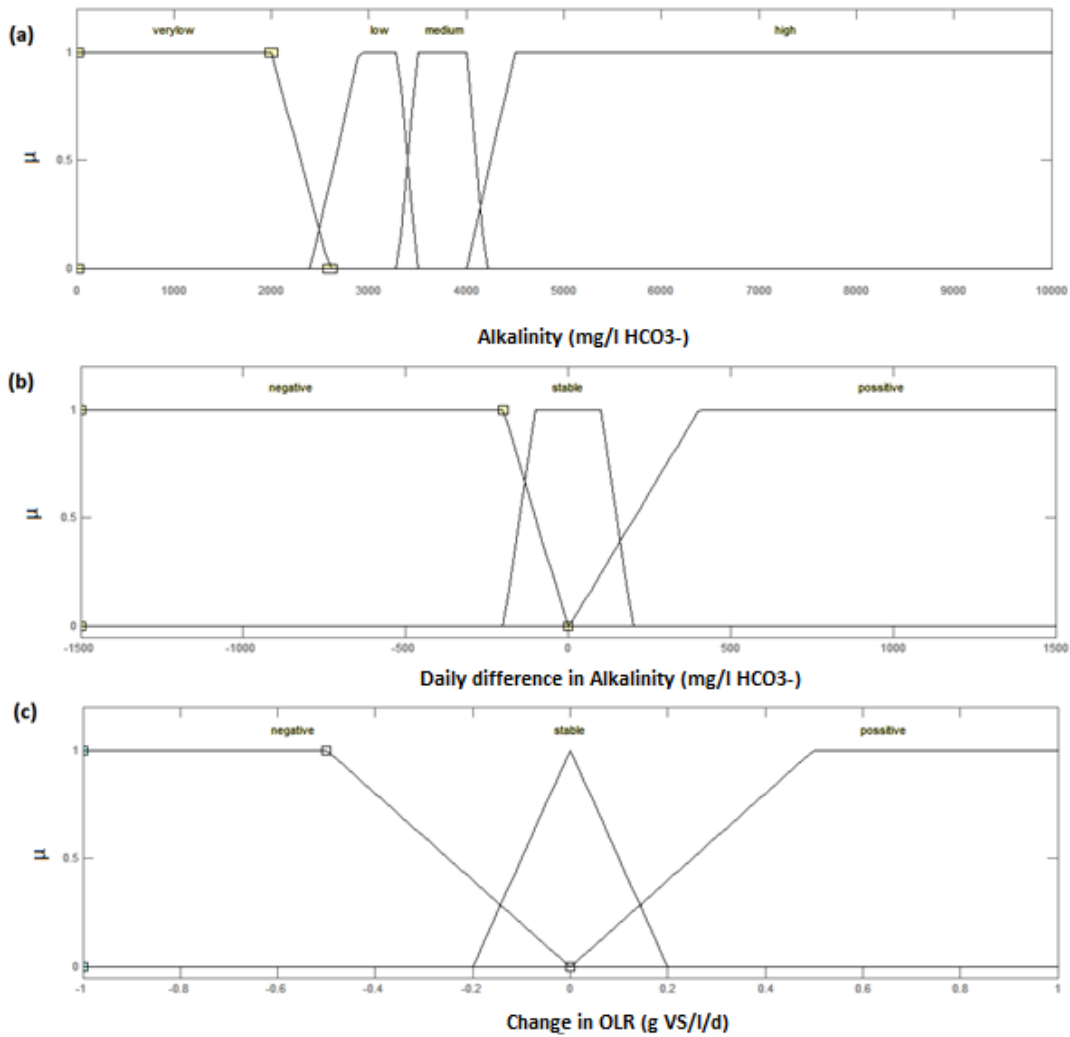
**Figure 5.3** Inputs and output variables of the Mamdani FIS

Four linguistic values corresponding to the same number of membership functions were assigned for alkalinity (very low, low, medium, and high). The very low membership function was assigned for alkalinity values below 2600 mg/l of  $\text{HCO}_3^-$  that are typical values of a system during the start-up process or a system that has become unstable. The low membership function included alkalinity values between 2400 mg/l of  $\text{HCO}_3^-$  and 3500 mg/l of  $\text{HCO}_3^-$  that characterise a digester that is either recovering, or is about to become unstable or has optimum gas production but can become unstable. The medium membership function was assigned for values ranging between 3300 mg/l of  $\text{HCO}_3^-$  and 4200 mg/l of  $\text{HCO}_3^-$  that can be indicative of a system that is about to become unstable (below 3500 mg/l of  $\text{HCO}_3^-$ ) or a stable system exhibiting optimum biogas production ( $3500 >$  mg/l of  $\text{HCO}_3^-$ ). The high membership function contained alkalinity values  $4000 >$  mg/l of  $\text{HCO}_3^-$  that signify a stable system that might have not reached the highest gas production rates.

Three linguistic values were set for the daily difference in alkalinity (negative, stable and positive) that could be indicative of the stability state of the system. The negative fuzzy set contained the negative changes in alkalinity values (a very low value signifies a digester that is overloaded). The stable set corresponded to changes in alkalinity values between -200 mg/l of  $\text{HCO}_3^-$  and 200 mg/l of  $\text{HCO}_3^-$ . The positive set included positive alkalinity value changes.

Three linguistic values were assigned for the change in OLR (negative, stable and positive) that ranged between -1 g VS/l/d and +1 g VS/l/d (Figure 5.4).





**Figure 5.4** Membership functions of (a) alkalinity, (b) daily difference in alkalinity, (c) change in OLR

Ten rules were established based on the experience that was obtained while conducting the experiments presented in Chapter 4. For simplicity alkalinity is symbolised as A, daily difference in alkalinity is symbolised as DDA and change in OLR as C. For the same reason, an abbreviation of each linguistic value is used in the rule-base description (VL= very low, L= low, M= medium, H= high, N= negative, S= stable, P= positive) as follows:

- If A is VL and DDA is N then C is S
- If A is VL and DDA is S then C is S
- If A is VL and DDA is P then C is P
- If A is L and DDA is N then C is N
- If A is L and DDA is S then C is S

If A is L and DDA is P then C is P

If A is M and DDA is N then C is S

If A is M and DDA is S then C is S

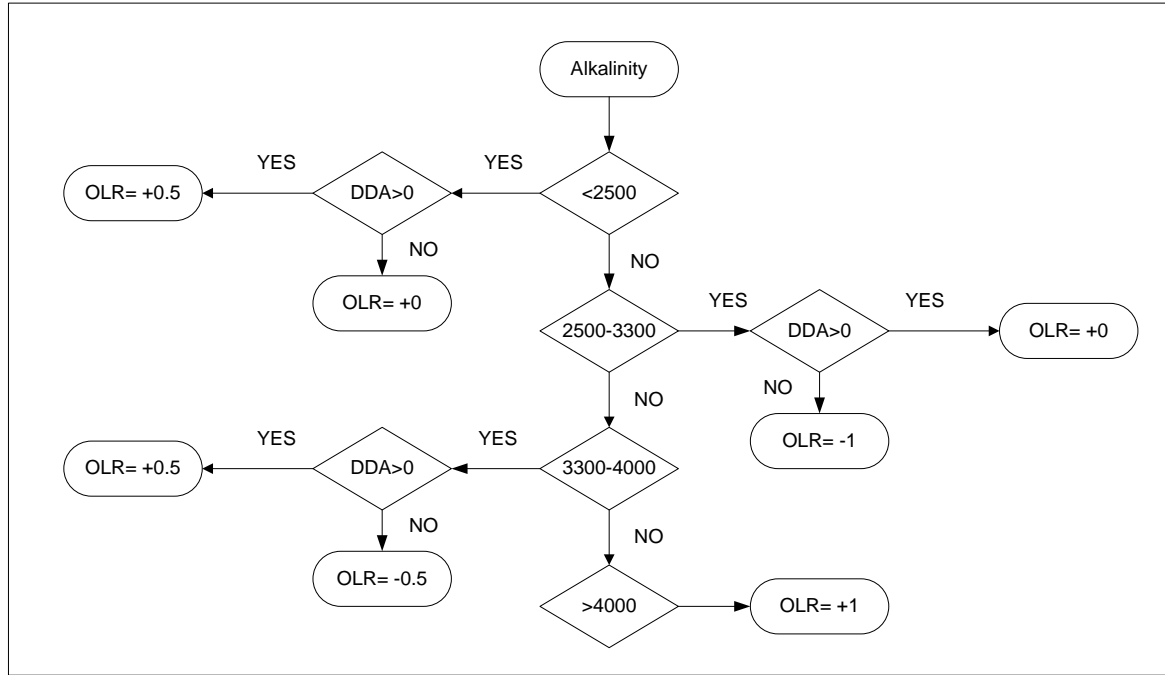
If A is M and DDA is P then C is P

If A is H then C is P

For experiment CT2 the OLR was kept below 5.5 g VS/l/d to ensure that the reactors would not be massively overloaded and so avoid system failure. Therefore, in case the rate of change in OLR was suggesting an increase above the 5.5 g VS/l/d, which never happened during the fuzzy control application, the OLR would remain stable until a decrease would be applied by the fuzzy system. The minimum OLR was set at 1.5 g VS/l/d. This value would also provide a starting OLR point for the initial system operation.

#### **5.2.10 Control strategy II (Rule-based)**

A rule-based system was implemented based on the same principles with which control strategy I was formulated. The same inputs (alkalinity and daily difference in alkalinity) were used to predict the rate of change in OLR. The major differences between Control strategy I and Control strategy II are that the alkalinity partition ranges and the OLR variations are crisp, and that the daily difference in alkalinity can be either positive or negative. The flow chart of the rule-based system is depicted in Figure 5.5.



**Figure 5.5** Flow chart of the rule-based system where DDA= daily difference in alkalinity, OLR (g VS/l/d), Alkalinity (mg/l of  $\text{HCO}_3^-$ )

The rule-based system was implemented based on the analysis of the data acquired from the experiments that were conducted in Chapter 4. It was ensured that during CT1 the OLR would not exceed 5.5 g VS/l/d even if the rule-based system suggested a higher increase although this action was never suggested by this system as will be presented later in this chapter. The minimum OLR of the rule-based system was set at 1.5 g VS/l/d.

## 5.3 Results and discussion

### 5.3.1 FT1 reactor operation

Experiment FT1 was performed for nine weeks to evaluate FIS II model to determine alkalinity using sensors and a digester of different size and configuration. The FIS II model was also tested by disturbing the reactor by dilution with water, addition of  $\text{NaHCO}_3$  and by temperature fluctuations. The OLR was varied manually based on the experience acquired from the operation and the data recorded during the experiments presented in Chapter 4. The aim was to maximise biogas productivity while maintaining a stable digester environment. Also, based on the

reactor performance controller strategy I and controller strategy II were formulated. However, stability was not always achieved due to circumstances that are explained below.

Following week one (Figure 5.6) where OLR was kept to zero to allow the inoculation of the biomass support media, OLR averaged around 3.3 g VS/l/d during week two and week three. At the beginning of week four, the reactor gas outlet blocked and 16.5l of the working inoculum was lost and was replaced with cow slurry with TS%= 3.9 and VS%= 3.25. The loading process was reinitiated during week five when 2.5 g VS/l/d were added in the reactor on a daily basis. During week six, a 1/5<sup>th</sup> water dilution and a NaHCO<sub>3</sub> addition of 3 g/l were performed. An average loading rate of 2.4 g VS/l/d was applied on week six till week nine. OLR was kept at low levels averaging at 2 g VS/l/d especially during weeks eight and nine due to temperature fluctuations that were a result of the water bath heater malfunction.

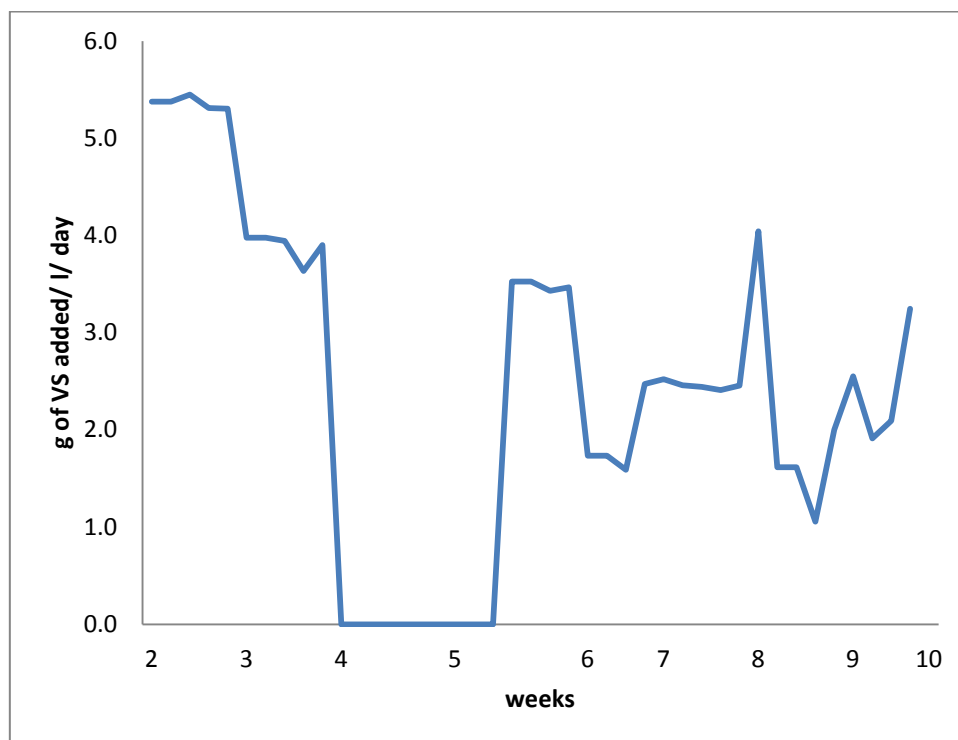
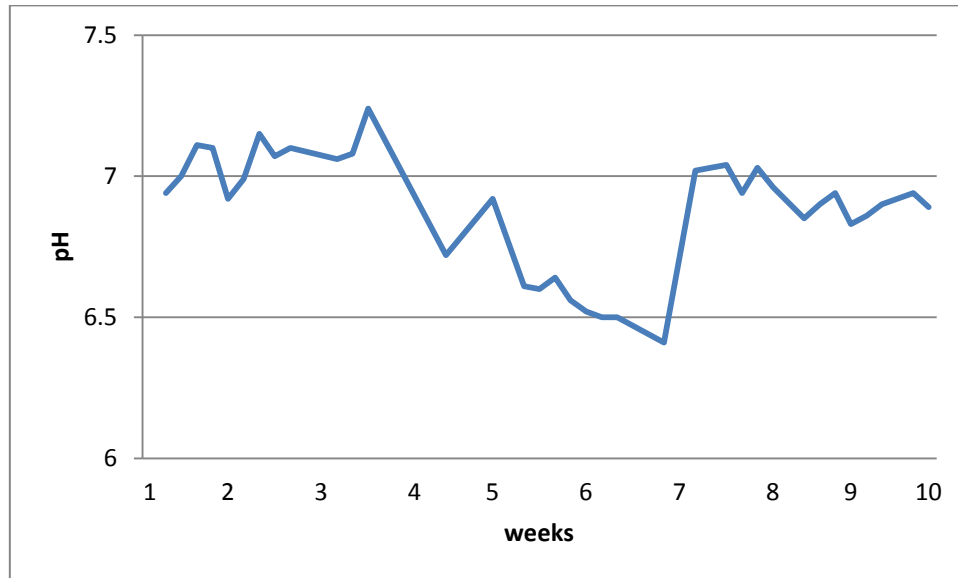


Figure 5.6 OLR during FT1 operation

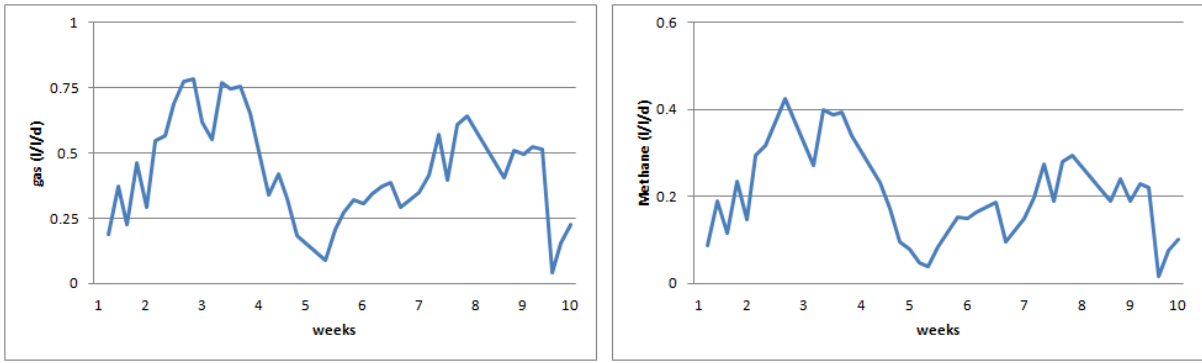
pH was kept within values indicating stability varying between 6.9- 7.24 until the beginning of week four when most of the working volume was lost (Figure 5.7). Following restart, the reactor was quite acidic with pH values varying between 6.41-

6.92. On week six, water dilution and NaHCO<sub>3</sub> addition boosted pH that reached a value of 7. After week seven severe temperature fluctuations destabilised pH that varied from 6.83- 6.94.

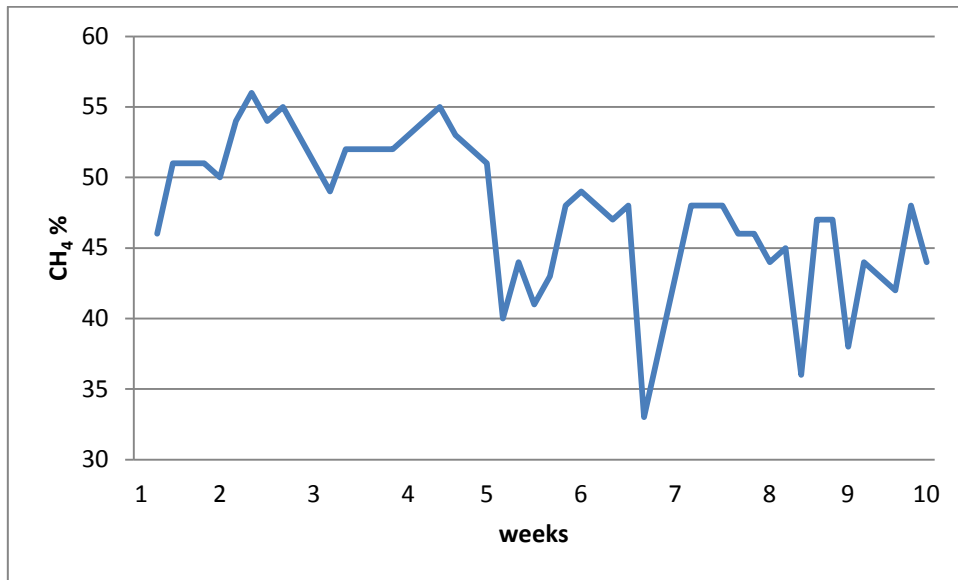


**Figure 5.7** pH during FT1 operation

Gas and methane production levels (Figure 5.8) were on average 0.56 l/l/d and 0.28 l/l/d respectively until week four before the process restart. During weeks four and five when pH values were low, gas and methane production values were halved, averaging at 0.27 l/l/d and 0.13 l/l/d respectively. Water dilution and NaHCO<sub>3</sub> addition boosted these levels to 0.5 l/l/d and 0.23 l/l/d until temperature fluctuations (weeks eight and nine) heavily impacted gas productivity resulting in a drop in both gas and methane production to 0.36 g/l/d and 0.16 g/l/d. Methane percentages (Figure 5.9) remained stable at quite high levels averaging at 47% throughout the entire reactor operation. This suggests that methane percentage might be indicative of an effective anaerobic fermentation but only when used in conjunction with other process parameters. This is validated by the fact that after the process restart and before the water dilution and NaHCO<sub>3</sub> addition, methane percentage was kept stable around 47%. However, pH, daily gas volume production and alkalinity indicated that the system was not stable by having values outside the stability range that was identified during Chapter 4 for stable operation.

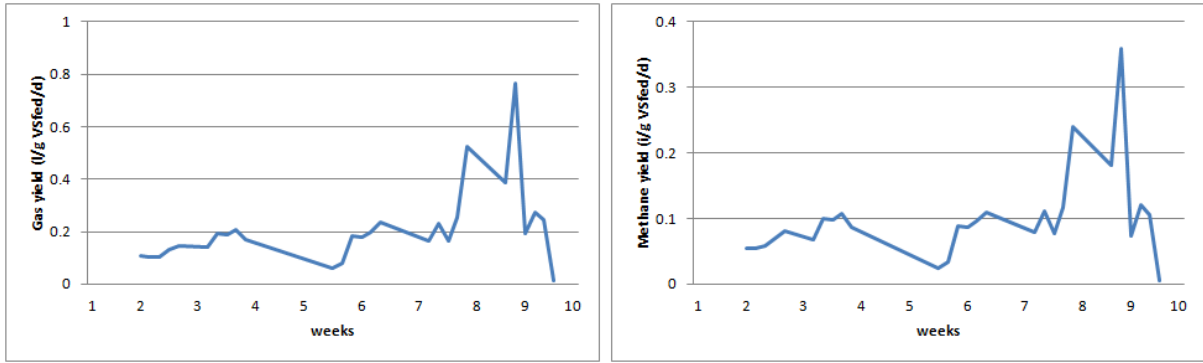


**Figure 5.8** Gas and methane production during FT1 operation



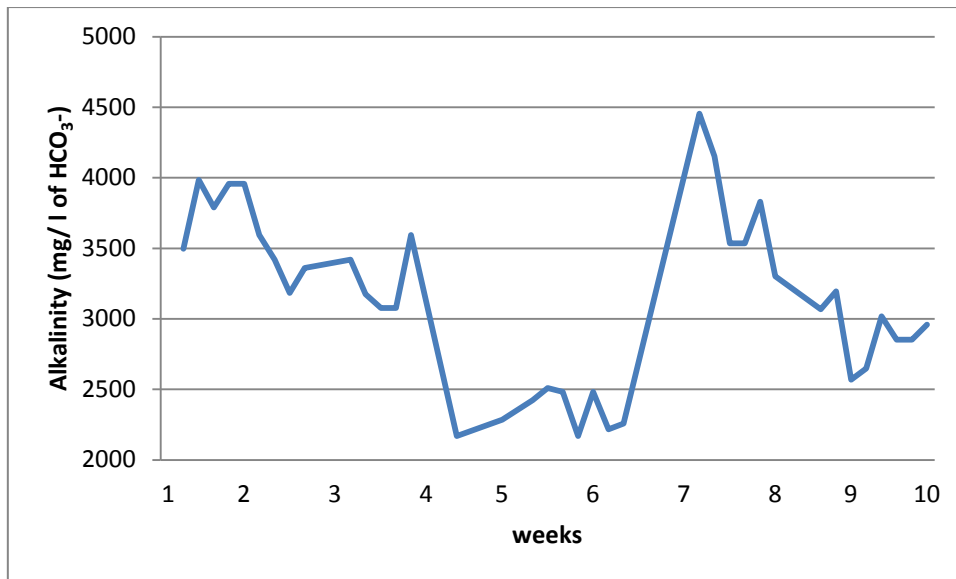
**Figure 5.9** Methane percentage during FT1 operation

Gas and methane yield (Figure 5.10) remained below 0.2 l/g of VSfed/d and 0.11 l/g of VSfed/d respectively even after week four. Buffer addition resulted in a yield increase that reached 0.76 l/g of VSfed/d and 0.36 l/g of VSfed/d respectively during week eight. However, temperature fluctuations occurring in week eight resulted in minimisation of biogas yield.



**Figure 5.10** Gas and methane yield during FT1 operation

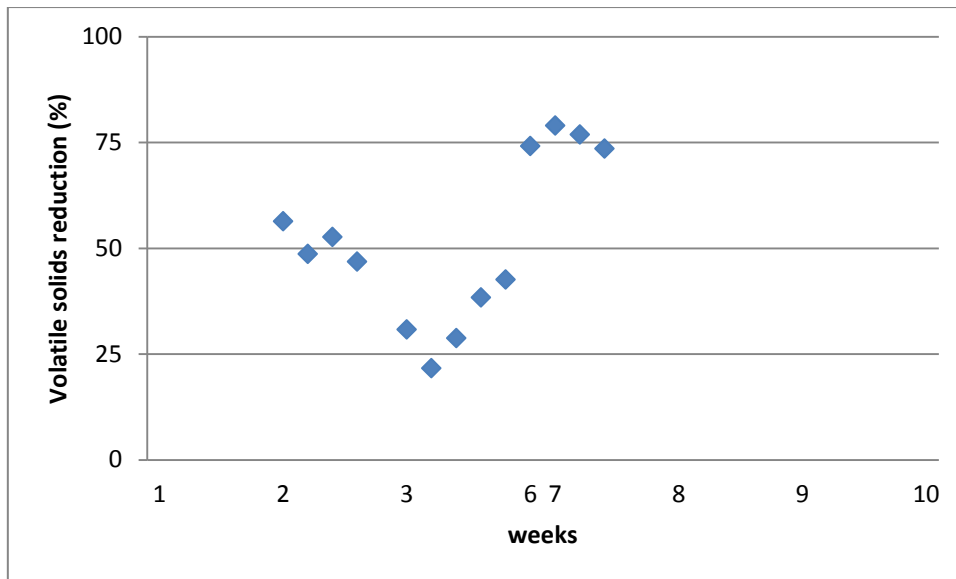
Since the beginning of the experiment alkalinity (Figure 5.11) was below 4000 mg/l of  $\text{HCO}_3^-$  and during the first three weeks remained between 3077 mg/l of  $\text{HCO}_3^-$  and 3985 mg/l of  $\text{HCO}_3^-$ . These alkalinity values are lower than values that dictated stable operation in Chapter 4 where digester stability occurred when alkalinity was above 3500 mg/l of  $\text{HCO}_3^-$ . However, in this case gas production was steadily increasing until the reactor had to be restarted (week four) and methane percentages averaged at 52% not indicating process imbalance. This suggests that a biogas reactor with higher capacity is more stable and can maintain stability at lower alkalinity values than smaller scale reactors. From week four to week six alkalinity values were below 2500 mg/l of  $\text{HCO}_3^-$  and followed the positive pH trend when  $\text{NaHCO}_3$  and water dilution were applied to the system reaching 4454 mg/l of  $\text{HCO}_3^-$ . Temperatures fluctuations had a negative impact on alkalinity that started to drop after week eight below 3000 mg/l of  $\text{HCO}_3^-$ .



**Figure 5.11** Alkalinity during FT1 operation

Volatile solids reduction (Figure 5.12) ranged between 22%-79% after the water dilution and NaHCO<sub>3</sub> addition. An OLR reduction from 5.3 g/l/d to 3.9 g/l/d during weeks two and three resulted in a VSR decline which is in accordance with the results from Chapter 4 where higher reduction rates had the same loading rate trend especially during stable operation. However, after week seven VSR reached 79 % when OLR was around 2.5 g VS/l/d much less than during the first weeks of operation. This is probably due to the buffering addition and water dilution that aimed to boost digester recovery.

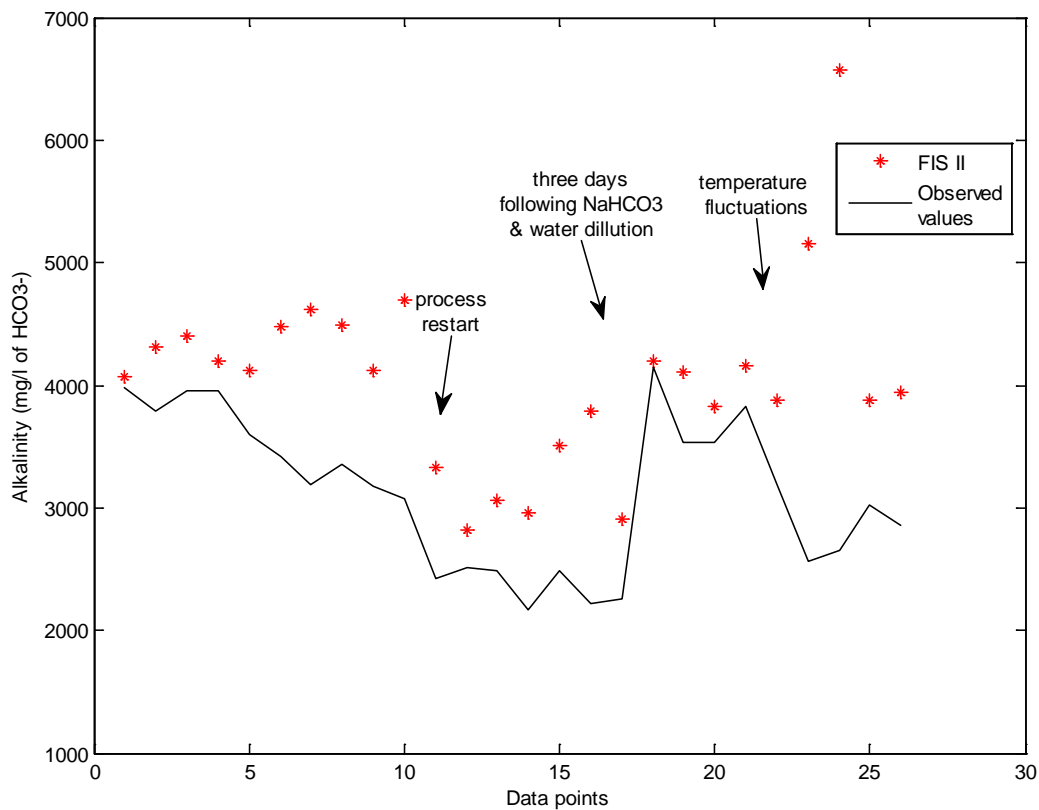




**Figure 5.12** Volatile solids reduction percentage during FT1 operation

### 5.3.2 FIS II evaluation during FT1 operation

FIS II that was introduced in Chapter 4 was utilised to predict alkalinity levels during FT1. FIS II predicted alkalinity based on daily pH, daily gas volume/reactor volume, daily pH difference and daily difference in gas volume/reactor volume. FIS II performance was evaluated based on the coefficient of determination, MAE, Bias, IA, NMSE and FB (Equations 3.4- 3.9). Data from process restart due to accidental loss of working volume, data following water dilution and  $\text{NaHCO}_3$  addition and alkalinity evolution during severe temperature fluctuations were utilised during the evaluation. Fuzzy model predictions are depicted in Figure 5.13.



**Figure 5.13** Observed and predicted alkalinity values using FIS II

FIS II alkalinity predictions are slightly higher than actual alkalinity values. This might be due to the fact that the system appeared to be stable even while alkalinity was lower than 3500 mg/l of HCO<sub>3</sub><sup>-</sup> as mentioned in 5.3.1. By utilizing the complete dataset, FIS II performance is adequate (Table 5.2) by having a poor R<sup>2</sup> value, acceptable MAE and bias values, a relatively normal IA=0.45 and slightly high NMSE and FB values. However, since temperature fluctuations result in a cessation of biogas production it can be clearly seen that severe differences in prediction values exist mainly when system operation under temperature fluctuations occurred. The fuzzy model might not have responded in a positive manner after the process was restarted, but was able to closely predict alkalinity the days that followed the combination of water dilution and NaHCO<sub>3</sub> addition. Therefore, if the five last data points are removed from the evaluation process, FIS II performance can be characterised as good. Considering the fact that it was designed based on data from

different digester set-up,  $R^2 = 0.54$  and  $MAE = Bias = 587$  indicate a slight deviation from the actual data.

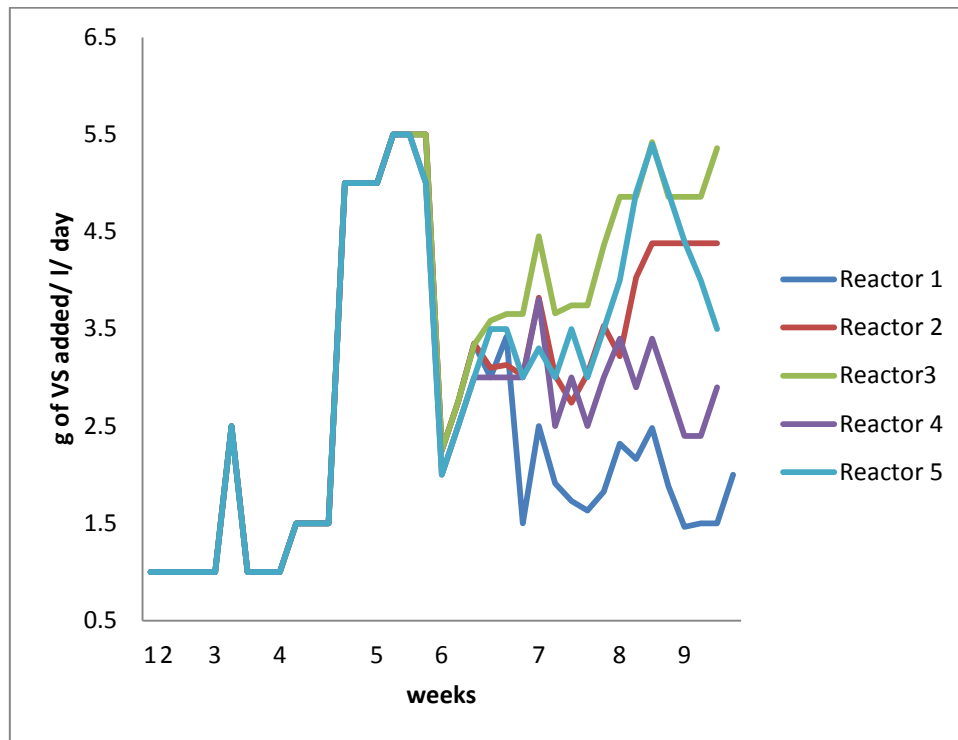
Performance indices	FIS II	
	A	B
$R^2$	0.099	0.54
MAE	762	587
Bias	762	587
IA	0.45	0.646
NMSE	0.089	0.046
FB	0.2587	0.2

**Table 5.1** Alkalinity performance for FIS II ((A) full dataset, (B) dataset without temperature fluctuation data)

### 5.3.3 CT1 and CT2 reactor operation

Experiments CT1 and CT2 lasted for approximately nine weeks. The aim was to test FIS II and the proposed control approaches (fuzzy logic for CT2 and rule-based for CT1) using reactors of different configuration and inputs as substrate. This would test the FIS II ability to predict alkalinity in reactors of different configuration than the ones utilised in its design and also to check the suitability of the proposed controllers.

OLR was varied manually during the first five weeks of operation and control approaches I and II were applied during the next four weeks of operation. The loading rates were identical for all five reactors during the first five weeks after which the proposed control methodologies were applied (Figure 5.14). From week five to week nine, reactors 1-3 were controlled by control strategy I and reactors 4-5 by control strategy II. OLR averaged at 1.25 g VS/l/d during the first three weeks to allow sufficient time for the immobilisation of the support media and was gradually increased to 5.5 g VS/l/d until week five aiming to increase biogas production. From week five to week nine OLR was varied individually for each reactor based on the two control strategies ranging between 1.5 g VS/l/d and 5.42 g VS/l/d.



**Figure 5.14** OLR during CT1 and CT2 operation

The impact of OLR to pH was positive in all reactors. pH remained at values below 7 (Figure 5.15) during the first ten days of operation despite the fact that OLR was kept at minimum (1.25 g VS/l/d). This was expected since the reactors were still trying to reach a stable state. pH started rising and reached a value between 7.07 and 7.18 in all reactors at the beginning of week three. From week three to week nine (when experiments CT1 and CT2 ended) the controllers were able to maintain stable pH values inside all reactors. pH remained above the bottom pH limit of 6.9 and within the range where maximum gas production occurs (6.9-7.42). These limits were established based on the experiments presented in Chapter 4. The only exceptions were the pH values recorded for reactor 1 during week six when pH was between 6.81 to 6.86. These low pH values were probably the result of an accident that took place the day that the first drop in pH was recorded. The gas outlet tube got blocked due to which half of the substrate inside the reactor was lost since a part of the reactor got detached. This was the probable cause for the pH decrease from a value of 7.12 to 6.86.

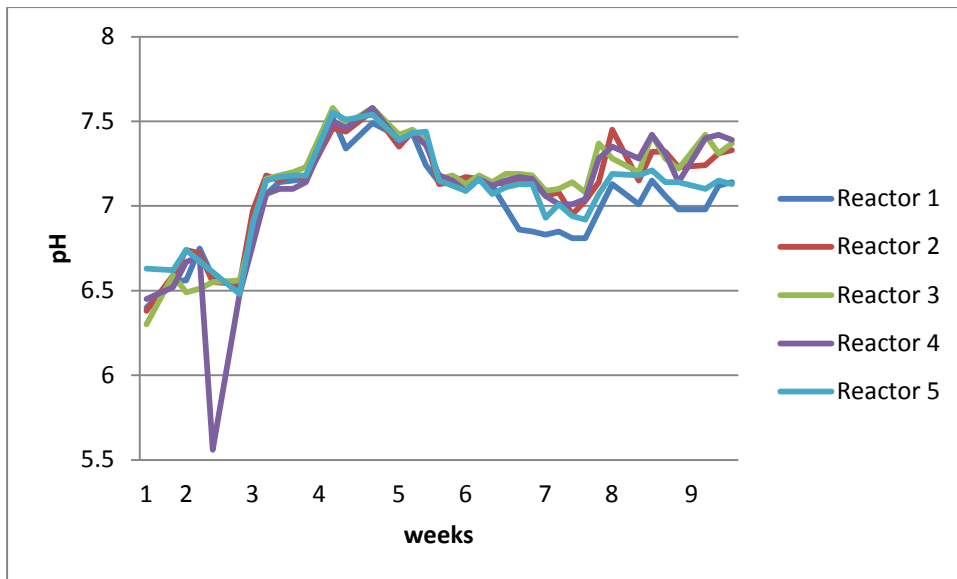


Figure 5.15 pH during CT1 and CT2 operation

Gas and methane production (Figure 5.16) remained at relatively low levels averaging between 0.26-0.35 l/l/d and 0.07-0.12 l/l/d respectively after the controller application. Additionally, methane percentages (Figure 5.17) were kept below the limit where a biogas system is considered stable (around 45%). The average methane values for all reactors were between 30%-33% CH<sub>4</sub> from week four to week nine. These values indicate that the reactors never reached a stable state that would enable the maximisation of biogas production. Even in reactor 1 (CT2) and reactor 4 (CT1) where the OLR was kept at lower levels than the other reactors (with average values of 2.17 g VS/l/d and 2.87 g VS/l/d respectively) gas production was equally low.

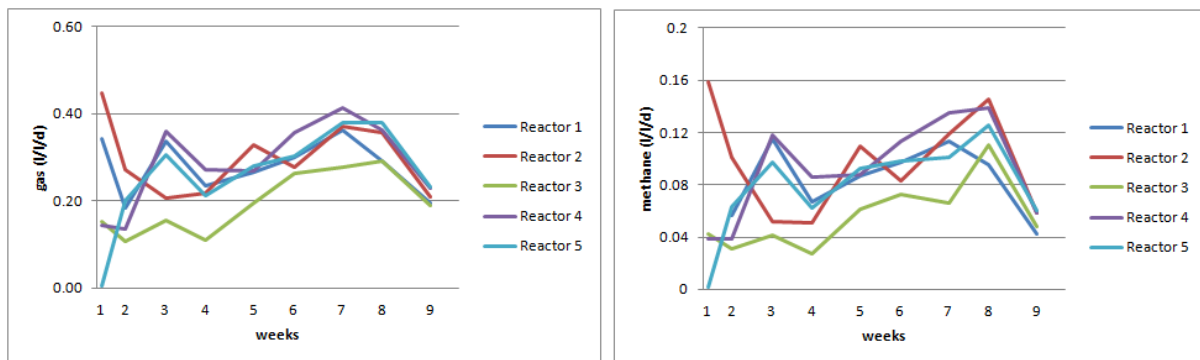
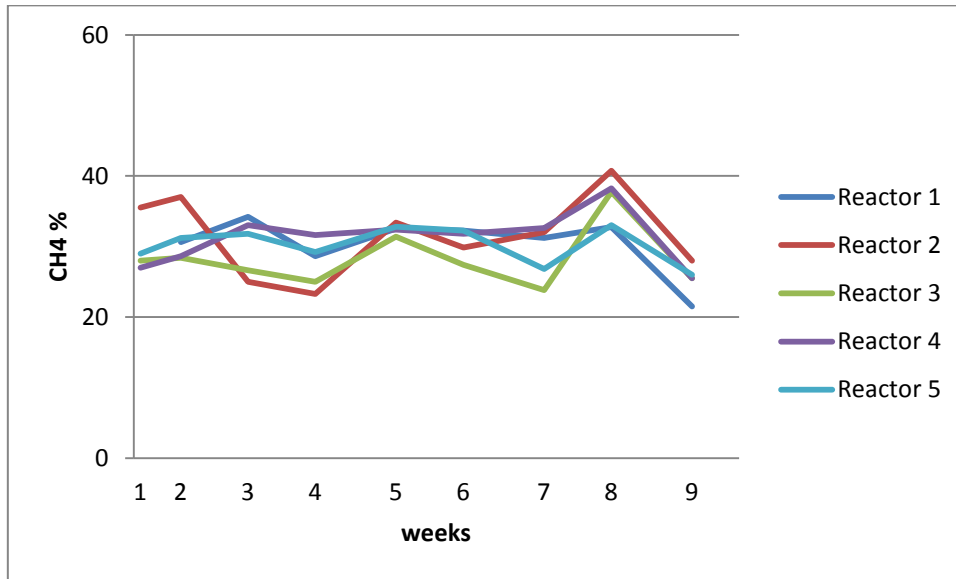


Figure 5.16 Weekly average gas and methane production during CT1 and CT2 operation



**Figure 5.17** Weekly average methane percentages during CT1 and CT2 operation

Alkalinity levels (Figure 5.18) started rising after two weeks of operation peaking between 5673 mg/l of  $\text{HCO}_3^-$  and 6349 mg/l of  $\text{HCO}_3^-$  after three weeks of CT2 and CT1 operation. Alkalinity remained at similar levels during week four. When controller strategy I and controller strategy II were applied, alkalinity remained at desired stability levels (>3500 mg/l of  $\text{HCO}_3^-$ ) for reactors 2-3 (CT2) and reactor 4 (CT1). A drop in alkalinity was recorded for reactor 1 that was not due to the controller application after the sixth week when the loss of substrate occurred as explained above. A similar drop in alkalinity was observed for reactor 5 (CT1) later during the same week. This drop was initially caused by oxygen intake. Gas pressure created by the outlet gas tube blockage resulted in the removal of the top of the reactor. However, alkalinity remained below 3000 mg/l of  $\text{HCO}_3^-$  for the remainder of the experiment. Such low values were due to relatively high loading rates that were applied by the rule-based controller averaging at 3.88 g VS/l/d.

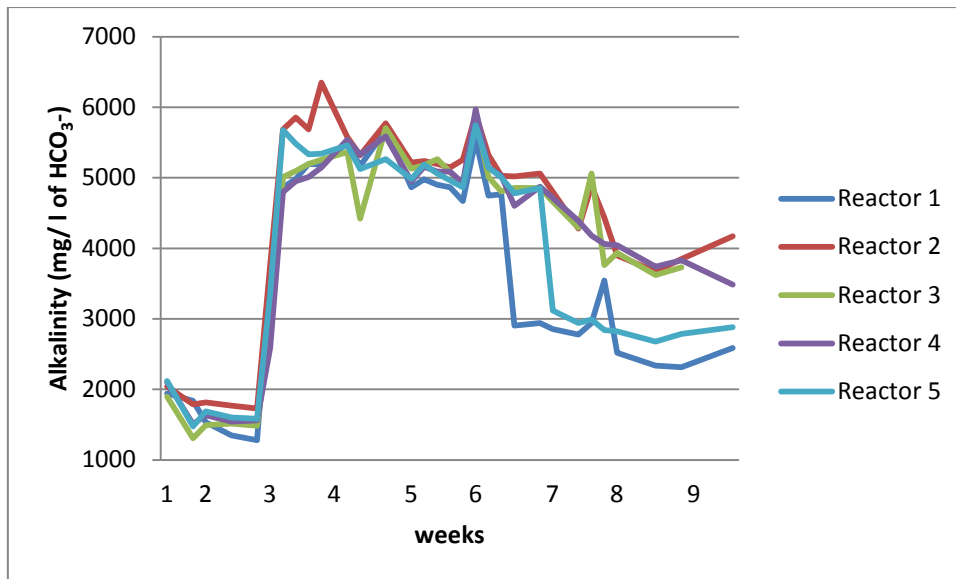


Figure 5.18 Alkalinity during CT1 and CT2 operation

### 5.3.4 Control strategy and FIS II evaluation during CT1 and CT2 operation

Alkalinity predictions of FIS II despite the accidents that occurred during the operation of reactors 1, 3 and 5 were quite accurate. The FL soft sensor was able to provide both the fuzzy logic controller and the rule-based controller with predicted values that deviated slightly from the observed values. Increased accuracy was present when alkalinity was between 2900 mg/l of HCO<sub>3</sub><sup>-</sup> and 4660 mg/l of HCO<sub>3</sub><sup>-</sup> in all reactors. Predicted alkalinity values outside this range might suggest that changes in the fuzzy structure should be applied. Especially for alkalinity values below 2700 mg/l of HCO<sub>3</sub><sup>-</sup> the maximum differentiation between observed and predicted values recorded was approximately 1700 mg/l of HCO<sub>3</sub><sup>-</sup> for reactors 1, 2 and 5 and 3450 mg/l of HCO<sub>3</sub><sup>-</sup> for reactors 3 and 4. However, this fact was taken into consideration during the formulation of both controller approaches and when the predicted alkalinity is at such low levels minimum OLR changes are applied.

The goal of the controller OLR variations was to keep alkalinity within the optimum operating limits for system stability and biogas maximisation as suggested in Chapter 4 (3500 mg/l of HCO<sub>3</sub><sup>-</sup> and 4300 mg/l of HCO<sub>3</sub><sup>-</sup>). The performance indices for FIS II when applied to all reactors indicated relative small variations between predicted and observed values (Table 5.3). These indices are definitely improved as far as alkalinity values within the optimum alkalinity operating range are concerned. FIS II application was most successful in reactor 2 as suggested by a

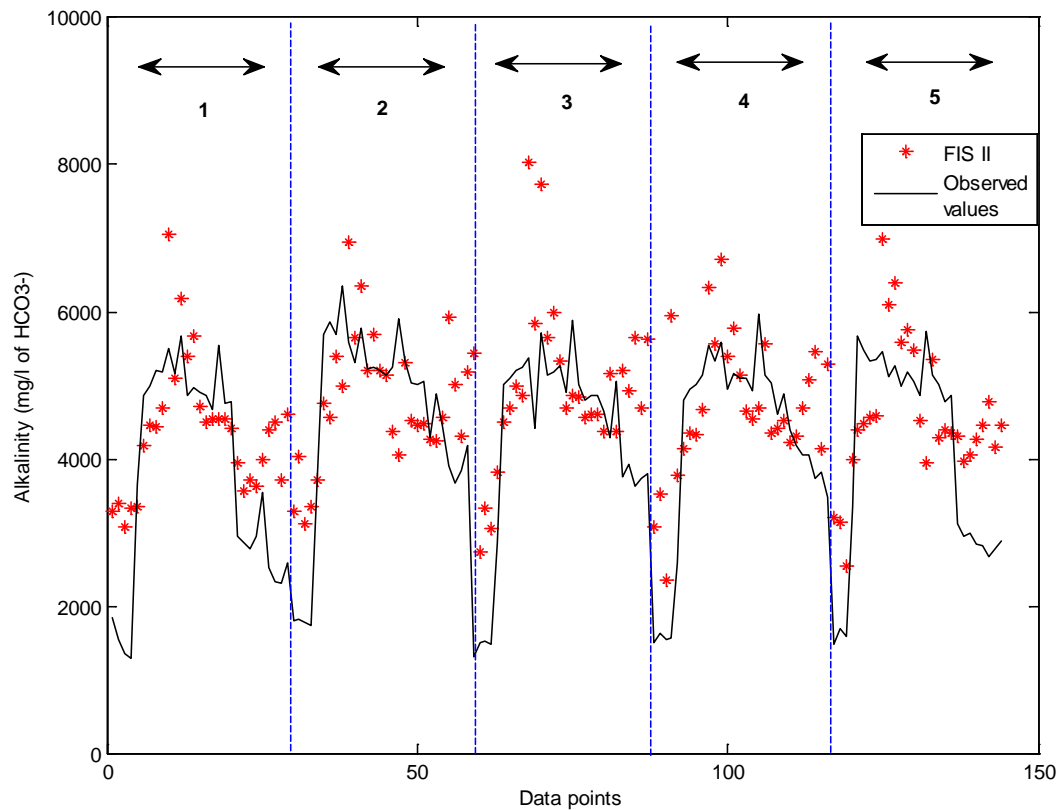
very low bias value of 154 and an equally low value for MAE of 660. An IA value of 0.72 and a NMSE value of 0.06 for all reactors indicate a pretty good correlation between predicted and observed values. FB was not as low as expected as it not very close to 0 (0.1).

Performance indices	Reactors					
	1	2	3	4	5	1-5
R <sup>2</sup>	0.541	0.389	0.294	0.284	0.43	0.381
MAE	754.39	660.75	845.501	706.617	853.722	763.574
Bias	468.876	154.12	584.53	373.508	411.376	398.392
IA	0.763	0.748	0.678	0.701	0.741	0.727
NMSE	0.063	0.046	0.072	0.062	0.06	0.06
FB	0.14	0.04	0.155	0.101	0.116	0.11

**Table 5.2** Alkalinity performance for FIS II during CT1 and CT2 operation

Predicted and observed alkalinity values for reactors 1-5 are depicted in Figure 5.19. All data appear in chronological order for each reactor where it can be clearly seen that alkalinity followed a similar trend in all reactors.





**Figure 5.19** Observed and predicted alkalinity during CT1 and CT2 operation

During CT2 and CT1 when controller strategy I and controller strategy II were implemented pH and alkalinity remained within the desired operated ranges for digester stability and increased biogas production. When values deviated from the desired range it was a result of accidents that took place during the operation of reactor 1 and reactor 5. Therefore it can be stated that both controller applications were successful. However, the system was never stabilised which is reflected in the low biogas production levels. This is probably due to the fact that no mixing was present and the fact that loading rates should differ when operating a biogas reactor with cellulose instead of cow slurry (Chapter 4).

It was quite difficult for cellulose to be dissolved especially when loading rates were above 3.5 g VS/l/d. During the feeding incidents and sampling, substrates appeared to contain huge amounts of undissolved cellulose, as suggested by substrate colour and the sediment that remained after centrifuging samples while preparing them for alkalinity analysis. Even reactor 4 (CT1), where the lowest loading rates were applied compared to the other reactors, exhibited the same

behaviour. On the other hand, feeding rates of less than 1.5 g VS/l/d may not maximise biogas production. Especially since a slightly lower average OLR value of 1.25 g VS/l/d was applied during the first two weeks to drive the system to start producing gas.

To sum up, both controller strategies were able to maintain alkalinity and pH within the desired ranges. Biogas production was not maximised due to the absence of mixing, the fact that different loading rates should be probably applied to reactors treating cellulose or the fact that the digesters should be supplemented with other nutrient sources to enhance their operation and stability. These conclusions are in accordance with other studies that showed:

- Micro and macro nutrients can influence the degradation rate and bacterial activity under thermophilic conditions (Golkowska & Greger 2013).
- Cellulose must be first liquefied or hydrolysed to produce methane and the successful initial hydrolysis function highly contributes to the rate of stabilisation and methane fermentation (Parkin & Owen 1986).
- Mesophilic cellulose digestion can be more effective than thermophilic anaerobic digestion (Yang et al. 2004).
- Gas production is limited for high OLRs both for mesophilic and thermophilic cellulose digestion (batch experiments with organic loading >16.3 g VS/l) (Golkowska & Greger 2013). This digestion retard combined with low degradation levels (77-89%) lead to the conclusion that system overloading can be more easily achieved for single-component substrates. In addition, these systems are susceptible to undesired system conditions that include nutrient shortage and ammonia inhibition.
- Permanent acidosis can be avoided even under extremely high loading rates (up to 34.3 g VS/l) applied during batch tests (Golkowska & Greger 2010). High OLRs result in prolonged degradation times and OLRs higher than 22.9 g VS/l resulted in dropping pH up to 6.83.

Therefore, although both controllers achieved what was expected in terms of maintaining process parameters within the desired ranges by supplying each reactor with different OLRs, neither system reached stability or was even close to maximisation of biogas production. So, loading rate adjustment should probably be

performed in reactors treating cellulose since system performance is different compared to reactors treating manure.

## 5.4 Conclusions

An evaluation of a fuzzy logic inference system (FIS II) to determine alkalinity using different digester configurations was conducted. During experiment FT1 a larger reactor than those used in the development of FIS II was utilised to test the proposed FIS. FIS II was able to predict alkalinity values with sufficient accuracy even when the digester was diluted with water and buffered with bicarbonate. It was also concluded that since gas production was used as an input, the system can be affected by temperature fluctuations. Therefore temperature has to be kept stable to in order to obtain meaningful alkalinity predictions.

Two controller strategies were implemented based on FIS II predictions. Control strategy I included a Mamdani FIS with two inputs (alkalinity and daily difference in alkalinity) and control strategy II was based on a rules-based system that worked with the same inputs. Both controllers were developed using the same design principles and both controlled the rate of change in OLR. They were tested against reactors containing cow slurry treating cellulose (experiments CT1 and CT2). Both control approaches worked in the same way and managed to maintain alkalinity within the ranges that were identified in Chapter 4 as optimum operating ranges (>3500 mg/l of  $\text{HCO}_3^-$ ) for process stability and biogas maximisation. Only interruptions for reactor 1 and reactor 5 caused alkalinity to drop below operational levels. However, gas production was relatively poor. This was most probably not due to the controller operation, but can be attributed to the absence of a consistent mixing system (since the mixing pump failed), the need for additional micro and macro nutrients, the absence of an initial hydrolysis function for cellulose and the fact that gas production for high OLRs is limited and can lead to prolonged degradation times in systems treating cellulose (5.3.4).

## Chapter 6 Conclusions and future work

Improving the performance of anaerobic digestion by process control proved to be a challenging task since changes in environmental conditions can heavily impact process stability, especially when operating under thermophilic temperatures. Process stability and optimisation can be achieved through the development of an online monitoring and control system. Reliable process monitoring indicators, as identified in Chapter 1, have to be utilised in the development of a cost-effective process control system. Process indicators should easily provide useful information and ideally indicator selection should lead to successful control of anaerobic digestion processes operating across different feedstocks. The conclusions drawn from the present work are as follows:

- Alkalinity was identified as an important process stability indicator especially for systems with low buffering capacity which was supported by the literature review presented. Also, fuzzy logic was identified as the data-driven control technique suitable to infer alkalinity. Data collected during the work presented in (Partner N° 2, Rothamsted Research 2010) were used in Chapter 3 to design a fuzzy logic system that inferred alkalinity based on pH, electrical conductivity (EC) and organic redox potential (ORP). Two first order Sugeno fuzzy inference systems (FIS1 and FIS2) were developed and compared with the two multiple linear regression models (MLR 1 and MLR2) that were used to infer alkalinity in (Partner N° 2, Rothamsted Research 2010). FIS1 and FIS2 alkalinity predictions were proved to be more accurate compared to MLR1 and MLR2 predictions. It was proven that by increasing the training fuzzy model database the predicted model values achieved a better convergence with the observed values. A FIS trained with less data-points was proven to be more accurate than a multiple linear regression model designed with a larger database. Also, systematic recording (ideally daily) of observed alkalinity values was suggested as a means to improve fuzzy model predictions.
- Reactors without support media were able to withstand maximum loading rates between 3- 3.5 g VS//d whereas reactors with support could produce

higher amounts of gas when the loading rate varied between 4- 5 g VS/l/d without being destabilised. Optimum pH operating ranges were between 7.1-7.3 and 6.9-7.2 for reactors with and without support media respectively. pH values <6.9 indicated process imbalance in all reactors. It was found that biomass media do not have a huge impact in enhancing biogas production but can highly enhance stability. However, sponge provided a more suitable environment for the growth of methanogens. Stability was guaranteed in all reactors for alkalinity values above 3500 mg/l of  $\text{HCO}_3^-$  and biogas production maximisation occurred between 3500 mg/l of  $\text{HCO}_3^-$  and 4500 mg/l of  $\text{HCO}_3^-$  for reactors without biomass support media and between 3480- 4300 mg/l of  $\text{HCO}_3^-$  for reactors with support media.

- Two first order Sugeno fuzzy systems were developed during different periods throughout the experiments presented in Chapter 4 trying to capture alkalinity behaviour. Instead of having pH, EC and ORP as inputs, pH, gas volume/reactor volume, daily pH difference and daily gas volume/reactor volume difference were selected as the new inputs. The second FIS (FIS II) that was developed using a larger database than the first FIS (FIS I) provided more accurate alkalinity predictions for future applications. FIS II was characterised by quite good MAE and bias values of 466.53 mg/l of  $\text{HCO}_3^-$  and an acceptable value for  $R^2= 0.498$  for the reactor containing sponge. NMSE was close to 0 with a value of 0.03 and a slightly higher FB= 0.154 than desired. During the design of FIS I and FIS II the fuzzy systems that exhibited a high  $R^2$  were characterised by a high MAE value and vice versa. Since low bias and MAE values are considered to be more desirable, the developed FISs presented in this work focused more on keeping these values as low as possible. Also, FIS II responded positively to disturbances such as  $\text{NaHCO}_3$  reactor addition but predicted alkalinity values declined by 1300 mg/l of  $\text{HCO}_3^-$  on average when water dilution was performed.
- FIS II was tested by operating a 25l reactor treating cow slurry supported by sponge (Chapter 5). Data from process restart due to accidental loss of working volume, data following water dilution and  $\text{NaHCO}_3$  addition and

alkalinity evolution during severe temperature fluctuations were utilised during the evaluation. Temperature fluctuations resulted in poor alkalinity predictions. However, predicted alkalinity values followed the observed values closely during the days following water dilution and  $\text{NaHCO}_3$  addition. By excluding the temperature fluctuation period from the evaluation process,  $R^2 = 0.54$  and  $\text{MAE} = \text{Bias} = 587$  indicated a slight deviation from the actual data.

- A rule based system and a fuzzy logic system were designed to regulate the OLR during the operation of 6.46l cylindrical reactors treating cellulose that used sponge as the biomass support media (Chapter 5). Alkalinity and daily difference in alkalinity were used to set the daily OLR variation. The goal of the controller OLR variations was to keep alkalinity within the optimum operating limits for system stability and biogas maximisation as suggested in Chapter 4. An IA value of 0.72, a NMSE value of 0.06 and  $\text{MAE} = 763$  for all reactors indicate a good correlation between predicted and observed values. Both controllers managed to maintain alkalinity and pH within the desired stability and biogas maximisation ranges. However, the systems were never stabilised which is reflected in the low biogas production levels. Poor cellulose degradation was also reflected in the substrate colour and the sediment that remained after centrifuging samples for alkalinity analysis.

Further research should focus on developing individual fuzzy systems that would predict alkalinity for hydrolyzed substrates other than cow slurry. Then depending on the type of substrate and after reformulating OLR variations, the fuzzy system should drive the controllers in a similar manner to the one presented in this work aiming to maximise biogas productivity while maintaining stability. More specifically, since different substrates have different optimum process parameter operating ranges (higher gas production volumes, buffering capacity and pH), OLRs should be adjusted based on alkalinity predictions and according to the substrate utilised. Also, embedding EC and ORP in the inference mechanism might have a positive impact on alkalinity predictions since these parameters might not exhibit such value variations as pH and gas production. Finally, this work should be expanded to two stage systems where hydrolysis takes place in the first stage and

methanogenesis in the second stage. By controlling both stages separately, a more robust system operation can be achieved.

## **Appendix A: Operation of a high-rate biogas reactor**

### **A.1 Introduction**

Two different digester designs were implemented to investigate the AD process. Data collection of important process parameters: pH, ORP, EC, gas volume, gas composition, and solid destruction rate aimed to provide sufficient information for the design of a controller. Based on the online measurements (pH, ORP, EC, temperature) and the evaluation of off-line measurements (gas volume, gas composition, solid destruction rate) a fuzzy logic based software sensor that would infer alkalinity could lead to the development of a controller that would regulate the OLR. However, system operational failures led to unsuccessful implementation of that design. The designs and lessons learnt from the unsuccessful digester operation are presented in this section.

### **A.2 Digester design I**

The anaerobic biogas reactor consisted of three 120l cylindrical reactors that were connected in series through a system of pipes. Support surfaces in the form of reticulated polyurethane foam were attached to the sides of the each reactor and also to an inner cylinder through which the substrate would be pumped and mixed (Figure A.1).

An immersion heater with a 3 kW thermostat was used to heat up the system with a system of induction heating coils, located inside each tank, with hot water to enable the reactors to reach and maintain the required temperature. Altech CPS 130-5 pumps were responsible for regulating the hot water circulation. Horstmann F222M motorised valves were selected to control the water stream from entering the coil systems present in each tank.

Each digester was mixed with a Zoeller Waste-Mate 260 series submersible pump (Zoeller Pump Company, 3649 Cane Run Rd., Louisville, KY 4021) that was located at the bottom of each tank inside a 28.85l cylindrical shaped box. Experiments conducted with water showed that the pumping speed was 170l water/min.



Ch-air CH042 pneumatic actuators (Figure A.1) were programmed accordingly to control the flow of the substrate between and within the three tanks during mixing and while applying the software specified substrate transferring methods.

Two reed switches were installed in each tank to provide measurements with respect to the amount of substrate present in each tank. Since the working volume (90l) would be less than the size of the reactors to allow headspace, the level sensor located at the top would be activated when  $88393\text{cm}^3$  of substrate were inserted in each tank. The bottom level sensor corresponded to  $66786\text{cm}^3$ . So, by having two level sensors the OLR could be calculated and the tanks would be protected from inserting higher amounts of material.

One sample port was available in each reactor to allow for sample collection and one port at the top would allow for gas volume measurement.

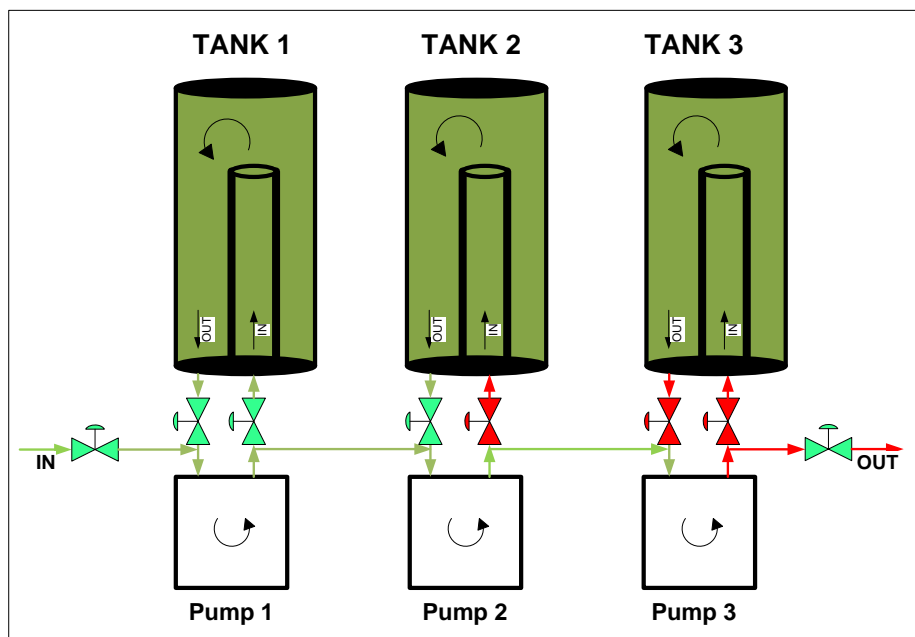


Figure A.1 Anaerobic digester design I

### A.2.1 Sensors- Data acquisition

Sensorex S8000 series pH and ORP electrode platforms were used to measure pH and ORP. Sensorex CS 650TC series model (Sensys Limited, Unit 9 Pond Close, Walkern Road, Stevenage, Herts, SG1 3QP) was used to measure electrical conductivity and provide measurements through a CT1000 PT transmitter.

Nine sensors (three of each kind) were located inside the boxes that contained the pumps.

Three thermocouple sensors were located at different places inside each cylinder to provide an accurate temperature profile (Figure A.2).

Gas volume measurements were conducted off-line using KumHo Metertech Inc. KG-2 gas meters and CH<sub>4</sub>% values were also recorded off-line using a Crowcon Triple + plus IR gas monitor (Crowcon Detection Instruments Ltd, 2 Blacklands Way, Abingdon Business Park, Abingdon, Oxfordshire OX14 1DY, UK).

All the sensor outputs were connected to Measurement Computing data acquisition devices. Measurement Computing USB-1208LS data acquisition devices received level and process sensor readings and were the means through which all control actions were applied. Temperature sensors were connected to Measurement Computing USB-TEMP temperature measurement devices. Finally, process monitoring, control and recording was carried out using National Instruments Labview version 11.0.

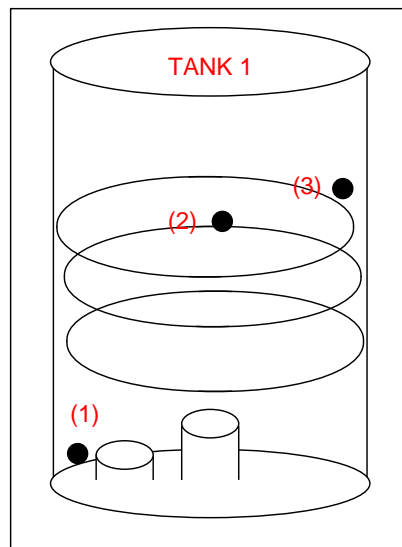


Figure A.2 Temperature sensors located in each tank

### A.2.2 Labview Architecture- System operation

Labview software was designed to operate on a manual feeding regime. The user could specify how often the unit would be fed and regulate the OLR by specifying the amount of time required between each feeding. The space between

the two level sensors provided information regarding the quantity of the material that was substituted in the digesters. The pumps and valves of the system were programmed to function automatically for the unloading and loading operation of the unit.

The developed software program recorded and displayed process parameter values (pH, ORP, EC and temperature) every minute and 30 seconds respectively. It allowed the user to have manual control over all stages of the process (tank mixing, feeding, temperature control, initial loading of the unit) to ensure that any process could be overridden at all times. Also, a live visualisation of the material inside the tanks was provided based on the pumping speed (the user could see the level of the liquid changing during loading and unloading the tanks).

All initial experiments were conducted with water and mixing was used to maintain the temperature at the required level of 55° C allowing for a strict  $\pm 0.5^{\circ}$  C deviation. This deviation is suggested for thermophilic anaerobic digestion processes (Tchobanoglous et al. 2003). A combination of ON-OFF and FL control was used to control temperature levels by stopping or allowing the hot water supplying the heating coils to flow and by varying the pump operating time respectively. The press of a button allowed the user to record the time of sampling from each tank and on-line measurements were recorded every second for 10 minutes since the press of the button in order to obtain accurate measurements during sampling.

The operating process flow chart is depicted in Figure A.3 and all the tabs that formed the front panel of the Labview software designed are available Figure A.4- Figure A.6.

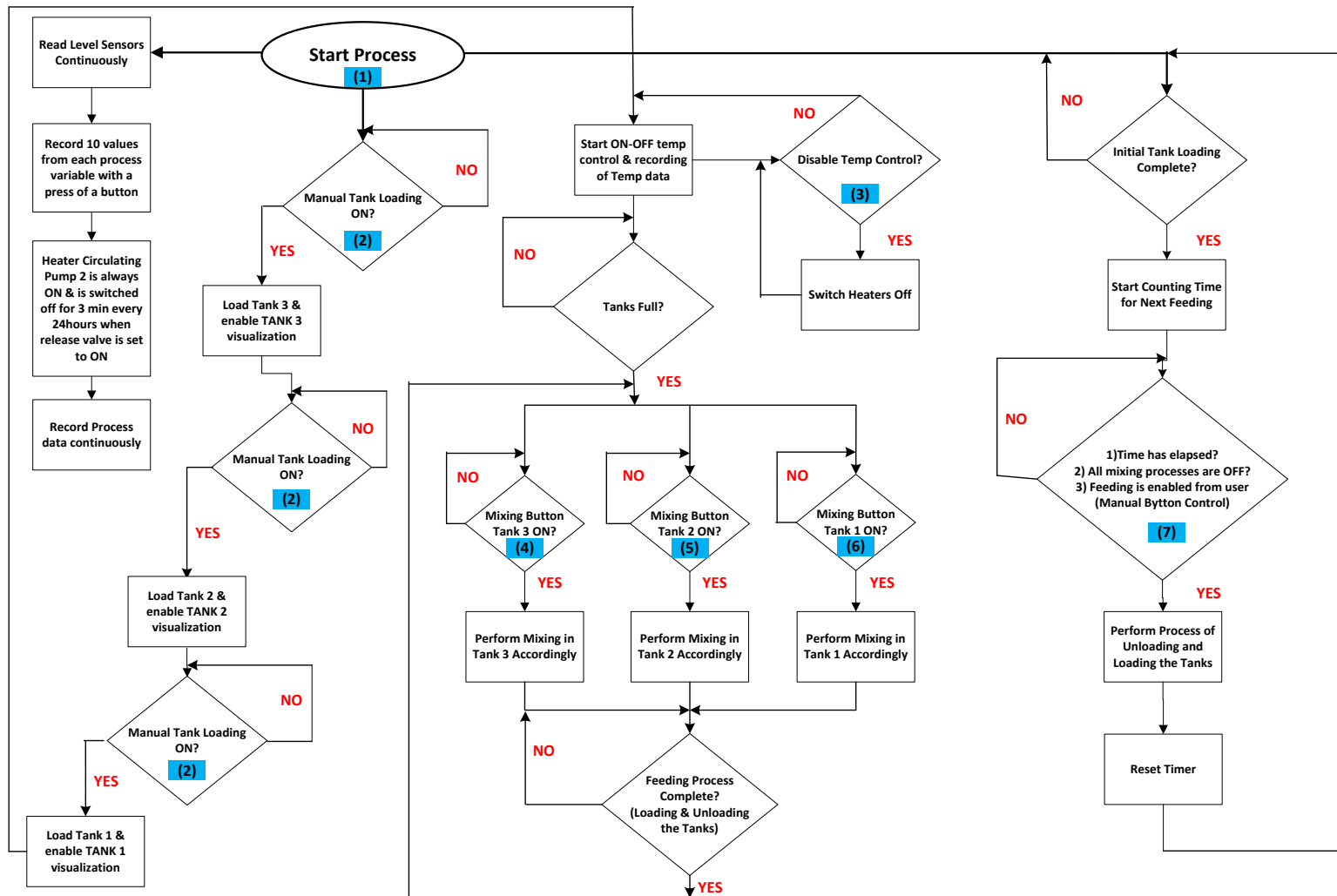


Figure A.3 Flow chart of the process operation. (1)- (7) correspond to the manual control buttons that appear in Figure A.4

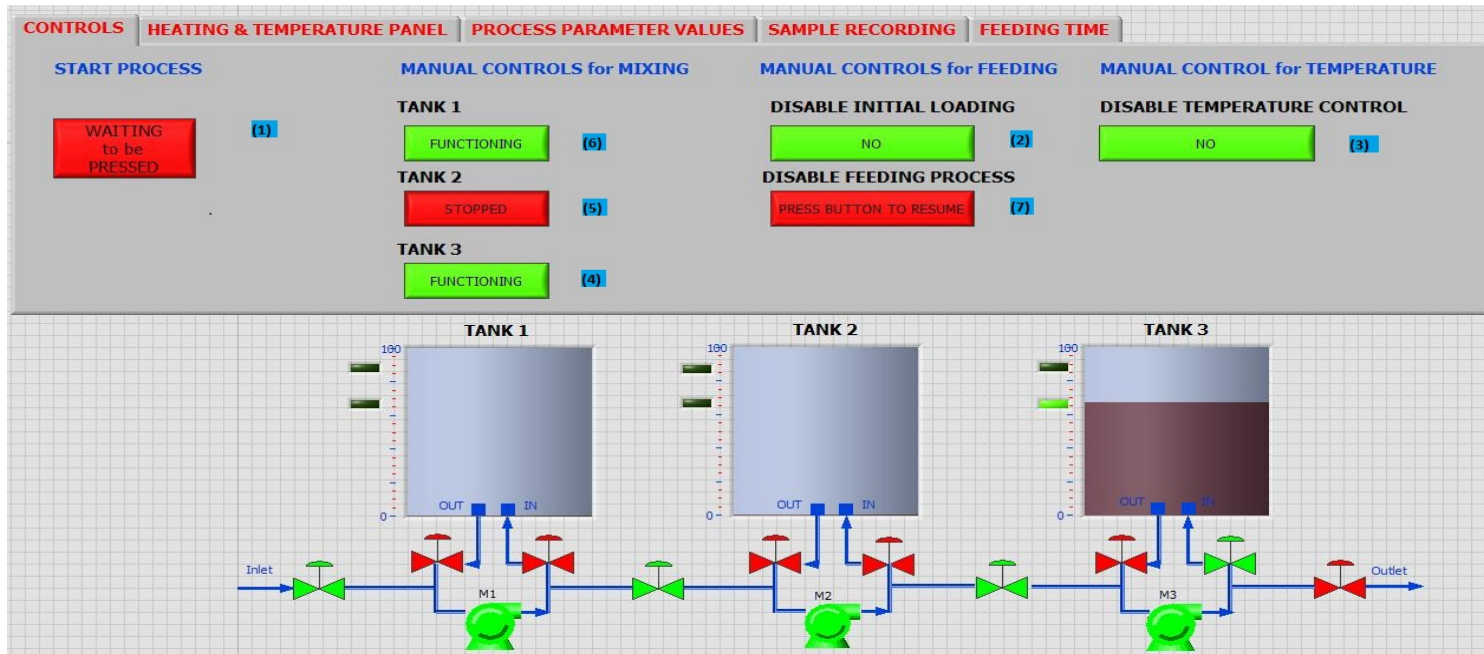


Figure A.4 Labview front panel showing the manual control buttons and process operation. When manual controls are disabled automatic control actions appear on the screen. Operating valves, pumps, level sensors are displayed in green color

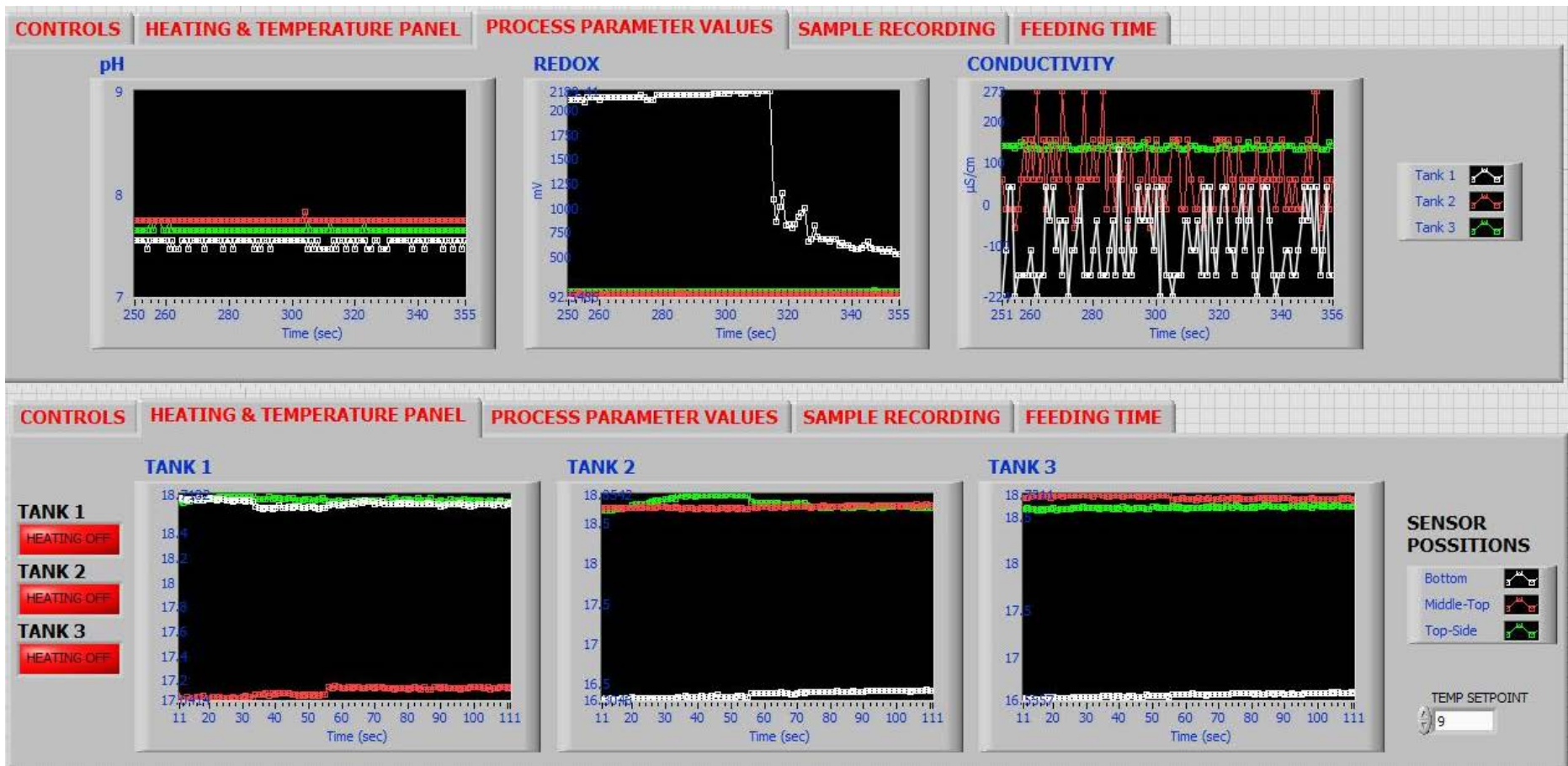


Figure A.5 'Heating and temperature' panel displays the heating status of each tank on the left (Heating ON, Heating OFF), and the temperature values of each sensor. The user can specify the set point that is normally set at 55°C. 'Process parameter values' tab displays pH, ORP and EC values for each tank.



**Figure A.6** 'Sample recording' tab contains a button to record continuous process parameter data while sampling and 'Feeding time' tab displays the elapsed time since last feeding. The 'Start feeding?' button enables the user to override the feeding regime that is configured at the background.

### **A.2.3 Results**

After the installation of the AD plant the pumps were unable to pump any material. Manure tends to form lumps. As a result, the submersible pumps would block and were unable to mix any of the cylindrical tanks. Therefore, the temperature inside the tanks was not uniform and the process of either loading or unloading had to be carried out manually by opening the top of the tanks.

Since mixing was the only way to maintain a stable temperature, temperature values varied between 40°C to 65°C inside each tank since the sensors were installed at different places inside the digesters. The sensors that were located at the top and close to the heating coil system displayed high temperature values.

Also, process parameter values could not be recorded. All the sensors were located inside the boxes that contained the pumps and with the absence of mixing they could not provide any accurate measurement. Therefore a need to replace the submersible pumps and to redesign the method of mixing arose.

### **A.3 Digester design II**

The anaerobic digester set-up was modified. The three submersible pumps were replaced by one progressive cavity pump made by Mono (model CML 263) that would be responsible for mixing all the tanks. The new system configuration is depicted in Figure A.7.



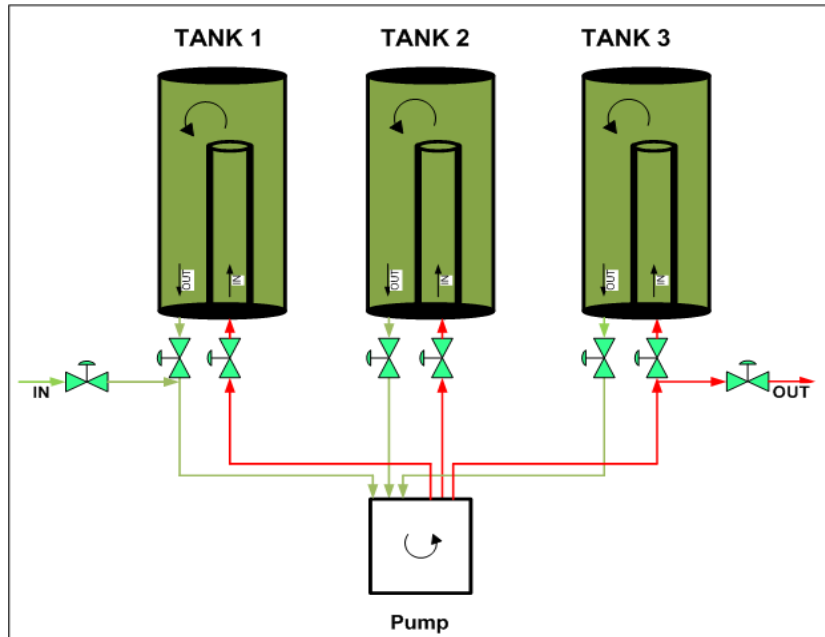


Figure A.7 Anaerobic digester design II

### A.3.1 System operation -Sensors- Data acquisition

The system operation was the same as for the first design with some slight modifications to accommodate for changes in the mixing regime since only one pump was available for mixing. Labview software was modified and ON-OFF control was used for temperature and all tanks were mixed for 20 minutes every hour. The new mixing regime was due to the new pump's lower flow rate (23 l/min).

Only three process parameter sensors were connected to the system (pH, ORP, EC) and data from each tank were recorded when the contents of a specific tank were mixed.

### A.3.2 Results

The unit was operated unsuccessfully for a short period of time (19/05/2012-12/08/2012) and consistent measurements could not be recorded. The experimental design operation failed due to the following reasons:

- The low speed of the pump resulted in having huge temperature variations inside the tanks. The temperature set-point was 55° C, however the temperature varied from 45°C to 60°C inside all tanks. Also, the temperature variation in all three tanks was different for similar mixing regimes which made it even harder to establish a uniform mixing approach.
- The substrate temperature profile in all tanks started to vary shortly after initializing the experimental work with cow manure. Therefore, a uniform strategy aiming to maintain stable temperature levels in all three tanks could not be applied. This behavioural difference was probably due to either leakages in the coil heating system or the unreliable performance of one of the Altech CPS 130-5 pumps.
- Since three sensors (pH, ORP, EC) were responsible for measuring the process parameters for all tanks, similar values were recorded for all tanks at all times mainly due to the flow rate of the pump.
- The level sensors were unable to detect the level of the slurry because they blocked due to slurry and scum accumulation on a regular basis. Even after cleaning the level sensors, one mixing cycle of maximum 10 minutes was enough to cause blockages and resulted in irresponsive behaviour.
- The level sensor failure resulted in abandoning the automatic loading regime and instead the feeding and unloading process had to be done manually by opening the top of the lids and exposing the anaerobic microbes to oxygen at regular intervals. This was a result of several overflowing incidents.
- The level sensor failure combined with the long pipe network that connected the feeding tank and the tanks together made it impossible to accurately calculate the OLR.
- The EC sensor was either not suitable for slurry operation or very sensitive to changes and provided a huge range of values even when samples were continuously recorded for a period of 2-5 minutes.

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