A MULTI-PARAMETER EMPIRICAL MODEL FOR MESOPHILIC ANAEROBIC DIGESTION

by

EMMANUEL CHUKWUMA OGBONNA

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Foreword

The work presented in this thesis is conducted at the School of Engineering and Technology, Science & Technology Research Institute, University of Hertfordshire, UK, from October 2010 to January 2015, under the supervision of Dr Rajnish Calay, Dr Rashid Ali, Dr Zhijun Peng and Dr Georgios Pissanidis.

The research work reported in this thesis sets out to develop a black-box (empirical) model for the mesophilic anaerobic digestion (AD) process. The goal is to have a better understanding of how physico-environmental operating parameters influence biogas and methane production. Experimental work is carried out to determine the effects of four operating parameters on biogas and methane production in a laboratory scale. In addition, the data acquired from the experiments is utilised to construct the mesophilic AD models for this research study, in MATLAB through System Identification method.

The introduction of this thesis in Chapter 1 presents the theme of the research work. It highlighted the irreversible impact of global energy production and consumption, especially, fossil fuels, on the environment and all life forms, as well as discussed the possible alternative energy sources that can mitigate these risks. In addition, this part discussed the environmental and health risks resulting from increasing municipal solid waste (MSW) generation due to rising human population and industrial advancement. Furthermore, it introduced the concept of AD, which is the process by which bacteria breakdown organic materials in the absence of oxygen to produce biogas and digestate (biofertiliser). Massarutto (2010) suggests that AD is the ideal technology for sustainable management of organic materials to produce a source of

renewable energy, and an effective waste management technique that provides economic, health and environmental benefits.

Following the introduction is a review of scientific articles relating to AD process (Chapter 2). This discussed some of the operating parameters that influence the activities of anaerobic bacteria. The improvement and current state of in AD are also reviewed in Chapter 3. Chapter 4 discusses the complex interaction of different species of anaerobic bacteria that constitute the four steps of digestion process as well as the experimental work conducted in this work. Chapter 5 discusses the collection and analysis of the experimental results, considering the effects of temperature, pH, mixing speed and pressure on biogas and methane production while Chapter 6 discusses the construction of black-box model through MATLAB System Identification Toolbox. Two nonlinear model structures, autoregressive with exogenous input (NARX) and Hammerstein-Wiener (NLHW) with different nonlinearity estimators and model orders are chosen by trial and error and utilised to estimate the models. The performance of the models is determined by comparing the simulated outputs of the estimated models and the output in the validation data. The approach is used to validate the estimated models by checking how well the simulated output of the models fits the measured output. The best models for biogas and methane production are chosen by comparing the outputs of the best NARX and NLHW models (each for biogas and methane production), and the validation data, as well as utilising the Akaike information criterion (AIC), to measure the quality of each model relative to each of the other models. The NLHW models mhw2 and mhws2 are chosen for biogas and methane production, respectively. The identified NLHW models mhw2 and mhws2 represent the behaviour of the production of biogas and methane, respectively, from mesophilic AD. Of all the candidate models studied, the nonlinear models provide a superior reproduction of the experimental data over the whole analysed period. (Chapter 6).

The thesis also discussed the scalability of black-box models. Finally, conclusions and recommendation for further research work are covered in Chapter 7.

This thesis presents the most vital outcomes of this work, which has been partially published as a conference paper in a journal, and the citation of the paper is as follows:

Ogbonna E. C., Ali R., Pissanidis G. (2013): Simulation Model for Mesophilic Anaerobic Digestion Heating System. Renewable Energy Research and Applications (ICRERA), 2013 International Conference: 505-510, doi: 10.1109/ICRERA.2013.6749807. The full copy of this paper is located in Appendix C.

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Abstract

Anaerobic digestion, which is the process by which bacteria breakdown organic matter to produce biogas (renewable energy source) and digestate (biofertiliser) in the absence of oxygen, proves to be the ideal concept not only for sustainable energy provision but also for effective organic waste management. However, the production amount of biogas to keep up with the global demand is limited by the underperformance in the system implementing the AD process. This underperformance is due to the difficulty in obtaining and maintaining the optimal operating parameters/states for anaerobic bacteria to thrive with regards to attaining a specific critical population number, which results in maximising the biogas production. This problem continues to exist as a result of insufficient knowledge of the interactions between the operating parameters and bacterial community. In addition, the lack of sufficient knowledge of the composition of bacterial groups that varies with changes in the operating parameters such as temperature, substrate and retention time. Without sufficient knowledge of the overall impact of the physico-environmental operating parameters on anaerobic bacterial growth and composition, significant improvement of biogas production may be difficult to attain.

In order to mitigate this problem, this study has presented a nonlinear multi-parameter system modelling of mesophilic AD. It utilised raw data sets generated from laboratory experimentation of the influence of four operating parameters, temperature, pH, mixing speed and pressure on biogas and methane production, signifying that this is a multiple input single output (MISO) system. Due to the nonlinear characteristics of the data, the nonlinear black-box modelling technique is applied. The modelling is performed in MATLAB through System Identification approach. Two nonlinear model structures, autoregressive with exogenous input (NARX) and Hammerstein-Wiener (NLHW) with different nonlinearity estimators and model

orders are chosen by trial and error and utilised to estimate the models. The performance of the models is determined by comparing the simulated outputs of the estimated models and the output in the validation data. The approach is used to validate the estimated models by checking how well the simulated output of the models fits the measured output. The best models for biogas and methane production are chosen by comparing the outputs of the best NARX and NLHW models (each for biogas and methane production), and the validation data, as well as utilising the Akaike information criterion to measure the quality of each model relative to each of the other models. The NLHW models mhw2 and mhws2 are chosen for biogas and methane production, respectively. The identified NLHW models mhw2 and mhws2 represent the behaviour of the production of biogas and methane, respectively, from mesophilic AD. Among all the candidate models studied, the nonlinear models provide a superior reproduction of the experimental data over the whole analysed period. Furthermore, the models constructed in this study cannot be used for scale-up purpose because they are not able to satisfy the rules and criteria for applying dimensional analysis to scale-up.

Table of Contents

List of Figures

List of Tables

List of Abbreviations

AD	Anaerobic Digestion
ADM1	Anaerobic Digestion model one
CaO	Calcium oxide
CFD	Computational Fluid Dynamic
CH_4	Methane
CO_2	Carbon dioxide
COD/N	Chemical Oxygen Demand/Nitrogen
C/N ratio	Carbon to Nitrogen ratio
CSTR	Continuous Stirred Tank Reactor
DA	Dimensional Analysis
DEFRA	Department for Environment, Food and Rural Affairs
DAE	Differential Algebraic Equation
DE	Differential Equation
DN	Dimensionless Number
EC	European Commission
EEA	European Environment Agency
EPSAC	Extended Prediction Self-Adaptive Control
EU	European Union
Fig.	Figure
FOA	Food and Agriculture Organisation
FW	Food Waste
GoF	Goodness-of-Fit

GUI	Graphical User Interface
H_2	Hydrogen
HCL	Hydrochloric Acid
H_2O	Water
HPA	Health Protection Agency
HRT	Hydraulic Retention Time
H_2S	Hydrogen Sulphide
IEA	International Energy Agency
IEO	Information Energy Administration
IWA	International Water Association
LAP	Laboratory Analytical Procedure
LCFA	Long Chain Fatty Acid
MIMO	Multiple Input Multiple Output
MISO	Multiple Input Single Output
MO	Measured Output
MPC	Model Predictive Control
MSW	Municipal Solid Waste
MVs	Manipulated Variables
N_2	Nitrogen gas
NaOH	Sodium Hydroxide
NARX	Nonlinear Auto Regressive with Exogenous input
NH_3	Ammonia
NI	National Instrument
NLHW	Nonlinear Hammerstein-Wiener
NNFCC	National Non-Food Crop Centre

NWCC	National Wind Coordinating Committee
OECD	Organisation for Economic Co-operation and Development
OFMSW	Organic Fraction of Municipal Solid Waste
OLR	Organic Loading Rate
pH	Power of Hydrogen
PhD	Doctor of Philosophy
Ref	Reference
SCADA	Supervisory Control and Data Acquisition
SNH	Scottish Natural Heritage
SSR	Sum of Squares of the Regression
SST	Total Sum of Squares
TOC	Total organic carbon
TS	Total Solids
UNFPA	United Nations Fund for Population Activities
VFAs	Volatile Fatty Acids
VS	Volatile Solids
WID	Waste Incineration Directive
WIR	Waste Incineration (England and Wales) Regulations
WWS	Wastewater Sludge

Nomenclature

α	Intersection of line meets the y-axis,
β	Slope of the equation and,
ε	Variation or random error of the actual data
y	Output
X	measure of time or dosage
A	Starting or initial quantity
B	Relative growth rate
D	Dilution rate
h^{-1}	Per hour
V =	Volume of digester
m^3	Metre cube
F	Flow rate
mg	Milligram
t	Time
l	Length
μ	Specific growth rate
μ_{max}	Maximum specific growth rate
S	Substrate concentration
k_s	Half-velocity constant
Y	Bacterial yield coefficient
G_s, G	Biogas production
T	Lag time
S	Second

Ts	Sampling time
P	prediction horizon
$y(t)$	Output at time t
f	nonlinear function
$\varepsilon(t)$	noise signals
$u(t)$	Input at time t
n_y	maximum lags of past output
n_u	maximum lags of past input
$w(t)$	input internal variables
x(t)	output internal variables
mL	millilitre
rpm	revolution per minute
L	Litre
^oC	degree celcius
kWh	Kilowatt hour
Press	Pressure
Temp	Temperature
MS	Mixing Speed

Chapter 1 **Introduction**

1.1 Introduction

This chapter presents the research work theme, highlighting the impact of global energy production and consumption, especially, the effects of fossil fuels on the environment and all life forms. It also discussed the possible alternative energy sources that can mitigate the environmental and health risks linked to fossil fuels. In addition, it discussed the environmental and health risks due to increasing municipal solid waste (MSW) generation. Furthermore, the concept of AD is introduced, including its suitability for providing a sustainable energy source as well as implemented as an ideal organic waste management system. Finally, the problem statement, aim and objectives of this research work are discussed.

1.2 Background

1.2.1 Energy and environmental problems

Energy and its impact on the environment are interrelated (Ahuja and Tatsutani, 2009). The environmental impacts of energy production and energy utilisation are irreversible (Sidik et al., 2013, Budiyono et al., 2013; Abdullahi et al., 2011; Espinoza-Escalante et al., 2009; Tejada and Gonzalez, 2006). First, energy consumption continues to escalate year after year due to increasing human population, rising living standards and rapid growth of energy intensive industries in emerging economies (OECD, 2011; Ahuja and Tatsutani, 2009). Global reserves of fossil fuels, our primary source of energy, are finite and are being depleted (Sidik et al., 2013; Budiyono et al., 2013, OECD, 2011).

Second, the conversion of the chemical energy of fossil fuels to useful work involves combustion, the products of which include greenhouse gases such as CO_2 and NO_2 (NRC,

2010; Tsoutsos et al., 2005). Emissions of these gases are not only harmful to many life forms but also contribute to the cause of climate change (Budiyono et al., 2013, OECD, 2011). With ever increasing energy consumption we are exhausting our reserves of fossil fuels, intensifying the deterioration of the quality of the air we breathe and increasing global warming.

In order to mitigate the two aforementioned risks, there is a need to explore alternative sources of energy that are sustainable. The potentials of sustainable energy sources such as solar, wind, geothermal and biomass, if fully harvested, could provide satisfactory solutions to the global energy and environmental challenges (EC, 1997; EC, 1995). The renewable energy sources are discussed as follows.

Solar energy:

Solar could be the world's ideal source of energy, it is free and inexhaustible (Tudorache and Kreindler, 2010). It has the capacity to provide more than 10,000 times the global annual energy requirement (Tsoutsos et al., 2005; Greenpeace, 2005). Not only is solar abundant and infinite, it also offers substantial environmental benefits relative to fossil fuels, our primary energy sources (Tsoutsos et al., 2005; Tsoutsos et al., 2003a; Tsoutsos, 2001; Boyle, 1996; Johansson and Burnham, 1993). However, solar systems are linked to a number of adverse environmental impacts such as land use, visual impact and effects on buildings (Hestnes, 1999; OECD/IEA, 1998; Boyle, 1996).

In addition, solar energy is not available at all time. To generate power from solar, there must be sunlight, which makes it impossible to produce power at night or even in some hours of daylight in places situated in the temperate, continental and polar climates. Solar energy in these climates decreases during the winter months as there are fewer sunlight hours, resulting

in the reduction of the intensity of solar radiation. In these circumstances, energy must be stored or obtained elsewhere at night or during the winter months when there is no sunshine.

Wind energy:

The Wind is another potential source of energy that is gaining prominence in the world (UCS, 2013). It is one of the ideal sources of renewable energy (UCS, 2013). It is infinite, plentiful and produces no toxic contamination or greenhouse gases (UCS, 2013). Although the wind offers many possibilities to replace the conventional sources of energy, yet it has a number of undesirable impacts on the environment. These include land use that varies from site to site (NREL, 2012; NREL, 2010; Denholm et al., 2009; Michel et al., 2007), impact of wind turbines on wildlife and habitat, most especially bats and birds' death (NWCC, 2010) and effect on public health, especially sound and visual disturbances.

Like solar energy, wind energy has its own limitations; it is unpredictable. Wind turbines cannot generate electricity when any wind is blowing, thus making wind energy not suitable for a single source energy solution (Rasmussen, 2010). In addition, wind energy is sited typically in coastal and hilly areas (SNH, 2014). Cities situated within the plane topographies will not be able to harness wind energy as much as in coastal and hilly geographies, making wind energy non-universal.

Geothermal energy:

Geothermal energy is another source of renewable energy. It is from the heat confined inside the earth crust (Gagel et al., 2007; Barbier, 2002). It can be utilised to generate electricity when the temperature is above 150 ^{o}C. On the other hand, geothermal energy can be applied directly for space heating and industrial processes when the temperature is below 150 ^{o}C (Fridleifsson,

2003; Barbier 2002; Fridleifsson, 1996). Geothermal resources are estimated to be 2,000±140 Terawatts hour per annum (TWh/a) worth of electricity, as well as more than 7,000 TWh/a worth of heat that can be used for direct space heating or industrial applications (Fridleifsson, 2003; Fridleifsson, 2001; Stefansson, 1998). The International Energy Outlook 2013 (IEO, 2013) estimated that the total energy consumption of the world would be 184,634.73 TWh in 2020 and 240,318.22 TWh in 2040. This means that the global geothermal energy can only contribute about 5 % of the projected total world energy requirement for 2020 and 3.8 % for 2040 (IEO, 2013).

It is apparent that geothermal energy cannot meet the global energy requirement. There are a number of geological environments that support exploitable geothermal energy. The geothermal fields with a temperature above 150 ^{o}C suitable for electricity generation are mainly trapped in locations where there is young volcanism, seismic and magmatic action (Fridleifsson, 1996), which do not occur in every country. On the other hand, the geothermal resources with a temperature below 150 ^{o}C that could be utilised directly are found in many parts of the world (Fridleifsson, 1996). Furthermore, the exploitation of geothermal energy results to some environmental threats, such as emission of gases like, CO_2, H_2S, NH_3, N_2, H_2, and CH_4 (Barbier, 2002; Fridleifsson, 2001), wildlife extinction, destruction of natural vegetation and land use (Barbier, 2002; Fridleifsson, 2001).

Biomass energy:

Biomass is also another source of renewable energy that has the promise to mitigate the impact of global energy production on the environment and resource conservation (Wang, 2014). The use of biomass to produce sustainable energy can replace fossil fuels and also minimises CO_2 and CH_4 emission, which contribute significantly to global warming (Kusch et al., 2011). The

conversion of biomass to energy has a variety of techniques that are currently applied, which is broadly classified into thermochemical and biochemical/biological processes (Cheng et al., 2014; Cheng, 2013; Tonini, 2013; Caputo et al., 2005; McKendry, 2002). Thermochemical conversion process consists of combustion, gasification, pyrolysis, and liquefaction (Ciubota-Rosie et al., 2008; Caputo et al., 2005; Demirbas, 2004; McKendry, 2002; Demirbas, 2001; Demirbas, 1998; Overend, 1998; Solantausta, 1995). While biochemical/biological conversion method is made up of aerobic fermentation, anaerobic digestion (AD) and mechanical extraction (Ciubota-Rosie et al., 2008; Caputo et al., 2005; McKendry, 2002; Demirbas, 2001; WEC, 1994). However, AD is the ideal biomass energy conversion technology due the advantage of not only producing renewable energy source but also well suited for organic waste management in the municipalities and rural areas (Kusch et al., 2011).

Every energy conversion process, including biomass conversion concepts, have some degree of negative impacts on the environment. Biomass conversion techniques also have a number of environmental concerns, such as air pollution, deforestation, as well as the impact on the cultivation of food crops (Ciubota-Rosie et al., 2008). But, unlike any other source of sustainable energy, biomass energy can achieve a neutral CO_2 contribution to the environment (Ciubota-Rosie et al., 2008; Jefferson, 2006; Robu, 2005).

This is attained by creating a balance between the harvested biomass for energy and the consumption of the CO_2 produced in the process of energy conversion and utilisation by new biomass plant growth (Ciubota-Rosie et al., 2008; Jefferson, 2006; Robu, 2005). According to Miner (2010), carbon neutral activity is the one that balances the release of CO_2 into the atmosphere and the absorption from the atmosphere. The carbon neutral cycle is illustrated in Figure 1.1 (Miner, 2010).

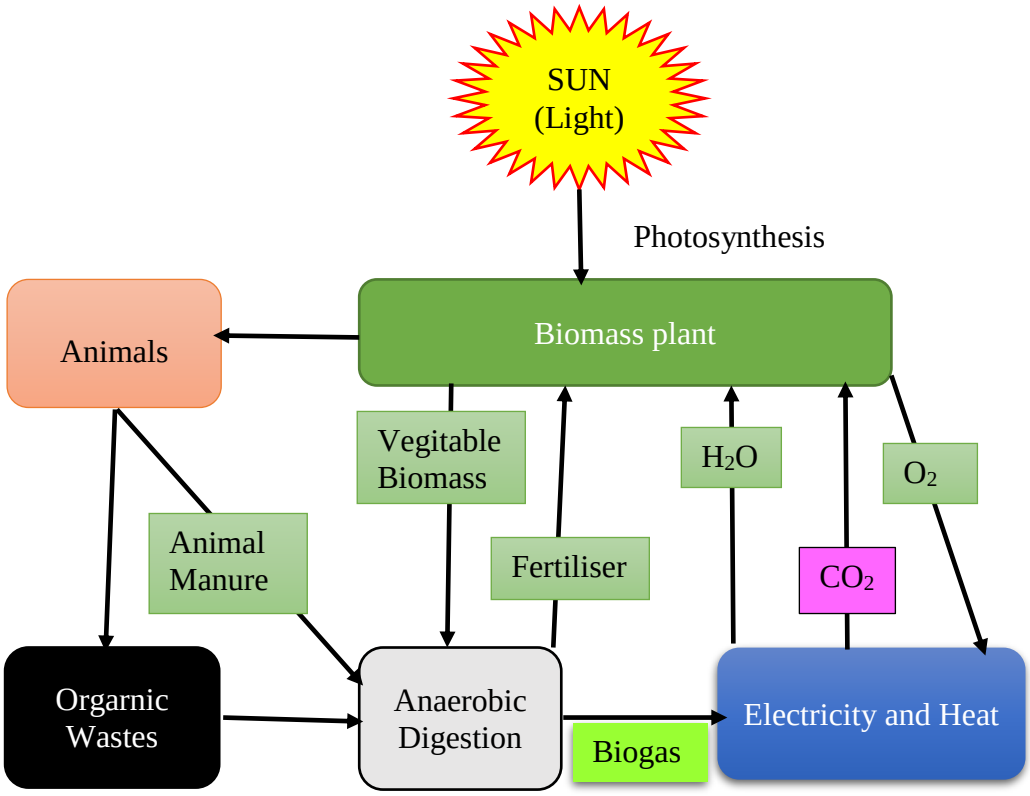


Fig.1.1. Carbon neutral cycle of biomass conversion through AD

1.2.2 Municipal solid waste generation and the world population

The United Nations (UN) in 2007 projected that the world population will increase by 2.5 billion between 2007 and 2050, that is, from 6.7 billion in 2007 to 9.2 billion in 2050 (UN, 2007). This increase is equivalent to the population of the world in 1950 and will be absorbed by the less developed parts of the world, whose population is likely to rise from 5.4 billion in 2007 to 7.9 billion in 2050, whilst the developed nations are expected to remain marginally constant at 1.2 billion people (UN, 2007). Similarly, UNFPA (2007) forecasted that about 5 billion people would live in the municipal areas by 2030.

As a result of the continuous growth of the world population as well as improvement in the standard of living of people and industrial advancement, the amount of municipal solid waste (MSW) generated is on a constant rise. The Organisation for Economic Corporation and Development (OECD, 2004), stated that between the year 1980 and 2000 the amount of municipal waste generated in its member states increased by about 54%. In another report, OECD stated that its member states are generating over 4 billion tonnes of MSW annually (OECD, 2013). In addition, it reported that in 2012 each person within the 28 EU member states generated an average of 492 kg of MSW (Eurostat, 2014). In the same report, each person in the UK generated an average of 472 kg of MSW (Eurostat, 2014).

Similarly, DEFRA reported that the UK generated 31.1 million tonnes of MSW in 2012 (Themelis and Bourtsalas, 2013). It is established that MSW is a global problem, but the improper waste management in developing countries increases the susceptibility to environmental and health hazards. These potential risks include air pollution, emission of greenhouse gases, underground water contamination, cholera, typhoid fever and bubonic plague (Antonis, 2013; Boadi and Kuitunen, 2005; DEFRA, 2004)

1.2.3 Evolution of waste management concepts

Before the advent of industrial revolution, the nature of waste produced was mainly biodegradable, such as vegetable, human waste and ashes from the incineration of other waste materials (Chandler et al., 1997; Ascari et al., 1992). The management of waste was not complex due to the utilisation as fertiliser or soil conditioner on farmland (Sangodoyin and Ipadeola, 2000; Chandler et al., 1997; Ascari et al., 1992; Nightingale, 1954). The industrial evolution in the 19th century created a significant increase in the global economic activities, resulting in mass migration of people to the industrial cities from the rural areas (Ayomoh et

al., 2008). The increase in industrial activities and the constant growth in human population, especially in the cities led to a significant increase in the quantity of solid waste generation (Ziadat and Mott, 2005). As a result, waste management becomes a critical issue to humanity (Articlebase, 2011; Melosi, 2005; Chandler et al., 1997). The different techniques used to minimise the challenges of waste management include landfill, incineration, recycling and biological reprocessing.

Landfill and incineration are the oldest and most common form of waste management techniques (Themelis and Ulloa, 2007). Many countries, particularly the developing countries, still apply landfill as the main waste management system (EEA, 2009; Themelis and Ulloa, 2007; Massarutto, 2001; Buclet and Godard, 2000). Landfills had been linked to a number of environmental and health hazards, like air contamination (Davoli et al., 2010), strong odours and smells perceived over 1 km from landfill sites (HPA, 2011), congenital malformation and low birth weight among babies born to women living within 2 km from landfill sites (Forastiere et al., 2011).

Due to the risks associated with landfill, many countries instituted frameworks on landfill regulation. One of the most prominent frameworks legislated by the EU is the Waste Frameworks Directive (Directive 75/442/EEC, replaced by the modified Directive 2008/98/EC). The framework emphasises the reduction of waste that goes to landfills (EEA, 2009). Similarly, the United States Environmental Protection Agency (USEPA) on October 9, 1991, publicised a revised minimum technical requirement for MSW landfills (USEPA, 1991). This regulation led to the closure of many smaller landfills in the USA, reducing the total number from 6,500 in 1988 to 1,767 in 2003 (USEPA, 2003). In addition, the USEPA (1991)

framework include guidelines for remodelling old landfills, construction of new landfill systems and the type of waste that go into them (Daskalopoulos et al., 1997).

The remodelled and new landfill systems are equipped with impermeable liners and caps as well as a subsystem that collects and treats leachate. They also have gas wells and pipes that collect and transport landfills gas, mainly methane, to where it is utilised for electricity or heat generation (Themelis and Ulloa, 2007). Furthermore, the landfill systems consist of impervious layer covers applied when full to prevent rainwater as well as facilitate biodegradation process (Themelis and Ulloa, 2007).

The application of incineration to reduce the excess quantity of household and agricultural waste dates back to thousands of years ago (Petts, 1994). Incineration involves the combustion of solid waste to produce ash residue (Petts, 1994). It is an effective way of reducing the volume of MSW and the demand for landfill space. However, it is related to possible environmental and health risks. These include airborne emission of dioxins (hazardous chemicals) (HPS, 2009), Sulphur dioxide (SO_2) (Roberts and Chen, 2006, also cited by Fewtrell, 2012) and nitrogen dioxide (NO_2) (Forastiere et al., 2011), which can cause cancer and death.

Similarly, a number of countries enacted frameworks for incineration systems due to the potential dangers. The Waste Incineration Directive (WID) 2000/76/EC of European Parliament and of the Council (WID, 2000), and the Waste Incineration (England and Wales) Regulations (WIR) 2002 (WIR, 2002) are some of the frameworks instituted. The aim of the directives is to innovate incineration systems in order to minimise the environmental and health risks (DEFRA, 2009). The recycling method of waste management is not discussed further in this thesis, but the biological reprocessing is discussed as follows.

1.2.4 Motivation of anaerobic digestion for organic waste management

Appropriate waste management practice is crucial for any sustainable society. It prevents air, soil and water pollution as well as improves public health, decreases greenhouse gas emission and preserves natural resources. The AD system, which is a type of biological reprocessing, is the ideal waste management technique (Bohn, 2010; Hartmann et al., 2004). AD is the process by which bacteria breakdown organic material to produce biogas (renewable energy source) and digestate (biofertiliser) in the absence of oxygen. It involves not only the collection and safe disposal of organic waste but also sustainable management of organic material to create a source of renewable energy as well as provide economic, health and environmental benefits (Massarutto, 2010). According to Bohn (2010), AD reduces greenhouse gas emission more than any other waste management systems. It is an effective method of recovering energy and nutrients from organic material (Hartmann et al., 2004). The following are some of the benefits of AD system (DEFRI, 2009).

- Contribution towards mitigation of climate change and other environmental targets;
- Treatment of biodegradable wastes to generate biogas, a renewable energy that can be utilised to produce electricity and heat from combined heat and power (CHP) or for vehicle fuel;
- Diversion of organic wastes from landfills and capturing of methane emission from organic wastes;
- Provision of organic fertiliser and soil conditioner for agriculture and land use; and
- A source of revenue generation to farmers and other practitioners, as excess energy and digestate are sold, which adds to the nation's gross domestic product (GDP).

1.3 Problem statement

The enhancement of biogas production is crucial in recent years due to increasing demand for biogas, resulting from the global quest for alternative sources of energy that are sustainable (Krustok et al., 2013; van Foreest, 2012; Rajendran et al., 2012; Ploechl et al., 2010). However, the production of biogas to keep up with the global demand is limited by the underperformance in the system implementing the AD process. This underperformance is due to the difficulty in obtaining and maintaining the optimal operating parameters/states for anaerobic bacteria to thrive with regards to attaining a specific critical population number, which results in maximising the rate of biogas production. This problem continues to exist as a result of insufficient knowledge of the interactions between the operating parameters and bacterial community. In addition, the lack of sufficient knowledge of the composition of bacterial groups that varies with changes in the operating parameters such as temperature, substrate and retention time. Without sufficient knowledge of the overall impact of the physico-environmental operating parameters on anaerobic bacterial growth and composition, significant improvement of biogas production may be difficult to attain.

In order to mitigate this problem, the construction of a suitable dynamic mathematical model is required that enables a better understanding of the relationship between the operating parameters and biogas production. This facilitates the means of predicting the behaviour of bacteria in AD, under different physico-environmental operating parameters

A number of AD models have been developed by previous studies (Batstone et al., 2002; Amon et al., 2007; Keymer and Schlicher, 2003; Angelidaki et al., 1999; Boyle 1976; Buswell and Mueller, 1952). However, the current models are mainly dependent on assumptions that

consequently result in neglect of real-life scenarios, which limits a more realistic study on AD process.

1.4 Aim and objectives

An effective digester operation is one that maintains the optimal operating parameters/states for anaerobic bacteria to thrive with regards to attaining a specific critical population number which results in maximising the rate of biogas production.

The aim of this PhD study is to develop a black-box (empirical) model for mesophilic AD process, in order to understand better, how physico-environmental operating parameters influence biogas and methane production. More specific there is a need to consider the control limits for the controlled parameters necessary for improving methanogenesis reaction while ensuring an enhanced digestion operation.

In order to accomplish this goal, the following objectives are addressed.

1. Identify and understand the performance variables of mesophilic AD system (Chapter 2);
2. Conduct a critical review of the operating parameters that influence the production of biogas and methane under mesophilic condition. For this research work the following four operating parameters are considered; temperature, pH, mixing speed and pressure (Chapter 2);
3. Determine the limits for the four operating parameters and their impact on biogas and methane production. This includes a series of laboratory experiments with each of the operating parameters. The data obtained from the investigations are analysed and the

range of values for each operating parameter that recorded the highest performance in terms of biogas quantity production and quality are determined (Chapters 4 & 5);

4. Construct a black-box model each for biogas production and methane production. The limits of the four operating parameters considered are analysed further in order to establish their correlation with biogas and methane production. Subsequently, two nonlinear multi-parameter black-box models are constructed using System Identification method through MATLAB functions. The models are able to predict the behaviour of the real system with sufficient accuracy (Chapter 6); and
5. Determine the scalability of black-box models. This is to determine the possibility of scale-up of the black-box models constructed in this research study.

1.5 Summary

This chapter has presented the concept of this research work. It highlighted the impact of energy production and consumption on the environment and all live forms, as well as the global need for an alternative energy source that is sustainable. It also discussed the risks of increasing MSW on the environment and public health. In addition, the chapter introduced AD as the ideal technology for both a sustainable energy source and organic waste management. It then presented the problem statement, as well as the aim and objectives of this research work. The following chapter discusses the review of previous and relevant scientific articles in AD technology.

Chapter 2 **Development in Anaerobic Digestion: Literature Review**

2.1 Introduction

The application of AD was recorded in China and India over 2000 years ago when it was used for treating animal manure (Nayono, 2010; Veenstra, 2000). However, not until the 19th century was AD applied for wastewater and solid waste treatment (Nayono, 2010; Residua, 2009). The reason for the gap was due to the perception of AD as a slow and ineffective technology for organic waste treatment, especially for the increasing wastewater volume in industrialised and densely populated municipalities (Nayono, 2010; Gijzen, 2002; Polprasert, 2001). The first properly constructed AD plant was in a leper colony in Bombay, India in 1859 (Abbasi et al., 2012; Khanal, 2008; Meynell, 1976). Similarly, the first account of AD system in England, UK, was in 1895, during which biogas was generated from sewage treatment plant and utilised as fuel for street lamps in Exeter (Residua, 2009; McCabe and Eckenfelder, 1957). AD technology has continued to gain prominence in many countries, particularly, in the recovery of renewable energy from household and municipal waste (Holm-Nielsen, 2009). In addition, AD contributes to sanitation improvement, greenhouse gas mitigation, and production of organic fertiliser (Wang, 2014; Johansen et al., 2013, Vaneeckhaute et al., 2013; Wang et al., 2011; Zheng et al., 2010). This chapter presents a discussion of the various physico-environmental operating parameters that influence the performance of AD process, as it relates to anaerobic bacteria activity and biogas production.

2.2 Operating parameters that influence AD performance

There are a number of operating parameters that influence anaerobic bacterial activities such as specific growth rate, degradation rate, substrate utilisation and biogas production (Rajendren

et al., 2012; Deublein and Steinhauser, 2008; Al Seadi et al., 2008). For improved performance of AD process, it is necessary to maintain the appropriate range of the operating parameters that support optimal growth of anaerobic bacteria (Rajendren et al., 2012; Deublein and Steinhauser, 2008; Al Seadi et al., 2008). In order to maintain the stability of anaerobic reaction, it is crucial to maintaining a balance between the rate of organic acid formation and methane production (Bernard, 2012; USEPA, 2008). Maintaining a proper equilibrium between acidogenic and methanogenic bacteria requires that the operating parameters are kept within their different optimal ranges (Bernard, 2012; USEPA, 2008). The operating parameters discussed in this thesis include pH, temperature, substrate characteristics, hydraulic retention time (HRT), organic loading rate (OLR), mixing and carbon to nitrogen (C/N) ratio (Rajendren et al., 2012; Deublein and Steinhauser, 2008; Yadvika et al., 2004; Gerardi, 2003).

2.2.1 pH

The performance of AD process is affected by variation of pH in the digester (Cioabla et al., 2012; Khalid et al., 2011; Romano and Zhang, 2011; Kumar, 2010; Wu et al., 2009). Each bacterial group in AD process has a specific range of pH values at which they optimally multiply and are most active (Khalid et al., 2011). Various studies have shown that anaerobic bacteria can function in a pH range of 5.5 - 8.5 (Nayono, 2009; Al Seadi et al., 2008; Lay et al., 1998; RISE-AT, 1998; Stronach et al., 1986). On the other hand, Cioabla et al., (2012) limited the pH range at which anaerobic bacteria can withstand to 6.5 – 8.0. However, it is reported that the optimum pH range for AD is 6.8 - 7.2 (Cioabla et al., 2012; Ward et al., 2008), which is the pH range for optimal growth of methanogenic bacteria. Other studies reported different optimal pH ranges for methanogenic bacterial growth, such as 6.8 - 7.6 (Nayono, 2009; Lay et al., 1998; Stronach et al., 1986), 7.0 - 8.0 (Al Seadi et al.'2008) and 6.5 - 8.2 (Lee et al., 2009b). The ideal optimal pH value for methanogenesis is 7.0, the neutral pH (Wang,

2014; Khalid et al., 2011; Yang and Okos, 1987; Huber et al., 1982), which should be the target pH value for biogas production. Furthermore, Hydrolytic and acidogenic bacteria are found to multiply optimally at lower pH levels of 5.5 and 6.5, respectively (Khalid et al., 2011; Kim et al., 2003).

Methanogenic bacteria are sensitive to the acidic environment; their growth, as well as methane production, are inhibited in acidic condition (Wang, 2014; Kangle et al. 2012). Organic acids produced during acidogenesis can decrease the pH level of the reaction below 5.0 and consequentially, limit the growth of methanogenic bacteria (Kangle et al. 2012). Since methanogenic bacteria utilise the organic acids, decrease in their population would lead to excess volatile fatty acids (VFAs) accumulation, resulting in digester sour – a situation whereby methanogenic bacteria fail to keep pace with acidogenic bacteria and the digester becomes acidic (Kozani, 2014). The acidic condition can be caused by any of or a combination of the following factors, temperature change, toxic compound or sudden addition of a large quantity of digestible organic matter. However, when there is less population of acidogenic bacteria than methanogenic bacteria in AD process, the pH value can increase beyond 8.0, which can cause an increase in the concentration of ammonia, and result in digester failure (Lusk, 1999). Since methanogenesis is the rate-limiting stage of AD, it is important to keep the pH value of digester within methanogenesis optimal range of 6.8 - 7.2.

2.2.2 Temperature

Temperature is characterised as the most important parameter that influence AD process (Cioabla et al., 2012; Chae et al., 2008; Choorit and Wisarnwan, 2007). The reason for this is due to the impact of temperature on all the processes involved in AD, which includes physico-chemical properties of substrates in the digester as well as the kinetics and thermodynamics of

biological processes (Çalli, 2012; Kangle et al. 2012; Boe, 2006). The different groups of anaerobic methanogenic (methane forming) bacteria can multiply under various temperature limits (USFDA, 2012; Lamprecht, 2009; Bisschops et al., 2009; Lettinga et al., 2001). Figure 2.1 shows the rate of growth of different methanogens at varied temperature ranges (Lettinga et al., 2001).

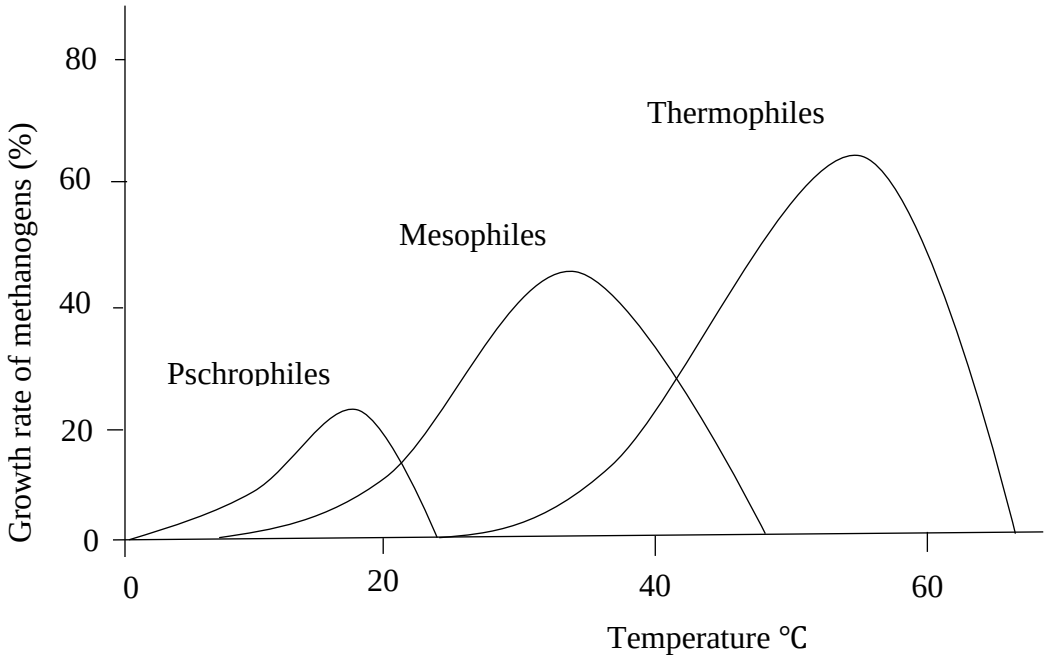


Fig. 2.1. Relative growth rate of different methanogenic bacteria

As seen in Figure. 2.1, different methanogenic bacteria grow in varied temperature ranges. Psychrophilic methanogens can multiply under temperature between 0 – 20 ^{o}C, with optimum temperature range of 10 – 15 ^{o}C (Schmidt and Schaecheter, 2012; Willey et al., 2008; Lettinga et al., 2001; Hoyle, 2003; Temper et al, 1983; Winter et al, 1982; Morita, 1975). Mesophilic bacteria can populate at temperature between 10 – 45 ^{o}C, but grow optimally at temperature range of 35 – 40 ^{o}C (Schmidt and Schaecheter, 2012; Willey et al., 2008; Lettinga et al., 2001; Hoyle, 2003; Temper et al, 1983; Winter et al, 1982; Morita, 1975). Whilst thermophilic bacteria can withstand a higher temperature range of 25–65 ^{o}C, and the optimum temperature

range is between 50–60 ºC (Schmidt and Schaecheter, 2012; Willey et al., 2008; Hoyle, 2003; Lettinga et al., 2001; Morita, 1975). In addition, AD process can operate at temperatures above 60 °C, which is referred to as extreme-thermophilic (Frock and Kelly, 2012; Abreu et al., 2010). Mackie and Bryant (1981) reported that low rates between these optimal temperatures could be attributed to lack of adaptability of the different methanogenic bacteria.

AD is predominantly operated at a mesophilic temperature range (Li et al., 2014; Wang et al., 2010). This is because there are more variety of bacteria that multiply at a mesophilic temperature range, thus mesophilic digestion is considered more stable than thermophilic and psychrophilic systems (Li et al., 2014; Wang et al., 2010). Similarly, mesophilic bacteria adapt to changing environmental conditions more than thermophilic bacteria (Li et al., 2014; Wang et al., 2010; Cheremisinoff 2008). In addition, thermophilic AD systems require more energy to maintain the required temperature, thereby making it more expensive to operate (OECD, 2010; Cheremisinoff 2008). However, thermophilic anaerobic process has faster degradation rate than mesophilic digestion, resulting in shorter HRT, higher rate of methane production and kill pathogens better than mesophilic AD process (Li et al., 2014; Zhao, 2011; Luostarinen, 2005; Mata-Alvarez, 2002; Lettinga et al., 2001; NREL, 1992).

2.2.3 Carbon to Nitrogen (C/N) ratio

The relationship between the proportion of carbon and nitrogen in a substrate is referred to as the carbon to nitrogen (C/N) ratio (Kangle et al., 2012). Different substrates have dissimilar C/N ratios. It is essential to maintain a suitable C/N ratio that can facilitate optimum bacterial growth in the digestion process (Kangle et al., 2012). Higher concentration of nitrogen in AD leads to excess ammonia production and can increase the pH level beyond 8.5, which is toxic to methanogenic bacteria (Kangle et al., 2012; Mata-Avarez, 2000). However, low nitrogen

concentration can result in acidic digester content, leading to insufficient ammonia production, which in effect, inhibits methane production (Kangle et al., 2012; Hartmann and Ahring, 2006; FOA, 1992). The nitrogen content in the substrate is converted to ammonia, which neutralises the VFAs produced by acidogenic bacteria, thereby maintaining the pH level at near neutral that is favourable for methanogenic bacterial growth and improved methane production (FOA, 1992; Speece and McCarty, 1964). Kimchie (1984) suggested that C/N ratios lower than 10:1 will be inhibitory to digestion process, but higher than 23:1 will not support optimum digestion process. In order to keep the optimal balance between carbon and nitrogen, substrates with high or low C/N ratios can be co-digested, such as animal manure and organic solid wastes (Kumar, 2012; Zaher et al., 2007).

2.2.4 Substrate characteristics

The performance of AD can be affected by the characteristics of the substrate such as composition, degradability, homogeneity, fluid dynamics, particle size and C/N ratio (Nayono, 2009; Steffen et al., 1998). Solid waste, especially the organic content of MSW may differ due to a number of reasons, including weather condition, cultural habits, seasons of the year and manner in which they are collected (Nayono, 2009).

The proportion of substrate composition (carbohydrates, proteins and fats) influences its biodegradability as well as methane yield potential (Hartmann and Ahring, 2006). Substrates that contain a greater amount of fats yield more methane than the same quantity of carbohydrates and proteins (Angelidaki et al., 1990; Hanaki et al., 1981). This is because more energy is stored in the bonds of fat molecules (Fernandes et al., 2009). When electrons migrate from the atom, like carbon, with a low affinity for electrons to atom, such as oxygen, with a high affinity for electrons, energy is given off (Fernandes et al., 2009). So, more energy is

released when fatty acids are oxidised than when the same process takes place in carbohydrate and proteins (Fernandes et al., 2009; Mosier et al., 2005). However, fat-rich substrates degrade slower than that of carbohydrates and proteins. This is due to the slower hydrolytic phase of fat catabolism compared to that of carbohydrates and proteins, which are more readily hydrolysed (Neves et al., 2008; Vidal et al., 2000).

The fluid dynamics of the substrate, which changes with seasons of the year, can influence the performance of anaerobic digester (Motte et al., 2013). Substrate with high water content may increase digester volume and require higher heat input per m^3 of digester content, thereby reducing digester effectiveness (Motte et al., 2013; Steffen et al., 1998). However, a substrate with low water content, meaning that the total solids (TS) of the substrate is high, can cause ineffective mixing performance, scum layer formation, solid sedimentation and clogging (Motte et al., 2013; Steffen et al., 1998). Steffen et al. (1998) suggested that for conventional Continuous Stirred Tank Reactor (CSTR), a type of digester, TS concentration of substrate should be about 6–10 %. TS is the ratio measured in percentage of the weight of solids left after the substrate sample was dried in an autoclave at 105 ℃ for approximately 24 hours and the weight of the original substrate sample (Sluiter et al., 2008). TS is represented mathematically in Eq. 2.1 (Sluiter et al., 2008):

$$\%TS = \frac{((DPW+DSW)-DPW)}{OSW} \times 100 \qquad (2.1)$$

Where,

DPW, DSW, OSW represent dry pan weight, dry sample weight and original sample weight, respectively.

Furthermore, the particle size of the substrate can significantly affect AD performance (Hajji and Rhachi, 2013; Hartmann and Ahring, 2006). Particle size reduction increases the available

specific surface area that facilitates biodegradation, especially in hydrolysis stage, resulting in an increase in methane production as well as a reduction in the quantity of residue generation (Hajji and Rhachi, 2013; Hartmann and Ahring, 2006; Palmowski and Müller, 2000; Hills and Nakano, 1984). Mshandete et al. (2006) found that by reducing the size of sisal fibre waste to 2mm, the methane production potential and total fibre biodegradation improved by approximately 20 % and 31–70 %, respectively. This has shown that pre-treating the particle size of the substrate can enhance both the performance of anaerobic digester as well as improve methane production.

2.2.5 Hydraulic retention time (HRT)

The hydraulic retention time (HRT) is the time it takes for complete digestion of substrate (Hartmann and Ahring, 2006; Lu and Ahring, 2005). The HRT of AD depends on some factors, which include process temperature, substrate composition, particle size, and digester configuration (Hartmann and Ahring, 2006; Lu and Ahring, 2005). The HRT for substrates digested in mesophilic AD process varies from 10 – 40 days, while that treated in thermophilic digester can take up to 3 – 14 days (Hartmann and Ahring, 2006; Lu and Ahring, 2005). The HRT can be determined analytically from the relationship between digester volume and the volume of substrate fed per unit time as shown in Eq. 2.2 (Al Seadi et al., 2008).

$$\text{HRT} = \frac{V_R}{V} \tag{2.2}$$

Where,

HRT = hydraulic retention time (days)

V_R = digester volume (m^3)

V = volume of substrate fed per unit time (m^3/d

Due to the relatively slow growth of methanogenic bacteria, sufficient HRT is required for repopulation in order to make up for the bacteria discharged with the effluent (liquid discharged from the digester) (Zaher et al., 2007). Thus, maintaining bacterial population to cope with any inconsistency in the organic loading rate (OLR) (Zaher et al., 2007).

2.2.6 Organic loading rate (OLR)

OLR is the quantity of organic material (substrate) that is to be digested by a particular volume of the digester in a particular period of time (Al Seadi et al., 2008). Loading anaerobic digester above its sustainable capacity can cause accumulation of fatty acids, which inhibits AD process, resulting in process failure and low or no methane yield (Al Seadi et al., 2008; Vandevivere, 1999; Rise-at, 1998). OLR can be used as a measure of how much dry matter that can be loaded into a digester per volume of a unit time as presented in Eq. 2.3 (Al Seadi et al., 2008).

$$B_R = m \cdot \frac{c}{V_R} \tag{2.17}$$

Where,

B_R = organic load (kg/d $\cdot m^3$)

m = mass of substrate fed per time unit (kg/d)

c = concentration of organic matter (%)

V_R = digester volume (m^3)

However, improper OLR can affect biogas production (Al Seadi et al., 2008). As mentioned above, if the concentration of organic material loaded to the digester is high, it can lead to the high production of organic acids during acidogenesis, which decreases pH level below the acceptable level and in effect, decreases the population of methanogenic bacteria. Since methanogenic bacteria utilise the organic acids, a decrease in their population will result in

VFAs accumulation and consequently digester sour (Lusk, 1999). However, the lack of sufficient organic material in the digester leads to a shortage of VFAs, as a result, reduces the population of methanogenic bacteria. The effect of the reduced number of methanogenic bacteria results in the rise of the digester pH beyond the optimum level, thereby, increases the ammonia content that leads to digester failure (Lusk, 1999).

2.2.7 Mixing

The anaerobic digesters require mixing/stirring to improve contact between bacteria and substrate, facilitating bacterial access to nutrients in substrate (Meroney and Colarado, 2009; Wards et al., 2008). Mixing prevents scum formation and thermal stratification in the digester (Meroney and Colarado, 2009; Karim et al., 2005). By mixing digester contents, a denser particulate such as heavy solids particles and sand are kept suspended to avoid sedimentation (Wards et al., 2008; Kaparaju et al., 2007).

There are different types of mixing systems, such as gas mixing, pumped mixing, long shafted paddle mixing, wall mounted draft tube mixing and submersible mixers (Wards et al., 2008; Karim et al., 2005; Burton and Turner, 2003). The AD process does not always require continuous mixing, in practice, intermittent mixing is often applied; which can vary from a few times a day to several times an hour. Excessive mixing upsets bacterial population (Lindmark, 2014). However, moderate mixing is preferable for improved digestion (Lindmark, 2014), which is supported by the findings of this study and reported in Chapter 5 of this thesis.

2.3 Types of anaerobic digester

The anaerobic digester is the main component of AD. It is an airtight tank that can be of any shape (cylindrical, rectangular, square and egg-shaped), where biodegradation of substrate and

biogas production take place (Ward et al., 2008). The design of anaerobic digester is required to contain the following basic objectives; to make a sustainable and continuous high organic load rate possible, to achieve a short HRT as possible and to optimise the production of methane (Khalid et al., 2011; Ward et al., 2008). The anaerobic digester can be broadly categorised according to the feeding mode (batch and continuous) and by the solid content of the substrate (dry and wet digestion) (Ward et al., 2008).

2.3.1 Feeding mode (batch and continuous)

The batch digester is considered the simplest to design, construct and operate (Khalid et al., 2011). The digester is fed with substrate mixed with water to form a slurry, then sealed and allowed for a length of time. During the HRT, biogas production progresses to a maximum and then decreases slowly as bacterial effectiveness decreases. Not only that the batch digester is simple to construct and operate, it is also not expensive. It can perform rapid digestion process and be utilised easily to measure the rate of digestion (Khalid et al., 2011; Koppar and Pullammanappallil, 2008; Weiland, 2006; Parawira et al., 2004; Vandeviere et al., 2002; Ouedraogo. 1999). However, batch digester has some limitations, including volume restriction, the inconsistent rate of biogas production and varying the proportion of methane content in biogas (Linke et al., 2006).

Unlike batch system, continuous fed digester requires daily loading of an organic substrate (Ward et al., 2008). The volume is designed large enough to contain the daily substrate feeding throughout the HRT (Ward et al., 2008). Continuous feed system can be divided into two types, namely, single-stage and two-stage or multistage digesters (Vandevivere et al., 2002; Lissens et al., 2001). In one-stage continuous feed system, all the four steps of AD process take place simultaneously in one digester. One major disadvantage of this system is that the entire

biochemical processes are kept under the same operating parameters, despite the fact that the growth rate of the various bacterial groups involved and their optimal pH ranges are different (Geradi, 2003; Vandevivere et al., 2002).

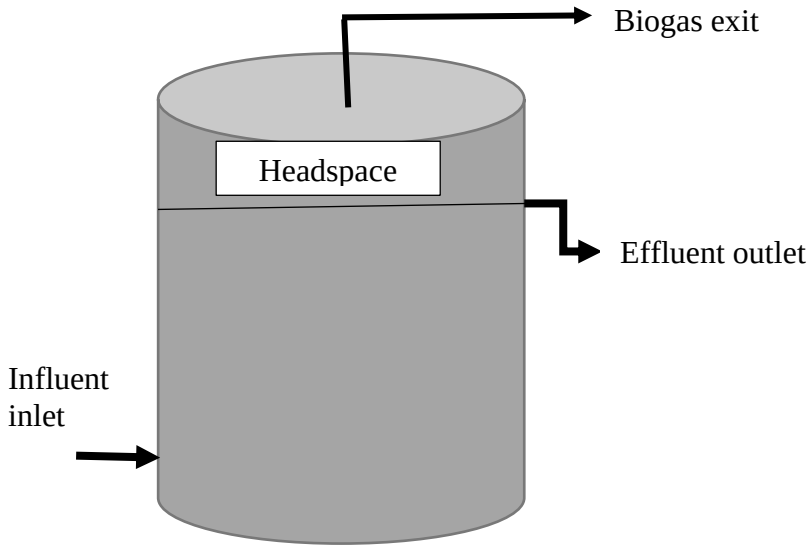


Fig. 2.2. Single-stage digester

However, a two-stage or multistage continuous feed system have the four stages of AD process take place in two separate phases in two digesters; hydrolysis/acidification take place in one digester and acetogenesis/methanogenesis processes occur in another digester. The product of the first stage (hydrolysis/acidification) in the first digester is fed into the second digester for the acetogenesis/methanogenesis step (De Baere, 2000; Schober et al., 1999). The separation of hydrolysis and acidification processes from acetogenesis and methanogenesis processes is mainly because they do not perform optimally under the same optimal pH range (Liu et al., 2006a; Liu et al., 2006b; Zoetemeyer et al., 1982).

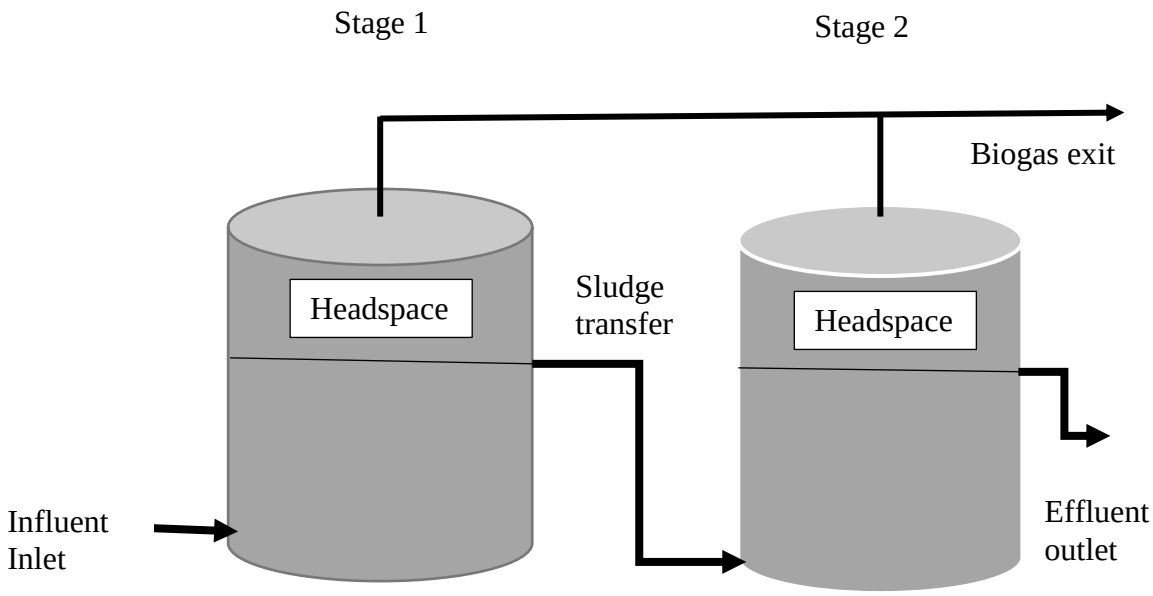


Fig. 2.3. Two or multi-stage digester

The two or multi-stage system maintains better process stability than the one-stage system, particularly during the metabolism of substrates that can be easily hydrolysed (Ward et al. 2008; Bouallagui et al., 2005; Mata-Alvarez, 2002). Although multi-stage system costs more to construct and maintain, it is more effective compared to the single-stage method. A study reported by Nielsen et al, (2004), where a one-stage and a two-stage thermophilic system fed with cattle dung were compared. The result of the study shows that the two-stage system produced 6 - 8% higher specific methane as well as effectively removed 9% more volatile solids than the one-stage process. In another study, MSW was utilised as the substrate, a two-stage system was found to produce 21% methane more than a single-stage system (Liu et al. 2006a). The multi-stage system is capable of handling variation in OLR and quality as well as maintains optimal conditions for the entire process than single-stage system (Rapport et al., 2008). Despite the various advantages of the multi-stage system over the single-stage system,

yet the multi-stage system has not been widely applied for commercial purposes (Rapport et al., 2008; Vandevivere et al., 2002; de Baere, 2000). This is attributed to the added design complexity and extra costs in constructing and operating a multi-stage anaerobic digester (Rapport et al., 2008; Vandevivere et al., 2002; de Baere, 2000).

2.3.2 Solid content (wet and dry)

The AD system can also be categorised as wet or dry digestion system, depending on the concentration of the total solids of substrate (Ward et al. 2008). Wet AD system is intended to process a dilute substrate, like slurry, which has its total solids concentration less than 15%. Whilst dry AD system is designed to digest substrate that contains 15 - 40% total solids (Lissens et al., 2001).

In wet digestion system, if the incoming substrate contains total solids higher than 15%, it can be diluted to make the appropriate slurry by adding deionised water, recirculated process water, or co-digested with another substrate with a lower total solids concentration (Hartmann and Ahring, 2006). For instance, Organic Fraction of Municipal Solid Waste (OFMSW) can be blended with sewage sludge to form the suitable slurry for wet digestion (Hartmann and Ahring, 2006; Luning et al., 2003). Wet digestion system is useful in processing low solid organic materials such as food industry liquid waste and sewage sludge (Hartmann and Ahring, 2006). However, the disadvantages of wet digestion system include the requirement for continuous mixing of slurry in the digester, which results in extra energy consumption by the mixer; the digester volume may be reduced as heavy and inert materials accumulate at the digester base due to sedimentation (Banks and Stentiford, 2007; Vandevivere et al., 2002). In addition, it requires larger digester volume compared to dry digestion system, more robust

water pumping and piping and utilises more energy to heat the extra digester volume (Banks and Stentiford, 2007; Vandevivere et al., 2002).

The dry digestion system has the advantage of minimising the process risk posed by the suspension of fibrous organic materials, like straw or straw-containing animal dung in the digester, which is common with wet AD process (Kalia and Singh, 1998). Furthermore, dry digestion system requires less energy for mixing and heating of digester content, less digester volume as well as reduced water consumption (Kottner, 2002; Vandeveivere et al., 2002; Hoffmann, 2001). However, due to the high solid concentration of such substrates, dry AD system requires a higher technical equipment for substrate handling, mixing devices, pumping and pre-treatment compared to the wet system (Sigrid et al., 2011; FNR, 2009; Lissens et al., 2001).

2.4 Summary

This chapter has discussed a number of operating parameters that influence anaerobic bacterial activity and subsequently, biogas and methane production. It is found that in order to improve biogas production; the system is required to maintain the appropriate limits of the various operating parameters that support the optimal growth rate of anaerobic bacteria. In addition, the different categories of anaerobic digesters, which are characterised by either the feeding mode or the solid content of the substrate. The review of the relevant scientific articles is continued in the next chapter with a focus on the improvement and the current state in AD technology.

Chapter 3 **Improvement and current state in AD: Literature Review**

3.1 Introduction

Many studies have been going on for the past decades in the field of AD with the aim of improving biogas productivity (Syngellakis, 2014; Montgomery and Bochmann, 2014; Brebbia, 2010; Ahring, 2003; Van Lier et al., 2001). The review of the relevant scientific articles in AD technology discussed in Chapter 2 above covered the physico-environmental operating parameters and their influence on AD performance, as well as the different types of anaerobic digesters based on the feeding mode and substrate solid content. However, this chapter discusses the relevant improvements and current state in AD technology, which include pre-treatment processes, co-digestion of different organic materials and process modelling.

3.2 Pre-treatment for process enhancement

Pre-treatment is the means of improving biogas yield per volume or mass unit of input substrate, by treating a substrate in advance before loading it into a digester (Malatak, 2013). In AD, especially when digesting complex substrate, it is not the entire substrate that is converted to biogas. This is due to the inability of the bacteria to gain access to the whole substrate (Malatak, 2013). Simple substrates such as cellulose and hemicellulose are easily biodegradable in AD. However, when combined with lignin, like lingo-cellulosic, they become complex (long chain polymer) and reduce in biodegradability (Van Liere, 2011). In digesting complex substrates, hydrolysis becomes another limiting stage (Zhang and Cai, 2008; Zhang et al., 2007; Noike et al., 1985). Meaning that apart from methanogenesis, biogas production also depends on the rate of hydrolysis - biodegradability (Fernandes, 2009).

A number of pre-treatment methods have been developed for AD process, which can be applied separately or combined (Donoso-Bravo and Fdz-Polanco, 2013; Quinones et al., 2012; Luo et al., 2012; Bishnoi, 2012; Erden and Filibeli, 2009; Perez-Elvira et al., 2009). The methods are broadly characterised into mechanical, chemical, biological, thermal and ultrasonic.

Mechanical method is a means of pre-treatment that is utilised mainly to reduce substrate particle size, by grinding and pressing in order to break the cell walls (Quinones et al., 2012). The advantages of reducing substrate particle size include an increase in the available specific surface area that facilitates biodegradation, increase in methane production and a decrease in the quantity of residue generation.

Chemical is the second method of pre-treatment process, which is applied by acidifying or alkalising the substrate (Quinones et al., 2012). Mata-Alvarez et al. (2000) reported the pre-treatment of fibres with N_aOH, NH_4OH and a combination of both chemicals resulted in improving biogas and methane production. In addition, Lopez-Torres and Espinosa-Llorens (2008), reported that increase in methane potential occurred when fibre was mixed with CaO.

In biological pre-treatment, the substrate is broken down aerobically in advance before feeding it to the digester. This process has shown to reduce solid particles, improve degradation rate and methane production (Mata-Alvarez et al., 2000; Capela et al., 1999). In another development, enzymes were used to catabolise substrate prior to feeding it to the digester, which is found to enhance degradation, while improving biogas production (Donoso-Bravo and Fdz-Polanco, 2013; Luo et al., 2012; Quinones et al., 2012; Petersson et al., 2007).

The thermal process is another kind of pre-treatment method used in AD. It utilises high temperature (130 – 180 ^{o}C) in conjunction with pressure (5 - 8 bar) to break the cell structure of organic substrate (Buddle et al., 2008). De-coupling and hydrolysing the protein and starch, make the complex substrate more readily available for bacterial consumption (Bishnoi, 2012; McCartyet al., 1986; Haug et al., 1983). According to Jolis et al. (2008), thermal pre-treatment achieved a 55 to 60 % volatile solids destruction.

Furthermore, ultrasonic pre-treatment is applied in the catabolism of complex substrate like sewage sludge (Carrere et al., 2010). In this process, cell structure of sludge is mechanically disrupted due to induced cavitation (Carrere et al., 2010; Tiehm et al., 1997). It is found that cell disintegration changes the characteristics of sludge, resulting in an increase in biodegradability and improving volatile solid removal (Tiehm et al., 2001; Tiehm et al., 1997). In addition, it enhances biogas production (Erden and Filibeli, 2009; Perez-Elvira et al., 2009; Salsabil et al., 2009; Ward et al., 2008; Braguglia et al., 2008; Neis and Nickel, 2008; Xie et al., 2007; Bougrier et al., 2005; Bien et al., 2004; Chu et al., 2002; Onyeche et al., 2002; Wang, et al., 1999).

3.3 Co-digestion of different substrates

Co-digestion is a process in AD by which two or more substrates are homogenously blended and simultaneously digested (Agdag and Sponza, 2007). The AD was initially applied to a single substrate, single purpose treatment plant (Wu, 2007). However, studies have shown that AD process is more stable and achieves more potential benefits as a result of co-digestion (Wu, 2007; Hartmann and Ahring, 2005; Murto et al., 2004; Mata-Alvarez et al., 2000). Co-digestion enhances biogas production due to the supply of the required nutrients by the co-substrates involved (Mata-Alvarez et al., 2000). This achievement in nutrient balance leads to better

digester performance and improved biogas production (Wu, 2007; Hartmann and Ahring, 2005). Murto et al. (2004) reported that co-digestion of solid slaughterhouse waste, fruit, vegetable and manure improved the buffering capacity of digester – the ability of digester to react to changes in pH. In other words, the buffering capacity of the digester is the measure of the amount of alkalinity present in the digester.

In addition, co-digestion is found to maintain the appropriate balance of C/N ratio in AD (Desai et al., 1994) and to reduce the concentration of nitrogen (Cuetos et al., 2008). For instance, the mixture of substrate with low nitrogen content and lipid is found to enhance biogas production (Castillo et al., 2006) as well as reduce the risk of acid accumulation and high concentration of ammonia that cause digester failure (Khalid et al., 2011; Castillo et al., 2006). In another study, co-digestion of the substrate that contains high moisture (liquid manure or sewage sludge) and substrate of poor moisture content is found to enhance the total solids (TS) content of digester (AgSTAR, 2012). Similarly, co-digestion involving substrate with high moisture content is found to dilute toxic compounds, thereby improving digester performance and biogas production (AgSTAR, 2012; Khalid et al., 2011; Braun, 2002).

Furthermore, a study by Macias-Corral et al., (2008) revealed that blending and digesting cow manure and organic fraction of municipal solid waste (OFMSW) enhances methane production. Other benefits derived from co-digestion include increase in biodegradation of organic materials (AgSTAR, 2012), stability and digestion rate improvement (Cornell et al., 2012) as well as higher mass conversion (Kangle et al., 2012), resulting in lower weight and volume of digestate (effluent). However, co-digestion has some disadvantages such as, an increase in digester effluent COD (chemical oxygen demand), need for additional pre-treatment, higher mixing requirement and higher energy requirement (Braun, 2002). Co-

digestion is a well-established practice in Europe, especially in Germany and Scandinavia (Appels et al., 2011; Environment Agency, 2010; Braun and Wellinger, 2009). Co-digestion plants for treating food waste and sewage sludge have been built in Germany, Switzerland and Denmark (Braun and Wellinger, 2009). The first co-digestion plant built in the UK is for the digestion of animal manure and food waste (Environment Agency, 2010). However, the co-digestion of food waste and sewage sludge is not in existence yet in the UK due to the different regulatory and management regimes guiding the digestion of the two substrates (Iacovidou et al., 2012; Environment Agency, 2010).

3.4 Process modelling

Process models have brought about simplification of processes and utilised to describe and predict the behaviour of processes as well as their required outputs (Sulaiman et al., 2011). Process models play an essential role in process understanding, process development, online diagnostic, and process automation (Craven et al. 2012; Sulaiman et al., 2011; Tham, 2000). Choosing the appropriate model depends on a number of factors such as the intended application, quality, quantity and the nature of the available experimental data (Craven et al. 2012; Lombardozzi and Sparks 2012; Müller et al., 2009; Bernacchi et al., 2009; Gómez et al., 2005; Hoffman et al., 2004; Boomen et al., 2002). Process models can be categorised as mathematical, statistical and quantitative. Mathematical models are further divided into mechanistic (white-box) and empirical (black-box) model as illustrated in Figure 3.1 (Vázquez-Cruz et al, 2014; Ljung, 2001).

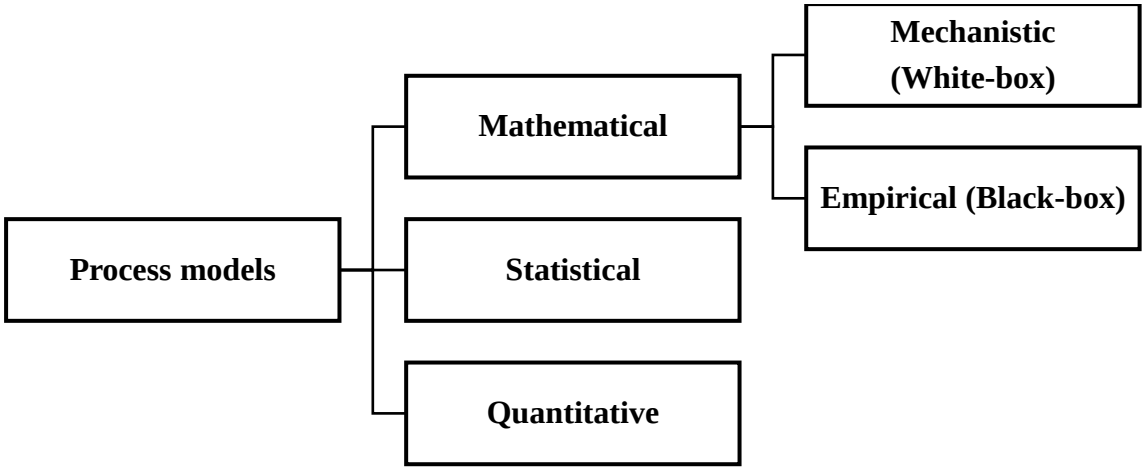


Fig. 3.1. Classification of process models

3.4.1 Mathematical models description

The complexity associated with bioprocess, which includes AD process, means that modelling is required in order to describe and predict the behaviour of the process in real life. As mentioned above, mathematical models are classified into mechanistic (white-box) and empirical (black-box) models (Vázquez-Cruz et al, 2014; Ljung, 2001). However, there is also another type of mathematical model known as a gray-box model, which combines the characteristics of white-box and black-box models (Vázquez-Cruz et al, 2014; Ljung, 2001). Henceforth in this thesis, mechanistic and empirical models are referred to as white-box and black-box models, respectively.

White-box model: This is the type of mathematical model that is constructed based on the prior knowledge of the chemical, physical and biological sub-processes of the system (Vázquez-Cruz et al, 2014; Ljung, 2001). It is constructed based on the kinetics and stoichiometry of the individual identified reaction (Fang, 2010). White-box model usually employs a state-space model construction method to define the internal structure of the complex biochemical and physico-chemical processes (Fang, 2010). White-box model can rely

on many assumptions and can be difficult to construct due to the complex nature of biochemical reactions (Yu et al., 2013; Fang, 2010)

The Monod kinetics model, which is one of the most used mathematical models for bioprocess is a white-box model (Craven et al. 2012; Liu et al., 2008; Zeng et al., 1998; Mitsdorffer, 1991; Glacken et al., 1989; Bree et al., 1988; Bergter, 1983; Chen and Hashimoto, 1978; Powell, 1967; Contois, 1959; Moser, 1958). The Monod kinetics model for bacterial growth depends on the relationship between specific growth rate and substrate concentration (Gerber and Span, 2008).

The relationship between the substrate flow rate and the volume of the digester is the dilution rate (Stanbury et al. 1995).

$$D = \frac{F}{V} \tag{3.1}$$

Where,

D is the dilution rate (h^{-1}), which is the ratio of the volumetric flow rate of nutrient supplied to the digester and the volume of the digester

V is the volume of digester (m^3)

F is the volumetric flow rate (m^3h^{-1})

In addition, the rate of change of bacterial concentration is as follows:

$$\frac{dX}{dt} = (\mu - D)\, X \tag{3.2}$$

Where,

X is the bacterial concentration (mg/l)

t is the time of incubation (day)

μ is the specific growth rate (day^{-1})

The bacterial growth is μX, while the bacterial output is DX.

Under steady-state conditions, the new substrate fed into the digester balances the loss of bacteria from the digester.

$$\frac{dX}{dt} = 0 \tag{3.3}$$

Then, Eq. 3.2 becomes,

$$\mu = D \tag{3.4}$$

According to Monod (1949), the nonlinear relationship between substrate concentration and specific bacterial growth rate is:

$$\mu = \mu_{max} \frac{S}{K_s + S} \tag{3.5}$$

Where,

μ is the specific growth rate (day^{-1})

μ_{max} is the maximum specific growth rate (day^{-1})

S is the substrate concentration (mg/l)

K_s is the half-velocity constant (g/l), meaning the substrate concentration at one-half of the maximum growth rate ($\mu_{max}/2$).

The Monod model for bacterial growth shows that the specific rate of bacterial growth increases rapidly at low substrate concentration and vice versa until a bacterial saturation level is attained, as illustrated in Figure 3.2 (Gerber and Span, 2008). This indicates that the substrate concentration is the limiting factor for the bacterial growth rate. The saturation point is the maximum specific bacterial growth rate (μ_{max}).

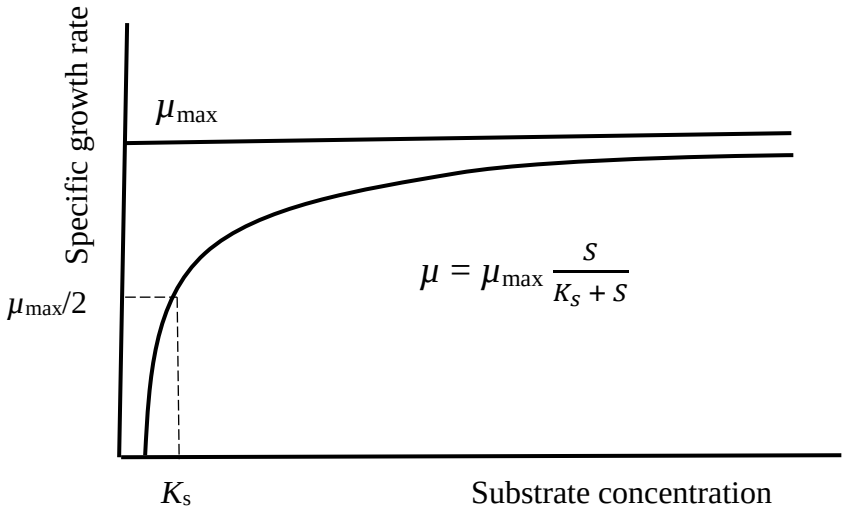


Fig. 3.2. Monod specific growth rate

According to Contois (1959), the accuracy of the Monod model is high for pure bacterial cultures and simple substrates. In addition, Grady (1969) concluded that μ_{max} is exclusive for individual bacterial cultures. However, Pfeffer (1974) suggested that the Monod model is insufficient to describe the degradation of complex substrate like municipal wastes. te Boekhorst et al. (1981) supported the finding of Pfeffer (1974) and added that the Monod model is sufficient for homogenous bacterial cultures, but cannot be used to describe processes involving heterogeneous bacterial cultures. Due to the shortfalls associated with Monod kinetics model, other authors modified the model as shown in Table 3.1.

Table 3.1. List of the modified Monod models

Model	Author	Eq. No.
$\mu = \mu_{max} \frac{S^n}{K_s + S^n}$	Moser, 1958	(3.6)
$\mu = \mu_{max} \frac{S}{K_c + X + S} = \mu_{max} \frac{S}{\frac{K_c \cdot X}{S} + 1}$	Contois, 1959	(3.7)
$\mu = \mu_{max} \frac{(K+L+S)}{2 \cdot L} \cdot \left(1 - \sqrt{1 - \frac{4 \cdot L \cdot S}{(K+L+S)^2}^2}\right)$	Powell,1967	(3.8)
$\mu = \mu_{max} \frac{S/S_1}{K + \frac{(1-K) \cdot S}{S_1}}$	Chen and Hashimoto, 1978	(3.9)
$\mu = \mu_{max} \frac{S}{K_s + S} \cdot \left[1 - \exp\left(-\frac{t}{T}\right)\right]$	Bergter, 1983	(3.10)
$\mu = \mu_{max} \frac{S^n}{S^{n \cdot} (1 + K_b \cdot G_s \cdot S^n)}$	Mitsdorffer, 1991	(3.11)

However, despite the various modifications of the Monod model for bacterial growth, it is concluded that these models with only one set of kinetic parameters are insufficient to describe biological processes of any retention time or degradation of complex substrates (Gerber and Span, 2008; Rao and Singh, 2004). Hence, the first-order models, which are also a type of white-box model, are developed (Gerber and Span, 2008; Rao and Singh, 2004). An example of the first-order model is the model developed by Rao and Singh, (2004).

$$k\left(\frac{\mathrm{d}S}{\mathrm{d}t} = -k\,.S\right) \tag{3.12}$$

Where,

k is the kinetics of bacterial growth due to enzyme activity

The first-order kinetics were applied in the hydrolytic step of the models developed by Bryers (1985), Angleidaki et al. (1999), Knobel and Lewis (2002) and Siegrist et al. (2002). However, Shin and Song (1995) applied the first-order kinetics for all the steps of the process. Although, the first-order models are easy to deal with, the accuracy is for confined requirements only (Hashimoto et al., 1981). Hashimoto et al. (1981) concluded that it is not possible to use first-order models for the prediction of optimum conditions of maximum biological activity or process failure.

Furthermore, the Anaerobic Digestion Model 1 (ADM1) developed by the International Water Association (IWA) task group in 2002 is a white-box, state-space model (Batstone et al., 2002). It utilised the theoretical concepts of material conservation and biochemical reactions to construct the structure of the model (Fang, 2010). The constituent bacterial population and processes of ADM1 are based on several assumptions in order to keep the model simple (Yu et al., 2013; Fang, 2010). In addition, the ADM1 requires several input parameters

implemented in the form of differential algebraic equations (DAE) and differential equations (DE) (Yu et al., 2013; Batstone et al., 2002)

Grey-box model: This is a combination of the white-box and black-box model. It is applied when there is not enough theoretical knowledge of the system to construct a white-box model. Then, the construction of the model is based on the prior knowledge of the system dynamics and the estimation of the unknown model parameters from measured input-output data (Vázquez-Cruz et al, 2014; Krishna, 2010; Brus, 2005). The disadvantage of grey-box modelling is that it requires a large number of parameters for a detailed complex system such as AD process (Brus, 2005).

Black-box model: These models utilise data generated from the process to define the relationship between input and output variables (Vázquez-Cruz et al, 2014; Krishna, 2010). The block diagram of black-box model is shown in Figure 3.3.

Fig.3.3. Block diagram of black-box process

Black-box models depend on the availability of data for model estimation and validation (Vázquez-Cruz et al, 2014). According to Krishna (2010), the main disadvantage of black-box models is that the functions of the parameters lack deep physical significance – unable to describe process parameters such as mass transfer coefficients, heat and reaction kinetics. However, black-box models can adequately represent the trends in process behaviour and are as effective as the white-box models. In addition, black-box models have the advantage of

sufficiently modelling a process when knowledge about the process is vague or in the case of a complex process whereby it is impossible to solve the resulting equations (Krishna, 2010). Furthermore, in a number of situations, it is impractically feasible to apply white-box models due to financial and time constraints; hence, black-box models are utilised (Krishna, 2010).

The Black-box models are further classified into linear and nonlinear forms. The linear category is made up of the main transfer function and time-series models, and the nonlinear type is composed of neural network and time-series, as illustrated in Figure 3.4 (Vázquez-Cruz et al, 2014; André, 2013; Kumar and Zhao, 2011; Krishna, 2010; Stein et al., 2007; Aufhammeret et al., 2006).

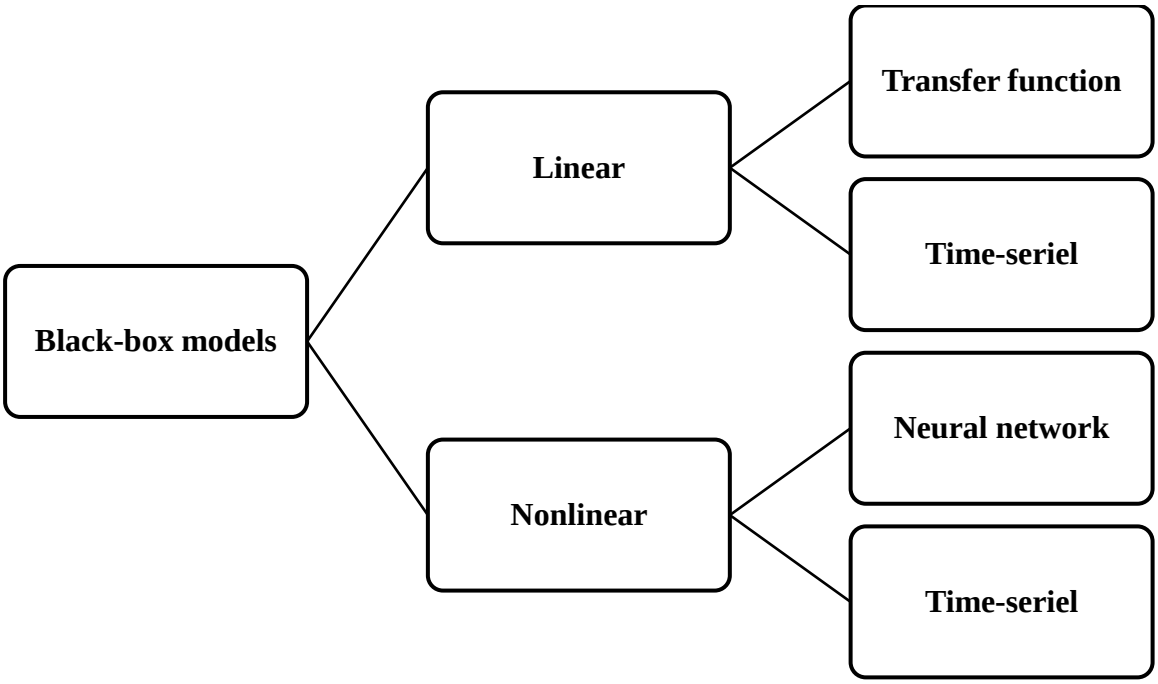


Fig.3.4. Classification of black-box models

3.4.2 Linear black-box models

For general linear models in time-domain, the following equation describes the model.

$$y(t) = G(z^{-1}, \theta)u(t) + H(z^{-1}, \theta)\,\varepsilon(t) \qquad (3.13)$$

Where,

$u(t)$ is the system input at time t

$y(t)$ is the system output at time t

$G(z^{-1}, \theta)$ is the transfer function of the deterministic part of the system

$H(z^{-1}, \theta)$ is the transfer function of the stochastic part of the system

$e(t)$ is the disturbance of the system which is usually zero-mean white noise

z^{-1} is the backward shift operator

The $G(z^{-1}, \theta)$ specifies the relationship between the input and output variable. Whilst the $H(z^{-1}, \theta)$ specifies the effects of the random disturbance on the output variable (Ljung, 2001).

For linear black-box models, G and H in Eq. 3.13 are rational transfer functions in the delay operator with unknown numerator and denominator polynomials defined by the following equations (Ljung, 2001):

$$G(z^{-1}, \theta) = \frac{B(z,\theta)}{A(z,\theta)F(z,\theta)} \tag{3.14}$$

Similarly,

$$H(z^{-1}, \theta) = \frac{C(z,\theta)}{A(z,\theta)D(z,\theta)} \tag{3.15}$$

The perimeter vector θ is the set of model parameters that contain the coefficients b_i, c_i, d_i and f_i of the transfer functions, which are eliminated in the following equations to make them appear simpler. Hence, Eq. 3.13 becomes;

$$A(z)y(t) = \frac{B(z)}{F(z)}u(t - n) + \frac{C(z)}{D(z)}\varepsilon(t) \tag{3.16}$$

Where,

n is the system delay

$A(z)$, $B(z)$, $C(z)$, $D(z)$ and $F(z)$ are polynomials in connection with the backward shift operator and described by the following equations.

$$A(z) = 1 + a_1 z^{-1} + \cdots + a_{na} z^{-na} \tag{3.17}$$

$$B(z) = 1 + b_1 z^{-1} + \cdots + b_{nb} z^{-nb} \tag{3.18}$$

$$C(z) = 1 + c_1 z^{-1} + \cdots + c_{nc} z^{-nc} \tag{3.19}$$

$$D(z) = 1 + d_1 z^{-1} + \cdots + d_{nd} z^{-nd} \tag{3.20}$$

$$F(z) = 1 + f_1 z^{-1} + \cdots + f_{nf} z^{-nf} \tag{3.21}$$

Where *na*, *nb*, *nc*, *nd*, and *nf* are structural parameters.

However, when one or more of $A(z)$, $C(z)$, $D(z)$ and $F(z)$ are set to 1, then simpler models are created such as Box-Jenkins (BJ), output error (OE), autoregressive with exogenous terms (ARX) and autoregressive-moving average with exogenous terms (ARMAX) models. For instance, when $C(z)$, $D(z)$ and $F(z)$ are set to one, ARX model is created.

$$A(z)y(t) = B(z)u(t - n) + \varepsilon(t) \tag{3.22}$$

When $A(z)$, $C(z)$ and $D(z)$ are equal to1, the resultant model is the OE model.

$$y(t) = \frac{B(z)}{F(z)} u(t - n) + \varepsilon(t) \tag{3.23}$$

When $A(z)$ is equal to1, the general linear model becomes the BJ model.

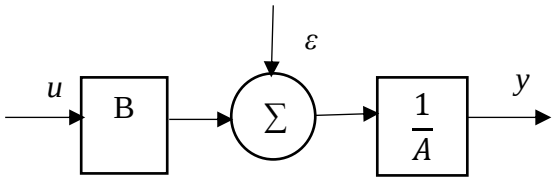
$$y(t) = \frac{B(z)}{F(z)} u(t - n) + \frac{C(z)}{D(z)} \varepsilon(t) \tag{3.24}$$

When $D(z)$ and $F(z)$ are equal to1, the general linear model is reduced to ARMAX model.

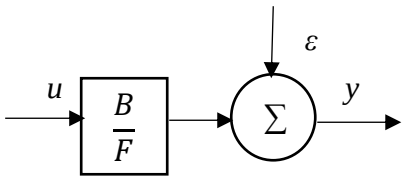
$$A(z)y(t) = B(z)u(t - n) + C(z)\, \varepsilon(t) \tag{3.25}$$

Figure 3.5 represents the different linear black-box models.

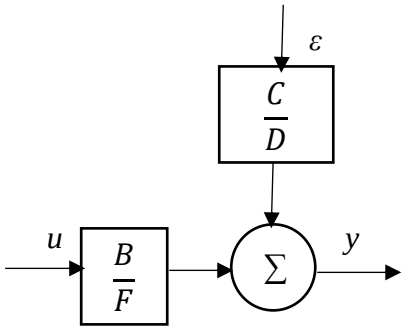
ARX



OE



BJ



ARMAX

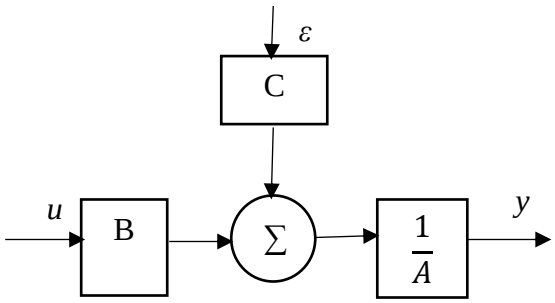


Fig. 3.5. Different linear black-box models

Furthermore, the predictor associated with Eq. 3.16 is of "pseudo-linear" regression form (Sjoberg et al., 1995; Liung and Soderstrom, 1983).

$$\hat{y}(t|\theta) = \theta^{T} \varphi(t, \theta) \tag{3.26}$$

The regressors, which are the components of $\varphi(t, \theta)$ are in the following general forms (Sjoberg et al., 1995):

- $u(t-k)$ is associated with the B-polynomial
- $y(t-k)$ is associated with the A-polynomial

- $\hat{y}_u(t|\theta)$ is the simulated output from past input (u) only and is associated with the F-polynomial
- $\varepsilon(t-k) = y(t-k) - \hat{y}(t-k|\theta)$ is the prediction error associated with the C-polynomial
- $\varepsilon_u(t-k) = y(t-k) - \hat{y}_u(t-k|\theta)$ is the simulation error associated with the D-polynomial

In addition, linear state-space models are common representations of dynamic models. They are similar to the ARX models, describing the same kind of linear relationship between inputs and outputs (Ljung, 2002).

$$x(t+1) = Ax(t) + Bu(t) + K\varepsilon(t) \quad (3.27)$$

$$y(t) = Cx(t) + Du(t) + \varepsilon(t) \quad (3.28)$$

Where,

$x(t)$ is the vector of state variables

A, B, C and K are the matrices

If $K = 0$, it means that noise source $\varepsilon(t)$ affects only the output. Also, if $D = 0$ it means that there is no direct influence from $u(t)$ to $y(t)$.

3.4.3 Nonlinear black-box models

The nonlinear black-box models are subdivided into time-series and neural network based models as shown in Figure 3.1. In the nonlinear time-series models, the behaviour of the process is modelled by combining the weighted cross-products and powers of the variables in the representation (Krishna, 2010). However, the perimeters of the functions remained linear, thereby facilitating the identification process.

Black-box models can be dynamic or static depending on their relationship with time. If the observed inputs (u) and outputs (y), form a dynamic system:

$$u^t = [u(1), u(2), \ldots, u(t)] \tag{3.29}$$

$$y^t = [y(1), y(2), \ldots, u(t)] \tag{3.30}$$

Then, the general nonlinear dynamic black-box model is as follows:

$$y(t) = g(u^{t-1}, y^{t-1}) + \varepsilon(t) \tag{3.31}$$

Where,

$y(t)$ is a vector that contains the outputs at time t

$u(t)$ is a vector that contains the inputs at time t

$g(u^{t-1}, y^{t-1})$ is the model structure to be selected

$\varepsilon(t)$ is the noise on the output at time t

The additional term $\varepsilon(t)$ in Eq. 3.31 denotes that the subsequent output, $y(t)$, is not an exact function of the past data. However, $\varepsilon(t)$ must be small so that given the past data, $g(u^{t-1}, y^{t-1})$ is a good predictor of $y(t)$.

In order to find the nonlinear function g in Eq. 3.31, $g(u^{t-1}, y^{t-1})$ is parameterised with a finite-dimensional parameter vector θ (Ljung, 2001; Sjoberg et al., 1995). Then, $g(u^{t-1}, y^{t-1})$ becomes $g(u^{t-1}, y^{t-1}, \theta)$. According to Sjoberg et al, (1995), this parameterisation is usually an approximation of the quality assessed by means of the fit between the model and the data.

Though, mapping the increasing number of past observations (u^t, y^t) into a finite dimensional vector $\varphi(t)$ of fixed dimension,

$$g(u^{t-1}, y^{t-1}, \theta) = g(\varphi(t), \theta) \tag{3.32}$$

Where,

$$\varphi(t) = \varphi(u^{t-1}, y^{t-1}) \tag{3.33}$$

In Eq. 3.33, the vector $\varphi(t)$ is called regression vector, while its components on the right-hand of the equation are referred to as regressors (Sjoberg et al., 1995). Parameterising the regressors gives:

$$\varphi(t) = \varphi(u^{t-1}, y^{t-1}, \theta) \tag{3.34}$$

In addition, similar to the linear black-box regression structure in Eq. 3.26, the nonlinear case is of the following form.

$$\hat{y}(t|\theta) = g(\varphi(t), \theta) \tag{3.35}$$

For the input and output case, $u(t-k)$ and $y(t-k)$ are measured variables. While $\hat{y}_u(t|\theta)$, $\varepsilon(t-k) = y(t-k) - \hat{y}(t-k|\theta)$ and $\varepsilon_u(t-k) = y(t-k) - \hat{y}_u(t-k|\theta)$ are based on preceding outputs from the black-box model $\hat{y}(t-k|\theta)$. If the measured outputs $y(t-k)$ in the regressors are substituted by the last simulated output $\hat{y}(t-k|\theta)$ then, the output from Eq. 3.35 becomes equal to $\hat{y}_u(t|\theta)$ (Sjoberg et al., 1995). The following are examples of nonlinear black-box model structures.

- Nonlinear autoregressive with exogenous terms (NARX) models use $u(t-k)$ and $y(t-k)$ as regressors
- Nonlinear output error (NOE) models use $u(t-k)$ and $\hat{y}_u(t-k|\theta)$ as regressors
- Nonlinear autoregressive moving average with exogenous terms (NARMAX) models use $u(t-k)$, $y(t-k)$ and $\varepsilon(t-k|\theta)$ as regressors
- Nonlinear Box-Jenkins (NBJ) models use $u(t-k)$, $\hat{y}(t-k|\theta)$, $\varepsilon(t-k|\theta)$ and $\varepsilon_u(t-k)$ as regressors
- Nonlinear Hammerstein-Wiener (NLHW) models use $u(t-k)$ and $\hat{y}_u(t-k|\theta)$ as regressors

However, the NOE, NARMAX and NBJ as well as the nonlinear state-space models are considered recurrent structures (Ali, 2010; Sjoberg et al., 1995). This is because the past

outputs from the models have to be fed back into the model computation, thereby required more considerable computation to fit the data (Ali, 2010; Sjoberg et al., 1995). In addition, it is difficult to check the conditions under which the obtained predictor model is stable (Sjoberg et al., 1995).

The black-box modelling method has been applied in a number of AD systems, such as the model of Beck (1980), which utilised black-box method for the identification of a single-input/single-output (SISO) model for volatile acid concentration as input and volumetric gas flow rate as output. Also, black-box models were used to model the combustion process of the internal combustion engine (Maass, 2011).

Neural network

The neural network has gained prominence in nonlinear modelling (Billings, 2013a). It is a computational model that interconnects a large number of computational units called neurons, to form a network capable of performing complex computational tasks (Billings, 2013b; Sulaiman et al., 2011). Generally, with the use of certain learning algorithm, the neural network receives training to learn and to represent the data set (Billings, 2013a). The process of determining the weights that decide the strength of the connection between the neurons in the network and enable it to model the mechanism that yielded the data set involves learning a mathematical expression of the system (Billings, 2013a).

The neural network has the advantage of robustness, suitable for nonlinear models, very adaptive in nature and able to function even when there is a failure in one of the elements (Billings, 2013a). It does not require the prior knowledge of the interrelationships that existed between the input and output variables. Rather, what it needed to function is the specification

of the network design and enough quantity of steady input data (Yu et al., 2012). However, the limitation of a neural network for AD process model development is the difficulty in utilising the model to design a digester or scale up AD system (Yu, 2013). This is due to the various level of input-output relationships required to train the model for real life process application, which could be susceptible to overfitting or underfitting, as well as takes a long time to process large networks (Tabatabaei et al., 2010).

3.5 Summary

This chapter has presented some improvements made in AD technology. Pre-treatment is an important development in AD process as it reduces the substrate particle size, especially in hydrolysis. Consequently, it increases the available specific surface area that facilitates biodegradation, which in effect enhances biogas production, as well as reducing the quantity of residue generation. In addition, co-digestion of two or more substrates has shown to enhance biogas production. This is due to the combined positive effect achieved by the supply of the required nutrients from the co-substrates involved. Furthermore, the chapter discussed the different mathematical models, mainly the white-box and black-box models. The various mathematical models provide the possibility of describing and predicting the behaviour of AD process. The discussion in the next chapter focuses on the experiments conducted in this study.

Chapter 4 **Experimental Apparatus and Methodology**

4.1 Introduction

The AD is a multi-step process involving several groups of anaerobic bacteria. These groups of bacteria promote a series of sequential biochemical reactions/processes in converting organic material to biogas (Baker and Evans, 2009). The biochemical processes consist of the following four main steps; hydrolysis, acidogenesis, acetogenesis and methanogenesis (Korres et al., 2013; Zieminski and Frac, 2012; Nwuche and Ugoji, 2010; Amani, 2011; Al Seadi et al., 2008). This chapter presents the discussion of the biochemical processes of AD that result in biogas production. It also discussed the theoretical chemical conversion of complex organic compounds to molecules of methane and carbon dioxide. Furthermore, the chapter describes the laboratory experiments conducted in this study, which investigates the influence of temperature, pH, mixing speed and pressure on biogas and methane production.

4.2 Bacteriological and biochemical processes in AD

The AD is the process by which bacteria breakdown organic matter in the absence of oxygen to produce biogas and digestate (NNFCC, 2011). Biogas is a mixture of methane (CH_4), carbon dioxide (CO_2) and traces of other gases, including ammonia (NH_3), hydrogen (H_2) and hydrogen sulphide (H_2S) (NNFCC, 2011; Hiremath et al., 2009; Gallert and Winter, 2005; Kelleher et al., 2002; McKendry, 2002; Veeken et al., 2000).

The AD is a process that depends on the complex interaction of different species of bacteria (Kangle et al., 2012; Baker and Evans, 2009). In order to achieve a stable AD process and optimal biogas production, it is important to maintain the balance between the various bacterial groups (Wang, 2014). This balance could be affected by changes in the physico-environmental

conditions, which could result in accumulation of intermediary products (short-chained organic acid molecules) that may inhibit the entire AD process (Hooshyari et al., 2009; Wong et al., 2009; Liu et al, 2009; de Bok et al, 2004). The short-chained organic acid molecules are called volatile fatty acids (VFAs) (Marchaim, 1992).

The conversion of organic material to biogas using AD technology is a complex biochemical reaction that is composed of successive multi-step processes and parallel reactions (Korres et al., 2013; Kangle et al., 2012; Khalid et al., 2011; McCarty, 1982). The degradation process of complex organic polymers involves interrelated steps that facilitate the catabolism of organic material to smaller units. Each of the stages is performed by a specific group of bacteria, which successively break down the product of the previous step. The putrefaction of organic material takes place in four sequential steps, which are hydrolysis, acidogenesis, acetogenesis and methanogenesis illustrated in Figure 4.1 (Korres et al., 2013; Zieminski and Frac, 2012; Al Seadi et al., 2008; Veeken et al., 2000; Marchaim, 1992).

Hydrolysis: This is the first stage of AD process, at which organic polymers are broken down into soluble monomers by hydrolytic enzymes excreted by hydrolytic bacteria (Kangle et al., 2012). At this step, carbohydrates are hydrolysed into glucose, proteins converted into amino acids while lipids are transformed into glycerol and fatty acids (Zieminski and Frac, 2012; Nayono, 2009; Al Seadi, 2008). The conversion of glucose into simple sugar is illustrated in Eq. 4.1 (Korres et al., 2013; Ostrem, 2004; Themelis and Verma, 2004).

$$C_6H_{10}O_4 + 2H_2O \longrightarrow C_6H_{12}O_6 + 2H_2 \qquad (4.1)$$

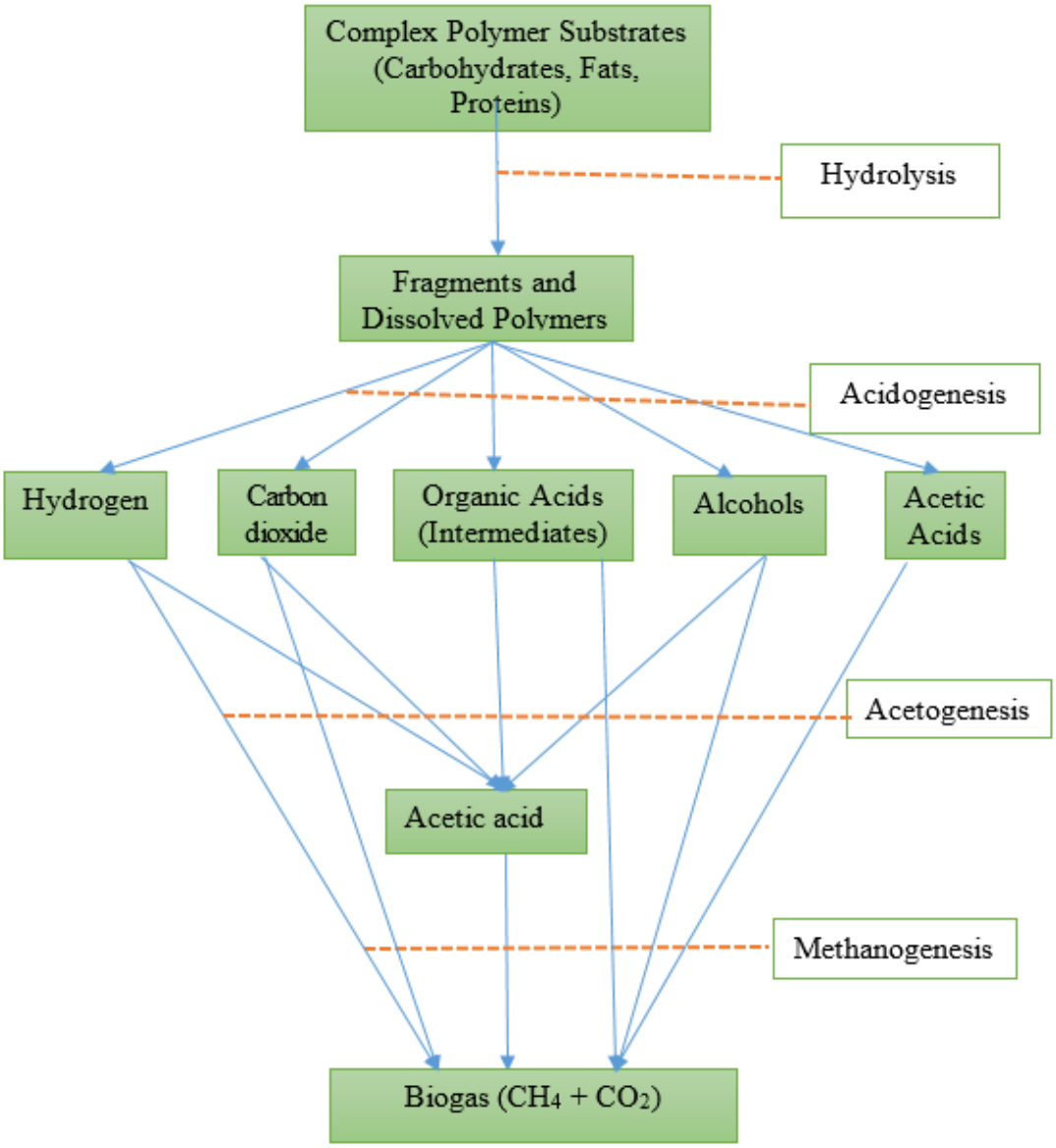


Fig.4.1. Stages in AD process

Acidogenesis: The second stage of AD process is also referred to as fermentation. The products of hydrolysis, simple sugars, amino acids and fatty acids are catabolised by acidogenic bacteria to form acetate, carbon dioxide, hydrogen, VFAs and alcohols. At this stage, acetate can be utilised by methanogenic bacteria and converted directly to methane (Nayono, 2009; Al Seadi, 2008). The following three chemical equations illustrate the three different conversions of

glucose by acidogenic bacteria into ethanol, propionate acid and acetic acid, as signifies in Eq. 4.2, Eq. 4.3 and Eq. 4.4, respectively (Korres et al., 2013; Ostrem, 2004; Themelis and Verma, 2004; Bilitewski et al., 1997; Marchaim, 1992).

$$C_6H_{12}O_6 \longleftrightarrow 2CH_3CH_2OH + 2CO_2 \quad (4.2)$$

$$C_6H_{12}O_6 + 2H_2 \longleftrightarrow 2CH_3CH_2COOH + 2H_2O \quad (4.3)$$

$$C_6H_{12}O_6 \longrightarrow 3CH_3COOH \quad (4.4)$$

Acetogenesis: At this stage, the products of acidogenesis that have not yet transformed to methane by methanogenic bacteria are converted into the methanogenic substrate by acetogenic bacteria. Low molecular VFAs are oxidised into acetate, hydrogen gas and carbon dioxide (Nayono, 2009; Al Seadi et al., 2008; Ostrem, 2004; Themelis and Verma, 2004). Also in acetogenesis, VFAs that have carbon chains longer than two units, as well as alcohols with carbon chains longer than one unit are oxidised into acetate or propionate and hydrogen gas (Al Seadi et al., 2008). The presence of hydrogen in this phase raises the hydrogen partial pressure that can inhibit the oxidation process (Al Seadi et al., 2008). For continuous conversion of VFAs into acetate, hydrogen gas and carbon dioxide, it is required to reduce the hydrogen partial pressure enough to thermodynamically let the process proceed (Nayono, 2009; Ostrem, 2004). Lowering the hydrogen partial pressure can be achieved by the presence of hydrogen-consuming bacteria in the digestion process, leading to the conversion of all the VFAs (Nayono, 2009; Ostrem, 2004). The concentration of hydrogen measured by its partial pressure can be an indicator of the performance of AD process (Mata-Alvarez, 2003). Examples of acetogenic reactions are illustrated in the conversion of propionate, glucose, ethanol and bicarbonate to acetate in Eq. 4.5, Eq. 4.6, Eq. 4.7 and Eq. 4.8, respectively (Korres et al., 2013; Ostrem, 2004; Marchaim, 1992; McInerney et al. 1981).

$$CH_3CH_2COO^- + 3H_2O \longleftrightarrow CH_2COO^- + H^+ + HCO_3^- + 2H_2 \quad (4.5)$$

$$C_6H_{12}O_6 + 2H_2O \longleftrightarrow 2CH_3COOH + 2CO_2 + 4H_2 \quad (4.6)$$

$$CH_3CH_2OH + 2H_2O \longleftrightarrow CH_3COO^- + 2H_2 + H^+ \quad (4.7)$$

$$2HCO_3^- + 4H_2 + H^+ \longleftrightarrow CH_3COO^- + 2H_2O \quad (4.8)$$

Methanogenesis: This is the final stage of AD process; the VFAs are converted to methane and carbon dioxide, which is biogas, by the activities of methanogenic bacteria. The conversion of acetate to methane and carbon dioxide is presented in Eq. 4.9 and Eq. 4.10, while the transformation of alcohol into biogas is shown in Eq. 4.11. Furthermore, the conversion of hydrogen and carbon dioxide to biogas is presented in Eq. 4.12 (Korres et al., 2013; Nayono, 2009; Al Seadi et al., 2008; United Tech, 2003; Omstead et al., 1980; Thauer et al., 1977).

$$2CH_3CH_3OH + CO_2 \longrightarrow 2CH_3COOH + CH_4 \quad (4.9)$$

$$CH_3COOH \longrightarrow CH_4 + CO_2 \quad (4.10)$$

$$2C_2H_5OH + CO_2 \longrightarrow 2CH_3COOH + CH_4 \quad (4.11)$$

$$CO_2 + 4H_2 \longrightarrow CH_4 + 2H_2O \quad (4.12)$$

The stability of anaerobic process depends on methanogenesis. This is due to the slower growth of methanogenic bacteria than other groups of anaerobic bacteria involved, besides very sensitive to changes in operating parameters (Wang, 2014; Zupancic and Grilc, 2012). Because of these reasons, methanogenesis is considered the rate-limiting step of AD process (Zupancic and Grilc, 2012). Methanogenic bacteria multiply and perform optimally at neutral pH condition (Nayono, 2009; Al Seadi et al., 2008; Gas Technology, 2003). Sudden temperature

change can inhibit the activity of methanogenic bacteria, resulting in less or no methane production (Çalli, 2012; Gao et al., 2011; Ahn and Forster, 2002). According to Davis and Cornwell (1998), the kinetics of methanogenesis determines the kinetics of the entire anaerobic biochemical process.

4.3 Further review of the operating parameters

The influence of operating parameters on biogas production is very challenging especially in the rural areas of developing nations (Pham et al., 2014; Cu et al., 2012). Millions of biogas production plants in countries like China, India, Tibet, Pakistan, Bangladesh and Vietnam are utilised mainly to produce biogas for cooking and lighting purposes (Bruun et al., 2014). Even in these simple biogas plants, the impacts of the operating parameters were critical. In addition to the review of the operating parameters in Chapter 2, the four operating parameters (temperature, pH, mixing speed and pressure) investigated in this research work are further discussed in this section.

For a reaction to occur, reactants require kinetic energy that facilitates their collusion with one another (Brown et al., 2009). Increasing the temperature of the reaction causes reactants' molecules to gain more kinetic energy that enables them to move about faster and collide frequently (Brown et al., 2009), thereby increasing the rate of reaction. Similarly, the rate of metabolism in bioprocess increases with temperature (Hoegh-Guldberg and Bruno, 2010). In AD process, the rate of anaerobic reaction increases as the operating temperature rises, but up to a certain temperature, known as the optimum temperature (Calli, 2012; Kent, 2000). Apart from increasing the rate of reaction by increasing the heat energy input, substrates utilised for biological process also require energy for the reaction to proceed (Starr and McMillan, 2014). The required energy, referred to as activation energy, is ideally supplied to the substrates by

the input heat energy (Dominiczak, 2007; Alters, 2000). In order to mitigate the influence of temperature, several simple biogas digesters in the countries like China and India are buried underground to maintain the temperature within the digesters (Pham et al., 2014, Kossman et al., 1997).

A number of researchers have investigated the effect of temperature on biogas production (Ma et al., 2013; Gavala et al., 2003; Chen and Hashimoto, 1978). Usman et al. (2012) investigated the influence of temperature on biogas production from two maize samples, which were digested at different temperatures, 30 ℃, 45 ℃, and 60 ℃. The goal of the study was to determine the most suitable temperature for the process, that is, the temperature that achieved the highest biogas production from the given maize samples (Usman et al., 2012). Pandey and Soupir (2012) also conducted a study to determine how the given temperatures (25 ℃, 37 ℃, and 52.3 ℃) influenced biogas production from AD of dairy manure. Furthermore, Zhao (2011) investigated how a change in temperature affected the production of biogas from the treatment of domestic wastewater. The results of the reviewed studies show that without the right digester temperature the production of biogas is limited and can make AD less reliable and unattractive as a means of sustainable energy source.

Similarly, the importance of stabilising the process pH within the optimum range in AD cannot be overemphasised. As discussed in section 2.2.1, the impact of pH variation on biogas production is very significant. It shows that a significant decrease in pH below 7.0, the neutral value, could result in the acidic slurry in the digester, which could inhibit biogas production. However, the significant rise in pH above the neutral level causes the slurry to become alkaline, thereby limiting or halting biogas production. For this reasons, many studies have been conducted with the goal of investigating the impact of pH variation on biogas production.

Sumardiono et al. (2013) investigated the effect of COD/N (Chemical Oxygen Demand/Nitrogen) ratios of the substrate and pH control on biogas production from vinasse. The author found that at COD/N ratio of 600/7, the controlled pH sample had about 10% COD removal more than that of the fluctuating pH. The author concluded that the biogas production at the controlled neutral pH is more than the biogas generated at the fluctuating pH.

Another study was carried out utilising four similar samples of dairy wastewater, but with variable starting pH values (Kheiredine et al., 2014). It was found from the biodegradation test that the highest COD removal of 90.8% was recorded for the sample with initial pH of 7.0, followed by 79.64% for 9.5 pH, 63.75% for 5.5 pH and 49.11% for 4.0 pH. The experiment further shows that the highest methane content was produced by the sample that has the starting pH of 7.0, and it followed the same sequence as the results obtained from the biodegradation test, with negligible methane production as the pH decreases below 5.5 (Kheiredine et al., 2014). The biodegradation process signifies the degree and rate at which organic material is broken down by bacteria, and in this case, to produce methane and carbon dioxide (Kheiredine et al., 2014). Furthermore, Budiyono et al. (2013) conducted a similar biodegradation test with three samples of bioethanol waste containing different pH values at the start of the digestion process. The results obtained indicated that the sample that started with pH of 7.0 generated the highest biogas production of 3.81 mL/g COD, while the other two samples with initial pH of 6.0 and 8.0 produced 3.25 mL/g COD and 3.49 mL/g COD of biogas, respectively (Budiyono et al., 2013).

Similar to temperature and pH, the significance of mixing in AD to improve biogas production has been widely acknowledged (Sindall et al., 2013). However, relatively, less emphasis has

been made on exploring the specific impact of mixing speed on the production of biogas (Sindall et al., 2013). A number of researches were carried out to determine the effect of mixing speed on biogas production. A study conducted by Hoffmann et al. (2013), on the effect of mixing intensities on biogas production, utilising cow manure as substrate. In that study, four continuously stirred digesters containing similar samples at four different mixing speeds of 1500, 500, 250 and 50 rpm are operated. The author found that the performance of the digester stirred at 1500 rpm was adversely affected, leading to lower biogas production and higher VFA concentration. In contrast, the digester that operated at 500 rpm achieved the highest biogas production. Also observed is that the other two digesters operated at 250 and 50 rpm produced biogas marginally at the same rate, but higher than the 1500 rpm digester (Hoffmann et al., 2013).

Furthermore, another research utilised velocity gradient, through computational fluid dynamics (CFD) to determine the influence of mixing speed on biogas production. Velocity gradient is the velocity variance between adjacent layers of fluid (CEE, 2012). The author used velocity gradient as a proxy for mixing intensity of turbulence (Sindall et al., 2013). The results from the study showed that as the velocity gradient was lowered from 9.7 s^{-1} to 7.2 s^{-1}, which represented a reduction in the mixing speed from 100 to 50 rpm, increase in biogas production is recorded. Nevertheless, as the velocity gradient increased from 9.7 s^{-1} to 14.3 , for increasing mixing speed from 100 to 200 rpm, biogas production was found to decrease (Sindall et al., 2013).

The operating pressure is the last parameter investigated; however, less emphasis has been made on evaluating its effect on biogas and methane production (Chen et al., 2014). Research conducted by Abe and Horikoshi (2001) and cited by Chen et al. (2014), reported that

methanogenic bacteria can be metabolically active at a pressure up to 100 bar. In addition, it is reported that pressurised anaerobic digester can improve methane production, thereby, refining the quality of biogas (Chen et al., 2014; Lindeboom et al., 2011; Hayes et al., 1990). Increasing digester pressure liquefies the CO_2 content of biogas, which decreases the overall biogas production, but improves the quality of biogas produced as it contains a higher proportion of methane (Lindeboom et al., 2011).

Similarly, a study performed by Chen et al. (2014) to investigate the impact of two different digester pressure, 1.5 and 9 bar, on biogas production, revealed that biogas generated by the 9 bar digester contained about 74.5 % methane. Whilst biogas produced by the 1.5 bar digester contained approximately 66.2 % methane. The outcome of this experimentation signifies that increasing the operational digester pressure can improve the methane content of the biogas produced.

The reason why CO_2 liquefied in the two studies cited is that it has a higher solubility in water compared to methane. From the solubility table, the solubility of CO_2 and methane in water (1ml/100ml) at 20 ℃ and 1atm is 0.782 and 0.032 g/kg, respectively (Yalkowsky et al., 2010). This means that CO_2 dissolves in water more than methane at the same temperature and pressure. According to Henry's law, the solubility of a gas in a liquid is directly proportional to the pressure of that gas above the surface of the solution (Yalkowsky et al., 2010). However, according to Japan-Agency for Marine-Earth Science and Technology (2007), it is difficult to explain the effects of pressure on complex metabolic systems, like AD, based on a simple volume law. Consequently, it is difficult to describe the influence of pressure on the actual production of biogas.

This study has shown the impact of each of the operating parameters evaluated not only on biogas production but also on the quality of the produced biogas. However, the following section presents the discussion of the laboratory experimentation of the combined effect of temperature, pH, mixing speed and pressure on biogas production and the methane content of the produced biogas under mesophilic condition.

4.4 Experimental procedures

This section contains the methodology and steps of all the laboratory experimentations conducted in this research work. It covers the preparation of the bacteria culture media and the substrate (organic samples), as well as the experimental setups. A total of 27 experiments were performed, involving the four operating parameters and each repeated four times. The experimental design of 26 of them is in such a way that only one parameter is manipulated while the other three operating parameters are maintained at the same given value throughout the investigation for all the variables of the manipulated parameter. The remaining experiment is performed with all the four investigated operating parameters manipulated simultaneously in three different intervals of eight hours each. Furthermore, this research work investigated the impact of co-digestion of wastewater sludge (WWS) and food waste (FW) on biogas and methane production. The study provided more insight on the behaviour of mesophilic anaerobic bacteria and the response of biogas production. The obtained results are presented in Chapter 5, which are utilised to construct the process model. In addition, all the data generated from the experiments are found in appendix A.

4.4.1 Preparation of bacterial culture media

Bacteria culture is a medium by which bacteria is supplied with all the elements (nutrients) they require for growth (Bauman, 2007). The media for this study is prepared by weighing out

1.0 g of glucose into a conical flask, mixed with 90 mL of deionised water. The solution was inoculated with 10 mL of WWS, obtained from Thames Water Rye Meads Sewage Treatment Plant, Stanstead Abbortts, UK, to make up the solution to 100 mL. The vigorously stirred flask was covered airtight with a rubber cork. The anaerobic environment was achieved within the conical flask by supplying it with nitrogen through a tube for 20 minutes, which displaced the dissolved oxygen present. Afterwards, the prepared bacteria culture was placed in an incubator operating at 39 ℃ for 12 hours. The prepared bacteria culture media was utilised to inoculate all the samples in this study, except for samples for co-digestion tests. The setup for bacterial culture preparation is shown in Figure 4.2.

Fig. 4.2. Bacterial culture preparation

4.4.2 Impact of variable temperature on biogas production

The experimentation was carried out with four units of 2 L volume New Brunswick BioFlo 111 Batch/Continuous fermentors (digesters), operated in batch mode under mesophilic condition. The sample fed to the digesters contains a mixture of 10 g of glucose, 13 g of nutrient

broth, 900 mL of deionised water and inoculated with 100 mL of the bacterial culture previously prepare, to make up the working volume to 1 L. In order to make the environment inside the digesters anaerobic (free from oxygen), nitrogen was supplied into the digesters for 30 minutes and dissolved oxygen contained in the digesters was displaced. The digesters were continuously stirred at 250 rpm while the digester pressure was controlled at 0.7 bar. The sample in the digester was retained for 24 hours.

The pH was maintained automatically at 7.2 by the supply of 2 M concentration of Sodium Hydroxide (NaOH) solution through the base peristaltic pump. The operating temperatures investigated are 32 ºC, 34 ºC, 36 ºC, 38 ºC, 40 ºC and 42 ºC. The biogas was collected through a tube inserted into the headspace of digesters to allow venting of biogas into a Tedlar gas sample bag attached to it. The cross-section of the digester and the experimental setup are shown in Figures 4.3 and Figures 4.4, respectively. The biogas and methane production were recorded every hour, using Biogas 5000 portable gas analyser connected to the gas collection tube. The experiments were repeated four times for each of the operating temperatures and the data obtained are analysed and discussed in Chapter 5.

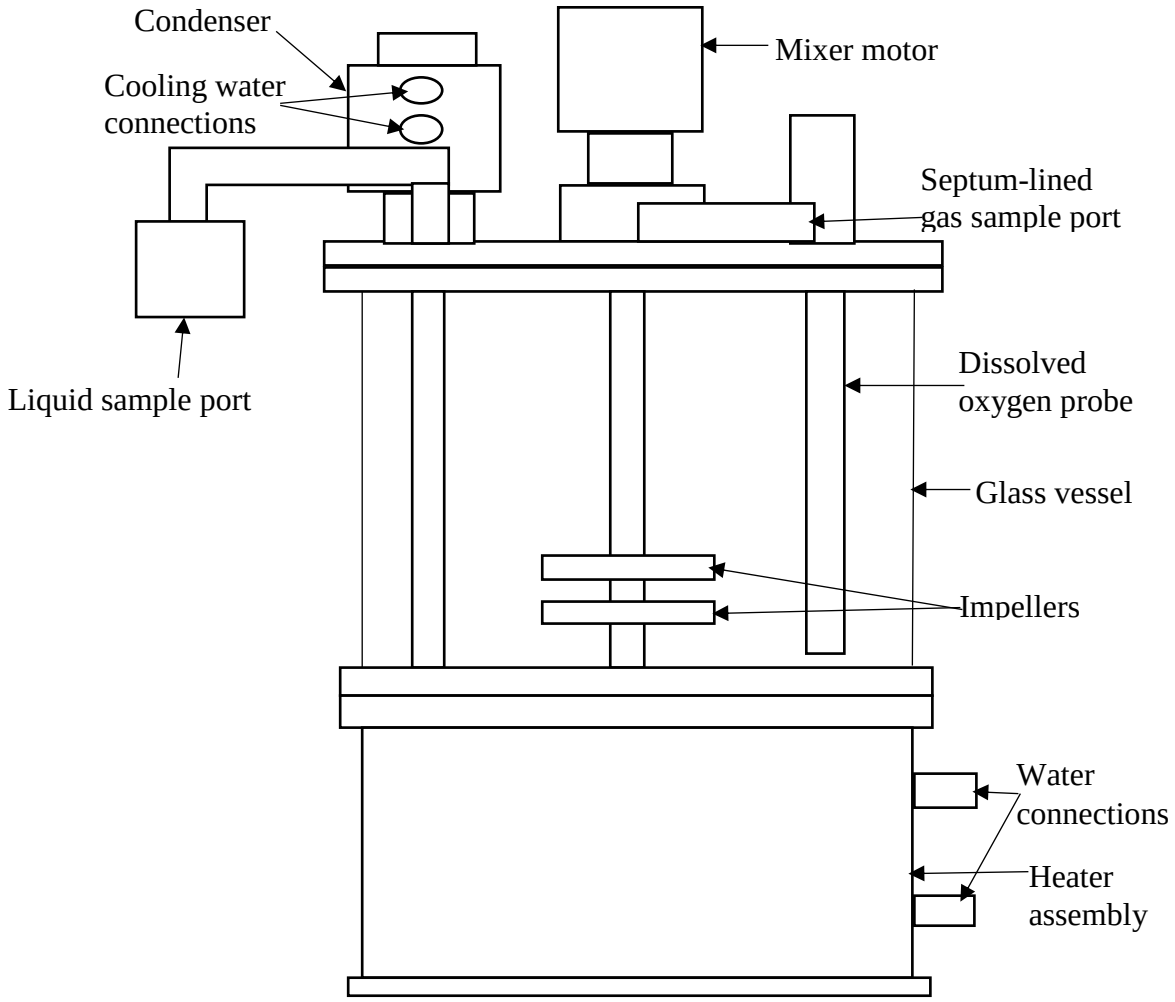


Fig. 4.3. Cross-section of New Brunswick BioFlo 111 Digester

Fig. 4.4. Setup of BioFlo 111 Digester

The technical specifications for both Biogas 5000 gas analyser (Geotech, 2012) and New Brunswick BioFlo 111 Batch/Continuous fermentor (New Brunswick, 2010) are presented in Table 4.1 and Table 4.2, respectively.

Table 4.1 Biogas 5000 technical specifications

Gas ranges				
Gases measured	CO_2 and CH_4	By dual wavelength infrared sensor with reference channel		
Standard gas cells	Cell	Range	Typical accuracy (range : accuracy)	Typical accuracy (range : accuracy)
	CH_4	0-100%	0-70% : ±0.5% (vol)	70-100% : ±1.5% (vol)
	CO_4	0-100%	0-60% : ±0.5% (vol)	60-100% : ±1.5% (vol)
Pump				
Flow	550 ml/min typically			
Flow fail point	-200 mbar vacuum - user settable			
Maximum vacuum restart	-375 mbar approximately with flow rate of approx 80ml/min			
Facilities				
Temperature measurement	-10°C to +75°C with optional probe			
Temperature accuracy	±0.5°C with optional probe			
Flow measurement	Via Pitot tube, orifice plate, or anemometer			
Alarm	User selectable alarms			
Communications	Via USB lead or wireless Bluetooth			
Relative pressure measurement	±500 mbar			
Relative pressure accuracy	±4 mbar typically (should be zeroed before reading) to ±15 mbar max			
Barometric pressure measurement	500 to 1500 mbar, ±5 mbar accuracy			
Available memory	10 IDs *, 500 readings			
Environmental conditions				
Operating temperature range	-10°C to +50°C			
Atmospheric pressure range	700 to 1200 mbar			
Relative humidity	0-95% noncondensing			
Certification rating				
ATEX	II 2G Ex ib IIA T1 Gb (Ta = -10°C to +50°C)			
MCERTS	MC/130240/00			
ISO17025	Calibration to UKAS certificate number 4533			
CSA	Ex ib IIA T1 (Ta= -10°C to +50°C) (Canada), AEx ib IIA T1 (Ta= -10°C to +50°C) (USA)			

Table 4.2. New Brunswick BioFlo 111 technical specifications

Vessel	Working Volume	0.48-1.12L
	Total Volume	2.0
Controller	Master Control Station	Controls 1-4 vessels, 32 control loops per vessel; stores 10 recipes & 8 process variables per vessel for trend graphing. Includes an industrial touchscreen monitor/user interface, 3 built-in pumps & connectors for all utilities & communications signals used by fermentor/bioreactor 1.
	Utility Station	One each required for optional 2nd, 3rd or 4th slave fermentors or bioreactors. Each includes 3 built-in pumps & connectors for all utilities & communications signals for its individual fermentor/bioreactor.
	Touchscreen Interface/Display	15-inch industrial monitor capable of supporting up to 4 fermentors/bioreactors. One is standard with the Master Control Station. Optional 2nd touchscreen available for use with slave fermentors/bioreactors, to replicate the image shown on the Master display.
Temperature	Indication	Digital display in 0.1°C increments
	Range	From 5°C above coolant temperature to 80°C (setting range: 4-80°C).
	Control	PI control employing PWM of heater and cooling water
	Sensor	Platinum RTD probe
Agitation	Drive	Permanent magnet motor with high torque input.
	Indication	Digital display in 1 RPM increments.
	Range	50-1200 RPM
	Control	PI-controlled
	Sensor	Optical photoplastic disc 500 lines/rev with quadrature output.
	Impellers	2 six-bladed Rushton turbine impellers provided
Exhaust	Filter	0.2µm disposable filter
	Condenser	Stainless steel, water-cooled in headplate
Aeration	4-Gas System	Up to 4 gases, including air, N2, CO2 & O2, delivered to ring sparger
	Sparger	Ring sparger
	Inlet Filter	0.2µm absolute disposable filter
	N2 Gas	For calibration of DO probe
pH	Indication	Digital display in 0.01 pH increments
	Range	2-12 pH
	Control	P&I
	Sensor	pH gel-filled probe
DO	Indication	Digital display in 0.1% increments
	Range	0-200%
	Control	P&I, Agitation, O2 Enrichment. Also GasFlow Rate if equipped with mass flow controller
	Sensor	Polargraphic probe
Other Sensors	Foam/Level	One foam/level sensor is standard
	Options	Redox or second pH and second DO probes available
Pumps	Pumps 1 & 2	Assignable peristaltic pumps Fixed speed (12 RPM) or variable duty cycle Available control modes: Off, Prime, Base, Acid, Foam, Levl2 Wet, Lvl2 Dry, Lvl 3 Wet or Lvl3 Dry.
	Pump 3	Assignable peristaltic pump Fixed speed (100 RPM) or variable duty cycle Available control modes: Off, Prime, Base, Acid, Foam, Levl2 Wet, Lvl2 Dry, Lvl 3 Wet or Lvl3 Dry.
Utilities	Water	10 PSIG maximum, 50 µm filtration
	Gas	10 PSIG maximum
Ambient Operating Conditions		10-30□C, up to 80% relative humidity, non-condensing

4.4.3 Effect of variable pH on biogas production

The goal of the experiment is to determine how variable pH values influence biogas and methane production in the anaerobic biodegradation of glucose. The experimental setup and procedure are similar to that described in section 4.4.2, except that temperature was controlled at 39 ^{o}C. In addition, the operating pressure and mixing speed were maintained at 0.7 bar and 250 rpm, respectively, with HRT of 24 hours. The experiment was repeated four times for each of the investigated pH values; 5.0, 6.0, 7.0, and 8.0. The variable pH values were achieved by automatically adjusting the digester pH to the set-point through the addition of 2 M of hydrochloric (HCL) acid and 2 M of NaOH into the digesters. The biogas and methane production were measured every hour by gas analyser similar to that of the temperature discussed above. The outcome of the experiments is analysed and discussed in Chapter 5.

4.4.4 Influence of digester pressure on biogas and methane production

The investigation carried out to ascertain the impact of digester pressure on biogas and methane production is described in this section. The experimental procedure, setup and HRT are similar to that described in sections 4.4.1 and 4.4.2. The temperature, pH and mixing speed were regulated at 39 ^{o}C, 7.2 and 250 rpm, respectively. The initial digester pressure was set at 0.7 bar using vacuum/pressure pump and monitored by a pressure gauge. The experiment was repeated with different operation pressures, 0.5, 0.3 and 0.1 bar. In addition, four repetitions of the experiment for each of the given operation pressures were performed. The biogas production and methane proportion were monitored as described in section 4.4.2 and the findings are reported and discussed in Chapter 5.

4.4.5 Impact of mixing speed on biogas production

The effect of mixing speed on the biogas production and its methane concentration are also investigated in this study. Mixing as highlighted in section 2.2.7, facilitates contact between bacteria and nutrients from substrates (Meroney and Colarado, 2009; Wards et al., 2008), thereby improving digester performance. However, inappropriate mixing speed can affect the performance of digester as found in this study.

The methodology and experimental setup are similar to that discussed in section 4.4.3. The operating temperature was set at 39 ^{o}C, while the digester pH and pressure were maintained at 7.2 and 0.7 bar, respectively. The operating speed of the mixer was initially set at 60 rpm and the experiment was retained for 24 hours. Biogas was collected as usual through the tube inserted into the headspace of digester into a Tedlar gas sample bag while online biogas and methane production were monitored every hour via the gas analyser connected to the tube. The experiment was performed with three other operating mixing speeds; 300, 600 and 2000 rpm. The experiment for each of the mixing speeds was repeated four times and the data obtained is discussed in Chapter 5.

4.4.6 Effect of simultaneous manipulation of operating parameters

An experiment is conducted to evaluate the impact of simultaneously manipulating the operating parameters investigated in this study on biogas production. The experiment was repeated four times under mesophilic conditions. The data obtained from this investigation is utilised to test the estimated model. The parameters were manipulated simultaneously with three different values at eight hours interval. The temperature, pH, mixing speed and pressure were initially set at 35 ^{o}C, 8.5, 900 rpm and 0.3 bar, respectively. After eight hours, the values of the parameters were changed to 37 ^{o}C, 6.2, 500 rpm and 0.4 bar for temperature, pH, mixing

speed and pressure, respectively. Then at the 17th hour, the values of the parameters were altered again, in the same order, to 41 ^{o}C, 6.8, 120 rpm and 0.5 bar.

4.4.7 Influence of co-digestion on biogas production

As mentioned in section 3.1.2, co-digestion can improve nutrient balance, biodegradability, as well as digester performance and biogas production in AD. The methodology for the study was as follows:

1. **Preparation of substrates**

The substrates utilised for this experiment are wastewater sludge (WWS), obtained from Thames Water Rye Meads Sewage Treatment Plant, Stanstead Abbortts, Hertfordshire, and food waste (FW) collected from the cafeteria of the University of Hertfordshire, all in the UK. The activated sludge used as inoculum was obtained from Thames Water Rye Meads Sewage Treatment Plant. The FW consists of fish, rice, chicken, beef, bread, fresh vegetables and scrambled egg, which were ground with a grinding machine to reduce their particle size. All the FW samples collected throughout the study are of the same basic content.

2. **Total solids (TS) determination**

The TS content of both FW and WWS were determined by applying the Laboratory Analytical Procedure (LAP) methodology, described in LAP Determination of Total Solids in Biomass and Total Dissolved Solids in Liquid Process Sample (Sluiter et al., 2008, cited by Sluiter et al., 2010, Shekiro 111 et al., 2014 and Ritchie, 2014). For this study, TS was determined by drying the samples at 105 ^{o}C for 12 hours in an autoclave.

3. Co-digestion experiment

The experiment was performed with 3 L glass vessels (digesters), operated in batch mode, under mesophilic condition. The aim of this experimentation is to investigate the influence of the co-digestions of WWS and FW on the biogas production. The digester temperature was maintained at 39 ^{o}C and the mixing was performed by fabricated rotating shakers at 150 rpm. The volume of each sample in the digester was 2 L and the influent and effluent pH values for all the samples were noted. Furthermore, the environment within the digesters was made anaerobic by displacing the dissolved oxygen with nitrogen, which was supplied by a rubber tube inserted through the digester rubber cork. Each of the samples was retained in the digester for seven days. The experimental setup is illustrated in Figure 4.5 and biogas was harvested by a tube inserted into the headspace of digesters, which transferred the gas to a Tedlar gas sample bag. Gas analyser coupled to the tube monitored the rate of biogas production per day and the corresponding methane content. In order to achieve the ideal mixing ratio of the co-substrates, three different mass ratios of FW: WWS were tested at 30:70, 50:50 and 70:30. In addition, FW and WWS were tested as a single substrate, which was utilised as a reference for the mixed samples. Each of the experiments was repeated five times and the results are discussed in Chapter 5.

Fig. 4.5. Setup of co-digestion experiment

4.5 Summary

This chapter has discussed the bacteriological and biochemical processes that make up the AD process. These are successive multi-step and parallel reactions such as hydrolysis, acidogenesis, acetogenesis and methanogenesis, which are the action of a specific group of anaerobic bacteria for each step/process. Of all the processes methanogenesis is found to be the limiting step as it determines the effectiveness of the entire AD. The chapter also discussed the different experimentations to evaluate the effects of temperature, pH, mixing speed and pressure on the biogas production. Furthermore, it discussed the influence of the co-digestion of different ratios of FW and WWS on the biogas production. The data obtained from all the experiments conducted in this chapter are analysed and discussed in Chapter 5.

Chapter 5 **Results and Discussions**

5.1 Introduction

The experimental procedures for the impact of four operating parameters and co-digestion, under the mesophilic condition, on biogas and methane production, are discussed in Chapter 4. Because of the huge amount of data collected from the investigations conducted in this study, this chapter could not accommodate such amount of data, rather they are documented in appendix A. This chapter contains the analyses and discussions of the results collected from the experiments, which are presented in graphical forms.

5.2 The impact of temperature on biogas and methane production

This section discusses the relationship between temperature variation and biogas production in mesophilic AD of glucose. The effect of this relationship in terms of average biogas production, cumulative biogas production and total biogas production are plotted. Similarly, the methane content of the biogas at different digester temperatures are also presented in graphs.

5.2.1 Average rate and cumulative biogas production

The average biogas production rate and average cumulative biogas production are presented in Figures 5.1 and 5.2, respectively.

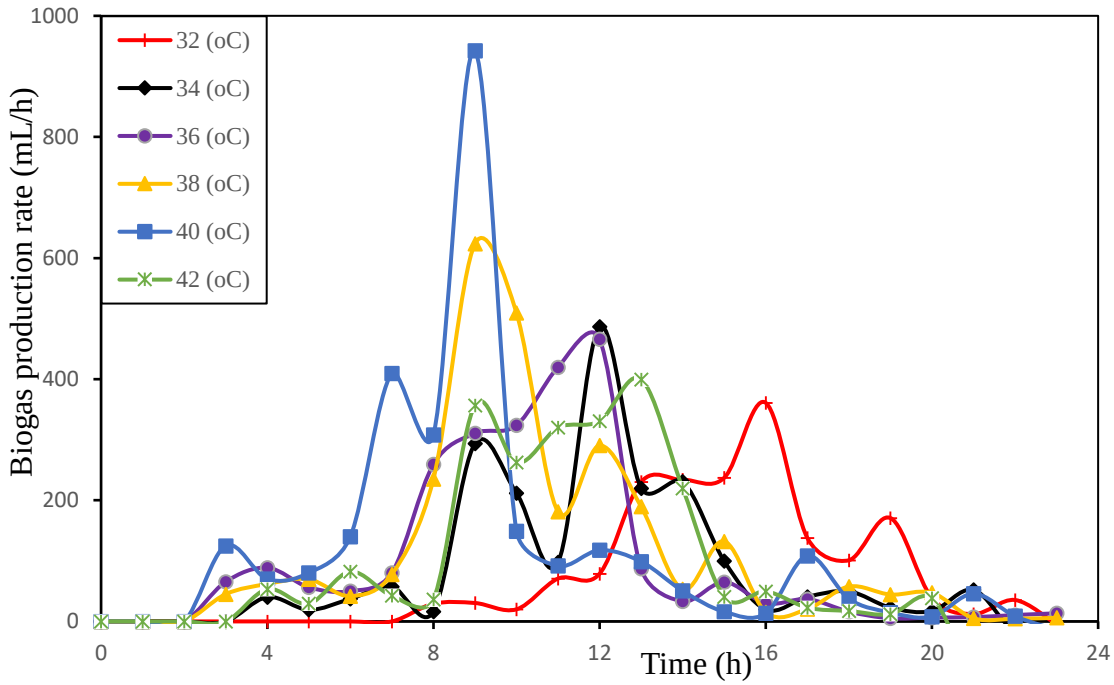


Fig. 5.1. Average rate of biogas production at different temperatures

Figure 5.1 presents the rate of biogas production from the digestion of glucose at 32, 34, 36, 38, 40 and 42 ^{o}C with HRT of 24 hours. As evident, the 36, 38, 40 and 42 ^{o}C digesters began biogas production at the 2nd hour, while the setups that operated at 34 ^{o}C, and 32 ^{o}C, commenced biogas production at the third and seventh hour, respectively. Indicating that the substrate and anaerobic bacteria received enough energy (activation energy) required for biodegradation to proceed from the 36, 38, 40 and 42 ^{o}C digesters within the second hour of the digestion process, which resulted in biogas yield within the second hour. Although the four digesters provided enough activation energy for the anaerobic reaction to commence, it is observed that the biogas production rate for 42 ^{o}C digester is the lowest at the beginning of the production, while the highest is recorded in the 40 ^{o}C digester, followed by the 38 ^{o}C digester and then the 36 ^{o}C digester. Indicating that 42 ^{o}C temperature is beyond the mesophilic optimal temperature range of the digester.

In addition, the 38, 40 and 42 ^{o}C digesters peaked at the ninth hour. Again, the digester that was operated at 42 ^{o}C generated the lowest peak, whereas the 40 ^{o}C digester produced the highest peak, followed by the 38 ^{o}C digester. The 36 ^{o}C digester is found to peak at the 12th hour, ahead of the 34 and 32 ^{o}C digesters that peaked at the 13th and 18th hour, respectively. This signifies that the 40 ^{o}C digester has the tendency to produce more biogas than the rest of the digesters within the first 10 hours of the digestion, followed by the digester that was operated at 38 ^{o}C.

Furthermore, the rate of biogas production remained positive for all the digesters up to the 20th hour, when the biogas production rate from the 42 ^{o}C digester became negative, meaning that the production of biogas was on the decline and lower than the production of the previous hour.

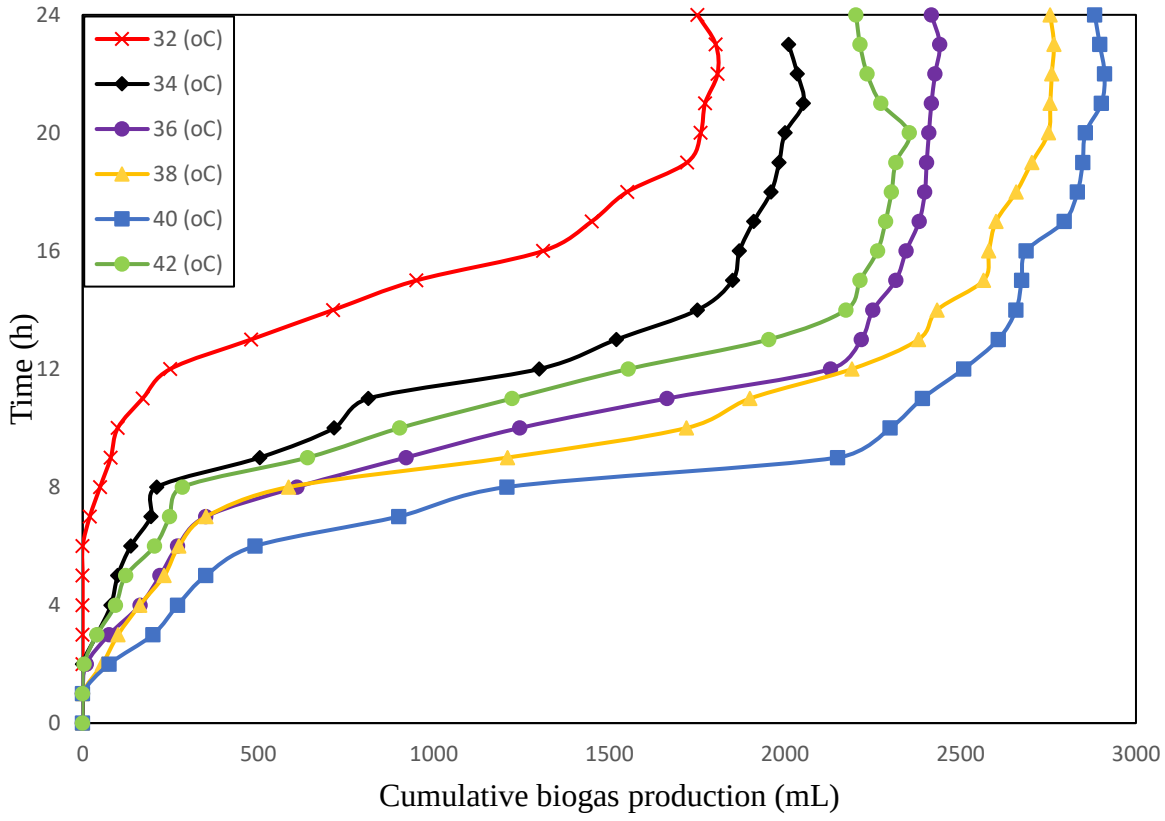


Fig. 5.2. Average cumulative biogas production at different temperatures

Similarly, the comparison of the average cumulative biogas produced at 32, 34, 36, 38, 40 and 42 ^{o}C as seen in Figure 5.2. It shows that the 40 ^{o}C digester-generated the highest average cumulative biogas production, followed by the 38 ^{o}C digester, then the third highest cumulative biogas is produced in the 36 ^{o}C digester. Whilst the 32 ^{o}C digester yielded the lowest average cumulative biogas, followed by the 34 ^{o}C digester and then the 42 ^{o}C digester. It is seen that the average cumulative biogas produced in the 42 ^{o}C digester is not the lowest, however, Figure 5.2 shows a significant drop in biogas production as digester temperature was elevated from 40 to 42 ^{o}C. This means that the temperature range that yielded the highest average cumulative biogas is 38 – 40 ^{o}C.

5.2.2 Total volume of biogas production

The average total volume of biogas production from the impact of variable operating temperatures is presented in Figure 5.3.

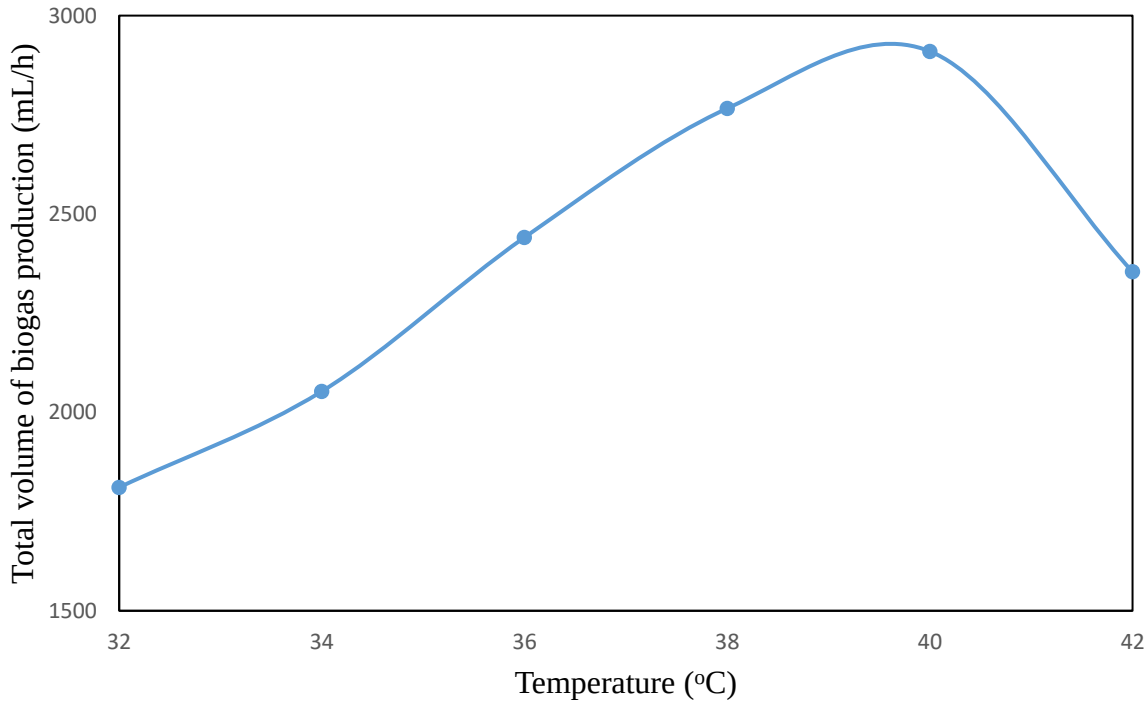


Fig. 5.3. Average total volume of biogas yield at different temperatures

As seen in Figure 5.3, as digester temperature increased from 32 to 39 ^{o}C, the average total biogas volume production increased, although not a linear relationship. As the temperature was further elevated from 39 to 40 ^{o}C, the production of biogas is relatively constant. However, as the temperature marginally increased beyond 40 ^{o}C, the volume of biogas yield is seen to decline. Implying that increase in temperature within the mesophilic range, but not beyond the higher optimal value, can improve biogas yield, but further temperature increase beyond the optimal range can cause a decline in biogas production. In addition, it is seen in Figure 5.3 that the digester temperature rose beyond 40 ^{o}C, supporting the reports of previous studies (Babaee et al., 2013; Usman et al., 2012; Chae et al., 2008; El-Mashad et al., 2004). Hence, it is deduced from Figures 5.1, 5.2 and 5.3 that the optimum temperature range for AD of glucose under the mesophilic condition for this research work is 37 - 40 ^{o}C.

5.2.3 Methane production at varying temperature

The relationship between variable temperatures and methane production is shown Figures 5.4 and 5.5 for average rate and average cumulative methane production, respectively.

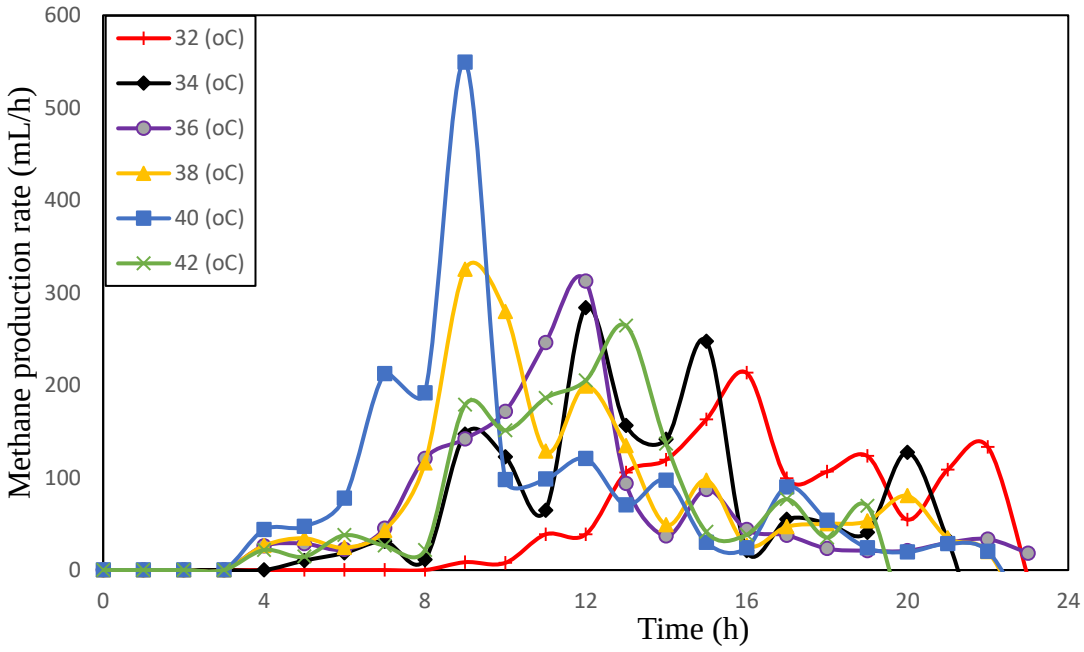


Fig. 5.4. Average rate of methane proportion at different temperatures

The rate of methane production presented in Figure 5.4 shows a similar behaviour to that of the biogas production rate in Figure 5.1. However, as seen in Figure 5.4, the 36, 38, 40 and 42 ^{o}C digesters began methane production at the third hour, while the setups that operated at 34 and 32 ^{o}C, commenced methane production at the fourth and eighth hour, respectively. In addition, the 38, 40 and 42 ^{o}C digesters peaked at the ninth hour. The digester that was operated at 40 ^{o}C produced the highest peak, followed by the 38 ^{o}C digester. The 36 and 34 ^{o}C digesters reached their peak at the 12th hour, ahead of the 42 ^{o}C digester, which peaked at the 13th hour. Then the 32 ^{o}C digester peaked at the 18th hour.

Furthermore, the rate of methane production remained positive for all the digesters up to the 19th hour, when the methane production rate from the 42 ^{o}C digester became negative. Other digesters, except the 34 ^{o}C digester, also produced negative methane production rate but at different times. The negative methane production rate means that the production of methane was on the decline and is lower than the methane production of the previous hour.

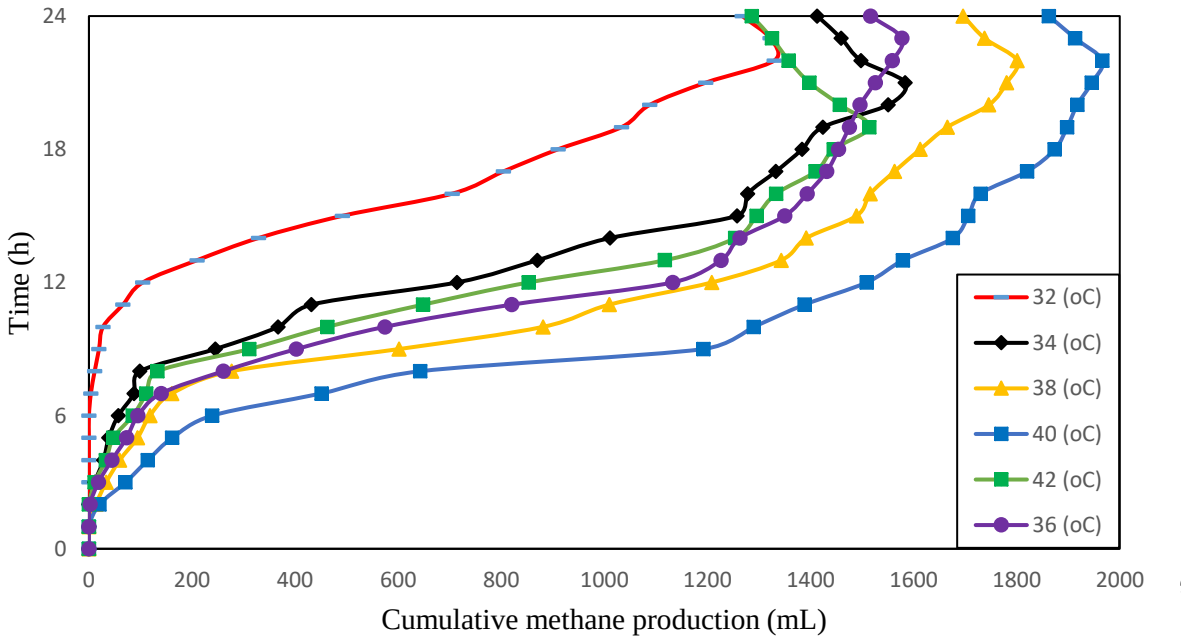


Fig. 5.5. Average cumulative methane production at different temperatures

The digester with a temperature of 36 ^{o}C provided the third highest average cumulative methane production, then the 42 ^{o}C digester. However, the 32 ^{o}C digester generated the least average cumulative methane after the 34 ^{o}C digester.

5.2.4 Average total volume of methane production at different temperatures

Figure 5.6 shows the average total methane production relative to temperature variation.

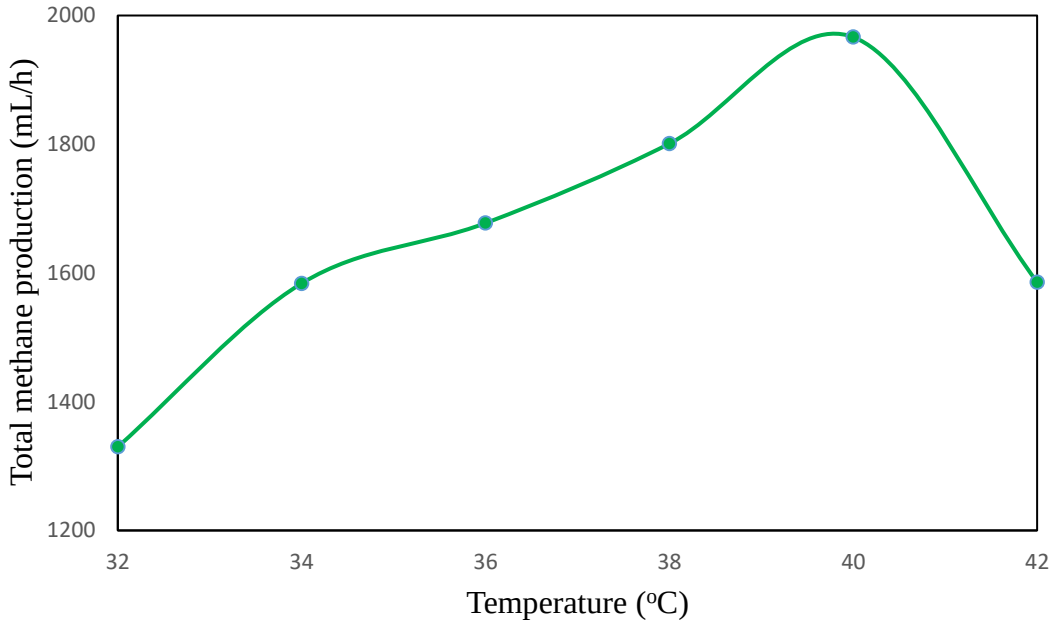


Fig. 5.6. Average total volume of methane yield at variable temperature

As seen in Figure 5.6, the impact of temperature on the total volume of methane yield is similar to the effect of temperature on the total volume of biogas production in Figure 5.3. It shows that as the operating temperature of the digesters increased from 32 to 39 ^{o}C, the total volume of methane production increased, though not a linear relationship. It is also seen that further increase in temperature from 39 to 40 ^{o}C, resulted in a relatively constant methane production. However, as the digester operating temperature slightly increased beyond 40 ^{o}C, the volume of

methane yield dropped. Indicating that 42 ^{o}C is above the temperature limit of which it supports the growth of the methanogenic bacteria.

5.3 Effect of pH

This section discussed the results obtained from the investigation conducted on the influence of pH variation on biogas production and methane concentration of the biogas.

5.3.1 Rate and cumulative biogas production at various pH values

The result of the experiment to determine the effects of pH variation on biogas and methane production are discussed in this section. The rate of biogas production and the cumulative biogas production are presented in Figures 5.7 and Figures 5.8, respectively.

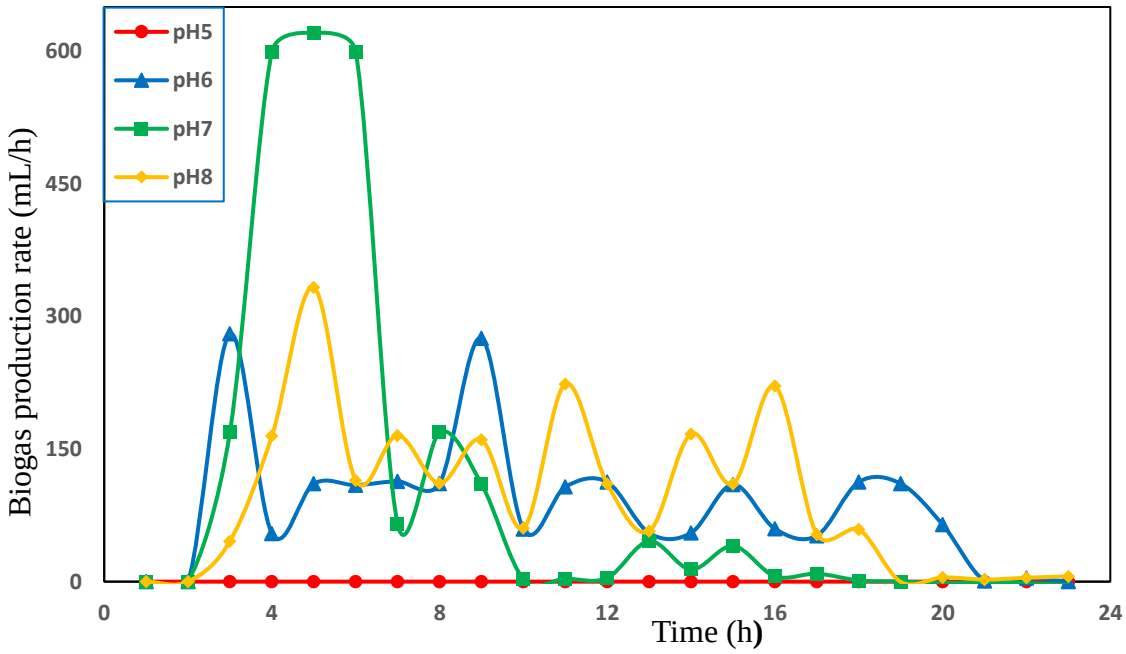


Fig. 5.7. Average rate of biogas production at different pH values

As seen in Figure 5.7, all the digesters commenced biogas production at the same time, except the digester that was operated at pH of 5.0. The reason for this is that all the digesters were operated at the same temperature of 39 ^{o}C. The digester that operated at pH of 5.0 recorded no biogas yield, as seen in both Figures 5.7 and 5.8 because the activity of methanogenic bacteria was halted due to the acidic condition. This finding supports the reports by previous studies, which states that AD failure occurs when the pH inside the digester is below 5.5 (Nayono, 2009; Al Seadi et al., 2008).

In addition, it is seen in Figure 5.7 that the rate of biogas production for pH6 digester is higher than the pH7 and pH8 digesters between the second and third hour. Afterwards, pH7 digester significantly increased biogas production rate to about 80% higher than the pH6 and pH8 digesters. The pH7 digester peaked at the fourth hour and remained at that peak for two hours, and then took a sharp drop between the sixth and seventh hour. Finally, the rate of biogas production for pH7 digester reached a steady state at the 10th hour. However, the pH6 and pH8 digesters came to steady state in the 21st and 19th hour, respectively. This indicates that the organic conversion of substrate to biogas was almost completed at the 10th, 21st and 19th hour for pH7, pH6 and pH8 digesters, respectively. It took the pH7 digester 10 hours to reach a steady state, meaning that the HRT to complete organic conversion process was about half of the time it took pH6 and pH8 digesters. Apart from the pH5 digester that produced no biogas, it is seen that pH6 digester recorded the lowest biogas production rate, followed by the pH8 digester while the highest rate is generated by the digester that was operated at pH of 7.0.

Similarly, the influence of pH on the cumulative biogas production presented in Figure 5.8, shows that the highest cumulative biogas yield is recorded by the pH7 digester, then followed

by the pH8 digester, whereas the pH6 digester produced the lowest biogas production apart from the pH5 digester that recorded no biogas production.

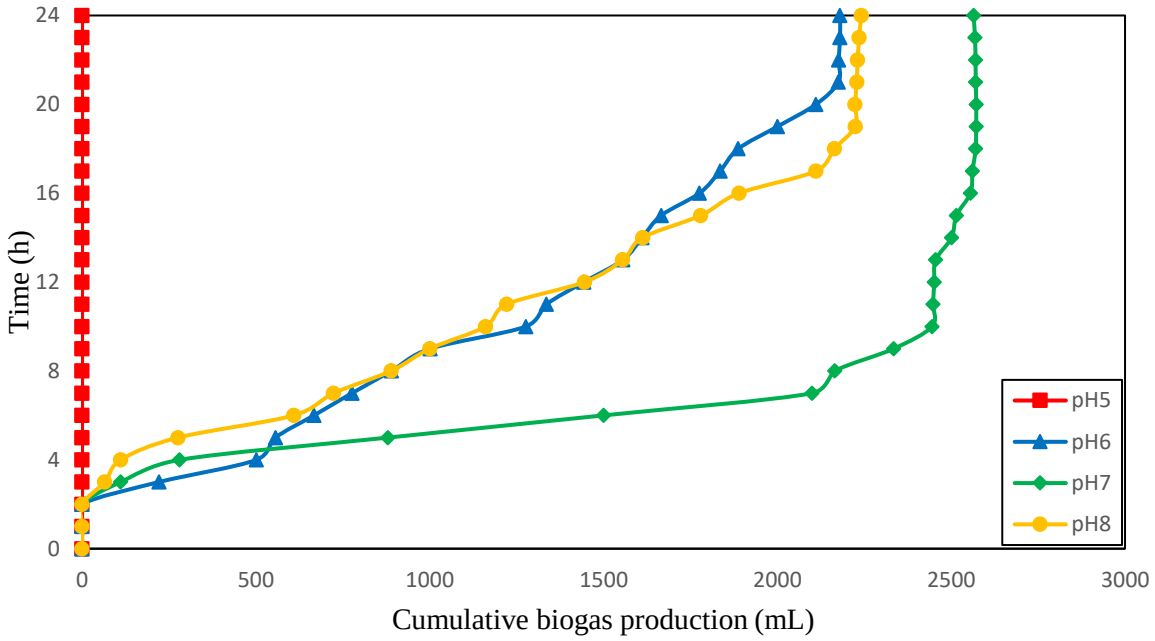


Fig. 5.8. Average cumulative biogas production at different pH values

5.3.2 Average total volume of biogas production at different pH values

This section discusses the impact of variable pH values on the total volume of biogas production. As seen in Figure 5.9, the average total volume of biogas yield decreased as pH value decreased as well as increased from the pH of 7. In addition, it is evident in Figure 5.9 that there was no biogas production until about 5.5 pH due to the acidic condition of the digester content, which inhibits methanogenic bacterial activity. This result supports the previous studies that suggest that the ideal pH value for methanogenesis is 7.0 and the optimal pH range is 6.8 – 7.2.

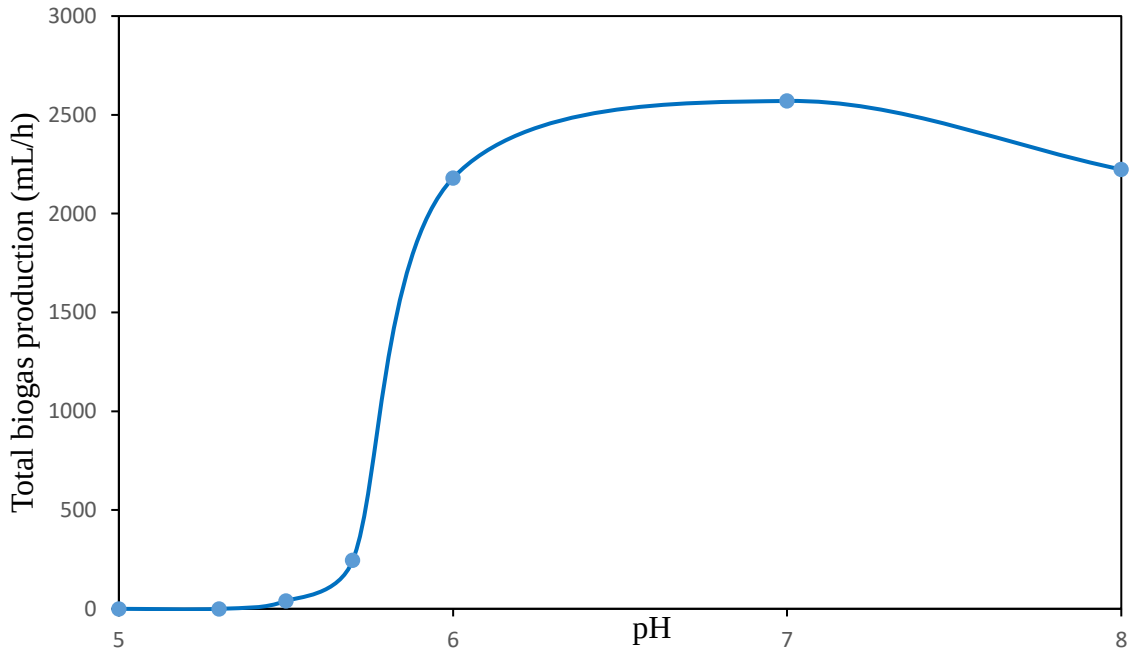


Fig. 5.9. Average total volume of biogas production at different pH values

5.3.3 Methane production rate and cumulative methane production at different pH values

The average methane production rate and cumulative methane production are presented in Figure 5.10 and Figure 5.11

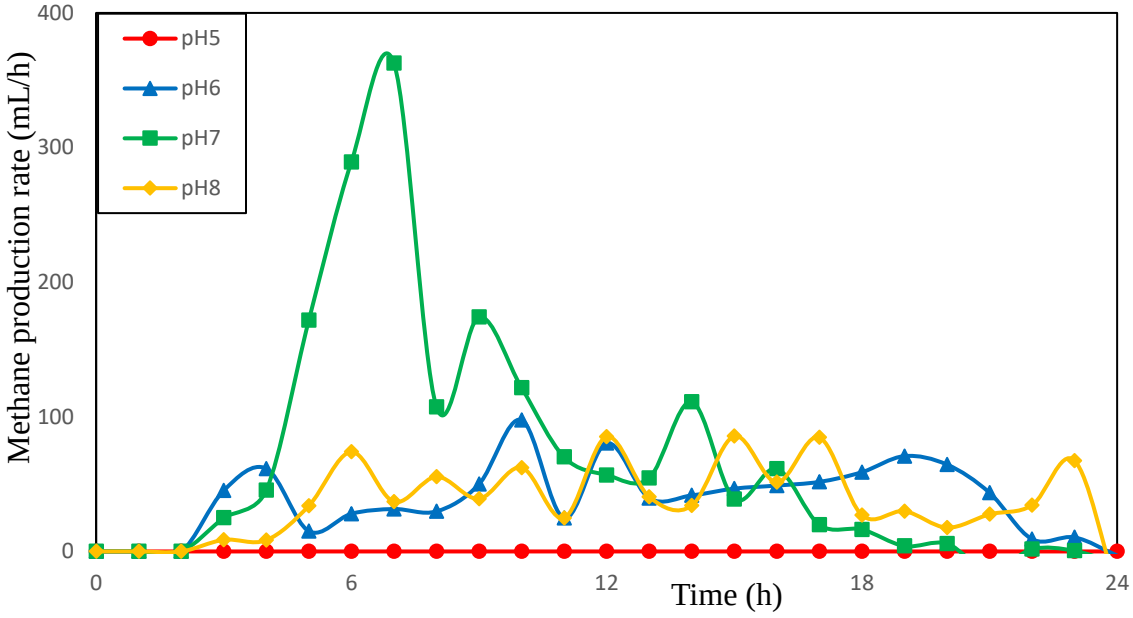


Fig. 5.10. Average methane production rate at different pH values

The methane production rate at variable pH as seen in Figure 5.10 is similar to the biogas production rate at variable pH seen in Figure 5.7. All the digesters commenced methane production at the same time, apart from the pH5 digester that produced no methane at all due to the acidic condition. In addition, in Figure 5.10, the rate of methane production for pH7 digester peaked about the sixth hour and then took a sharp drop. Finally, the rate of methane production for pH7 digester reached almost the steady state at the 19th hour. However, it is seen that the methane production rate for pH6 and pH8 digesters are close on average.

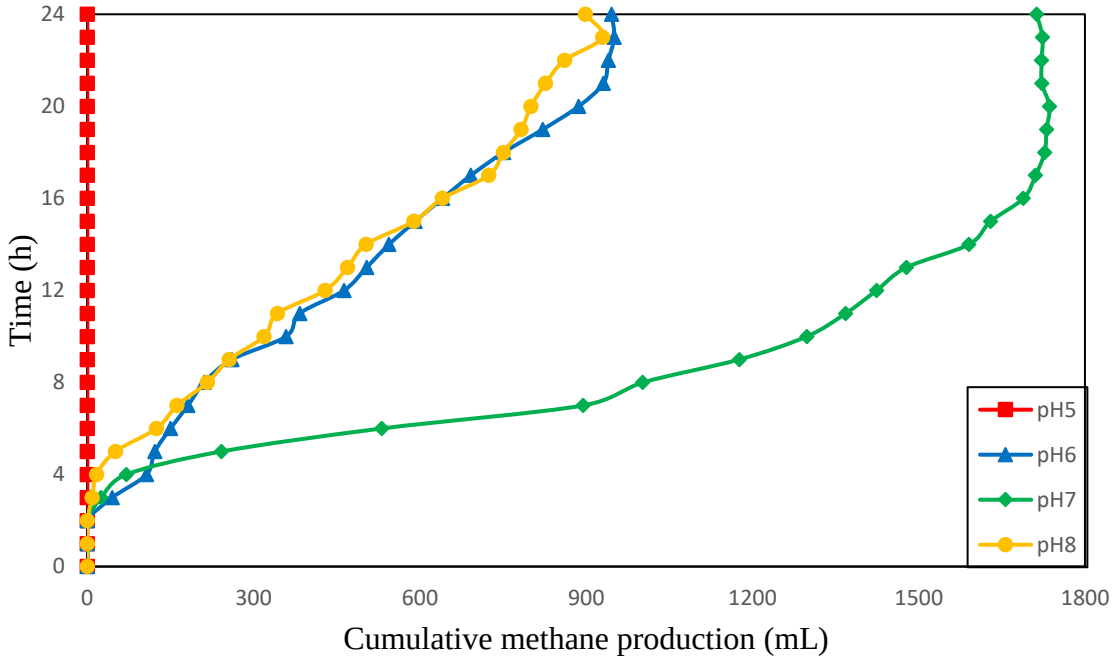


Fig. 5.11. Average cumulative methane production at different pH values

As seen in Figure 5.11, the average cumulative methane production is highest at digester pH of 7.0. In addition, the cumulative methane production for pH6 digester is slightly higher than the pH8 digester. Whilst the pH5 digester did not produce methane.

5.3.4 Average total volume of methane production at different pH values

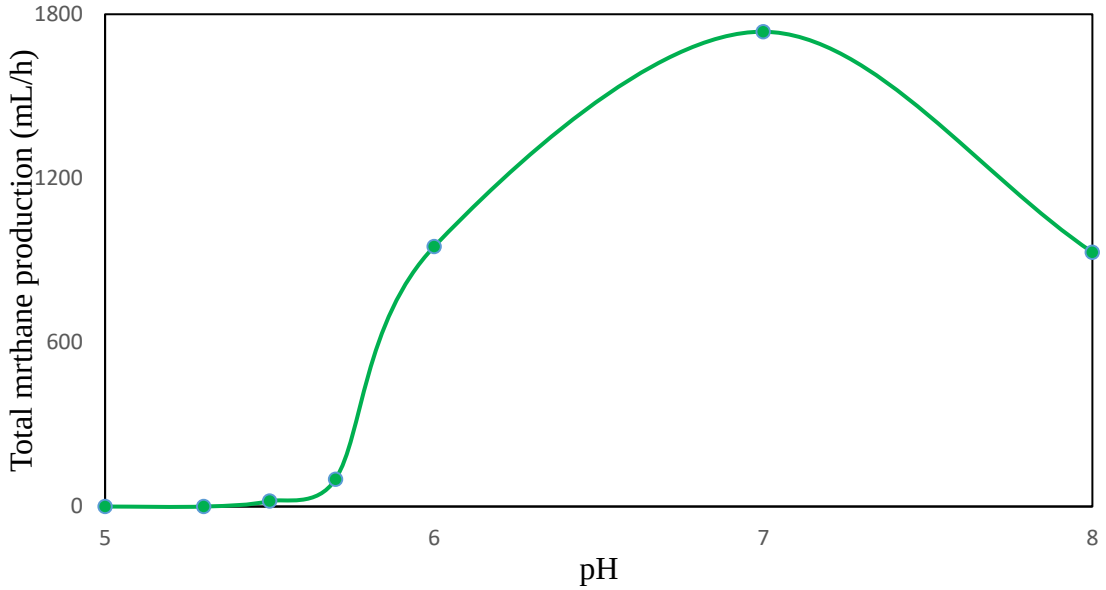


Fig. 5.12. Average total volume of methane production at different pH values

Figure 5.12 shows that from pH of 5.0 to 5.5 no methane was produced, but as digester pH increased from 5.5 to 6.8, the average total volume of methane production increased. However, methane production is seen to be relatively stable between pH 6.8 and 7.2. As digester pH exceeded 7.2 the average total volume of methane production is seen to decline, which supports the suggestion that the optimum mesophilic pH range for methanogenesis is 6.8 – 7.2.

It is not that no gas was produced at pH of 5.0, rather, there is no detection of methane in the generated gas due to the inhibition of methanogenic bacteria by the acidic condition in the digester. No further evaluation was carried out in the produced gas in order to determine its composition. Since there is no methane detection on the gas produced at this pH, no biogas is accounted for, as biogas is primarily the combination of methane and carbon dioxide. Hence, the absence of methane production in AD process means that no biogas is produced.

5.4 Influence of digester pressure

This section discusses the experimental results from the influence of digester pressure variation on biogas and methane production.

5.4.1 Rate and cumulative biogas production at different digester pressures

The average biogas production rate and average cumulative biogas production under different digester pressures are presented in Figure 5.13 and 5.14, respectively.

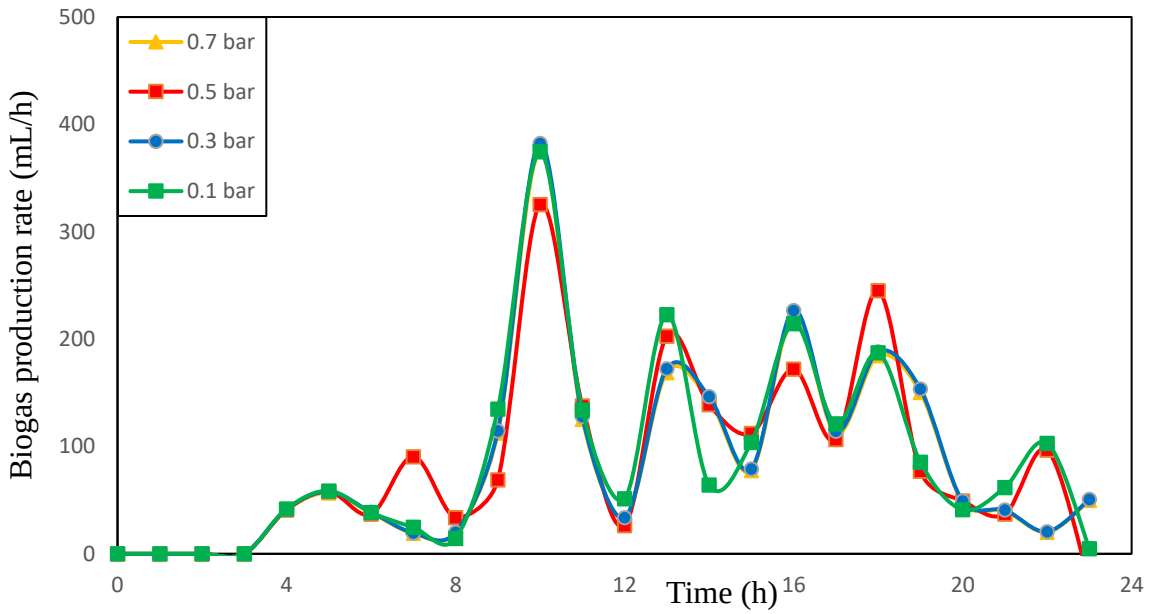


Fig. 5.13. Average rate of biogas production at different pressures

As seen in Figure 5.13, the rate of biogas production did not follow a particular pattern with respect to the varied operating pressures. It is difficult to explain why there is no particular response of biogas production rate to the increase or decrease in digester pressure. This result is in agreement with the work of Japan-Agency for Marine-Earth Science and Technology (2007), which highlights the difficulty in explaining the influence of pressure on complex metabolic systems, which includes the AD process.

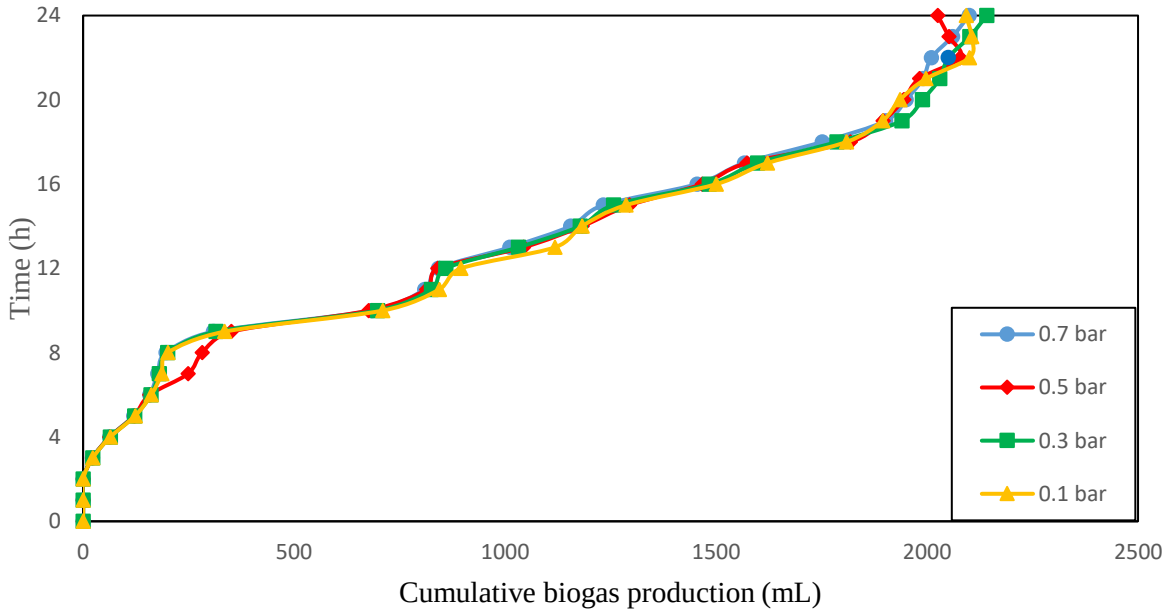


Fig. 5.14. Average cumulative biogas production at different pressure values

It is seen in Figure 5.14 that there is a marginal difference in the cumulative biogas yield of the different samples at as the digester operating pressure increases. This might be because of the small difference in the operating pressure. However, the studies by Chen et al. (2014), Lindeboom et al. (2011) and Hayes et al. (1990) shows that there is a decrease in biogas production as operating pressure increases. This is because part of the generated CO_2 in biogas liquefies with an increase in digester operating pressure, thereby reducing the overall biogas production while increasing the concentration of methane in the biogas. The phenomenon is based on gas solubility; the CO_2 being more soluble in water than methane liquefies at the same temperature and pressure (Yalkowsky et al., 2010), resulting in more concentration of methane in the biogas.

5.4.2 Average total volume of biogas production at variable digester pressure

The average total volume of biogas production at variable digester pressure is presented in Figure 5.15.

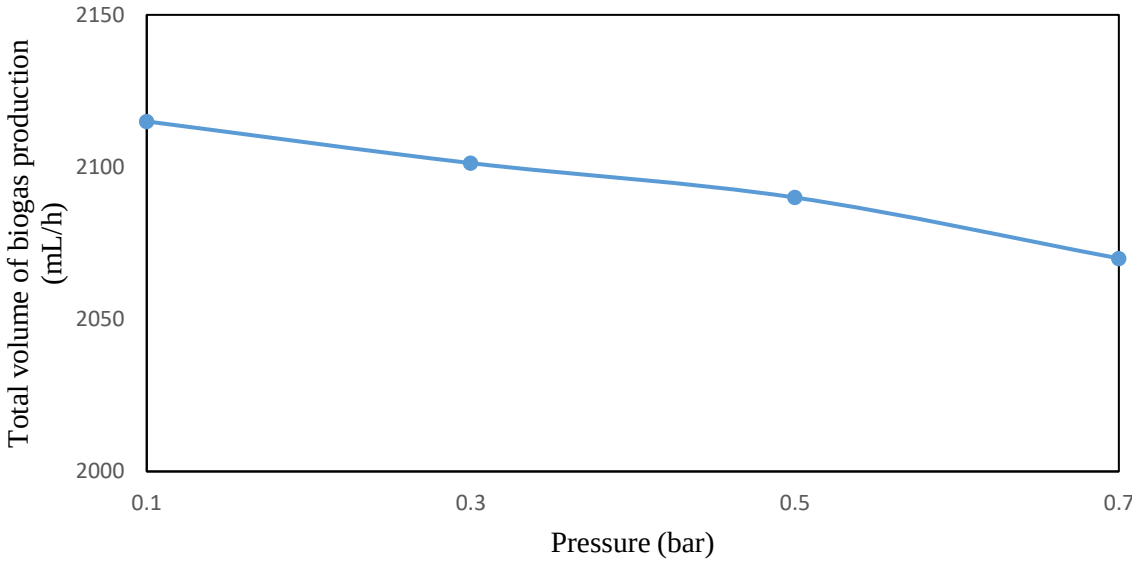


Fig. 5.15. Average total biogas production at different pressure values

As seen in Figure 5.15, the total volume of biogas yield marginally increases as digester pressure decreases from 0.7 to 0.1 bar. In Figure 5.15, it is seen that the 0.1 bar digester generated the highest total biogas production, followed by the 0.3 bar digester while the 0.7 bar digester yielded the lowest biogas behind the 0.5 bar digester. The reason for this is also based on the solubility of gases.

5.4.3 Methane production rate and cumulative methane yield at different digester pressure

The relationship between pressure and methane production rate and cumulative methane production are presented in Figure 5.16 and Figure 5.17, respectively.

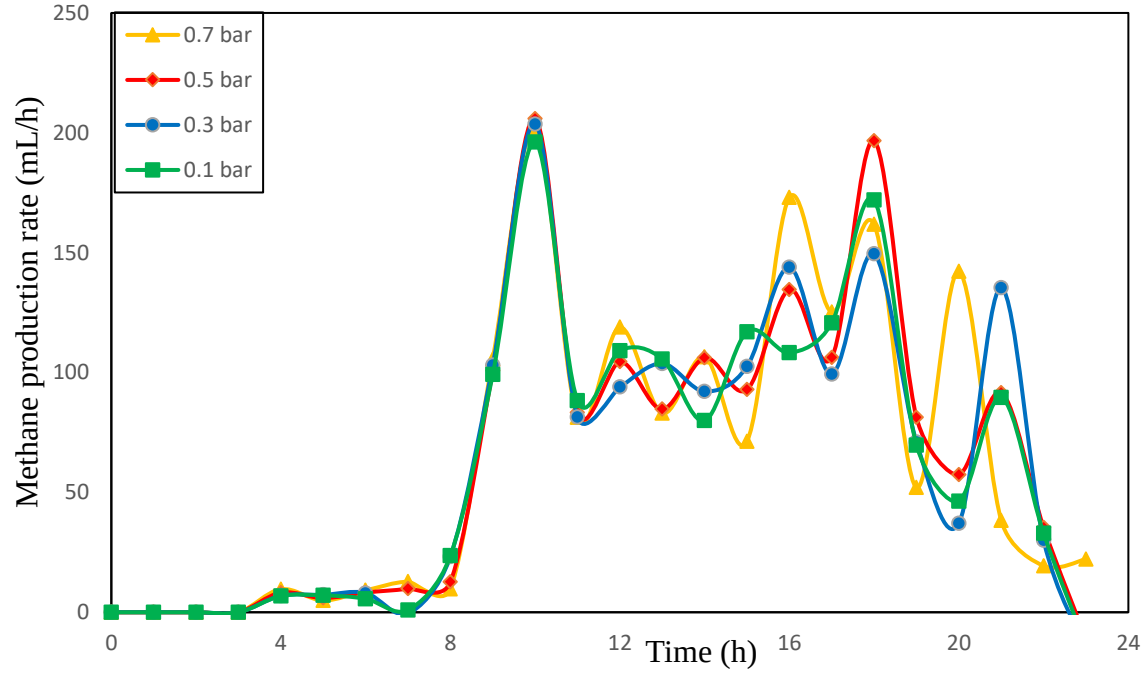


Fig. 5.16. Average methane production rate at different pressures

Figure 5.16 shows a similar behaviour as the biogas production rate at various digester pressure, with no clear pattern. This is also attributed to the difficulty in describing the effects of pressure on the complex metabolic process.

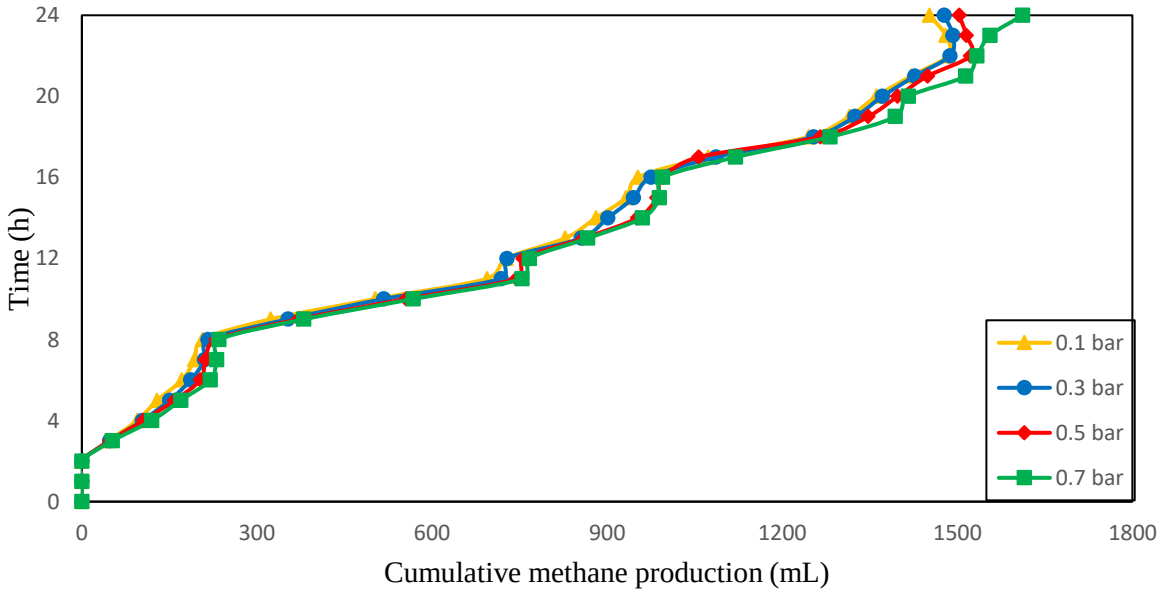


Fig. 5.17. Average cumulative methane production at variable pressure

As seen in Figure 5.17, the variation of digester pressure marginally influenced the amount of methane production. This is similar to the behaviour of biogas to the given operating pressures. The small difference in the digester operating pressures utilised in this study may have contributed in the reason why there is no significant change in cumulative methane production with an increase in the digester operating pressure.

5.4.4 Average methane concentration at different digester pressures

The influence of variable digester pressure on the methane proportion is seen in Figure 5.18.

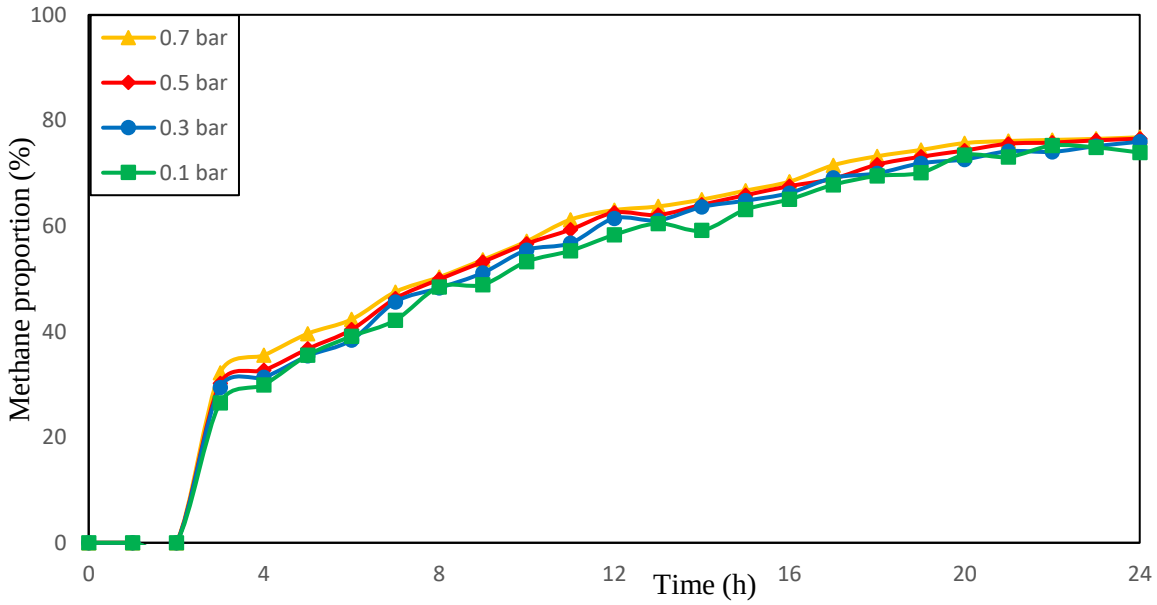


Fig. 5.18. Average methane proportion at different pressures

As seen in Figure 5.18, an increase in digester pressure resulted in an increase in methane proportion. Though the increment is marginal due to the small increase in the digester operating pressure. As the digester pressure increases from 0.1 to 0.7 bar, the methane proportion is found

to increase. The significant of this result highlights the importance of digester pressure in AD, with regards to improving the methane concentration in biogas.

5.4.5 Average total methane production at different digester pressures

The influence of variable digester pressure on methane production is seen in Figure 5.19.

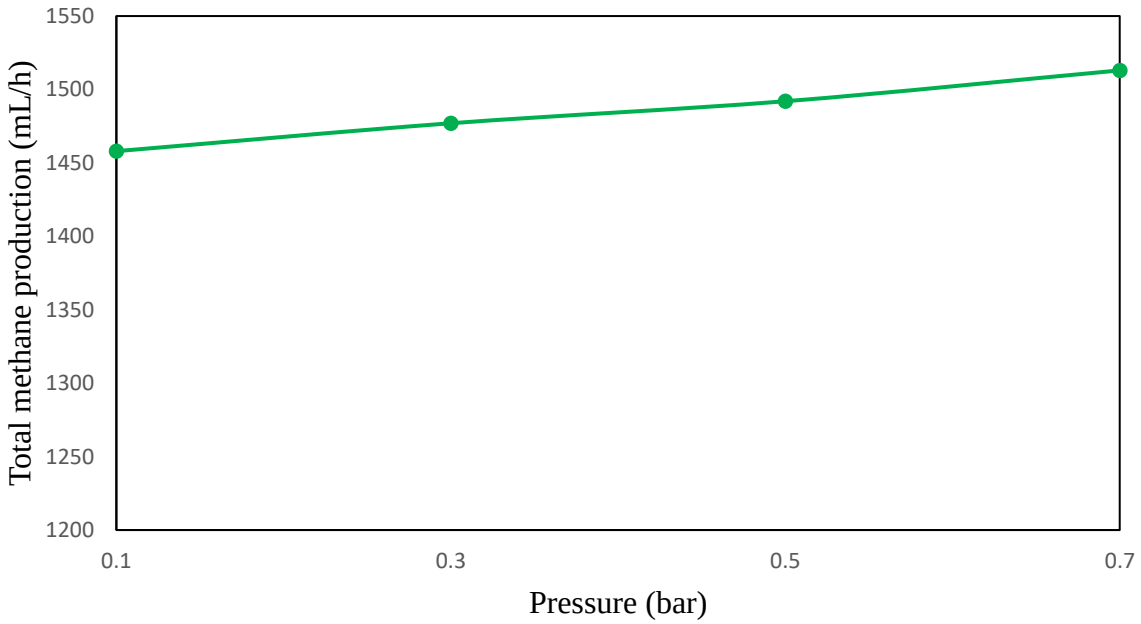


Fig. 5.19. Average total methane production different pressures

As seen in Figure 5.19, methane production increases as digester pressure increase from 0.1 to 0.7 bar. Though the increment is marginal due to the small increase in the digester operating pressure. The significant of this result highlights the importance of digester pressure in AD, with regards to improving methane production.

5.5 Effect of mixing speed

This section discusses the impact of various mixing speed on biogas and methane production.

5.5.1 Average rate and cumulative biogas production at various mixing speeds

The average biogas production rate and cumulative biogas production relative to different mixing speeds are presented in Figures 5.20 and 5.21, respectively.

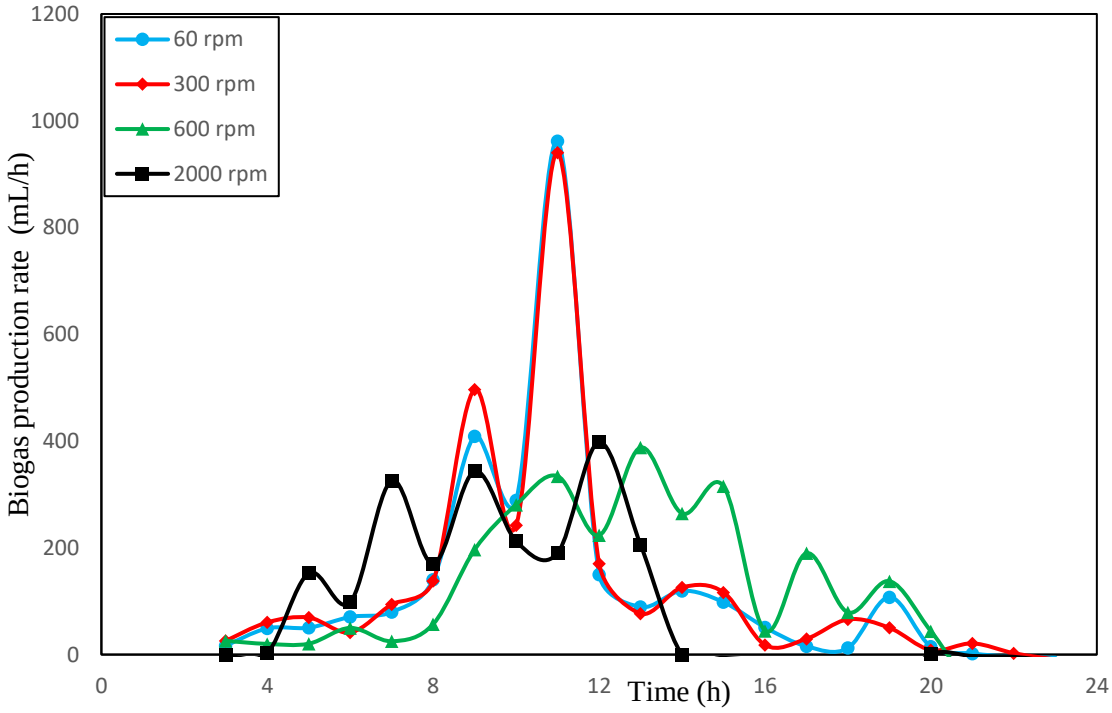


Fig. 5.20. Average rate of biogas production at various mixing speed

As seen in Figure 5.20, the 60 and 300 rpm digesters maintained a similar rate of biogas production throughout the digestion process. In addition, the 60 and 300 rpm digesters produced the highest peak between the eighth and the .11th hour of retention time. It is seen in Figure 5. 20 that the rate of biogas production for 600 rpm digester is higher than the 60, 300, and 2000 rpm digesters between the 12th and 20th hour. Furthermore, the plot shows the continuous decline in biogas production from the 2000 rpm digester for two hours, between the 16th and 18th, which is indicated by the negative biogas production rate. However, the biogas production picked up again for another two hours, before reaching the steady state at the 20th hour, signifying that no more increase in biogas production by the 2000 rpm digester.

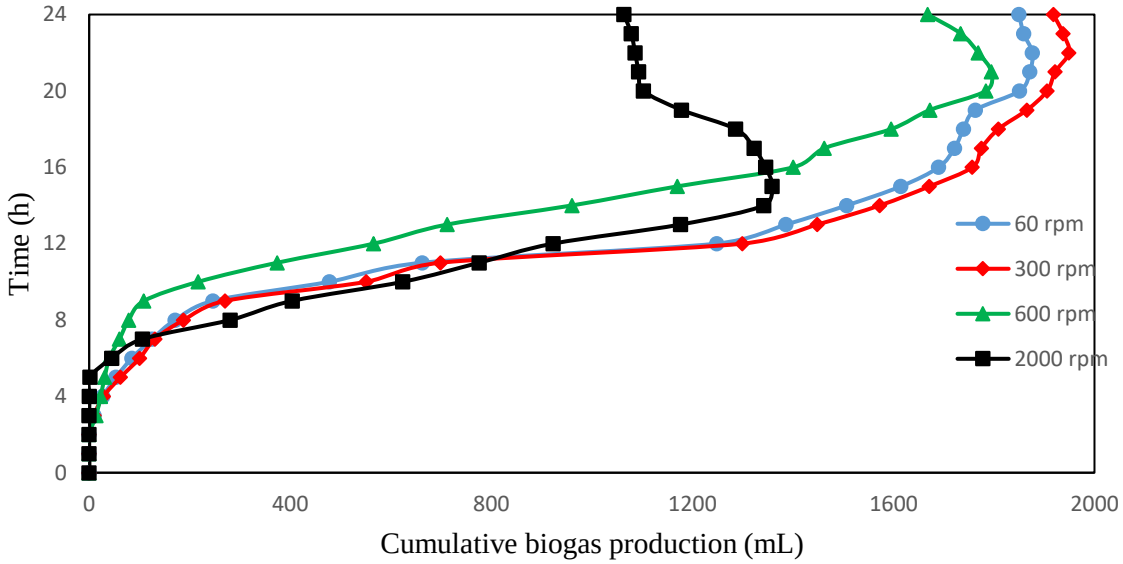


Fig.5.21. Average cumulative biogas production at different mixing speed

Similarly, in Figure 5.21 the 60, 300 and 600 rpm digesters maintained parallel biogas production pattern between the third and 10th hour while the 2000 rpm digester produced little or no biogas before the fifth hour. The 2000 rpm digester improved between the fifth and 15th hour, where the biogas production peaked between the 14th and 15th hour. It then gradually declined before reaching the steady state at the 20th hour. The cumulative biogas production by the 600 rpm digester is lower than the 60 and 300 rpm digesters and reached its biogas production peak at about the 20th hour before it the gradual decline. Contrarily, the cumulative gas generated by the 300 rpm digester is the highest for all the samples, but marginally higher than the 60 rpm digester. Making 300 rpm the optimal mixing speed relative to the other mixing speed investigated and for the quantity of the samples utilised in this study. It means that there is a marginal increase in biogas production as the mixing speed increased from 60 to 300 rpm. However, a further increase in the mixing speed to 600 rpm shows a decrease in the biogas production. In addition, as the mixing speed is further increased to 2000 rpm, there is even

much more reduction in biogas production relative to the biogas produced by the 60 and 300 rpm digesters.

5.5.2 Average total volume of biogas production at variable mixing speeds

The bar chart in Figure 5.22 presents the average total volume of biogas production at different operating mixing speeds.

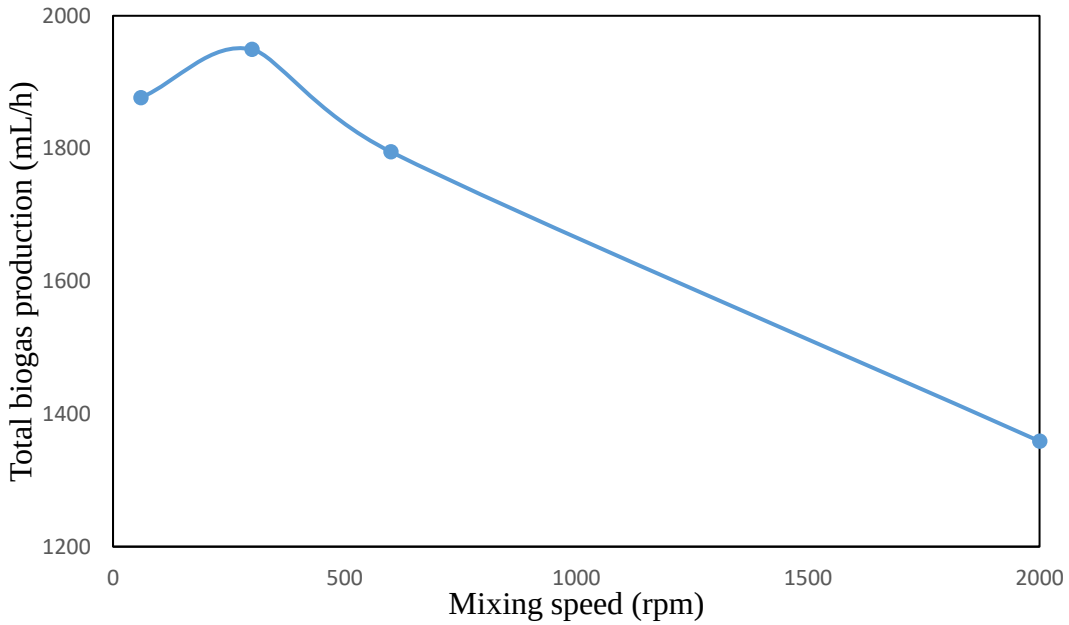


Fig. 5.22. Average total volume of biogas production at different mixing speeds

The average total volume of biogas production at different mixing speeds as seen in Figure 5.22, shows that the digester that was operated at 300 rpm produced the highest volume of biogas, followed by the 60 rpm digester. However, the 2000 rpm digester recorded the lowest total volume of biogas production, behind the 600 rpm digester. For this study, the optimal mixing speed range is 150 – 300 rpm. Further study is required in this area, with the goal of investigating the relationship between various mixing speeds and a specific quantity and total solids of substrates.

5.5.3 Average rate and cumulative methane production at various mixing speeds

The average biogas production rate and cumulative methane production relative to different mixing speeds are presented in Figures 5.23 and 5.24, respectively.

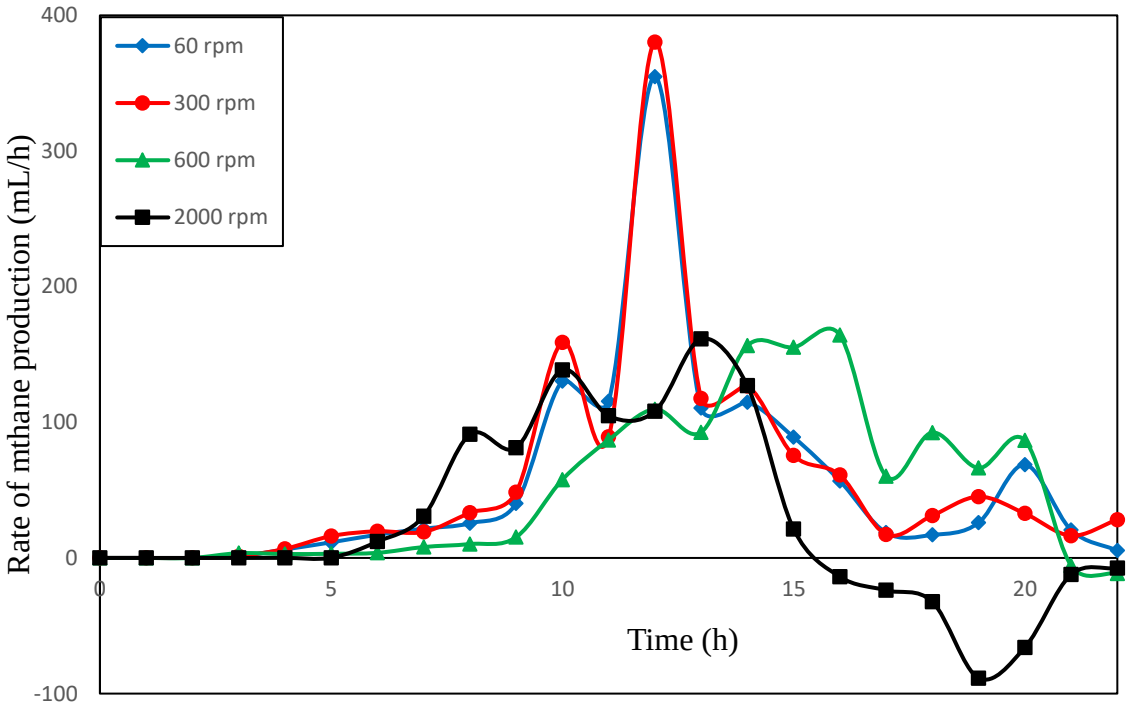


Fig. 5.23. Average rate of methane production at various mixing speeds

As seen in Figure 5.23, the 60 and 300 rpm digesters maintained nearly a similar pattern throughout the digestion process. In addition, the 60 and 300 rpm digesters produced the highest peak at the fourth hour. It is seen in Figure 5.23 that while the rate of methane production for the 60, 300 and 600 rpm digesters was declining, the methane production rate for the 2000 rpm digesters was increasing just after the fourth hour. Furthermore, the plot shows that the methane production rate for the 2000 rpm digesters was negative from the 16th hour, indicating the continuous decline in methane production from the 2000 rpm digester.

The influence of variable digester mixing speed on the cumulative methane production is presented in Figure 5.24.

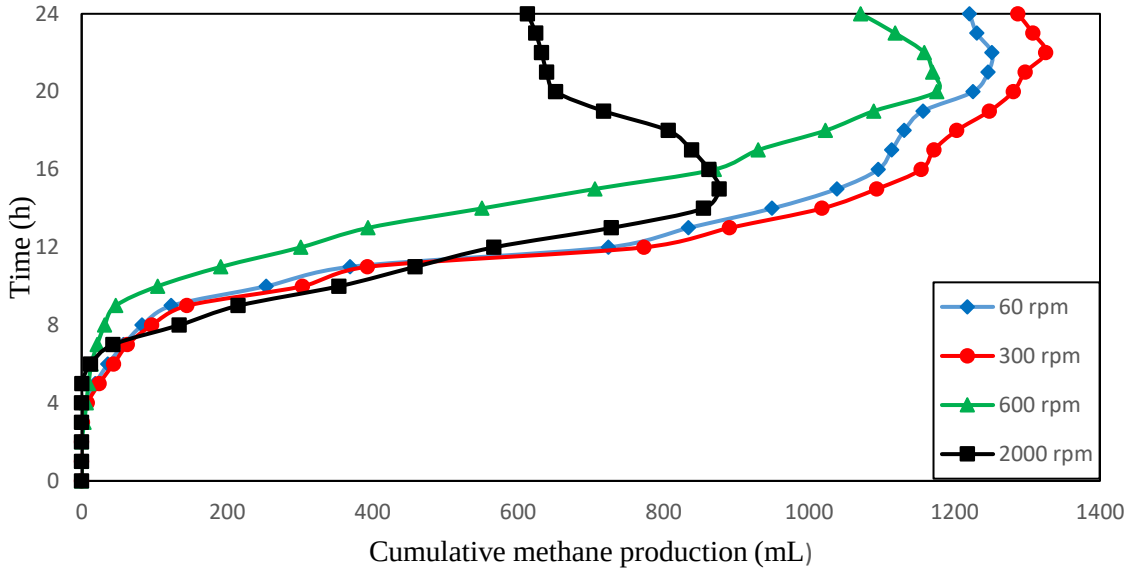


Fig. 5.24. Average cumulative methane production at different mixing speeds

As seen in Figure 5.24, the cumulative methane production to the different mixing speeds is similar to the cumulative biogas production was seen in Figure 5.21. This is because the methanogenic bacterial activity is responsible for the behaviour of the methane and biogas production.

5.5.4 Average total volume of methane production at variable mixing speeds

The average total volume of methane produced at various mixing speeds is presented in Figure 5.25. It shows a similar pattern as the average total volume of biogas production see in Figure 5.22. The 300 rpm digester produced the highest methane, followed by the 60 rpm digester, whereas, the 2000 rpm produced the lowest average total volume of methane. In addition, as the mixing speed increased from 300 rpm, the methane production is seen to decline, indicating that the optimal mixing speed is exceeded.

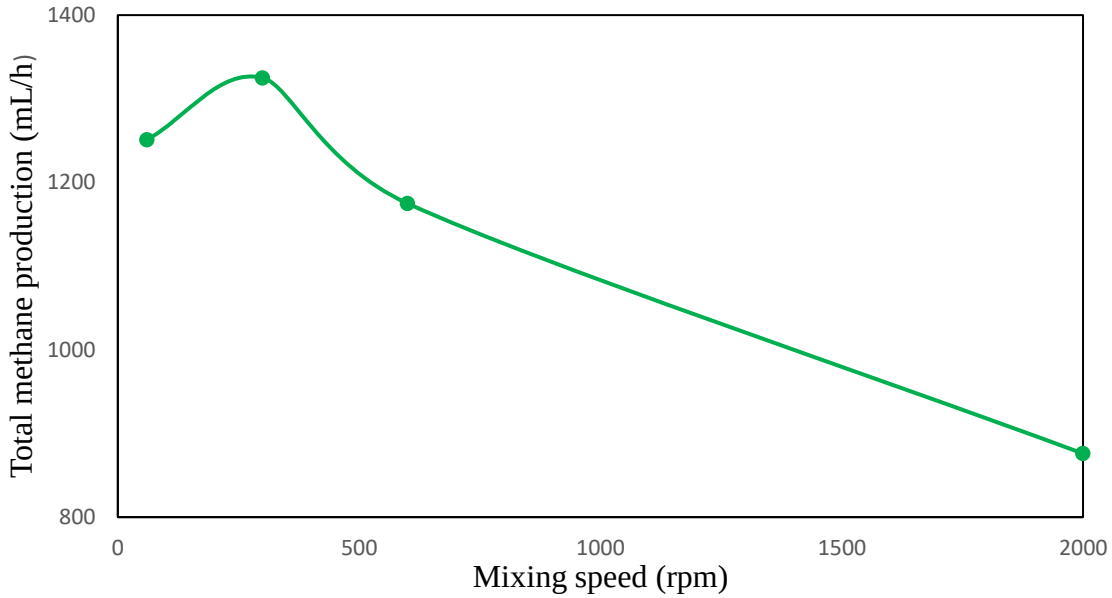


Fig.5.25. Average total volume of methane production at different mixing speeds

5.6 Influence of simultaneous manipulation of multiple inputs on biogas and methane production

The experiment is conducted to explore the significance of simultaneous manipulation of four operating parameters. The operating parameters are temperature, pH, mixing speed and pressure. The operating parameters were simultaneously altered at three different intervals of eight hours each. The data obtained from the experiment is used to test the black-box developed in this research study.

5.6.1 Biogas and methane production

The initial value for temperature, pH, mixing speed and pressure for the experiment are set at 35 ^{o}C, 7.6, 700 rpm and 0.3 bar. This is followed by another parameter adjustment after eight hours to 37 ^{o}C, 5.4, 500 rpm and 0.4 bar, and the final amendment made to temperature, pH, mixing speed and pressure are 41 ^{o}C, 6.8, 120 rpm and 0.5 bar, respectively. The impacts of

the simultaneous alteration of the operating parameters on biogas and methane production are presented in the following plots.

5.6.1a Average rate and average cumulative biogas production

The effect of this experimental design on biogas production rate is presented in Figure 5.26.

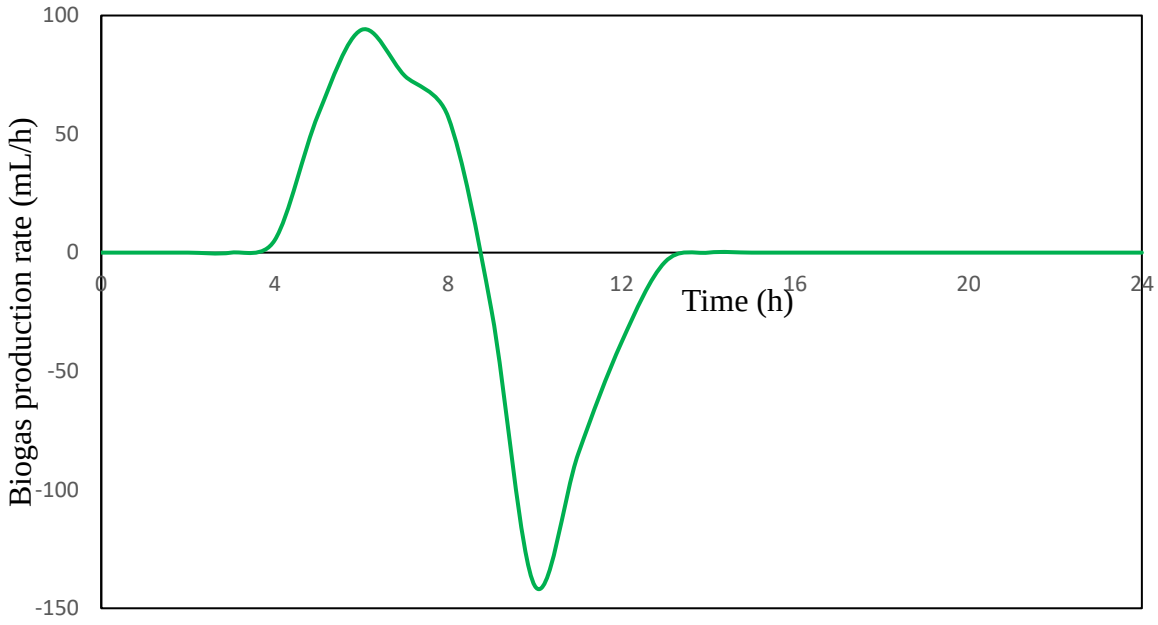


Fig. 5.26. Average rate of biogas production at simultaneous variance of input parameters

As seen in Figure 5.26, the rate of biogas production gradually increased from the fourth hour and peaked after two hours at the sixth hour. It then declines and becomes negative, indicating a continuous decline in biogas production that is lower than the biogas production of the biogas production recorded in the previous hour. Furthermore, the rate of production picked up again but was still on the negative axis for another 4 hours before biogas production ceased and did not resume until the end of the experiment. The reason why biogas production was halted from the 13th hour was due to the reduction of the digester pH to 5.4, at which the digester content becomes acidic and inhibited the activity of the methanogenic bacteria, consequently halting

methane production that combines with CO_2 to forms biogas. Despite increasing the pH to 6.8 at the 17th hour, the system did not recover for the rest of the experiment as seen in Figure 5.26. Indicating that pH inhibition of methanogenic bacteria is difficult to reverse. This result supports the study of Kangle et al. (2012).

The average cumulative biogas production is presented in Figure 5.27.

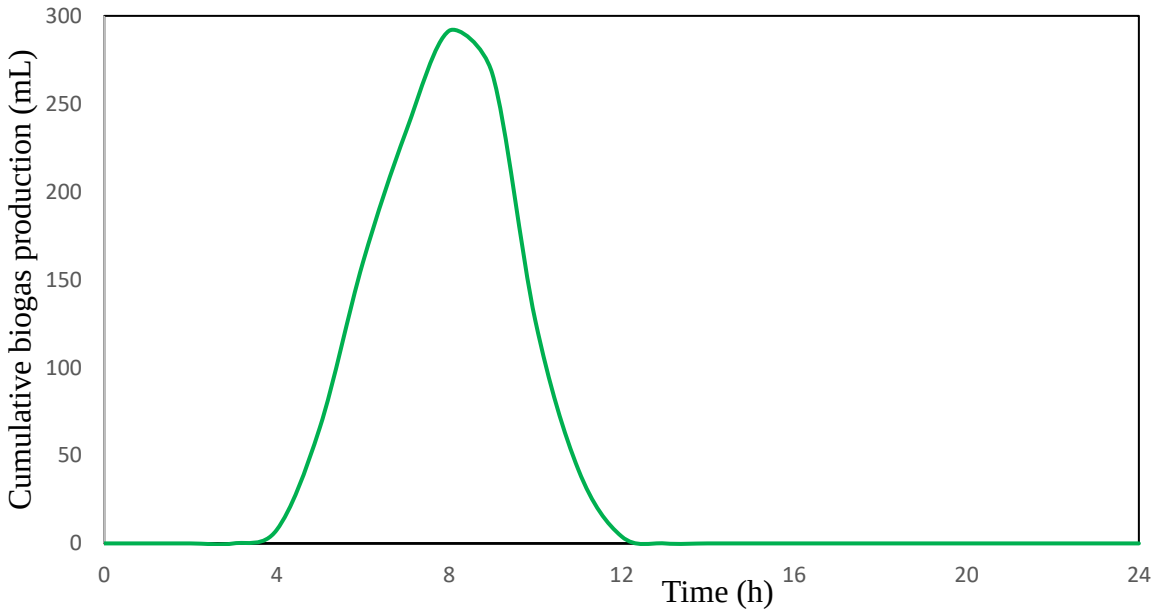


Fig. 5.27. Average cumulative biogas production at simultaneous adjustment of input parameters

As seen in Figure 5.27, the average cumulative biogas production peaked at the eighth hour. It then decreased to the 13th hour at which biogas production is halted. The reason for the termination of biogas production is similar to the one given for the biogas production rate in.

5.6.1b Average rate and average cumulative methane production for Expeiment31

The influence of the adjustment of the operating parameters on methane production is presented and discussed in this section.

In Figures 5.28, the average methane production rate is presented.

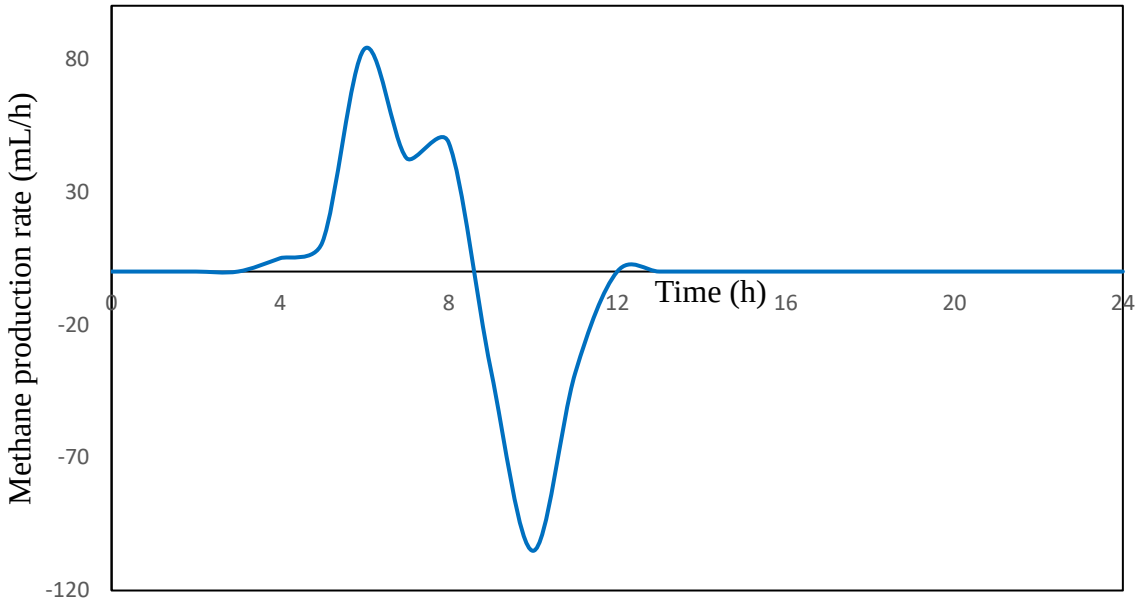


Fig. 5.28. Average rate of methane production at simultaneous adjustment of input parameters

The response of methane to the simultaneous adjustment of the operating parameters is similar to that of biogas pattern is seen in Figure 5.26. The rate of methane production as seen in Figure 5.28 reached the peak at the sixth hour. It then decreased to the minimum on the negative axis at the tenth hour. Then the rate of methane production increased again up to the 12th hour when it halted and did not recover. This is due to the acid condition caused by the adjustment of digester pH to 5.4, which inhibited the methanogenic bacteria.

Furthermore, the average cumulative methane production is presented in Figure 5.29.

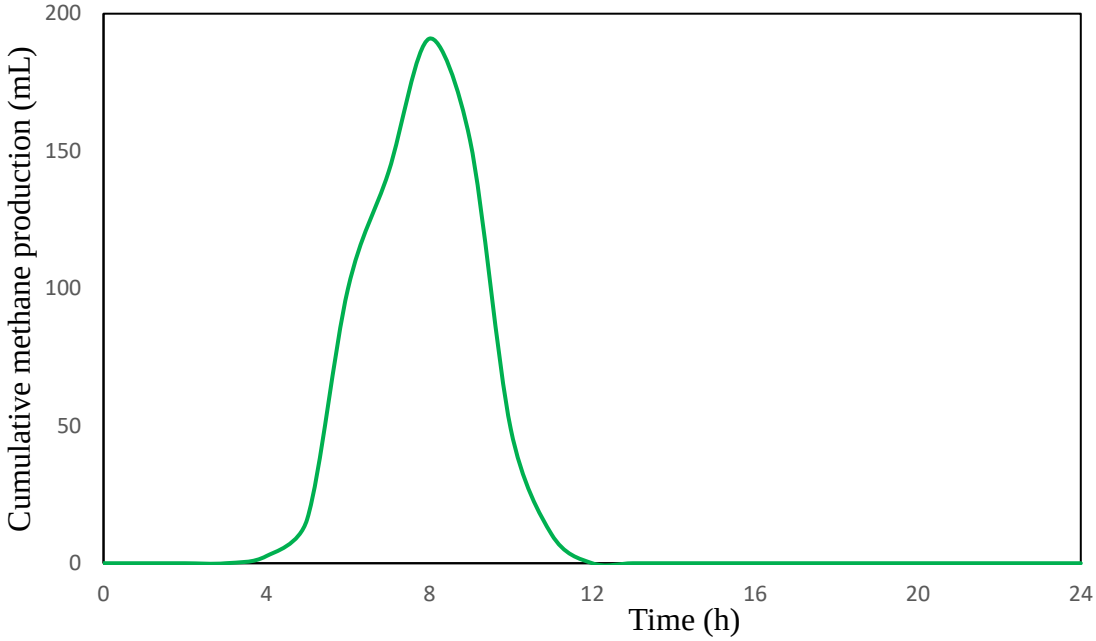


Fig. 5.29. Average cumulative methane production at simultaneous adjustment of the operating parameters

Similarly, the average cumulative methane production as seen in Figure 5.29 increased rapidly and peaked at the seventh hour as observed in Figure 5.27. It then dropped to the zero level at the 13th hour when methane production ceased, because of the same reason stated for the biogas production rate.

5.7 Influence of co-digestion on biogas and methane production

This section discusses the significance of the results obtained from the investigations carried out to determine the effect of co-digestion on biogas and methane production. Samples of Food waste (FW) and Wastewater sludge (WWS) are co-digested in the following ratio: FW only represented as (S1); WWS only (S2); 0.3FW+0.7WWS (S3); 0.5FW+0.5WWS (S4) and 0.7FW+0.3WWS (S5). The results obtained are presented in the following plots.

5.7.1 Average rate of biogas production at different co-digestion ratio

The biogas production rate is presented in Figure 5.30.

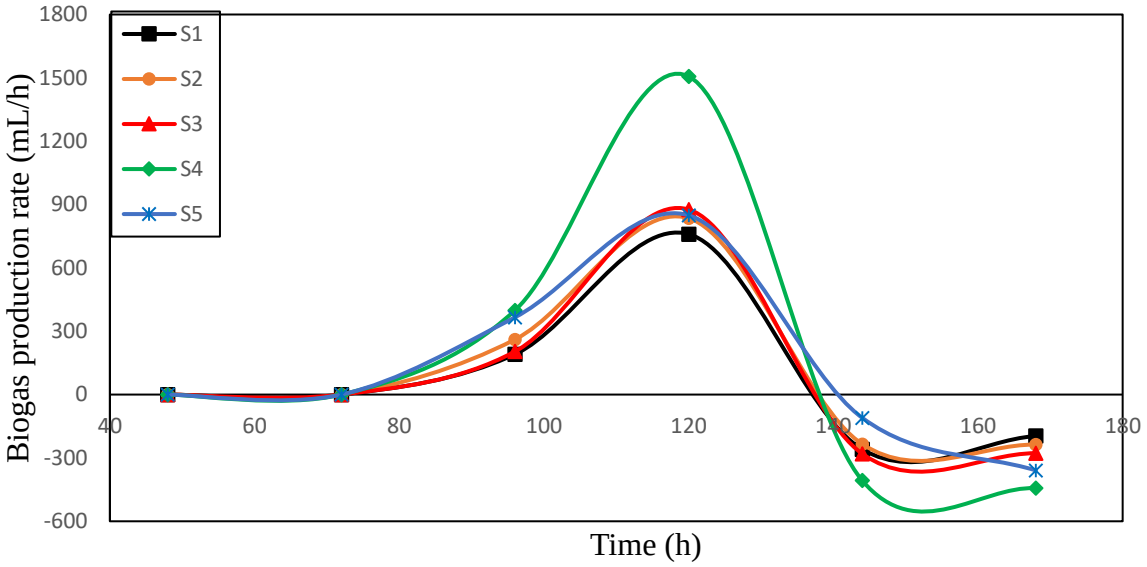


Fig. 5.30. Average rate of biogas production at different co-substrate ratio

As seen in Figure 5.30, the average rate of biogas production from co-digestion of substrates at different ratios followed nearly a similar pattern. This indicates that the response of the anaerobic bacteria to the operating parameters of all the samples is similar. The biogas production rate peaked on the fifth day for all the samples and the decreased. However, the S4 sample recorded the highest biogas production rate, followed by the S5 digester and the S3 digester. Whilst the S1 sample generated the lowest biogas production rate, followed by the S2 sample, which is the single-substrate samples. The result shows that co-substrates samples performed better than the single-substrate samples. The negative rate of biogas production indicates that the conversion of substrate to biogas was nearly completed, thereby resulting in a continuous decline in the rate of biogas production.

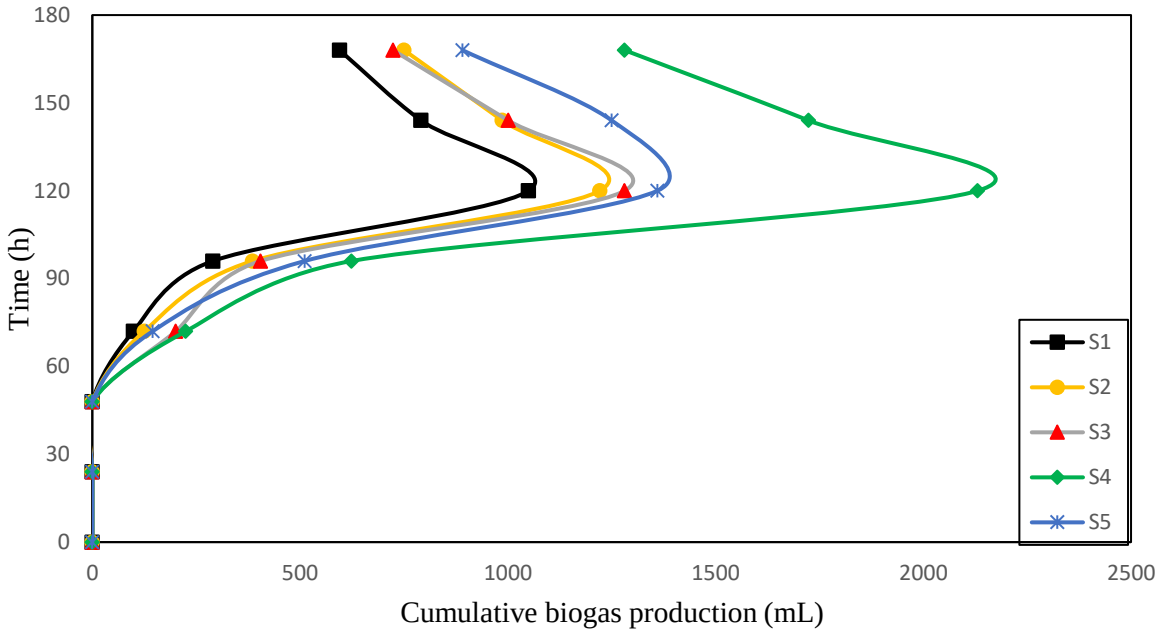


Fig. 5.31. Average cumulative biogas production at different co-substrate ratio

Similarly, Figure 5.31 shows that S4 digester produced the highest cumulative biogas followed by the S5 digester and then again, the S3 digester. Furthermore, it was observed that the digestion of WWS as a single substrate generated the lowest average cumulative biogas while the second lowest is produced by the digestion of FW only substrate, that is, SI and S2, respectively. Implying that co-digestion of FW and SSW improved the digestion process, as suppose to the digestion of FW and WWS as single substrates. In addition, S4 was found to be the best co-substrate ratio for co-digestion of FW and WWS for improved biogas production for this study.

5.7.2 Average methane production rate at different co-substrate ratio

As seen in Figure 5.32, the average rate of methane production from co-digestion of substrates at different ratios followed nearly a similar pattern. This indicates that the response of the anaerobic bacteria to the operating parameters in all the samples is similar. The methane production rate peaked at the fifth day for all the samples and the decreased. However, the S4

sample recorded the highest methane production rate, followed by the S5 digester and the S3 digester. Whilst the S2 sample generated the lowest biogas production rate, followed by the S1 sample, which is the single-substrate samples. The result shows that co-substrates samples performed better than the single-substrate samples. The negative rate of methane production indicates that the conversion of substrate to methane was nearly completed, thereby resulting in a continuous decline in the rate of methane production.

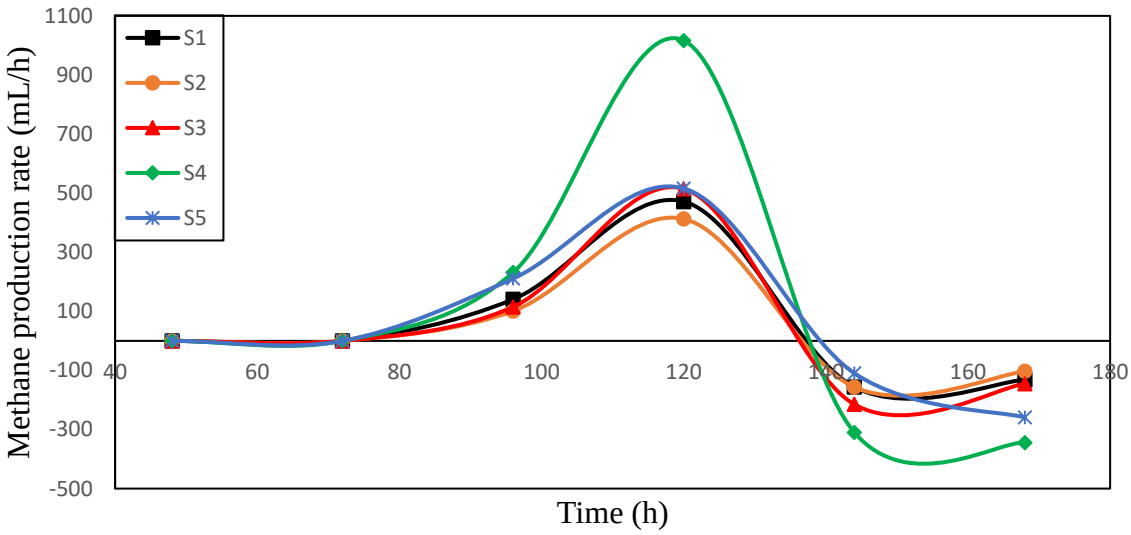


Fig. 5.32. Average methane production rate at various substrate ratio

5.7.3 Average cumulative methane production at different co-substrate ratio

The average cumulative methane production as seen in Figure 5.33, shows that all the samples recorded their individual highest methane production on the fifth day. It also indicated that the co-substrate digesters performed better than the single-substrate digesters. Similarly, figure 5.33 shows that S4 digester produced the highest cumulative methane, which implies that S4 is the best co-substrate ratio for the co-digestion of FW and WWS for this study. This study has shown that co-substrate digestion offers better digester performance than the single-

substrate digestion, which supports the work previously conducted on co-digestion (Wu, 2007; Mata-Alvarez et al., 2000).

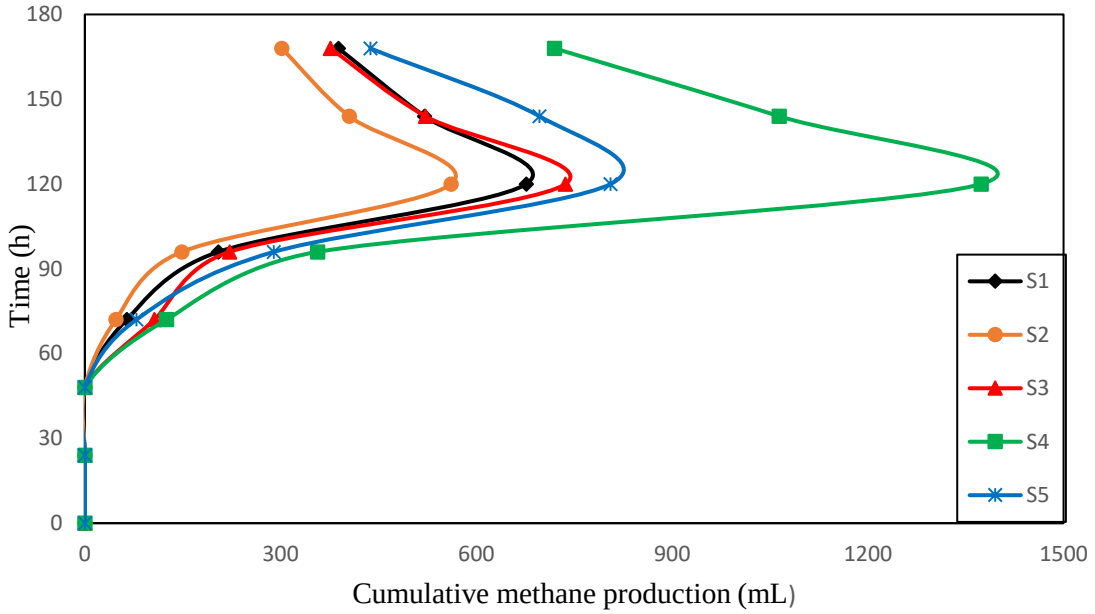


Fig. 5.33. Average cumulative methane production at different co-substrate ratio

5.7.4 Observed process characteristics

The observation of the responses of biogas and methane production to variable temperature, pH, mixing speed and pressure plotted and discussed in this chapter indicate that mesophilic AD process is a nonlinear dynamic system. The properties of the nonlinear system include the following.

1. The mesophilic AD process does not satisfy both superposition and homogeneity principles.

   Superposition - For a system *S* to satisfy superposition principle (Cuff, 2012):

   If $y_1 = Sx_1$ and $y_2 = Sx_2$, then $y_1 + y_2 = S(x_1 + x_2)$

   Homogeneity - For a system *S* to satisfy homogeneity principle (Cuff, 2012):

   If **y** = *S*x, and k is a constant then yk = *S*(xk)

2. The system has multiple equilibrium points. The curves of the plots in this chapter signify the multiple equilibrium points. The equilibrium point is a point x_0 in the state space of autonomous system $\dot{x} = f(x)$, when the state x reaches x_0 and stays at x_0 for all times (Canon, 2016). The equilibrium points for a nonlinear system is the solution to the equation:

$$f(x_n) = 0$$

Where, n nonlinear equations in n unknowns have to be solved and the solution is between 0 and infinity.

3. The pattern of some plots of the system demonstrated randomness irrespective of the deterministic nature of the system.

5.8 Summary

This chapter has presented the results of the experimentations conducted in this research work. It is found that the biogas production is influenced by the variation in the operating temperature, pH, mixing speed and pressure. The outcome of this study has also shown that for improved biogas production, all the operating parameters need to be maintained at their individual optimum range. In addition, the behaviour of the system under consideration shows that it is a nonlinear process. Furthermore, this chapter highlighted the importance of co-digestion of substrates as well as the importance of the appropriate ratio of the co-substrates in achieving improved biogas production. The next chapter presents the modelling of nonlinear multiparameter models for mesophilic AD process, which is based on the response of biogas and methane production to the overall impact of the operating parameters considered in this study.

Chapter 6 **Black-Box Model Construction**

6.1 Introduction

Chapters 4 and 5 discussed the method, generation and analysis of the data from the experimental investigation of the influence of temperature, pH, mixing speed and pressure on biogas and methane production. More specific, Chapter 4 discussed the experimental study to determine the effects of the multi-parameter input on biogas and methane production, while Chapter 5 present an analysis of the implications of these effects based on the data obtained from the experimentations - how the input variables influenced the production of biogas and methane. However, the discussion in this chapter centres on the construction of multivariable nonlinear mathematical models for mesophilic AD process from experimental measurement considering a multiple input single output (MISO) configuration. The purpose of the models is to predict the production of biogas and methane under mesophilic conditions. The models in this thesis are constructed using System Identification method. Figure 6.1 shows the block diagram of the model identification process.

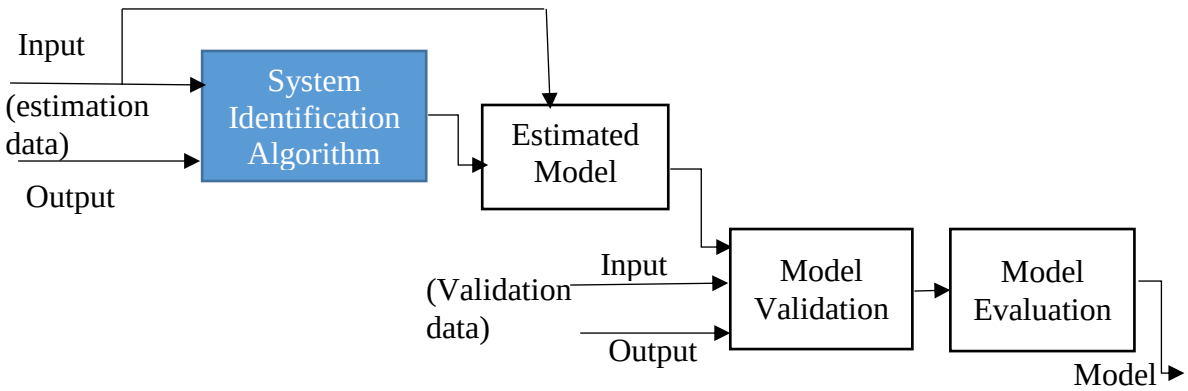


Fig. 6.1. Block diagram for model identification (Schoukens et al., 2012)

6.2 System Identification modelling method

System Identification is a method of estimating mathematical models of a dynamic system from measured input-output data (Schoukens et al., 2012). It is implemented under MATLAB functions (Ljung, 2015; Ljung, 2010). The procedure of estimating a model of a dynamic system from measured input-output data (based on the System Identification algorithms) consists of three main phases (Ljung, 2015).

- Design of the experimental setup and data collection,
- Selection of the most appropriate model structure, and
- Identification of method to select a particular model in the set based on the information in the input-output data

In more detail, System Identification modelling method is composed of a number of steps as shown in the flowchart in Figure 6.2a.

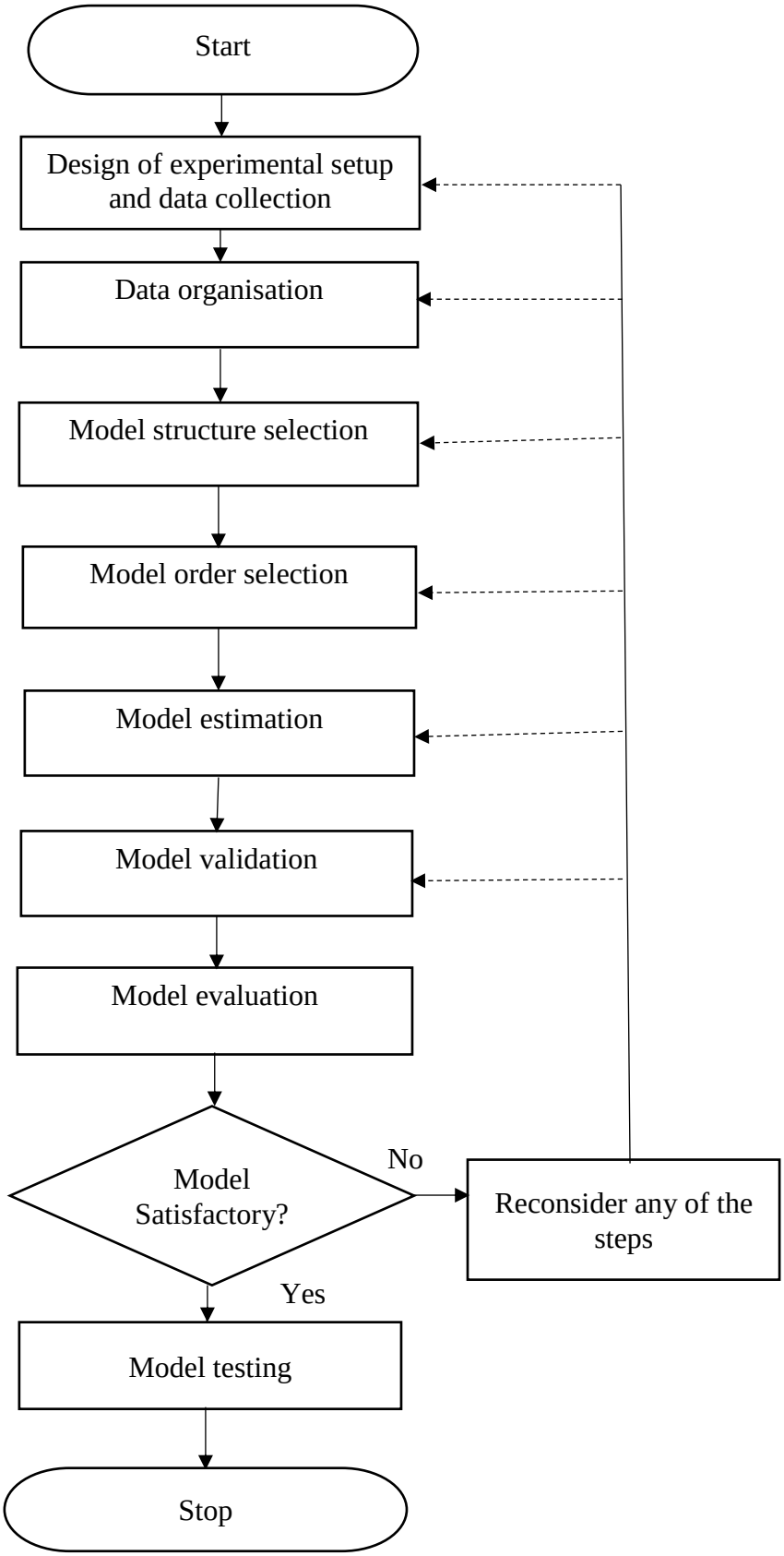


Fig. 6.2a. Flowchart of steps in System Identification modelling method

I. A Good design of experimental setup and data collection ensures that the relevant variables are measured and collected with sufficient accuracy in order to reflect the behaviour of the dynamic system under consideration. The input-output data from the dynamic system, which is acquired through experimentation, is used by System Identification Toolbox to estimate the values of adjustable parameters in a given model structure (Ljung, 2015). The quality of a model depends on the how well the data collected from experimental setup reflects the behaviour of the dynamic system (Ljung, 2015).

II. The data organisation involves splitting of sample data into two parts; one part is used for model estimation and the other part is used for model validation purpose (Ljung, 2015). The bigger the size of the data set for model estimation, the more information is obtained about the system and the better the model fit the data (Tangirala, 2015; Villanverde and Banga, 2013). Data organisation also include the separation of output vector and sampling time from the estimation and validation data sets, which are the information required for the construction of discrete-time models (Ljung, 2015). Furthermore, the organisation of data also includes the conversion of estimation and validation data sets into iddata objects, which is the representation of time-domain data and the required data structures by MATLAB for System Identification (Ljung, 2015)

III. The model structure is the mathematical relationship between input and output variables, with unknown parameters (Ljung, 2015). System Identification method requires the selection of model structure in order to determine the numerical values of the parameters of the model (Ljung, 2015).

IV. Model order is an important component in System Identification modelling method, which is represented by one or more numbers whose definition is a function of the model structure (Ljung, 2015; Pillonetto et al., 2013). The model order represents the set of a number of poles and number zeros, as well as delays for defining the type of model structured used for model identification (Ljung, 2015; Sugiki, 2014).

V. In model estimation, System Identification Toolbox uses estimation algorithms and the selected model structure to determine the numerical values of model parameters (Ljung, 2015).

VI. Model validation is used to determine how close the model is to the actual system (mesophilic AD process). This involves comparing the simulated output of the model and output of the measured data (Ljung, 2015; Proctor et al. 2013; Rahiman et al., 2009).

VII. Model evaluation is used to analyse the suitability of the estimated model with respect to how close the simulated output of the model compares with output of measured data of the actual system for a specific stimulus (Vazquez-Cruz et al., 2014). If the evaluation shows that the model output compares “reasonably” (accuracy evaluation) with the measurement of the actual system, based on the desired accuracy figure, then the modelling process is complete. However, if the results of the model evaluation are not satisfactory, then it is necessary to reconsider the previous steps of the model construction as seen in Figure 6.2a.

It is essential to consider the various levels of accuracy of data and information utilised in constructing the models in this study, especially when evaluating the quality of the models, which are affected by:

- The assumptions based on views from general knowledge and discussions with experts in the field;
- Knowledge of the stoichiometry and analytical relationships guiding the processes;
- The relationships between input and output variables being experimentally determined and presented in the form of data; and
- Statistical correlations that might exist

6.2.1 Choice of a suitable mathematical model

The choice of a suitable mathematical model is an important prerequisite to constructing a satisfactory model of a bioprocess. In section 3.2, the two main types of mathematical model (white-box and black-box models) were discussed. White-box models require an in-depth knowledge of the system under consideration in order to represent the system in a sufficiently detailed manner to reproduce the process behaviour (Krishna, 2010); however, the knowledge about AD process is limited. This is due to the poor understanding of the bacterial community responsible for AD process, particularly with respect to the composition of the bacterial community and the bacterial response to different substrates and operations (Li, 2013; Riviere et al., 2009).

Monod kinetic white-box model is inadequate to describe AD process because of the presence of a single set of kinetic parameters involved (Vázquez-Cruz et al, 2014). Again, white-box models such as ADM1 that are based on the first principal is computationally intensive to fit

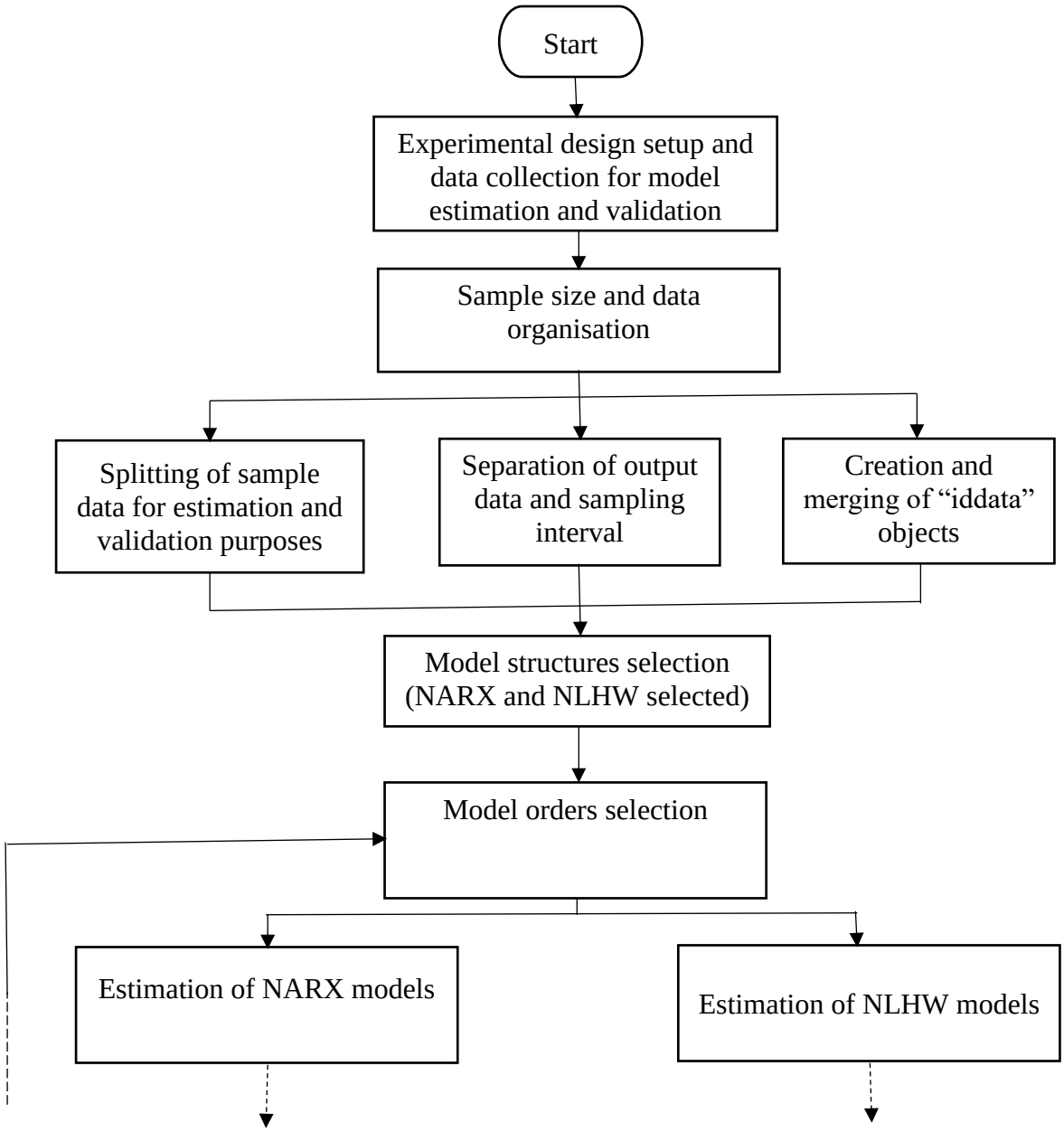
because of the requirement for a large number of kinetic parameters implemented in the form of DAE and DE set (Yu et al., 2013).

Furthermore, the financial and time constraint, which limits the construction of white-box models, especially when dealing with a complex process, such as AD, whose process knowledge is vague. According to Heams (2006), white-box models have little or no significance in AD because of their inadequacy to consider all the stages of the anaerobic process, operating parameters and history of operational data. Another drawback of white-box models is the lack of consideration for the actual physico-environmental operating parameters that influence anaerobic bacterial activity. Leading to the inability of the white-box model to describe AD process when physico-environmental parameters need to be considered for a more realistic study of the operation. Due to these limitations, it may not be practically feasible to apply white-box models to predict the production of biogas and methane from measured input-output data for this study.

However, black-box models are based on the availability of input-output data for model identification (Vázquez-Cruz et al, 2014). They require no in-depth knowledge of the system under consideration; thereby can be applied when there is little or no knowledge about the process. Though the parameters of the functions of black-box models lack physical significance, yet black-box models can adequately represent the trend in system behaviour even in a complex process where it is not possible to solve the resultant equations (Krishna, 2010). In addition, black-box models can adequately predict biogas and methane production from input-output data. Therefore, black-box models are considered for the construction of the models for mesophilic AD process from measured MISO data for this study.

6.2.2 Black-box model identification procedure

Given an overview of how the System Identification Toolbox "works" to construct models based on input-output data sets as seen in Figure 6.2a. The flowchart in Figure 6.2b shows the actual procedure followed in this study to estimate the multi-input-single-output (MISO) nonlinear black-box models from experimental data, which are discussed in more specific details as applied to this research work in the following steps. The models are estimated in MATLAB command line, and the execution codes for various processes are documented in Appendix B.



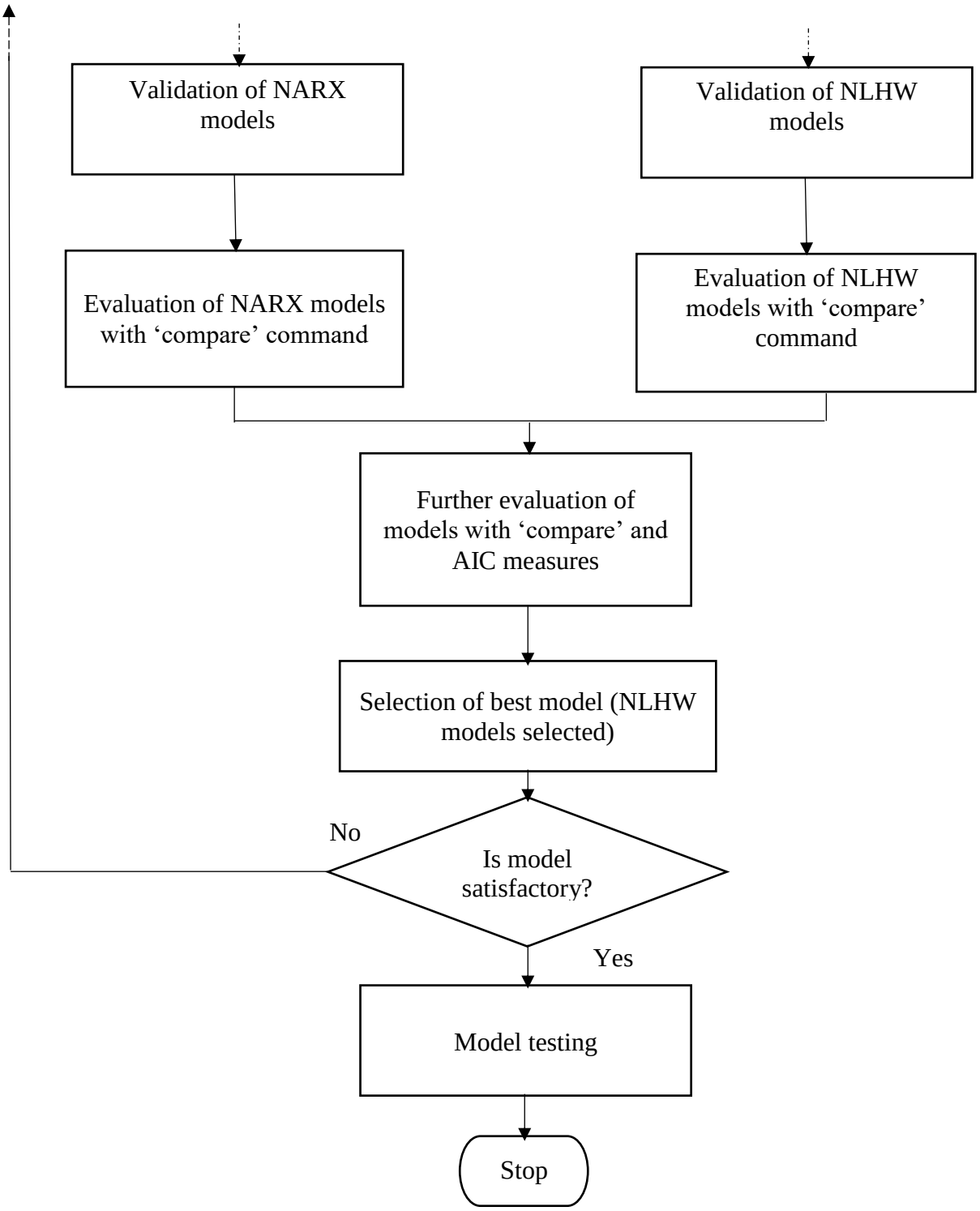


Fig. 6.2b. Flowchart of the actual black-box model identification procedure

Step 1: Design of experimental setup and data collection

The design of experimental setup for this research study, which is discussed in Chapter 4, is utilised to investigate the influence of multi-input parameters on biogas and methane production. The data set utilised for the model identification, which is in time-domain, contains 624 data samples, collected from 26 experiments with a sample time of 1 hour for a 24-hour duration, as described in Chapter 5. The inputs $u(t)$, are composed of 26 variables: eight variables of temperature (in ℃), six variables of pH, six variables of mixing speed (in rpm) and six variables of pressure (in bar). The output vector $y(t)$ contains a single variable: biogas or methane.

Step 2: Data organisation

The input-output data collected from the experiments are loaded into MATLAB Workspace. The input data sets contain a 4-by-25 matrix for each set and the output data is a vector. The output vector, as well as the time vector, are extracted from the 26 experiments in MATLAB command line. An iddata object is created for each of the 26 experimental data sets with the following syntax (Ljung, 2015).

iddata_object = iddata(y, u, Ts)

This creates an iddata object containing a time-domain output variable y, and input variable u. Where Ts specifies the sampling time of the experimental data (Ljung, 2015). An iddata object is a basic object and the required data structure by MATLAB for handling input-output variables in System Identification Toolbox (Ljung, 2015).

Step 3: Data quality analysis and data selection

The quality of the data collected from the experiments is analysed using the input-output data plot shown in Figure 6.3 and Figure 6.4 for biogas and methane production, respectively.

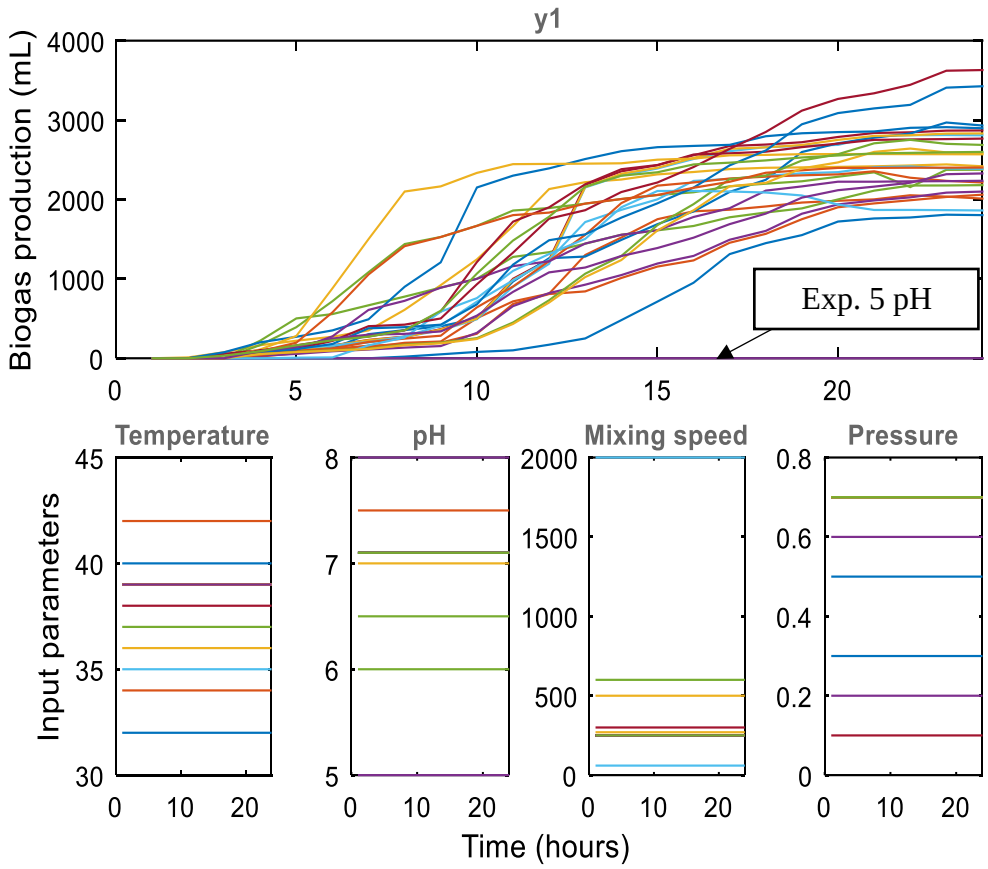


Fig. 6.3. Input-output data plot for biogas production

The plot seen in Figure 6.3 is used to inspect and clean data. The plot shows that the output data generated from the experiment with pH of 5 is zeros all through due to the none production of methane, which is the major constituent of biogas, resulting from the acid state of the digester sludge. Therefore, the data from the experiment with pH of 5 is dropped, leaving the total experimental data used for the estimation and validation of the nonlinear black-box models to 25.

Similarly, the plot in Figure 6.4 is used to inspect and clean the input-output data for methane production.

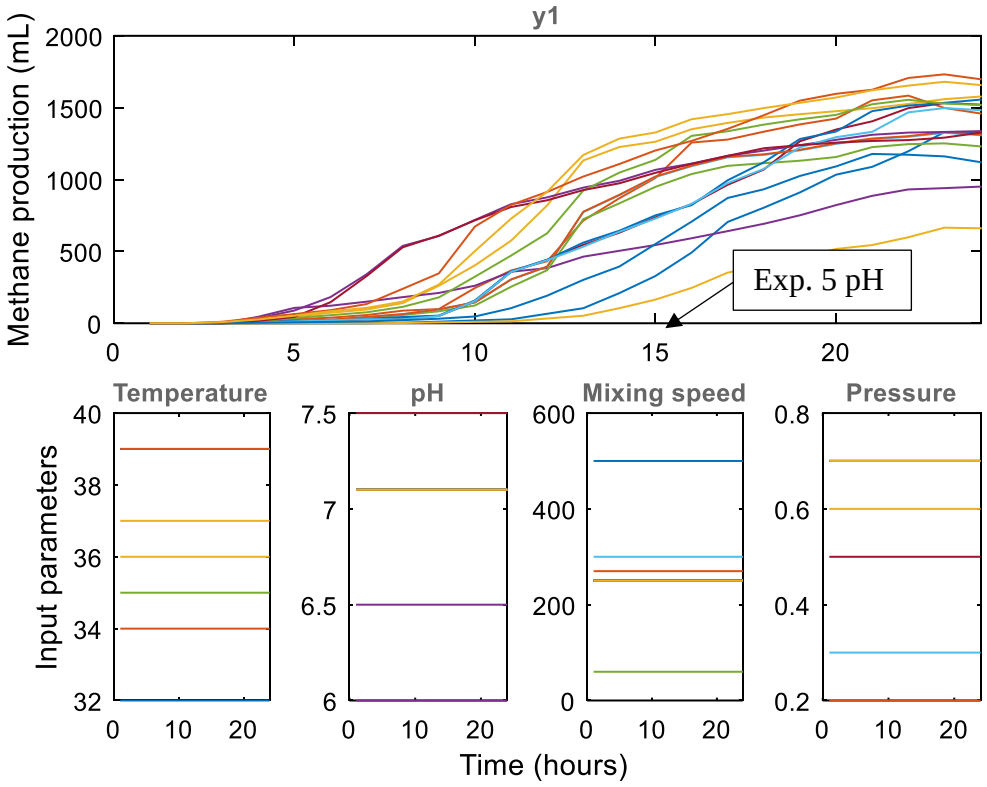


Fig. 6.4. Input-output data plot for methane production

The plot in Figure 6.4 shows that the output data generated from the experiment with pH of 5 is zeros all through due to non-production of methane at the acid state of digester sludge. Therefore, the data from the experiment with pH of 5 is also dropped, leaving the total experimental data used for the estimation and validation of the nonlinear black-box models to 25.

The 25 iddata objects from the 25 experiments for both biogas and methane production are split into two unequal subsets, 17 iddata objects are used for model estimation and eight iddata objects are used for model validation. All the iddata objects for model estimation are merged into a single iddata object; de for biogas production and ze for methane production. Similarly,

all the iddata objects for model validation are merged into one iddata object, dv for biogas production and zv for methane production, with the following syntax (Ljung, 2015).

de or dv = merge (iddata1, iddata2 …. IddataN)

The merged estimation and validation iddata objects contain individual iddata objects that represent all the four input parameters.

Step 4: Model structure selection

The choice of a suitable model structure is based on the understanding of the System Identification method as well as the dynamic systems under consideration (Murray-Smith, 2015). As discussed in section 6.2.1, mesophilic AD process is a nonlinear system, which can be adequately represented by black-box models. There are a variety of nonlinear black-box model structures that are available for modelling mesophilic AD process, such as NARX, NOE, NARMAX, NLHW and NBJ, discussed in section 3.2.1. However, NARX and NLHW model structures are often used for nonlinear black-box model identification (Rankovi´c et al., 2012). The NARX and NLHW are the two model structures used to estimate the models for biogas and methane production for this research work. The model structures have different nonlinearity estimators that are specific to the selected model structures.

The output plot for NARX and NLHW models do not show a good Fit with the incorrect complexity of the nonlinearity estimators (Alexandridis and Zapranis, 2014; Zhang, 1997, cited in Matlab 2016a). The complexity of the nonlinearity estimators is specified using the NumberOfUnits (number of units in nonlinear estimator) (Alexandridis and Zapranis, 2014; Zhang, 1997, cited in Matlab 2016a). This is a positive integer that can be chosen automatically when the number of units is determined from estimation data. Or interactively, when the number of units is determined during model estimation (Alexandridis and Zapranis, 2014;

Zhang, 1997, cited in Matlab 2016a). A higher number of units signifies a more complex nonlinearity estimator. A nonlinear estimator with a fewer number of units will result in underfitted model (Alexandridis and Zapranis, 2014). However, specifying more number of units will lead to overfitted model (Alexandridis and Zapranis, 2014).

Selecting the appropriate nonlinearity estimator complexity requires validating a low complex model first and then progressively increase the complexity and validate accordingly (Zhang, 1997, cited in Matlab 2016a). The quality of the models reduces as the nonlinearity estimator becomes too complex (Zhang, 1997, cited in Matlab 2016a). The reduction in quality of the performance of the models is only seen if independent estimation and validation data sets are used (Alexandridis and Zapranis, 2014; Zhang, 1997, cited in Matlab 2016a). Therefore, it is essential to select the suitable number of units in nonlinearity estimators for a particular model estimation. According to Alexandridis and Zapranis (2014), the simplest way to select the optimal number of units is by trial and error.

Step 5: Model orders selection

Model order is used for estimating nonlinear black-box models (Ljung, 2015). It is a set of positive integers that represent the poles, zeros and delays used for defining the regressor configuration for NARX models, and the linear subsystem transfer function for NLHW models (Ljung, 2015; Sugiki, 2014).

The model order for NARX models are in the form [na nb nk], where na signifies the number of past output terms used to predict the current output, nb represents the number of past input terms used to predict the current output, and nk specifies the delay from input to the output in terms of the number of samples (Ljung, 2015).

Similarly, the model order for NLHW models are in the form [na nf nk], where na signifies the number of past output terms used to predict the current output, nf specifies the number of poles of the linear transfer function, and nk specifies the delay from input to the output in terms of the number of samples (Ljung, 2015).

A poor model Fit can be as a result of the incorrect model order. Ideally, the lowest-order model that adequately describes the system dynamics well is preferred (Ljung, 2015; Sugiki, 2014). If the model order is too low, it can result in model underfitting. Similarly, If a model order is too high it can cause a model to be overfitted (Ljung, 2015; Sugiki, 2014). Both underfitting and overfitting are undesirable in a model, however, overfitting has more impact than underfitting in that it causes the model to fit the noise, instead of the actual behaviour of the system under consideration. The selection of model orders is by trial and error (Ljung, 2015; Sugiki, 2014).

The remaining steps in model identification, including model estimation, model validation and model evaluation are discussed more in the following sections as necessary due to the description being specific to the selected model structures.

6.2.3 NARX model estimation

The NARX model is characterised by regressors and a nonlinear estimator (Ljung, 2015; Shariff et al., 2014). The nonlinear estimator is composed of a parallel combination of nonlinear and linear functions. The regressors are described as delayed inputs and outputs from the nonlinearity identification, which are inputs to the nonlinear estimator functions, as shown in Figure 6.5 (Ljung, 2015; Shariff et al., 2014).

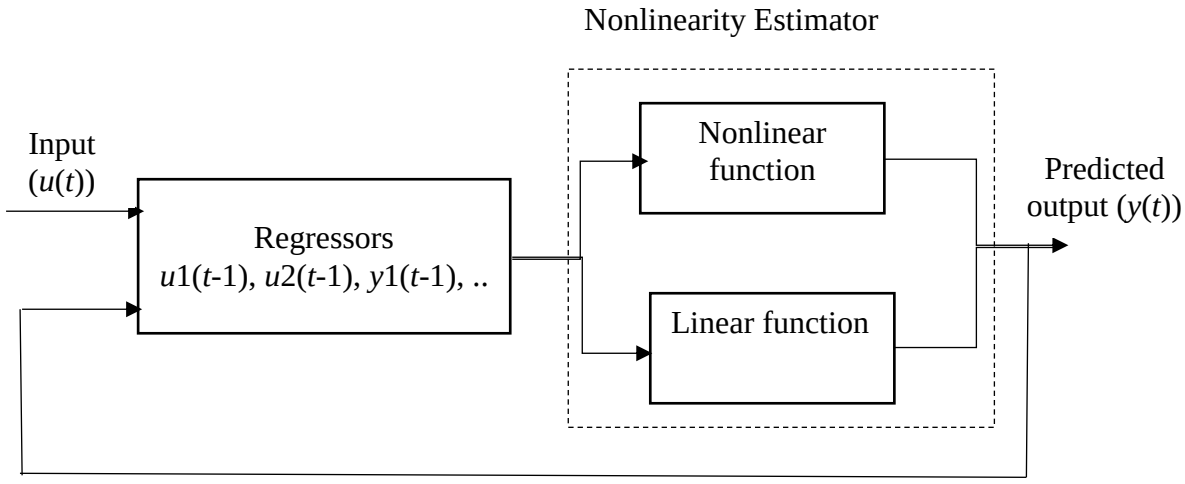


Fig.6.5. Block diagram of NARX models (Ljung, 2015; Shariff et al., 2014)

The NARX model is described by the following equation (Shariff et al., 2014).

$$y(t) = f\,[y(t-1), \ldots y(t-n_y), \ldots, u(t), u(t-1), u(t-n_u)] + \varepsilon(t) \qquad (6.1)$$

Where,

$y(t)$ is the output

$u(t)$ is the input

$\varepsilon(t)$ is the noise signals

n_y is the maximum lags of past output

n_u is the maximum lags of past inputs

f is the nonlinear function

$u(t - 1)$ and $y(t - 1)$ are the regressors

Step 6a: NARX model estimation for biogas and methane production

Two important elements, model order and nonlinearity estimators, which are required for the estimation of black-box models, are chosen by trial and error. The NARX models are estimated in MATLAB command line with the following syntax.

M = narx (iddata, model order, nonlinearity estimator),

where M is the NARX models.

The Fit (in %) of the estimated model is the mean square error (MSE) between the measured data and the simulated output of the model (Ljung, 2015). A model of perfect fit (no error), means that the Fit is 100 %. Similarly, a model that is not able to describe any of the variation of the output and only the mean level corresponds to a Fit of 0% (Ljung, 2015).

To get a measure of how good the Fit of the estimated model to the measured data is, the simulated output of the model is compared to the output of the measured data (Ljung, 2008; Ljung, 2015). This approach is used to validate the estimated models by checking how well the simulated output of the models fits the measured output (Ljung, 2008; Ljung, 2015). The model output plot comparing the simulated output of the models and the measured output, the percentage of the output the model reproduces, which is the 'Best Fit', is computed using the following equation (Ljung, 2008):

$$\text{Best Fit} = \left(1 - \frac{|y - \hat{y}|}{|y - \bar{y}|}\right) \text{x } 100 \tag{6.3}$$

Where, y is the measured output, $\hat{y}$ is the simulated output, and $\bar{y}$ is the mean of y. The Fit ranges from 100%, which indicates a perfect Fit, to 0% that corresponds to a very bad fit. The measured data is reffered to as the validation data.

Comparing the quality of different estimated models, a model is considered to describe the data well when the Fit to the validation data is high. If an estimated model has a higher Fit to the validation data than another model, then the first model is considered to describe the real system better. The comparison between the simulated output of the model and the output in the validation data is computed by the 'compare' command in MATLAB with the following syntax (Ljung, 2015).

Compare(M), where M is the estimated model

As seen in the plots in Figure 6.3, the responses of biogas production to the variables of Temperature, pH, Mixing speed and Pressure, indicate that mesophilic AD system is in the region of the third order. The first model order chosen for the estimation of NARX models for this study is [ones(1,1), ones(1,4), ones(1,4)], which is equivalent to [1 1 1] = [na nb nk]. It means that the output variable is predicted by the output and all the four input variables, and it is being delayed by one sample. With the MATLAB default 'wavelet' nonlinearity estimator, the function nlarx, which is used to facilitate the estimation of NARX models (Shariff et al., 2014), and the model order, the NARX model mx1 is estimated.

Similarly, five more models are estimated using the following five different model order:

[ones(1,1), ones(1,4), 2*ones(1,4)] for model mx2;

[2*ones(1,1), ones(1,4), 2*ones(1,4)] for model mx3;

[2*ones(1,1), 2*ones(1,4), 2*ones(1,4)] for model mx4;

[3*ones(1,1), ones(1,4), ones(1,4)] for model mx5; and

[3*ones(1,1), 3*ones(1,4), ones(1,4)] for model mx6.

The six estimated models are evaluated by comparing their simulated output and the output in the validation data, dv, as shown in Figure 6.6.

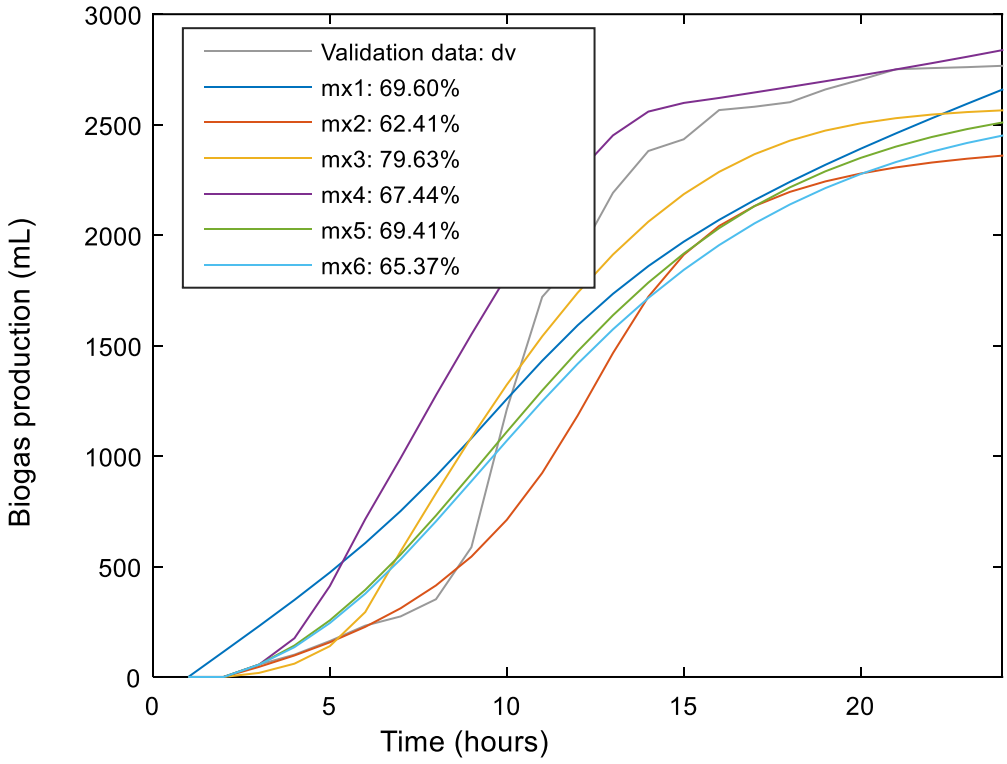


Fig. 6.6. Evaluation of estimated NARX models with different model moder for biogas production

As seen in Figure 6.6, the result of the evaluation shows that the best model is mx3 as the model has a higher Fit (79.63%) to the validation data than other estimated models. For the estimated model mx3, the number of units in 'wavelet' is automatically chosen by the estimation algorithm, which is 7. In order to explicitly specified the number of units in the 'wavelet', 7 is replaced with 6, 8 and 9, and are used to estimate models mx7, mx8, and mx9, respectively, with the same model order [2*ones(1,1), ones(1,4), 2*ones(1,4)] used to estimate model mx3. The models are evaluated with the validation data as shown in Figure 6.7.

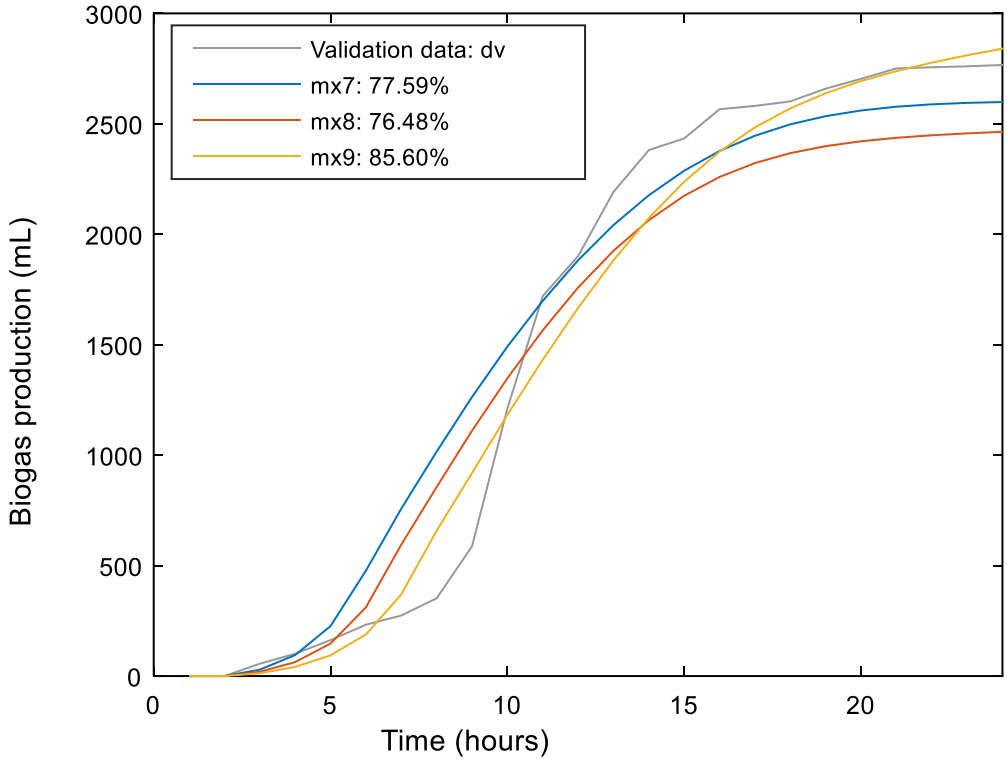


Fig. 6.7. Evaluation of estimated NARX models with different number of units in the 'wavelet' estimator for biogas production

The result of the evaluation as seen in Figure 6.7 shows that there is about 5% improvement of the previously estimated model mx3 when the number of units in the 'wavelet' is increased from 7 to 9. This indicates that model mx9 is a better model than models mx3, mx7 and mx8, and the best performing NARX model with 'wavelet' estimator for biogas production for this study.

Apart from 'wavelet' estimator two other nonlinearity estimators 'treepartition' and 'sigmoidnet' are used to estimate two new NARX models, mx10 and mx11, respectively. The models are estimated with the estimation data (de), model order [2*ones(1,1), ones(1,4), 2*ones(1,4)] and their respective nonlinearity estimator. The estimated NARX models mx9, mx10 and mx11 are evaluated with the validation data as presented in Figure 6.8.

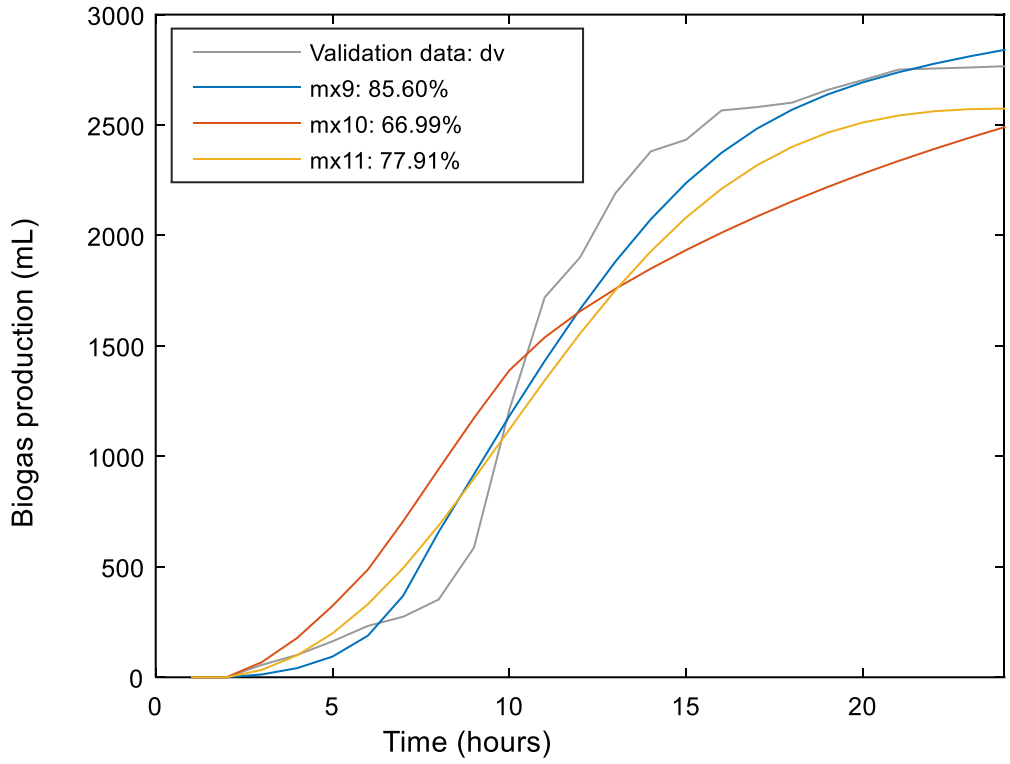


Fig. 6.8. Evaluation of NARX models with different nonlinearity estimators for biogas production

As seen in Figure 6.7, the evaluation shows that the model mx9 with 'wavelet' nonlinearity estimator performed better than the models mx10 and mx11 with 'treepartition' and 'sigmoidnet' nonlinearity estimators, respectively. Therefore, the estimated model mx9 is considered the best NARX model that describes the dynamics of mesophilic AD for biogas production for this research work.

Similarly, estimating the NARX model for methane production, involed all the steps followed to estimate the NARX model for biogas production. The data acquired from methane production, a constituent of biogas production, is split into two subsets, 17 data sets for the

estimation data (ze) and eight data sets for the validation data (zv). Six different NARX models are estimated with 'wavelet' nonlinearity estimators and the following model order:

[ones(1,1), ones(1,4), ones(1,4)] for model ms1;

[ones(1,1), ones(1,4), 2*ones(1,4)] for model ms2;

[2*ones(1,1), ones(1,4), 2*ones(1,4)] for model ms3;

[2*ones(1,1), 2*ones(1,4), 2*ones(1,4)] for model ms4;

[3*ones(1,1), ones(1,4), ones(1,4)] for model ms5; and

[3*ones(1,1), 3*ones(1,4), ones(1,4)] for model ms6.

The estimated models are evaluated by comparing their simulated output and the output in the validation data, zv, as shown in Figure 6.9.

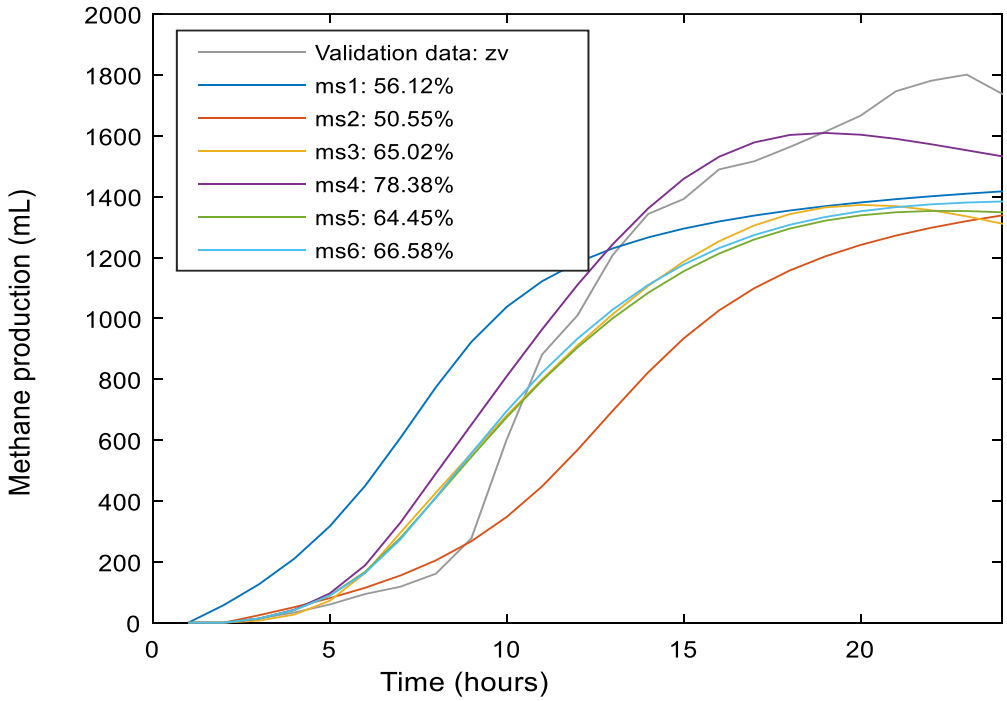


Fig. 6.9. Evaluation of NARX models with different model order for methane production

As seen in Figure 6.9, the result of the evaluation shows that the best model is ms4 as the model has a higher Fit (78.38%) to the validation data than other estimated models. The number of units in the 'wavelet' estimator is automatically chosen by the estimation algorithm as 10 for model ms4. In order to explicitly specified the number of units in 'wavelet', 10 is replaced with 9, 11 and 12, and are used to estimate models ms7, ms8, and ms9, respectively, with the same model order [2*ones(1,1), 2*ones(1,4), 2*ones(1,4)] used to estimate model ms4. The models are evaluated with the validation data as shown in Figure 6.10.

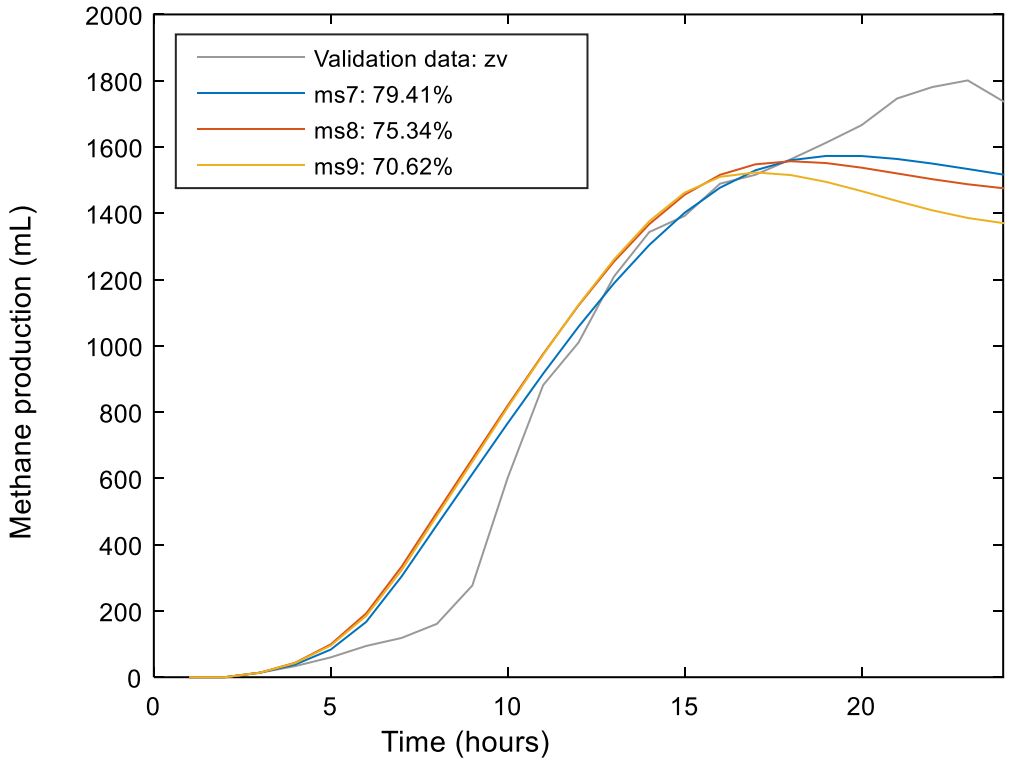


Fig. 6.10. Evaluation of estimated NARX models with different number of units in 'wavelet' estimator for methane production

The result of the evaluation as seen in Figure 6.10, shows that there is a marginal improvement of about 1% on the previously estimated model ms4 when the number of units in 'wavelet' is reduced to 9. This indicates that model ms7 is a better model than models ms4,

ms8 and ms9, and the best performing NARX model with ‘wavelet’ estimator for methane production for this study.

Likewise, apart from ‘wavelet’ estimator two other nonlinearity estimators ‘treepartition’ and ‘sigmoidnet’ are used to estimate two new NARX models, ms10 and ms11, respectively. The models are estimated with the estimation data (ze), model order [2*ones(1,1), 2*ones(1,4), 2*ones(1,4)] and their respective nonlinearity estimator. The estimated NARX models ms7, ms10 and ms11 are evaluated with the validation data as presented in Figure 6.11.

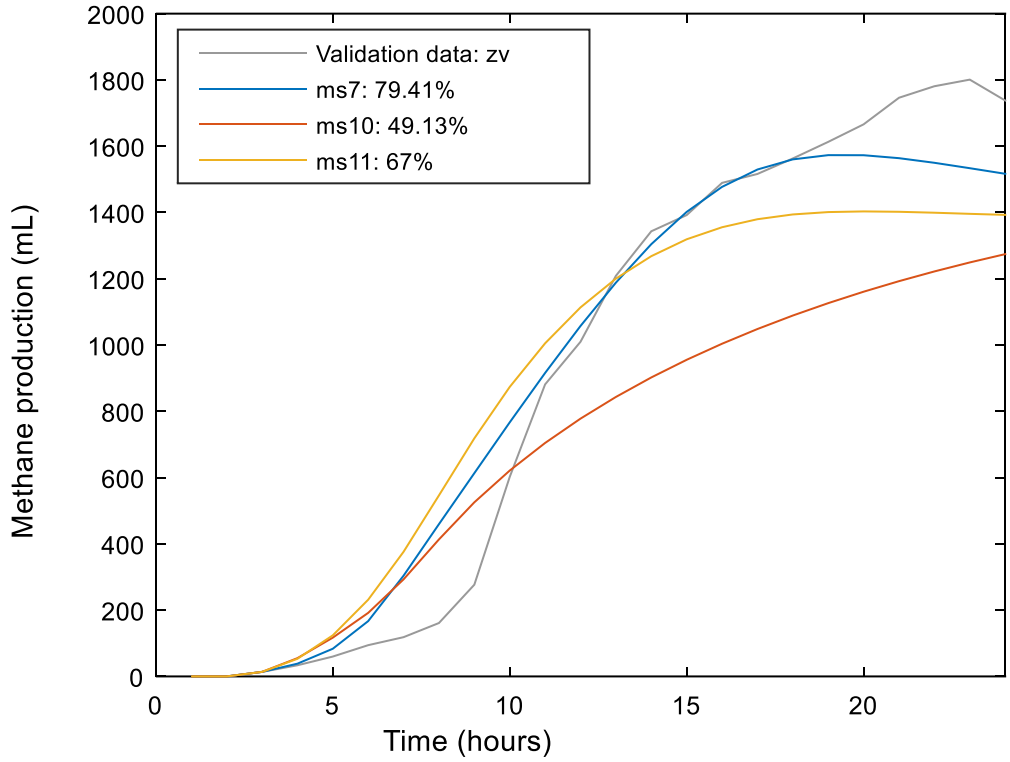


Fig. 6.11. Evaluation of NARX models with different nonlinearity estimators for methane production

The result of the evaluation as seen in Figure 6.11 shows that the model ms7 with ‘wavelet’ nonlinearity estimator performed better than the models ms10 and ms11 with ‘treepartition’

and ‘sigmoidnet’ nonlinearity estimators, respectively, when compared with the validation data. Therefore, the estimated model ms7 is considered the best NARX model that describes the dynamics of mesophilic AD for methane production for this research work.

6.2.4 Hammerstein-Wiener model estimator

The Nonlinear Hammerstein-Wiener (NLHW) model consists of three serial blocks, such that a dynamic linear block is placed in between the input and output static nonlinear blocks, as presented in Figure 6.12 (Abbasi-Asl et al., 2012).

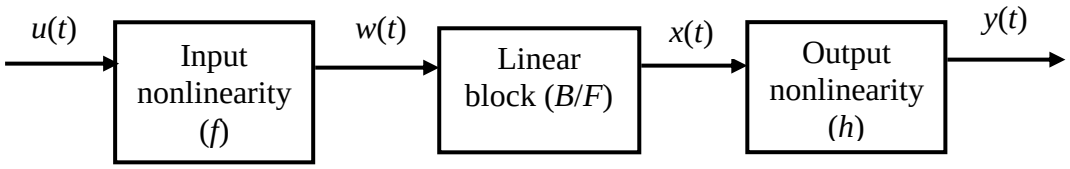


Fig. 6.12. Block diagram of Hammerstein-Wiener model (Abbasi-Asl et al., 2012)

The three components of Hammerstein-Wiener model are represented with the following three mathematical expressions (Abbasi-Asl et al., 2012).

For the static input nonlinear function,

$$w(t) = f\,(u(t)) \tag{6.4}$$

Where,

$w(t)$ is an input to the linear function B/F.

For the dynamic linear block,

$$x(t) = \frac{B}{F}\,w(t) \tag{6.5}$$

Where,

$x(t)$ represents a linear transfer function. The dimension of $x(t)$ is also the same as $y(t)$, while B and F have similar characteristics as polynomial in the linear OE model.

Considering the inputs n_u and outputs n_y, the dynamic linear block is a transfer function that has the matrix entries as (Abbasi-Asl et al., 2012):

$$\text{Transfer function} = \frac{B_{ji}(q)}{F_{ji}(q)} \tag{6.6}$$

Where,

$j = 1, 2, \ldots, n_y$ and $i = 1, 2, \ldots, n_u$, represent possible number of inputs and outputs, respectively

The output static nonlinear function is expressed mathematically as:

$$y(t) = h(x(t)) \tag{6.7}$$

The Eq. 6.7 is a nonlinear function that converts the output of linear block to the system output. Both $w(t)$ and $x(t)$ are internal variables that define the input and output of the linear block. The f function is known as the input nonlinearity because it acts on the linear block. While the h function signifies the output nonlinearity, because it acts on the output port of the linear block (Abbasi-Asl et al., 2012). However, in the absence of the input nonlinear block, the model becomes a Wiener model. Similarly, if the output nonlinear block is not present, then the model becomes a Hammerstein model (Choo et al., 2012).

Step 5b: NLHW model estimation for biogas and methane production

The NLHW models are estimated with the syntax (Ljung et al., 2007):

```
Mhw = nlhw(Data, Orders, InputNonlinearity, OutputNonlinearity);
```

Where Mhw is the NLHW model, model Order = [nb bf, nk], which specifies the orders and delay of the linear function, InputNonlinearity and OutputNonlinearity specify the nonlinearity estimators for two nonlinear blocks.

Similar to the NARX model estimation, the initial model order used for estimating the NLHW model mhw1 is [ones(1,4), ones(1,4), zeros(1,4)]. This means that in the linear block, the output is the sum of the four first order transfer function driven by the four inputs. The nonlinearity estimator is the pwlinear for both InputNonlinearity and OutputNonlinearity. The model mhw1 for biogas production is estimated using MATLAB command line. Two more NLHW models are estimated with the same InputNonlinearity and OutputNonlinearity, data de and the following model order:

[2*ones(1,1), 2*ones(1,4), 2*ones(1,4)] for model mhw2; and

[3*ones(1,1), ones(1,4), ones(1,4)] for model mmh3

The estimated models are evaluated by comparing their simulated outputs to the output in the validation data, dv, as shown in Figure 6.13.

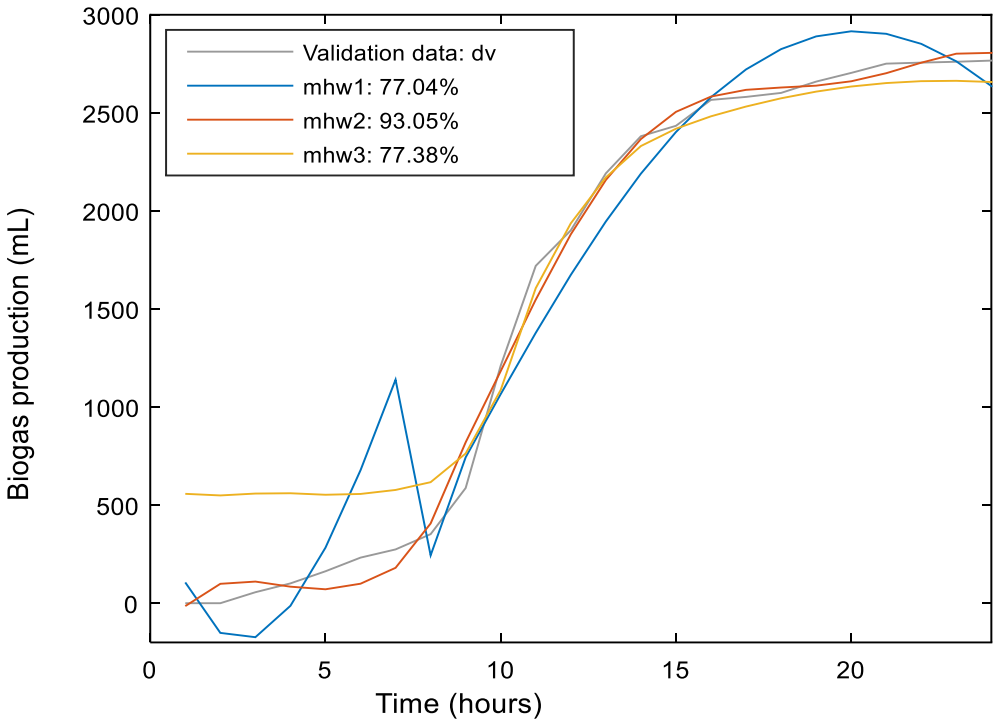


Fig. 6.13. Evaluation of NLHW models with pwlinear for both nonlinearities for biogas production

As seen in Figure 6.13, the result of the evaluation shows that the NLHW model mhw2, which has a Fit of 93.05% to the validation data, is considered a better model than models mhw1 and mhw3. This is because the Fit of mhw2 to the validation data is higher than that of mhw1 and mhw2.

Again, three other NLHW models, mhw4, mhw5 and mhw6, are estimated with the data de and model orders similar to the ones used to estimate models mhw1, mhw2 and mhw3 but with different nonlinearities for both InputNonlinearity and OutputNonlinearity - unitgain and saturation, respectively. The estimated models are evaluated by comparing their simulated outputs to the output in the validation data, dv, as shown in Figure 6.14.

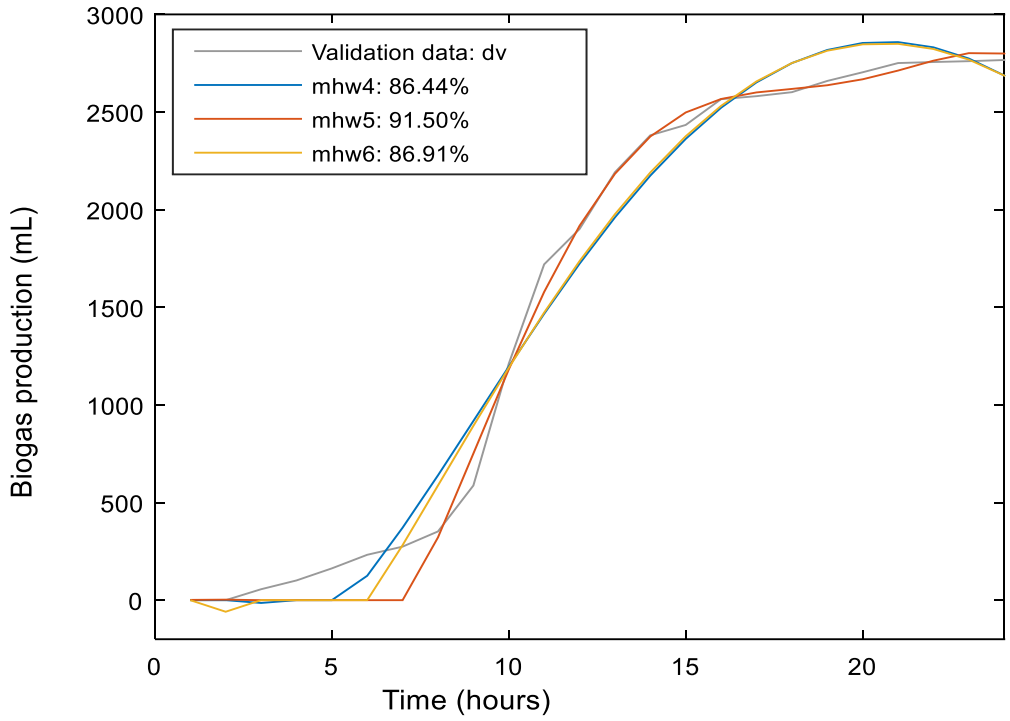


Fig.6.14 Evaluation of NLHW models with unitgain and saturation nonlinearities for biogas production

The result of the evaluation as seen in Figure 6.14 reveals that model mhw5 has a higher Fit to the validation data than mhw4 and mhw6, indicating a better NLHW model.

Furthermore, the InputNonlinearity and OutputNonlinearity are replaced with saturation and deadzone, respectively, and used to estimate models mhw7, mhw8 and mhw9. The estimated models are evaluated with the validation data dv as presented in Figure 6.15.

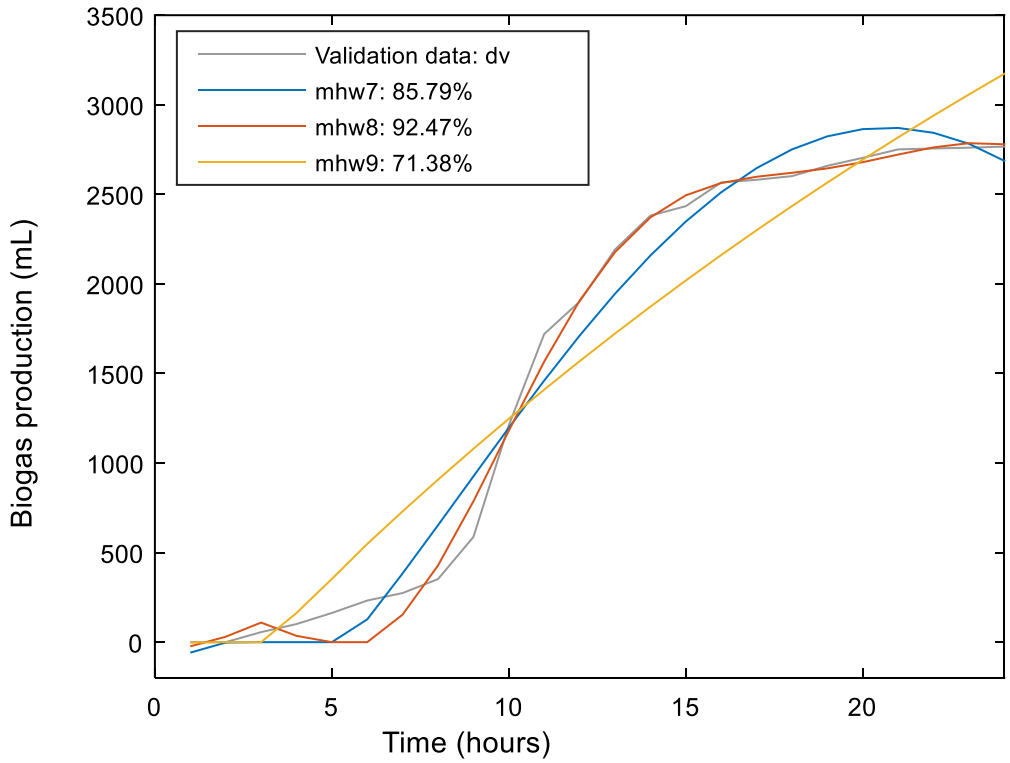


Fig. 6.15. Evaluation of NLHW model with saturation and deadzone nonlinearities for biogas production

The result of the evaluation as seen in Figure 6.15 reveals that model mhw8 has a higher Fit to the validation data than mhw7 and mhw9, which signifies a better NLHW model.

The selection of the best NLHW model for biogas production is performed by comparing the simulated output of the best model from each of the set of the nonlinearity combinations presented in Figure 13, Figure 14 and Figure 15 (models mhw2, mhw5 and mhw8) and the output in the validation data as plotted in Figure 6.16.

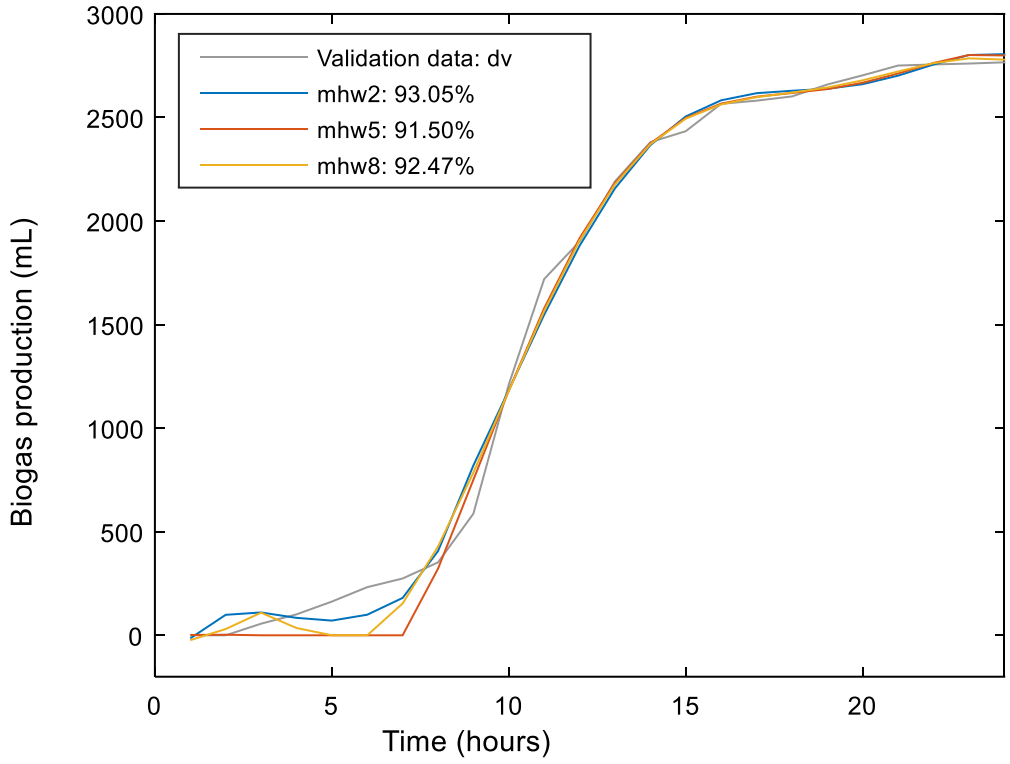


Fig. 6.16. Comparison of the best NLHW model for biogas broduction

The result of the analysis seen in Figure 6.16 shows that the three estimated NLHW models performed very well in comparison to the output in the validation data. In addition, the three models are estimated with identical model order, [2*ones(1,1), 2*ones(1,4), 2*ones(1,4)]. However, model mhw2 marginally describes the dynamic system better than the other two models, indicated by a slightly higher Fit to the validation data than models mhw5 and mhw8. Therefore, model mhw2 is the best NLHW model for biogas production for this study.

The estimation of NLHW model for methane production is performed using similar syntax used to estimate the best of the NLHW models for biogas production from each set of the nonlinearity combinations. Using the same model order, the three estimated NLHW models for

methane production are evaluated by comparing their simulated output and the output in the validation data zv as shown in Figure 6.17.

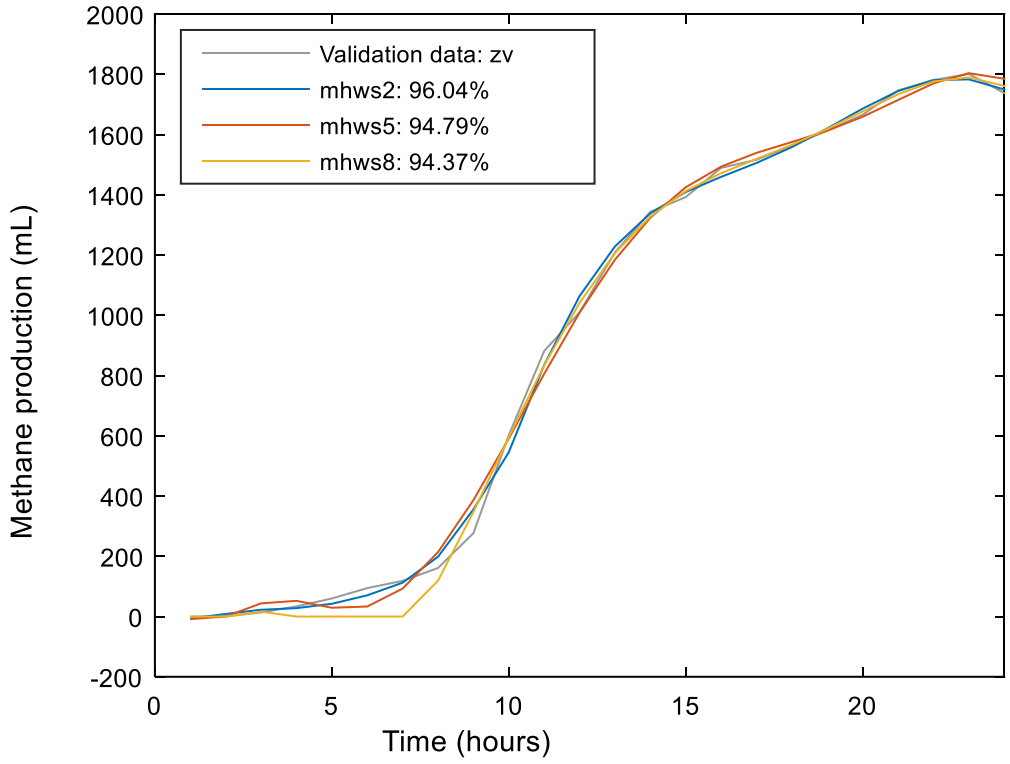


Fig. 6.17. Evaluation of estimated NLHW models with different nonlinearity combinentios for methane production

As seen in Figure 6.17, the evaluation indicates that model mhws2 is marginally better than models mhws5 and mhws8, because model mhws2 better fits the validation data (96.04% vs. 94.79% and 94.37%). Therefore, model mhws2 is the best performing NLHW model for methane production for this research work.

Step 6: Post-estimation analysis – model selection

The selection of the models for this study is carried out by evaluating the quality of the best estimated NARX and NLHW models for biogas and methane production, and choosing the one that best describes the mesophilic AD system behaviour within the acceptable bounds. This

research study utilises two different Goodness of Fit (GoF) approaches to measure the quality of NARX and NLHW models, in order to determine the best model each that describes well the production of biogas and methane. These approaches include the following:

- Comparing simulated output to measured output; and
- Comparing models using Akaike Information Criterion (AIC);

Comparing simulated output to measured output:

This approach is used already in this study to compare the performance of different models within the same model structure. The NARX and NLHW models for biogas and methane production are compared using the 'compare' command in MATLAB, in order to determine which model best fits the validation data.

For biogas production, the simulated outputs of the estimated NARX model (mx9) and NLHW model (mhw2) are compared to the output in the validation model (dv) as presented in Figure 6.18.

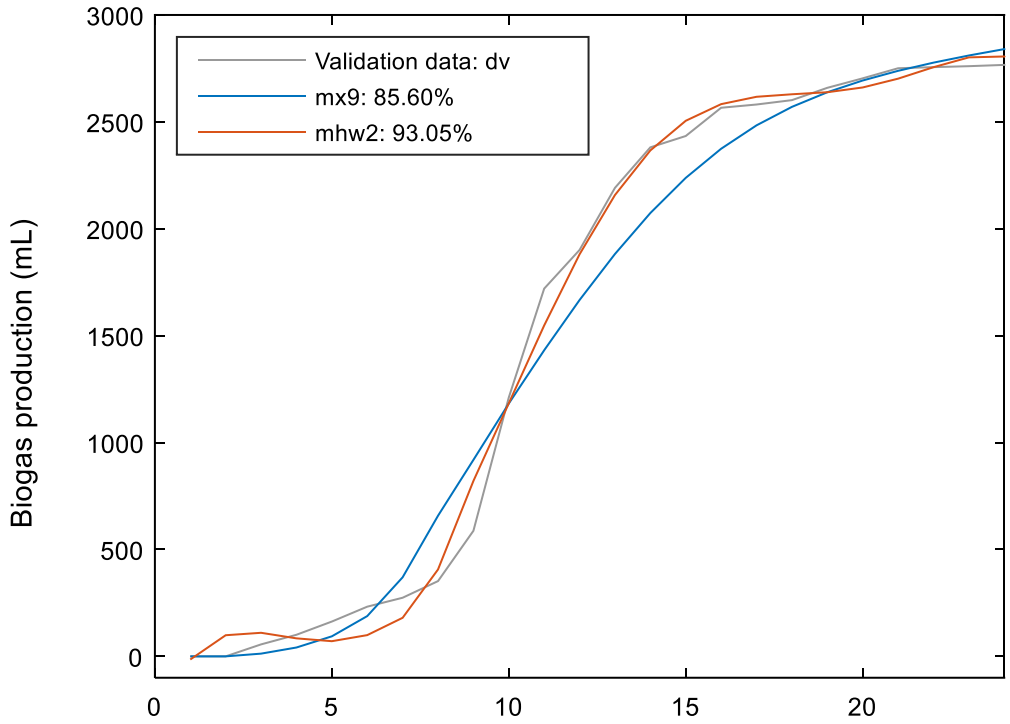


Fig. 6.18. Comparison of the estimated NARX and NLHW models for biogas production

As seen in Figure 6.18, the evaluation indicates that NLHW model mhw2 is better than NARX model mx9, because model mhw2 better fits the validation data (93.05% vs. 85.60%). Therefore, comparing the simulated outputs of the estimated models NARX and NLHW and the output in the validation data, model mhw2 is the best model that describes the system better for biogas production for this research work.

Similarly, the simulated outputs of estimated NARX model ms7 and NLHW model mhws2, and the output in the validation data are compared for methane production and the result is presented in Figure 6.19.

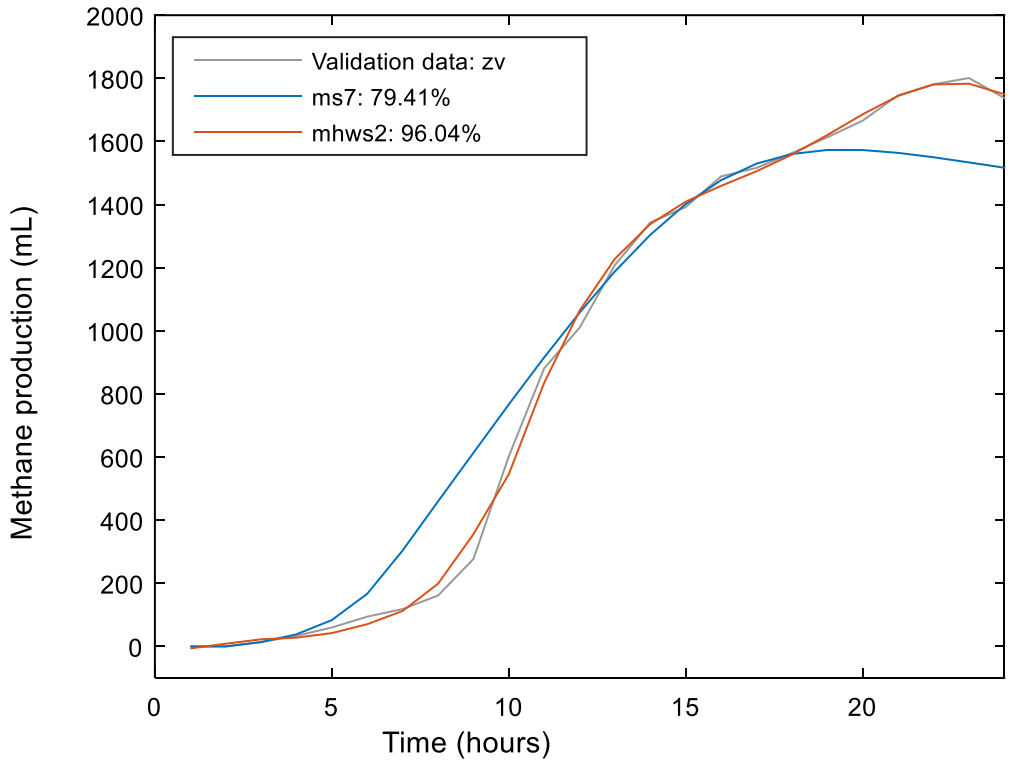


Fig. 6.19. Comparison of the estimated NARX and NLHW models for methane production

The result of how well the different models (NARX model ms7 and NLHW model mhws2) fits the validation data, as seen in Figure 6.19, indicates that NLHW model mhws2 is better than NARX model ms7 because model mhw2 better fits the validation data (96.04% vs. 79.41%). Once again, comparing the simulated outputs with the measured output approach, the NLHW model mhws2 is the best model for methane production for this study.

Comparing models using Akaike Information Criterion (AIC):

The second approach used to select the best models for this research study is the AIC, which is utilised to compare the quality of the best NARX model and the best NLHW model for biogas and methane production

The AIC provides a measure of model quality by simulating the situation where the model is tested on a different data set (Ljung, 2008). Akaike's theory states that the most accurate model is the one with the smallest AIC (Ljung, 2008, Ljung, 2015). The AIC is defined by the following equation (Ljung, 2008, Ljung, 2015):

$$AIC = \log V + \frac{2d}{N} \tag{6.8}$$

Where V is the loss function, d is the number of estimated parameters, and N is the number of values in the estimation data set.

The loss function V is defined by the following equation (Ljung, 2008):

$$V = \det\left(\frac{1}{N}\sum_1^N \varepsilon(t,\theta_N)(\varepsilon(t,\theta_N))^T\right) \tag{6.9}$$

where θ_N represents the estimated parameters.

For $d<<N$:

$$\text{AIC} = \log\left(V(1+\frac{2d}{N})\right) \tag{6.10}$$

Computing AIC in MATLAB, the aic command is used to compare the different models for biogas and methane production and the syntax is as follows (Ljund, 2008).

AIC = aic(m1, m2) for biogas and methane production.

For biogas production, the comparison of NARX model mx9 and NLHW model mhw2 using AIC approach indicates that NLHW model mhw2 is a more accurate model than NARX as it gives the smaller AIC (9.6273 vs. 13.8054).

Similarly, the execution of the aic command indicates that NLHW model mhws2 is a more accurate model than NARX ms7 as it gives the smaller AIC (8.0308 vs. 13.2119) for methane production.

The measure of the quality of the NARX and NLHW models for biogas and methane production using the two GoF approaches indicates that NLHW model mhw2 and NLHW model nhws2 are the best models for biogas production and methane production, respectively, for this study. Therefore, NLHW model mhw2 is chosen as the model for biogas production, and NLHW model mhws2 chosen for methane production.

The mathematical expression for NLHW models is as follows:

$$y(t) = [\frac{B(z)}{F(z)}]u(t) + \varepsilon(t) \tag{6.11}$$

Where, $y(t)$ is the output (biogas or methane production), $u(t)$ is the input (operating parameters) and $\varepsilon(t)$ is the noise signals

For biogas production, the domain-mathematical expression from the estimated nonlinear multi-parameter model, NLHW model mhw2, is as follows:

$$G_b(\mathrm{z}) = \frac{1 - 15.8\ \mathrm{z}\text{^}{-1}}{1 - 1.935\ \mathrm{z}\text{^}{-1} - 0.9871\ \mathrm{z}\text{^}{-2}} * \mathrm{Temp} +$$

$$\frac{0.8907 - \mathrm{z}\text{^}{-1}}{1 - 1.963\ \mathrm{z}\text{^}{-1} + 0.9837\ \mathrm{z}\text{^}{-2}} * \mathrm{pH} + \frac{-0.7488 + \mathrm{z}\text{^}{-1}}{1 - 1.811\ \mathrm{z}\text{^}{-1} + 0.8417\ \mathrm{z}\text{^}{-2}} * \mathrm{MS} +$$

$$\frac{-0.12 + \mathrm{z}\text{^}{-1}}{1 - 1.947\ \mathrm{z}\text{^}{-1} + 0.9988\ \mathrm{z}\text{^}{-2}} * \mathrm{Press} + \varepsilon(t) \tag{6.12}$$

Similarly, for methane production, the domain-mathematical expression from the estimated nonlinear multi-parameter model, NLHW model mhws2, is as follows:

$$G_m(\mathrm{z}) = \frac{1 - 0.7134\ \mathrm{z}\text{^}{-1}}{1 - 1.876\ \mathrm{z}\text{^}{-1} - 0.9137\ \mathrm{z}\text{^}{-2}} * \mathrm{Temp} +$$

$$\frac{-0.698 - z^{-1}}{1 - 1.913\, z^{-1} + 0.9218\, z^{-2}} * \text{pH} + \frac{1 - 0.9726 + z^{-1}}{1 - 1.993\, z^{-1} + z^{-2}} * \text{MS} +$$

$$\frac{1 - 0.2323 + z^{-1}}{1 - 1.908\, z^{-1} + 0.9307\, z^{-2}} * \text{Press} + \varepsilon(t) \quad (6.13)$$

Where, Temp (in ℃), pH, MS (in rpm) and Press (in the bar) are temperature, pH, mixing speed and pressure respectively, while $G_b(z)$ and $G_m(z)$ (in mL) represent biogas and methane production, respectively. The following are a range of the values of the operating parameters at which the Eq. 6.12 and Eq. 6.13 are based on. 34<Temp<42 ℃; 5.0<pH<8.0; 60<MS<2000 rpm; and 0.1<Press<0.7

Step 7: Model testing: simulation error analysis

Simulation error occurs as a result of inaccurate input data, inaccurate physical model and limited accuracy of the solutions of the governing equations (Kocak, 2012; Christie, 2005; Oberkamfa, 2002).

The input error is the error in the data utilised to specify the problem. It is the error from the experimental error, which is classified as random or systematic errors (Christie et al., 2005; Oberkamfa, 2002). Random and systematic errors are problems associated with measurements (Christie et al., 2005; Oberkamfa, 2002). The physical model error is due to the effects of the phenomena, which are inadequately represented in the simulation (Kocak, 2012; Christie et al., 2005). Solution error is the difference between the approximate solution of the equations obtained with the numerical algorithms used in the simulation and the exact mathematical solution of the governing equations for the model (Kocak, 2012; Christie et al., 2005; Oberkamfa, 2002).

It is assumed that the models constructed in this study are not affected by the measurement and physical errors. This is due to the care is taken in the experimental setup and acquisition of data, as well as following the principle and procedure for identifying nonlinear black-box models using System Identification method. However, there is the possibility of error that may exist in the constructed models.

To analyse the error, the Root Mean Square Error (RMSE) is used. The RMSE is a commonly used measure of the difference between the measured outputs of a system in consideration and the outputs predicted by a model of the system (Chai and Draxler, 2014). The individual differences are called residuals, and the RMSE is used to combine the errors into a single measure of productive power (Chai and Draxler, 2014).

The RMSE of a model prediction with respect to the estimated output is the square root of the mean squared error, which is computed as follows (Chai and Draxler, 2014).

$$RMSE = \sqrt{\frac{\sum_{i=1}^{n} (\hat{y}_i - y_i)^2}{n}} \text{ (mL)} \tag{6.14}$$

Where, n is samples of model errors, y is measured output values and $\hat{y}$ is simulated output values

The percentage error of the system for biogas and methane production is calculated by dividing the RMSE by the measured output values, as follows.

$$\%Error = \frac{RMSE}{\sum_{i=1}^{n}(y)} \text{ x } 100 \tag{6.15}$$

The Simulink model as shown in Figure 6.20a, which represents Eq. 6.15 and Eq. 6.16, is used to simulate some intervals of a range of variables of two of the operating parameters at which

the models are based on. The internal part of error estimator in Figure 6.16 is shown in Figure 6.21. The variables of temperature and pH which are used experimentally to generate the measured output values (biogas or methane), are also utilised to simulate the models for biogas and methane production, in order to compute the RMSE and the percentage error between the simulated output values of the models mhw2 and mhws2, and the measured output values of biogas and methane production.

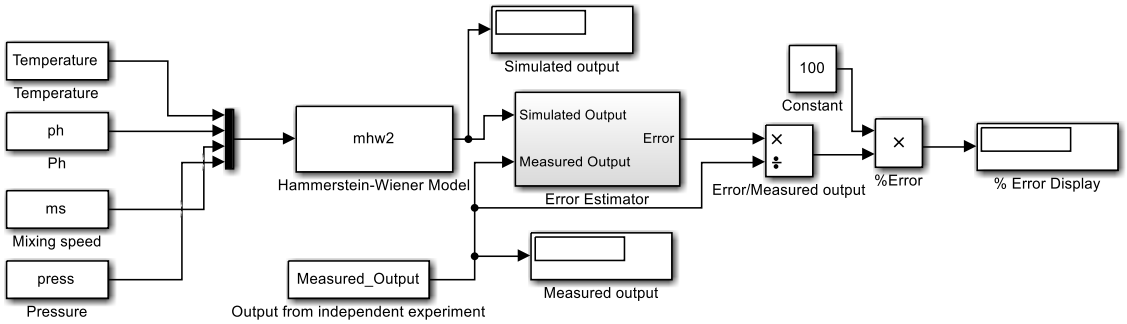


Fig. 6.20a. Simulation of percentage error from models for biogas and methane production

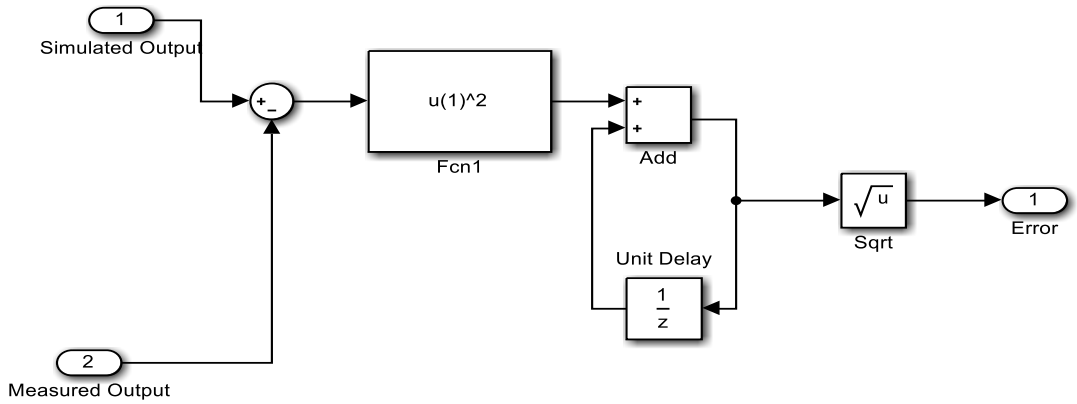


Fig. 6.20b Error estimator

The following Tables show the result of the Simulink model, containing simulated output, measured output and percentage error for biogas and methane production.

Table 6.1a. Test for biogas production model mhw2 for variables of temperature

Temperature (℃)	39	43
Simulated output (mL)	2809	2310
Measured outpit (mL)	2752	2460
Percentage error (%)	2.1	-6.5

Table 6.1b. Test for methane production model mhws2 for variables of temperature

Temperature (℃)	39	43
Simulated output (mL)	1830	1522
Measured outpit (mL)	1875	1553
Percentage error (%)	-2.4	-2.0

Table 6.2a. Test result for biogas production model mhw2 for variables of pH

pH	7.3	8.3
Simulated output (mL)	2620	2240
Measured outpit (mL)	2570	2149
Percentage error (%)	1.9	4.2

Table 6.2b. Test result for methane production model mhws2 for variables of pH

pH	7.3	8.3
Simulated output (mL)	1691	854
Measured outpit (mL)	1734	827
Percentage error (%)	-2.5	3.3

The analysis of simulation error as seen in Tables 6.1a – 6.2b, compares the simulated and measured output for biogas and methane production. It shows that the percentage error for biogas and methane production are less than 7% and 4%, respectively, signifying a small simulation error. The negative percentage error in some of the values of the tests indicates that the measured output is higher than the simulated output at those variables and vice verse.

6.2.5 Comparison between the biogas and the methane production models

The biogas and methane production models are constructed using the same nonlinear black-box model structure, NLHW models. The similarities of biogas production and methane production model support the response of biogas and methane production as seen in the plots in Chapter 5. This goes to strengthen the claim that methanogenesis is the limiting stage in AD process. Meaning that the behaviour of methanogenic bacteria determines the performance of AD process with regards to biogas production.

Although the NLHW models produced a high fit to the respective estimation data for biogas and methane production, there is the possibility of the following weaknesses in these models.

1. It is uncertain that these models can fit well data from the four input parameters that are significantly higher or lower than the data used in this study.
2. If the production of biogas and methane are considerably influenced by other operating parameters outside temperature, pH, mixing speed and pressure, it is unlikely that these models will give a good fit to the data, or able to predict the behaviour of mesophilic AD system well.

6.3 Bioprocess Scale-up

There are several ways of developing and implementing biological processes, which are operated at different scales, such as laboratory, pilot and large scales. Many laboratory-scale processes that are used for research work are developed with the goal of screening and developing better bacterial strains, improving existing culture and performing the process more effectively with enhanced productivity (Najafpour, 2006). Usually, the production design of large-scale bioprocesses is developed in the laboratory, where all the factors that influence the intended output production are evaluated through experiments (Najafpour, 2006). Many factors, such as physical and biochemical, influence the production of biogas and methane as discussed in Chapter 2. The results from the laboratory process are transferred to the pilot-scale for verification by a relatively simple scale-up, after which they are transferred to large-scale (Galindo and Ramirez, 2013; Najafpour, 2006).

In other to scale-up a bioprocess, a useful technique called dimensional analysis (DA) is applied (Galindo and Ramirez, 2013; Moreira and Wallece, 2012; Najafpour, 2006). DA is the analysis of the relationship between various physical quantities by identifying their basic dimensions and units, as well as tracking these dimensions as calculations or comparisons (Gibbings, 2011). In DA, only quantities with similar dimensions are subtracted, added or compared (Barnett, 2007). Applying DA to scale-up requires certain rules and criteria to be followed. These criteria include maintaining geometric similarities and the keeping dimensionless numbers (DNs) constant (Galindo and Ramirez, 2013; Moreira and Wallece, 2012; Najafpour, 2006).

One way of obtaining DNs is from a structured grouping of all the parameters and variables that influence bioprocess outcome (Galindo and Ramirez, 2013). However, this method is

prone to errors as the omission of one important variable can differ the outcome (Galindo and Ramirez, 2013). The other way to achieve the DNs is to work with the ratios of time constants, forces and velocities (Galindo and Ramirez, 2013). This method is less prone to errors and often used (Galindo and Ramirez, 2013). The DNs from the ratios include Reynolds number or momentum factors, power consumption per unit volume of liquid, impeller tip velocity, liquid mixing and recirculation time and volumetric of mass transfer coefficient, which are discussed in detail in the books written by Simpson and Sastry (2013), Moreira and Wallece (2012) and Najafpour (2006).

According to Najafpour (2006), the geometric similarities and constant power per unit volume have been applied in the scale-up of most fermentation processes for alcohol and organic acid production, which are some of the products of acidogenesis, the second stage of methane production in AD process. However, there are some limitations associated with applying DA for scale-up of bioprocess (Moreira and Wallece, 2012; Najafpour, 2006). It is practically impossible to keep all the DNs constant during scale-up (Moreira and Wallece, 2012; Najafpour, 2006). For instance, mixing and mixing time is a major concern in applying DA for scale-up, because large-scale digesters may not have uniform mixing, whilst mixing is not a problem in lab-scale and pilot-scale digesters (Najafpour, 2006).

In applying DA for scale-up, the operating parameters are well controlled and certain rules have to be followed in changing the digester size to meet special criteria (Najafpour, 2006). In addition, maintaining only digester similarities and keeping some DNs constant may not satisfy the requirements of bioprocess design (Moreira and Wallece, 2012; Najafpour, 2006). The basic concept of DA, which is the geometric similarity, is usually maintained in scale-up from

laboratory to pilot scale. However, in the large-scale, it presents a real design challenge because the configurations of the digester are often changed (Moreira and Wallece, 2012).

6.3.1 Scale-up of Black-box Model

The black-box approach is based on laboratory-scale or pilot-scale investigations, which focuses mainly on the manipulated and measured parameters (Heams, 2006). These parameters, such as physical, chemical and biochemical phenomena, can form the major factors of scale-up (Steel, 2007; Heams, 2006). The advantage of black-box model approach is that it allows for faster development of the determined configuration, by considering the target as several small black-box systems (Heams, 2006).

However, in opposition to nonlinear black-box model approach to scale-up, Heams (2006) reports that this approach offers less assurance as there are many physical, chemical, biochemical and microbiological phenomena that are not well explained (Heams, 2006). Meaning that the rules to maintaining the geometric similarities as well as keeping the DNs constant are not followed in nonlinear black-box model scale-up. Additionally, some authors reported that nonlinear black-box approach limits applications to the experimental conditions that generated the model, thereby, allows only the interpolation of the results and consequently, difficult to use for scale-up (Steel, 2007; Cybulski et al., 2001; Wentzel and Ekama, 1997).

The nonlinear black-box models constructed in this study focuses only on four manipulated parameters; temperature, pH, mixing speed and pressure. Although the geometric similarities required to apply DA to scale-up can be maintained, it is not possible to account for all the physical and biochemical parameters involved in a bacterial growth environment that are not considered in this study. It means that the nonlinear black-box models constructed in this study

lack the basis to apply DA to scale-up. Since the models are not able to satisfy the rules and criteria for DA, they cannot be used for the scale-up purpose.

One of the ways it can be possible to apply nonlinear black-box model to scale-up is to make assumptions about all the manipulated physical and biochemical operating parameters not considered in this study. Then the nonlinear black-box models can satisfy the criteria to apply DA to scale-up, however, the model would not be considered a black-box, rather, a grey-box model. Another way is if the digester geometric increase produces a corresponding linear output, then the target can be considered as a multiple of laboratory linear black-box model. Further study is encouraged in this area to explore the possibility of nonlinear black-box model scale-up in mesophilic AD process.

6.4 Summary,

This chapter has described how the nonlinear multi-parameter black-box models for biogas and methane production are constructed. The process includes the selection of black-box model technique due to inadequate knowledge of AD process. The estimation and validation of the experimental data are performed in MATLAB, through System Identification toolbox. The identified NLHW models mhw2 and mhws2 represent the behaviour of the production of biogas and methane, respectively, from mesophilic AD. Of all the candidate models studied, the nonlinear models provide a superior reproduction of the experimental data over the whole analysed period. Other appealing features of these HLHW models reside in the simplicity of the nonlinearities considered and the possibility to include new nonlinearities, as well as in its easy implementation. The models are prone to possible weaknesses and cannot be applied for scale-up purpose due to the inability of black-box models to satisfy the dimensional analysis criteria.

Chapter 7 **Conclusions**

7.1 Introduction

The study is set out to develop a black-box model for mesophilic AD process, in order to understand better, how physico-environmental operating parameters influence biogas and methane production. The research has also sought out to consider the control limits for the controlled parameters necessary for improving methanogenesis reaction while ensuring an enhanced digestion operation. The review of the relevant academic articles showed that much progress has been made in AD technology. However, much work is still needed to improve the knowledge of AD, specifically, the interactions between the operating parameters and bacterial community. In addition, the composition of bacterial groups that varies with changes in the operating parameters.

This research work sought to provide solution to the following objectives:

- Identify and understand the performance variables of mesophilic AD system (Chapter 2);
- A critical review of the operating parameters that influence the performance of biogas and methane production was undertaken. For this research work the following four operating parameters are considered; temperature, pH, mixing speed and pressure (Chapter 2);
- Determine the limits for the four operating parameters and their impact on biogas and methane production. This includes a series of laboratory experiments with each of the operating parameters. The data obtained from the investigations are analysed and the range of values for each operating parameter that recorded the highest performance in terms of biogas and methane production (Chapters 4 & 5);

- Construct a black-box model each for biogas and methane production. The limits of the four operating parameters considered are analysed further in order to establish their correlation with biogas and methane production. Two nonlinear multi-parameter black-box models are constructed through System Identification method in MATLAB. The models are able to predict the behaviour of the real system with sufficient accuracy (Chapter 6); and
- Determine the scalability of black-box models. The black-box models constructed in this study focused only on four manipulated parameters. Although the models can maintain the geometric similarities required to apply DA to scale-up, it cannot account for all the other physical and biochemical parameters involved in the bacterial growth environment that are not considered in this study, making it difficult to use the models for scale-up (Chapter 6).

7.2 Observed Outcomes

The main observed outcomes with respect to the individual objectives of the study are chapter specific. This section unifies the observed outcomes to provide a solution to the aforementioned objectives of this study.

- Identify and understand the performance variables of mesophilic AD system through a critical review of the operating parameters that influenced the process performance:

i. **Influence of the operating parameters on AD performance:** Various operating parameters influence the performance of AD system. Different operating parameters have a different degree of impact on biogas and methane production. The temperature is considered the most significant parameter in AD process as it influences the entire biochemical process. Anaerobic bacteria are grouped based on their temperature

preference and tolerance. The different types of AD are characterised by their bacterial group, which are based on the temperature favourite of the bacterial group. These include psychrophilic, mesophilic and thermophilic AD, which operates at 0 – 20 ^{o}C, 10 – 45 ^{o}C and 45 – 65 ^{o}C, respectively. The mesophilic AD process, which is the type of AD process considered in this study, performs optimally at a temperature range of 38 – 40 ^{o}C (Chapter 3).

Similarly, pH is an important parameter in AD, however, its variability does not impact on the entire anaerobic reactions. This is because different bacterial groups of the multi-step process of AD have variable pH preferences, irrespective of the type of AD implemented. In Chapter 5 of this thesis, it is found that there is no record of methane production at pH of 5.0. This is because the activity of methane forming bacteria is inhibited in acid condition. However, acidogenesis thrive in acidic condition up to a low pH of 4.0 (Chapter 5).

Furthermore, some of the operating parameters, such as mixing/mixing speed, hydraulic retention time, pressure and substrate characteristics; do not actually halt biogas and methane production as in the case of temperature and pH. However, they influence the enhancement of biogas and methane production (Chapter 5).

ii. **Anaerobic digestion process improvement:** Various enhancements have been achieved in AD technology in other to improve biogas and methane production. These include pre-treatment, co-digestion and system modelling.

iii. **Co-digestion of two or more substrates -** The simultaneous digestion of two or more different substrates improves digestion process as investigated in this research work. In co-digestion, the participating substrates supply the required nutrients, which improves the performance of AD. It is found that improving the nutrient balance resulted in improved biodegradation, process stability and biogas production. The experiments reported in Chapter 5 show that better biodegradation and conversion of substrates to biogas is achieved by co-digestion of wastewater sludge and food waste compared to the digestion of only wastewater sludge or food waste. The study also found that co-digestion of substrates produced more methane than the single-substrate digestion, thus, improving the quality of biogas. This indicates that co-digestion offers better process performance than single-substrate digestion (Chapter 5).

iv. **System Modelling -** A number of previous studies (Chapter 2) reviewed show that modelling is an important tool for a complex system like AD process. It is used to describe and predict the behaviour and output of systems in real life. Mathematical modelling is generally classified into white-box models and black-box models. However, when there is the availability of some information about the system under consideration but not enough to apply the white-box models, then the known information is combined with the input/output data generated from physical experimentation, which is known as a grey-box model. In other words, grey-box model is the combination of the white-box and black-box models (Chapter 3). The black-box models are selected (Chapter 6). Moreover, some of the previous models constructed for predicting biogas production are reviewed and the outcome contributed to shaping the basis for developing the models for this study (Chapter 3).

- Determining the limits for some key parameters that influence mesophilic AD:

  Several factors, which are categorised into the physical, biological and chemical need to be considered for achieving effective mesophilic AD. This study considers the bacteriological and biochemical processes involved in AD (Chapter 4). In addition, it investigated the impacts of temperature, pH, mixing speed and pressure on AD under mesophilic conditions. The goal is to measure and compare the biogas and methane production for all the variables of temperature, pH, mixing speed and pressure, in order to determine the variables that achieved the highest biogas and methane production (Chapter 4).

i. **Bacteriological and biochemical processes involve in anaerobic digestion:** The process of AD involves complex interactions of diverse species of bacteria organised in four different steps that function almost simultaneously. The four steps include hydrolysis, acidogenesis, acetogenesis and methanogenesis. In hydrolysis, complex organic polymers such as carbohydrates, fats and proteins are broken down to simple sugars, lipids and amino acids. The acidogenic bacteria convert the products of hydrolysis into hydrogen, carbon dioxide, alcohols and VFAs. At this stage, acetic acid contained in the VFAs are converted directly to methane by methanogenic bacteria. The remaining products of acidogenesis are transferred to the acetogenic stage, where they are converted to mainly acetic acid and finally, the methanogenic bacteria utilise the acetic acid as a substrate to produce methane.

   Methanogenesis is considered the rate-limiting stage of the AD process, thereby determining the stability of the entire process. Methanogenic bacteria are very sensitive to changes in operating parameters. Since they limit the kinetics of the entire anaerobic

biochemical process, the operating parameters of the whole process need to be maintained at the optimal range of methanogenesis (Chapter 4).

ii. **Investigation of the influence of multiple operating parameters:**

Temperature: The influence of mesophilic digestion of glucose for all the following variables of temperature, 32, 34, 36, 38, 40 and 42 ^{o}C on biogas and methane production is studied. Each of the experiments was kept at a constant temperature for the whole duration of 24 hours. The results show that the digester set at 32 ^{o}C for the entire duration generated the lowest biogas and methane production. Whereas the highest biogas and methane production are from the digester maintained at 40 ^{o}C, followed by the 38 and 36 ^{o}C digesters. The production of biogas and methane are found to decline as the digester temperature marginally exceeds 40 ^{o}C, indicating that the 42 ^{o}C temperature is beyond the temperature that supports the growth of mesophilic anaerobic bacteria.

From this study, the optimum temperature range is between 38 – 40 ^{o}C. This means that in order to achieve a constant improved biogas production in mesophilic AD, digester temperature needs to be maintained within the optimal temperature range for the entire duration of the process (Chapter 4).

pH: The influence of pH variation on the biogas and methane production was also studied. Different pH value is maintained for each digester throughout the retention time of 24 hours. The pH values investigated include 5.0, 6.0, 7.0 and 8.0. It is found that the pH of 5.0 inhibited the methanogenic process, thereby no methane is produced. However, the digester that was operated at pH of 7.0 generated the highest biogas and

methane production. The result also shows that as the pH moves below or beyond 7.0, the biogas and methane production decline. Indicating that as closer the pH value is to 7.0, the neutral value, the better the performance of the
methanogenic process. Thus, for continuously improved biogas and methane production, the pH needs to be kept close to the neutral value (Chapter 4).

Mixing speed: Mixing is important for optimal AD performance; however, excessive mixing can cause a number of problems such as lowering the surface tension of solution in the digester, and accumulation of solids over liquids within the digester. Results from the experimentation of the influence of mixing speed variation on biogas production show that digesters that operated at minimal or moderate mixing speed performed better than the digester with vigorous mixing. Among all the four mixing speeds experimented, the 60, 300 and 600 rpm are found to produce more biogas and methane than the 2000 rpm digester. In addition, the digester that operated at 2000 rpm mixing speed delayed biogas and methane production at the initial stage of the process due to the excessiveness of the mixing. The vigorous mixing does not only impact negatively on the performance of AD, but it also increases the cost of energy used by the mixing system, thereby increasing the overall operating cost of the system and making the AD technology less attractive (Chapter 4).

Pressure: The results of the experiment carried out to evaluate the influence of digester operating pressure on biogas and methane production shows that the 0.7 bar digester produced less biogas than the 0.1 bar digester. However, the methane production by the 0.7 bar digester is more than the 0.1 bar digester. This is because more carbon dioxide content in biogas from 0.7 bar digester is liquefied compared to biogas produced by

0.1 bar digester. It is therefore recommended that appropriate digester operating pressure need to be chosen, but not exceeding 100 bar, which is the maximum operating pressure that methanogenic bacteria can tolerate (Chapter 4).

- Constructing multi-parameter models for biogas and methane production:

  This study has presented two nonlinear multiparameter models for mesophilic AD for predicting the biogas production and the methane production. The modelling utilised raw data generated from lab experiments of the influence of four operating parameters, temperature, pH, mixing speed and pressure on the biogas and methane production. Due to the nonlinear characteristics of the acquired data, the nonlinear black-box modelling technique is used to construct the models.

  The black-box modelling offers some advantages over the white-box modelling technique; it requires no in-debt knowledge of the system under consideration, as well as adequately predict the biogas and methane production from input-output data. In addition, black-box modelling considers all the stages of the anaerobic process, operating parameters and history of operational data unlike the white-box modelling (Chapter 6).

  Two nonlinear model structures, autoregressive with exogenous input (NARX) and Hammerstein-Wiener (NLHW) with different nonlinearity estimators and model orders are chosen by trial and error and utilised to estimate the models. The performance of the models is determined by comparing the simulated outputs of the estimated models and the output in the validation data. The approach is used to validate the estimated models by checking how well the simulated output of the models fits the measured

output. The best models for biogas and methane production are chosen by comparing the outputs of the best NARX and NLHW models (each for biogas and methane production), and the validation data, as well as utilising the Akaike information criterion. The NLHW models mhw2 and mhws2 are chosen for biogas and methane production, respectively. The identified NLHW models mhw2 and mhws2 represent the behaviour of the production of biogas and methane, respectively, from mesophilic AD. Of all the candidate models studied, the nonlinear models provide a superior reproduction of the experimental data over the whole analysed period. (Chapter 6).

The favourable outcomes indicate that black-box simple models can estimate biogas and methane production, as well as contributed to improving the knowledge of mesophilic AD process. The constructed models can replicate the mesophilic AD process and forecast its response for a given multiple operating parameters used in the study with sufficient accuracy. Although the models may not have been the perfect representation of the behaviour of mesophilic AD process with regards to predicting the biogas and methane production. However, this study has opened up the opportunity for further research on modelling mesophilic AD process with multiple parameters. It is then inferred that this research work on the construction of mesophilic AD models for predicting biogas and methane production is successful.

- Determining the scalability of black-box model.

  This study considered only four operating parameters in the construction of the black-box models. Although geometric similarities required to apply DA (Dimensional Analysis) to scale-up can be achieved, but it is not possible to account for all the other

physical and biochemical operating parameters involved in a bacterial growth environment that are not considered in this study.

In addition, with the nonlinear black-box models, it is practically impossible to keep all the DNs (Dimensionless Numbers) constant, meaning that the models constructed in this study lack the basis to apply DA to scale-up. Since the models are not able to satisfy the rules and criteria for DA, they cannot be used for the scale-up purpose (Chapter 6).

7.3 Limitation of this study

The study has presented an empirical contribution to improving mesophilic AD process, which is achieved through experimentation and applying System Identification Toolbox in MATLAB to construct nonlinear black-box models. This research work encountered possible limitations that need to be considered. The limitations are categorised in terms of methodological and longitudinal effects that influenced the experimentation and interpretation of the study.

1. Methodological limitations:

- **Sample size** - The design of the experiment is such that only one operating parameter is manipulated at a time while the other three operating parameters are maintained at the same value throughout the investigation for all the variables of the manipulated operating parameter. This methodology is repeated for all the variables of temperature, pH, mixing speed and pressure. As a result, the number of sample units used for this study is reduced. The study is not able to capture the response of biogas and methane production if the design of the experiment is different. Because of the reduced sample size, it is difficult to find a comprehensive relationship between operating parameters and biogas as well as methane production.

- **Lack of available and reliable data** – The scope of analysis for this study is limited due to the insufficient evaluation of produced biogas sample to identify the entire gas composition. The reason for this is due to the gas analysed used in this study not equipped enough to identify and measure the proportion of other gases like carbon dioxide, hydrogen and ammonia present in biogas sample. Not only did it limit the scope of analysis conducted in this research work, but also significantly hindered the study of the relationship between operating parameters and other gases contained in biogas sample.

  In addition, there is the possibility of lack of data reliability, which is likely to be as a result of biogas sample contamination, resulting from transporting biogas sample from the laboratory in the University of Hertfordshire, where it is produced, to the laboratory in Imperial College London, where it is analysed. Another reason that could be responsible for data unreliability, in this research work, is the possible laboratory incompatibility that may alter the environmental conditions of the biogas samples.

- **Mode of data collection** – The sample interval for the experiments performed in this research work could also contribute to the possible limitations in this study. Reducing sample interval from 60 minutes (used in this study) to 60 seconds, would increase the sample size and reduce any possible random error effect. Also, the manual acquisition of data, due to the unavailability of computerised data acquisition system, may likewise affect the accuracy of data.

2. Researcher limitations:

- **Longitudinal effects** - The availability of time to conduct a thorough study is constrained by the due date of this research work. It did not only limit the scope of the study but also reduced the number of experiments conducted (sample size), which could have provided more information about mesophilic AD, thereby, improving the accuracy of the outcome of this study.
- **Insufficient equipment**: The lack of sufficient equipment is one of the major factors that limited this study. It adversely affected almost every stage of this research work, from experimental setup to sample size, data collection and data analysis.

7.4 Recommendations for Further Work

In other to construct models that can reflect the behaviour of mesophilic AD with improved predictive accuracy, additional studies are required, which include the following.

a) A further study is needed with the design of experimental setup in such a way that a variable of the operating parameters is investigated at a time with all the possible combinations of all the variables of other operating parameters. This methodology would require many experiments to be conducted. The purpose is to increase the sample size, as well as generate the best combination of the variables of the operating parameters at which the optimal production of biogas and methane is attained. In addition, it will increase the amount of information that can be used to improve the accuracy and robustness of the models.

b) Further investigation consisting of more operating parameters that influence biogas and methane production is encouraged. The models developed in this study are limited in their ability to predict the behaviour of mesophilic AD due to the influence of other operating parameters that are not considered in this study. Thus, a study that

incorporates more operating parameters would produce a model that is more dynamic and more robust, with improved predictive accuracy.

c) Additional study is also required to determine the entire gas content of biogas, as well evaluate how the individual gases respond to different combinations of the variables of the operating parameters. The study can be undertaken in conjunction with the previous recommended further studies or as a separate study utilising already acquired data. The proposed study is set out to find what combination of the variables of the operating parameters encourages the production of any of the gases that constitute biogas. The findings of the study can then be utilised to construct a model that improves methane production.

d) Further study is required to explore the possibility of black-box model scale-up, as it relates to mesophilic AD process.

This study has developed two pilot nonlinear multi-parameter black-box models for mesophilic AD process, which are based on the input-output data. This has demonstrated the need for developing multi-parameter models that reflect the behaviour of mesophilic AD process with sufficient accuracy. With adequate models in place, it is possible to evaluate the actual system, in order to reduce process disturbance that reduces or inhibits process efficiency.

Based on the models constructed in this research work, further studies are possible with the goal of improving biogas and methane production. The enhancement of methane production through mesophilic AD process is an appropriate answer to the global quest for alternative

energy source that is sustainable, as well as a satisfactory solution for environmental and health risks associated with fossils fuels.

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Appendices

Appendix A: Raw Experimental Data

The following tables are the experimental data generated from the various experiments conducted in this research work:

Average experimental data for temperature variables for biogas production:

Time (h)	32 (ºC)	34 (ºC)	36 (ºC)	38 (ºC)	40 (ºC)	42 (ºC)
	Average biogas production (mL)					
1	0	0	0	0	0	0
2	0	0	10.2	56	75.6	3.999
3	0	40.6	75.6	101	200.4	40.6
4	0	80.8	164.2	162.8	270.6	93.8
5	0	100	220.6	232.4	350.8	123
6	0	137.2	270.6	274.2	490.8	205.2
7	20.4	194.8	351	352	900.4	247.8
8	49.6	211	610.4	587.2	1208.4	284
9	80	504.6	921	1210.8	2150.8	640.9
10	100	716.4	1245	1720	2299.8	903.4
11	170.8	813.8	1664.8	1901	2391.6	1223.4
12	249.6	1300.4	2130.8	2191.4	2509.2	1554.4
13	480	1520.4	2217.8	2380.8	2608	1954
14	713.8	1751.4	2251	2433.8	2658.4	2173.8
15	951	1851	2316	2566	2674.4	2214.4
16	1312	1870.8	2345.8	2581.2	2687.8	2264
17	1450	1911.4	2382.6	2601.6	2796.2	2286.8

18	1551	1961	2398.8	2659.2	2833.6	2304
19	1721.8	1983.8	2404	2703.6	2848.4	2316
20	1760.6	2001	2410.4	2750.8	2856.2	2354.4
21	1772.8	2053	2417.4	2756	2902	2273.8
22	1808.2	2036	2428	2760.4	2910.6	2234.4
23	1803	2010.6	2441.4	2766.4	2896.8	2214
24	1751.2	2001.6	2417.6	2755.8	2882.6	2202.6

- Average experimental data for pH variables for biogas production:

Time (h)	pH5	pH6	pH7	pH8
	Average biogas production (mL)			
1	0	0	0	0
2	0	0	0	0
3	0	221.4	111.2	65.4
4	0	501.8	280.8	111.2
5	0	556.4	880	276
6	0	667.2	1500.6	609
7	0	776.4	2100	723.6
8	0	890	2165	889
9	0	1001	2334.4	1000.2
10	0	1276.4	2444.8	1160.6
11	0	1336	2447.8	1221.2
12	0	1443.2	2451.2	1444.8
13	0	1555.8	2455.4	1555.2
14	0	1610.8	2501.2	1612.4

15	0	1666	2515	1779.4
16	0	1775.8	2555.4	1890
17	0	1835.6	2561.4	2111.2
18	0	1887.6	2570.4	2164.6
19	0	2000.2	2571.8	2223.8
20	0	2111	2572	2223.6
21	0	2175.5	2570.6	2228.4
22	0	2176.4	2570	2230.8
23	0	2180.4	2568.4	2235.2
24	0	2180.4	2564.4	2241.2

- The experimental data for pH variables for methane proportion:

pH	5	6	7	8
Time (h)	Average methane proportion (%)			
	pH5	pH6	pH7	pH8
1	0	0	0	0
2	0	0	0	0
3	0	20.4	22.4	13.2
4	0	21.3	25.1	15.3
5	0	21.9	27.5	18.4
6	0	22.5	35.4	20.5
7	0	23.4	42.6	22.4
8	0	23.7	46.3	24.4
9	0	26.1	50.4	25.6
10	0	28.1	53.1	27.5
11	0	28.7	55.9	28.1
12	0	32.1	58.1	29.7
13	0	32.4	60.2	30.2
14	0	33.8	63.6	31.2
15	0	35.5	64.8	33.1
16	0	36.1	66.1	33.9
17	0	37.7	66.8	34.3
18	0	39.8	67.2	34.7
19	0	41.1	67.3	35.2
20	0	42	67.5	36
21	0	42.8	67	37.1

22	0	43.2	67	38.6
23	0	43.6	67.1	41.6
24	0	43.4	66.8	40.1

- Average experimental data for mixing speed variables for biogas production:

Time (h)	60 rpm	300 rpm	600 rpm	2000 rpm
	Average biogas production (mL)			
1	0	0	0	0
2	0	0	0	0
3	9.835	9.9	13.886	0
4	27.337	28.426	22.713	0.725
5	53.25	62.417	30.864	1.607
6	85.01	100.495	40.003	44.695
7	125.039	131.037	60.295	105.943
8	170.98	187.919	78.635	280.469
9	246.189	270.157	108.927	403.921
10	477.932	551.351	216.99	624.04
11	662.972	699.288	374.17	775.983
12	1248.44	1299.48	566.154	923.088
13	1386.243	1448.427	712.228	1176.463
14	1506.908	1572.857	960.577	1342.453
15	1614.934	1671.636	1170.302	1358.886
16	1690.114	1756.958	1400.976	1345.772
17	1721.566	1775.247	1462.721	1322.994
18	1739.69	1808.921	1595.478	1286.17
19	1763.341	1865.618	1672.81	1178.759
20	1851.276	1906.031	1784.045	1103.005
21	1871.194	1921.918	1794.752	1093.271

22	1876.462	1949.288	1769.024	1086.152
23	1859.193	1937.858	1734.061	1078.495
24	1849.748	1918.488	1668.545	1063.987

- Average experimental data for mixing speed variables for methane proportion:

Time (h)	60 rpm	300 rpm	600 rpm	2000 rpm
	Average methane proportion (%)			
1	0	0	0	0
2	0	0	0	0
3	11.7	13.2	25.3	0
4	27.1	28.2	28.2	9.8
5	35.5	38.7	30.7	15.2
6	42.4	43.6	33.1	27.4
7	46.1	48.1	35.3	40.6
8	48.7	51.3	40.1	47.8
9	50.1	53.6	43	53.3
10	53.1	55.1	48.2	56.7
11	55.7	56.2	51.2	59.1
12	58	59.5	53.2	61.4
13	60.2	61.5	55.3	61.9
14	63	64.7	57.3	63.7
15	64.3	65.4	60.3	64.5
16	64.8	65.7	62.1	64.1
17	64.7	66	63.6	63.4
18	65	66.5	64.1	62.7
19	65.6	66.9	65.1	60.9
20	66.2	67.2	65.9	59.1
21	66.6	67.5	65.2	58.5

22	66.7	68	65.5	58.2
23	66.2	67.5	64.5	57.9
24	66	67.1	64.2	57.6

- Average experimental data for pressure variables for biogas production:

Time (h)	0.7 bar	0.5 bar	0.3 bar	0.1 bar
	Average biogas production (mL)			
1	0	0	0	0
2	0	0	0	0
3	21.86	31.37	83.2	102.73
4	62.707	93.878	115.6416	118.41
5	119.45	123.28	179.6012	217.84
6	157.56	303.44	377.3172	403.82
7	176.64	354.72	391.5	423.31
8	196.2	402.89	422.864	501.3
9	308.67	501.24	678.5653	931.77
10	683.65	966.06	1178.766	1329.4
11	809.09	1260	1486.471	1758
12	842.11	1281	1560.58	1864.9
13	1011.1	1490.4	1771.199	2093.7
14	1154.8	1688.9	1951.596	2213.7
15	1232.4	1849.4	2149.482	2408.4
16	1454.7	2095.3	2429.584	2637
17	1566.8	2247.4	2616.3	2850.1
18	1751.4	2597.5	2946.807	3118.8
19	1901.9	2707.5	3088.064	3266.2
20	1950.1	2778	3146.516	3337.1
21	1990.1	2831	3191.4	3443.8

22	2010.3	2969.1	3408.339	3621.4
23	2060.1	2931.1	3425.823	3630
24	2099.3	2893.3	3430.801	3608

- Average experimental data for pressure variables for methane proportion:

Time (h)	0.7 bar	0.5 bar	0.3 bar	0.1 bar
	Average methane proportion (%)			
1	0	0	0	0
2	0	0	0	0
3	32.2	28.2	20.5	15.6
4	35.5	30.5	23.1	17.6
5	42.6	34.3	25.5	20.9
6	46.3	37.7	29	23
7	49.5	43.2	33.5	24.8
8	52.3	46.6	36.4	36.3
9	57.6	49.7	40.6	32.6
10	59.1	52.9	44.2	35.5
11	61.2	55.4	47.3	39.5
12	63	58.4	51.2	43.6
13	63.7	58	53.9	46.4
14	65	59.8	56	49.3
15	66.7	61.5	58	52.6
16	68.4	63.1	60.2	54.2
17	71.5	64.4	61.6	56.5
18	73.2	66.9	62.3	59.9
19	75.4	68.3	62.9	60.4
20	75.7	69.4	63.5	61.2

21	76.1	70.6	63.8	61.9
22	76.3	70.8	63.7	62.7
23	76.5	71.2	63.6	62.4
24	76.8	71.5	63.3	61.6

Appendix B: MATLAB Modelling Codes

MATLAB modelling codes:

```
%Data processing

%Load data containing all the parameters from mat files into your own variables.

load('Biogas_data.mat');


%extract from the K_data.mat the time vector.
Time=parameters(2:end,1);

Time=3600*Time;                %the time vector values in hours.

%From the data loaded extract  production for temperatures and pH, mixing speed and pressure.

Temperature32=parameters(2:end,2);    %Extract the measurements for the Temperature at 32 degrees.
pH5=parameters(2:end,3);               %Extract the measurements for pH of 5.
ms60=parameters(2:end,4);          %Extract the measurements for ms 60.
pressure700=parameters(2:end,5);       %Extract the measurements for pressure 700 mbar

Temperature34=parameters(2:end,6);    %Extract the measurements for the Temperature at 34 degrees.
pH6=parameters(2:end,7);               %Extract the measurements for pH of 6
ms270=parameters(2:end,8);         %Extract the measurements for ms 300
pressure600=parameters(2:end,9);

Temperature36=parameters(2:end,10);
pH65=parameters(2:end,11);
ms300=parameters(2:end,12);
pressure500=parameters(2:end,13);


Temperature38=parameters(2:end,14);
pH7=parameters(2:end,15);
ms500=parameters(2:end,16);
pressure300=parameters(2:end,17);

Temperature40=parameters(2:end,18);
pH75=parameters(2:end,19);
ms600=parameters(2:end,20);
pressure200=parameters(2:end,21);

Temperature42=parameters(2:end,22);
```

```
pH8=parameters(2:end,23);
ms2000=parameters(2:end,24);
pressure100=parameters(2:end,25);
Temperature35=parameters(2:end,26);
Temperature37=parameters(2:end,27);
% Creation of an iddata object for each of the experiments.
%Experiment 1
InputExp1=ones(25,4);
InputExp1(1:end,1)=32.*InputExp1(1:end,1);      %Temperature (C)
InputExp1(1:end,2)=7.1.*InputExp1(1:end,2);     %pH
InputExp1(1:end,3)=250.*InputExp1(1:end,3);     %Mixing speed (rpm)
InputExp1(1:end,4)=0.7.*InputExp1(1:end,4);     %Pressure (bar)
OutputExp1=Temperature32;
SystemEperiment1=iddata(OutputExp1,InputExp1,3600);
SystemEperiment1.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment1.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment1.OutputName='';
SystemEperiment1.OutputUnit='mL';

%Experiment 2
InputExp2=ones(25,4);
InputExp2(1:end,1)=34.*InputExp2(1:end,1);      %Temperature (C)
InputExp2(1:end,2)=7.1.*InputExp2(1:end,2);     %pH
InputExp2(1:end,3)=250.*InputExp2(1:end,3);     %Mixing speed (rpm)
InputExp2(1:end,4)=0.7.*InputExp2(1:end,4);     %Pressure (bar)
OutputExp2=Temperature34;
SystemEperiment2=iddata(OutputExp2,InputExp2,3600);
SystemEperiment2.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment2.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment2.OutputName='';
SystemEperiment2.OutputUnit='mL';

%Experiment 3
InputExp3=ones(25,4);
InputExp3(1:end,1)=36.*InputExp3(1:end,1);      %Temperature (C)
InputExp3(1:end,2)=7.1.*InputExp3(1:end,2);     %pH
InputExp3(1:end,3)=250.*InputExp3(1:end,3);     %Mixing speed (rpm)
InputExp3(1:end,4)=0.7.*InputExp3(1:end,4);     %Pressure (bar)
OutputExp3=Temperature36;
SystemEperiment3=iddata(OutputExp3,InputExp3,3600);
SystemEperiment3.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment3.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment3.OutputName='';
SystemEperiment3.OutputUnit='mL';

%Experiment 4
InputExp4=ones(25,4);
InputExp4(1:end,1)=38.*InputExp4(1:end,1);      %Temperature (C)
InputExp4(1:end,2)=7.1.*InputExp4(1:end,2);     %pH
InputExp4(1:end,3)=250.*InputExp4(1:end,3);     %Mixing speed (rpm)
```

```
InputExp4(1:end,4)=0.7.*InputExp4(1:end,4);     %Pressure (bar)
OutputExp4=Temperature38;
SystemEperiment4=iddata(OutputExp4,InputExp4,3600);
SystemEperiment4.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment4.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment4.OutputName='';
SystemEperiment4.OutputUnit='mL';

%Experiment 5
InputExp5=ones(25,4);
InputExp5(1:end,1)=40.*InputExp5(1:end,1);      %Temperature (C)
InputExp5(1:end,2)=7.1.*InputExp5(1:end,2);     %pH
InputExp5(1:end,3)=250.*InputExp5(1:end,3);     %Mixing speed (rpm)
InputExp5(1:end,4)=0.7.*InputExp5(1:end,4);     %Pressure (bar)
OutputExp5=Temperature40;
SystemEperiment5=iddata(OutputExp5,InputExp5,3600);
SystemEperiment5.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment5.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment5.OutputName='';
SystemEperiment5.OutputUnit='mL';

%Experiment 6
InputExp6=ones(25,4);
InputExp6(1:end,1)=42.*InputExp6(1:end,1);      %Temperature (C)
InputExp6(1:end,2)=7.1.*InputExp6(1:end,2);     %pH
InputExp6(1:end,3)=250.*InputExp6(1:end,3);     %Mixing speed (rpm)
InputExp6(1:end,4)=0.7.*InputExp6(1:end,4);     %Pressure (bar)
OutputExp6=Temperature42;
SystemEperiment6=iddata(OutputExp6,InputExp6,3600);
SystemEperiment6.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment6.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment6.OutputName='';
SystemEperiment6.OutputUnit='mL';

%Experiment 7
InputExp7=ones(25,4);
InputExp7(1:end,1)=39.*InputExp7(1:end,1);      %Temperature (C)
InputExp7(1:end,2)=5.*InputExp7(1:end,2);     %pH
InputExp7(1:end,3)=250.*InputExp7(1:end,3);     %Mixing speed (rpm)
InputExp7(1:end,4)=0.7.*InputExp7(1:end,4);     %Pressure (bar)
OutputExp7=pH5;
SystemEperiment7=iddata(OutputExp7,InputExp7,3600);
SystemEperiment7.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment7.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment7.OutputName='';
SystemEperiment7.OutputUnit='mL';

%Experiment 8
InputExp8=ones(25,4);
InputExp8(1:end,1)=39.*InputExp8(1:end,1);      %Temperature (C)
```

```
InputExp8(1:end,2)=6.*InputExp8(1:end,2);     %pH
InputExp8(1:end,3)=250.*InputExp8(1:end,3);     %Mixing speed (rpm)
InputExp8(1:end,4)=0.7.*InputExp8(1:end,4);     %Pressure (bar)
OutputExp8=pH6;
SystemEperiment8=iddata(OutputExp8,InputExp8,3600);
SystemEperiment8.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment8.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment8.OutputName='';
SystemEperiment8.OutputUnit='mL';

%Experiment 9
InputExp9=ones(25,4);
InputExp9(1:end,1)=39.*InputExp9(1:end,1);      %Temperature (C)
InputExp9(1:end,2)=7.*InputExp9(1:end,2);     %pH
InputExp9(1:end,3)=250.*InputExp9(1:end,3);     %Mixing speed (rpm)
InputExp9(1:end,4)=0.7.*InputExp9(1:end,4);     %Pressure (bar)
OutputExp9=pH7;
SystemEperiment9=iddata(OutputExp9,InputExp9,3600);
SystemEperiment9.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment9.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment9.OutputName='';
SystemEperiment9.OutputUnit='mL';

%Experiment 10
InputExp10=ones(25,4);
InputExp10(1:end,1)=39.*InputExp10(1:end,1);      %Temperature (C)
InputExp10(1:end,2)=8.*InputExp10(1:end,2);     %pH
InputExp10(1:end,3)=250.*InputExp10(1:end,3);     %Mixing speed (rpm)
InputExp10(1:end,4)=0.7.*InputExp10(1:end,4);     %Pressure (bar)
OutputExp10=pH8;
SystemEperiment10=iddata(OutputExp10,InputExp10,3600);
SystemEperiment10.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment10.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment10.OutputName='';
SystemEperiment10.OutputUnit='mL';

%Experiment 11
InputExp11=ones(25,4);
InputExp11(1:end,1)=39.*InputExp11(1:end,1);      %Temperature (C)
InputExp11(1:end,2)=7.1.*InputExp11(1:end,2);     %pH
InputExp11(1:end,3)=60.*InputExp11(1:end,3);     %Mixing speed (rpm)
InputExp11(1:end,4)=0.7.*InputExp11(1:end,4);     %Pressure (bar)
OutputExp11=ms60;
SystemEperiment11=iddata(OutputExp11,InputExp11,3600);
SystemEperiment11.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment11.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment11.OutputName='';
SystemEperiment11.OutputUnit='mL';

%Experiment 12
```

```
InputExp12=ones(25,4);
InputExp12(1:end,1)=39.*InputExp12(1:end,1);      %Temperature (C)
InputExp12(1:end,2)=7.1.*InputExp12(1:end,2);     %pH
InputExp12(1:end,3)=300.*InputExp12(1:end,3);     %Mixing speed (rpm)
InputExp12(1:end,4)=0.7.*InputExp12(1:end,4);     %Pressure (bar)
OutputExp12=ms300;
SystemEperiment12=iddata(OutputExp12,InputExp12,3600);
SystemEperiment12.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment12.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment12.OutputName='';
SystemEperiment12.OutputUnit='mL';

%Experiment 13
InputExp13=ones(25,4);
InputExp13(1:end,1)=39.*InputExp13(1:end,1);      %Temperature (C)
InputExp13(1:end,2)=7.1.*InputExp13(1:end,2);     %pH
InputExp13(1:end,3)=600.*InputExp13(1:end,3);     %Mixing speed (rpm)
InputExp13(1:end,4)=0.7.*InputExp13(1:end,4);     %Pressure (bar)
OutputExp13=ms600;
SystemEperiment13=iddata(OutputExp13,InputExp13,3600);
SystemEperiment13.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment13.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment13.OutputName='';
SystemEperiment13.OutputUnit='mL';

%Experiment 14
InputExp14=ones(25,4);
InputExp14(1:end,1)=39.*InputExp14(1:end,1);      %Temperature (C)
InputExp14(1:end,2)=7.1.*InputExp14(1:end,2);     %pH
InputExp14(1:end,3)=2000.*InputExp14(1:end,3);     %Mixing speed (rpm)
InputExp14(1:end,4)=0.7.*InputExp14(1:end,4);     %Pressure (bar)
OutputExp14=ms2000;
SystemEperiment14=iddata(OutputExp14,InputExp14,3600);
SystemEperiment14.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment14.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment14.OutputName='';
SystemEperiment14.OutputUnit='mL';

%Experiment 15
InputExp15=ones(25,4);
InputExp15(1:end,1)=39.*InputExp15(1:end,1);      %Temperature (C)
InputExp15(1:end,2)=7.1.*InputExp15(1:end,2);     %pH
InputExp15(1:end,3)=250.*InputExp15(1:end,3);     %Mixing speed (rpm)
InputExp15(1:end,4)=0.1.*InputExp15(1:end,4);     %Pressure (bar)
OutputExp15=pressure100;
SystemEperiment15=iddata(OutputExp15,InputExp15,3600);
SystemEperiment15.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment15.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment15.OutputName='';
SystemEperiment15.OutputUnit='mL';
```

```
%Experiment 16
InputExp16=ones(25,4);
InputExp16(1:end,1)=39.*InputExp16(1:end,1);      %Temperature (C)
InputExp16(1:end,2)=7.1.*InputExp16(1:end,2);     %pH
InputExp16(1:end,3)=250.*InputExp16(1:end,3);     %Mixing speed (rpm)
InputExp16(1:end,4)=0.3.*InputExp16(1:end,4);     %Pressure (bar)
OutputExp16=pressure300;
SystemEperiment16=iddata(OutputExp16,InputExp16,3600);
SystemEperiment16.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment16.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment16.OutputName='';
SystemEperiment16.OutputUnit='mL';

%Experiment 17
InputExp17=ones(25,4);
InputExp17(1:end,1)=39.*InputExp17(1:end,1);      %Temperature (C)
InputExp17(1:end,2)=7.1.*InputExp17(1:end,2);     %pH
InputExp17(1:end,3)=250.*InputExp17(1:end,3);     %Mixing speed (rpm)
InputExp17(1:end,4)=0.5.*InputExp17(1:end,4);     %Pressure (bar)
OutputExp17=pressure500;
SystemEperiment17=iddata(OutputExp17,InputExp17,3600);
SystemEperiment17.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment17.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment17.OutputName='';
SystemEperiment17.OutputUnit='mL';

%Experiment 18
InputExp18=ones(25,4);
InputExp18(1:end,1)=39.*InputExp18(1:end,1);      %Temperature (C)
InputExp18(1:end,2)=7.1.*InputExp18(1:end,2);     %pH
InputExp18(1:end,3)=250.*InputExp18(1:end,3);     %Mixing speed (rpm)
InputExp18(1:end,4)=0.7.*InputExp18(1:end,4);     %Pressure (bar)
OutputExp18=pressure700;
SystemEperiment18=iddata(OutputExp18,InputExp18,3600);
SystemEperiment18.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment18.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment18.OutputName='';
SystemEperiment18.OutputUnit='mL';

%Experiment 19
InputExp19=ones(25,4);
InputExp19(1:end,1)=39.*InputExp19(1:end,1);      %Temperature (C)
InputExp19(1:end,2)=7.1.*InputExp19(1:end,2);     %pH
InputExp19(1:end,3)=270.*InputExp19(1:end,3);     %Mixing speed (rpm)
InputExp19(1:end,4)=0.7.*InputExp19(1:end,4);     %Pressure (bar)
OutputExp19=ms270;
SystemEperiment19=iddata(OutputExp19,InputExp19,3600);
SystemEperiment19.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment19.inputUnit={'C', ' ', 'rpm', 'bar'};
```

```
SystemEperiment19.OutputName='';
SystemEperiment19.OutputUnit='mL';

%Experiment 20
InputExp20=ones(25,4);
InputExp20(1:end,1)=39.*InputExp20(1:end,1);      %Temperature (C)
InputExp20(1:end,2)=7.1.*InputExp20(1:end,2);     %pH
InputExp20(1:end,3)=250.*InputExp20(1:end,3);     %Mixing speed (rpm)
InputExp20(1:end,4)=0.6.*InputExp20(1:end,4);     %Pressure (bar)
OutputExp20=pressure600;
SystemEperiment20=iddata(OutputExp20,InputExp20,3600);
SystemEperiment20.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment20.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment20.OutputName='';
SystemEperiment20.OutputUnit='mL';

%Experiment 21
InputExp21=ones(25,4);
InputExp21(1:end,1)=39.*InputExp21(1:end,1);      %Temperature (C)
InputExp21(1:end,2)=(6.5).*InputExp21(1:end,2);     %pH
InputExp21(1:end,3)=250.*InputExp21(1:end,3);     %Mixing speed (rpm)
InputExp21(1:end,4)=0.7.*InputExp21(1:end,4);     %Pressure (bar)
OutputExp21=pH65;
SystemEperiment21=iddata(OutputExp21,InputExp21,3600);
SystemEperiment21.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment21.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment21.OutputName='';
SystemEperiment21.OutputUnit='mL';

%Experiment 22
InputExp22=ones(25,4);
InputExp22(1:end,1)=39.*InputExp22(1:end,1);      %Temperature (C)
InputExp22(1:end,2)=(7.5).*InputExp22(1:end,2);     %pH
InputExp22(1:end,3)=250.*InputExp22(1:end,3);     %Mixing speed (rpm)
InputExp22(1:end,4)=0.7.*InputExp22(1:end,4);     %Pressure (bar)
OutputExp22=pH75;
SystemEperiment22=iddata(OutputExp22,InputExp22,3600);
SystemEperiment22.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment22.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment22.OutputName='';
SystemEperiment22.OutputUnit='mL';

%Experiment 23
InputExp23=ones(25,4);
InputExp23(1:end,1)=39.*InputExp23(1:end,1);      %Temperature (C)
InputExp23(1:end,2)=7.1.*InputExp23(1:end,2);     %pH
InputExp23(1:end,3)=500.*InputExp23(1:end,3);     %Mixing speed (rpm)
InputExp23(1:end,4)=0.7.*InputExp23(1:end,4);     %Pressure (bar)
OutputExp23=ms500;
SystemEperiment23=iddata(OutputExp23,InputExp23,3600);
```

```
SystemEperiment23.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment23.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment23.OutputName='';
SystemEperiment23.OutputUnit='mL';

%Experiment 24
InputExp24=ones(25,4);
InputExp24(1:end,1)=39.*InputExp24(1:end,1);      %Temperature (C)
InputExp24(1:end,2)=7.1.*InputExp24(1:end,2);     %pH
InputExp24(1:end,3)=250.*InputExp24(1:end,3);     %Mixing speed (rpm)
InputExp24(1:end,4)=0.2.*InputExp24(1:end,4);     %Pressure (bar)
OutputExp24=pressure200;
SystemEperiment24=iddata(OutputExp24,InputExp24,3600);
SystemEperiment24.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment24.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment24.OutputName='';
SystemEperiment24.OutputUnit='mL';


% Experiment 25
InputExp25=ones(25,4);
InputExp25(1:end,1)=35.*InputExp25(1:end,1);      %Temperature (C)
InputExp25(1:end,2)=7.1.*InputExp25(1:end,2);     %pH
InputExp25(1:end,3)=250.*InputExp25(1:end,3);     %Mixing speed (rpm)
InputExp25(1:end,4)=0.7.*InputExp25(1:end,4);     %Pressure (bar)
OutputExp25=Temperature35;
SystemEperiment25=iddata(OutputExp25,InputExp25,3600);
SystemEperiment25.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment25.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment25.OutputName='';
SystemEperiment25.OutputUnit='mL';

% Experiment 26
InputExp26=ones(25,4);
InputExp26(1:end,1)=37.*InputExp26(1:end,1);      %Temperature (C)
InputExp26(1:end,2)=7.1.*InputExp26(1:end,2);     %pH
InputExp26(1:end,3)=250.*InputExp26(1:end,3);     %Mixing speed (rpm)
InputExp26(1:end,4)=0.7.*InputExp26(1:end,4);     %Pressure (bar)
OutputExp26=Temperature37;
SystemEperiment26=iddata(OutputExp26,InputExp26,3600);
SystemEperiment26.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment26.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment26.OutputName='';
SystemEperiment26.OutputUnit='mL';
%Using KI, K2, K3 as estimation data sets and K6 as validation data set
%merging all estimation iddata to a single iddata object;
%This is for all the measurements that will be considered for the model extraction.
Overall_iddata=merge(SystemEperiment1,SystemEperiment2, SystemEperiment3,
SystemEperiment8, SystemEperiment11, SystemEperiment12, SystemEperiment17,
SystemEperiment18, SystemEperiment19, SystemEperiment20, SystemEperiment21,
```

SystemEperiment25, SystemEperiment16, SystemEperiment22, SystemEperiment23, SystemEperiment24, SystemEperiment26);

%VALIDATION PROCESS

%merging data to a single iddata object;
%This is for all the measurements that will be considered for the model extraction.
Overall_iddata_val=merge(SystemEperiment4,SystemEperiment5, SystemEperiment6, SystemEperiment9, SystemEperiment10, SystemEperiment13, SystemEperiment14, SystemEperiment15);

%Estimation data and validation data have been entered and two iddata objects have been created:
%Overall_iddata contains the experiment results for the model identification process.
%Overall_iddata_val contains the experiment results for the model validation.
de=(Overall_iddata);
dv=(Overall_iddata_val);

%NALRX MODEL

%mx1 = nlarx(de, [ones(1,1), ones(1,4), ones(1,4)], wavenet)
%mx2 = nlarx(de, [ones(1,1), ones(1,4), 2*ones(1,4)], wavenet)
%mx3 = nlarx(de, [2*ones(1,1), ones(1,4), 2*ones(1,4)], wavenet)
%mx4 = nlarx(de, [2*ones(1,1), 2*ones(1,4), 2*ones(1,4)], wavenet)
%mx5 = nlarx(de, [3*ones(1,1), ones(1,4), ones(1,4)], wavenet)
%mx6 = nlarx(de, [3*ones(1,1), 3*ones(1,4), ones(1,4)], wavenet)

%compare(dv,mx1, mx2, mx3, mx4, mx5, mx6)

%The numbers of units (wavelets) of the two WAVENET estimators have been automatically chosen by the estimation algorithm. These numbers are displayed below. Notice the abbreviations 'nl'='Nonlinearity' and 'num'='NumberOfUnits'
%mx4.Nonlinearity(1).NumberOfUnits %using full property names

%nanbnk = [2*ones(1,1), ones(1,4), 2*ones(1,4)];

%The number of units in the WAVENET estimators can be explicitly specified instead of being automatically chosen by the estimation algorithm:
%mx7 = nlarx(de, nanbnk, [wavenet('num',6)]);
%mx8 = nlarx(de, nanbnk, [wavenet('num',8)]);
%mx9 = nlarx(de, [2*ones(1,1), ones(1,4), 2*ones(1,4)], [wavenet('num',9)])

%compare(dv,mx7, mx8, mx9)

%Nonlinear ARX Model - Trying Other Nonlinearity Estimators

%mx10 = nlarx(de, nanbnk, treepartition);

%The SIGMOIDNET estimator can also be used. Estimation options such as maximum iterations (MaxIter) and iteration display can be specified using NLARXOPTIONS command.
%opt = nlarxOptions('Display','on');
%opt.SearchOption.MaxIter = 2;
%mx11 = nlarx(de, nanbnk, sigmoidnet);

%compare(dv, mx12, mx13)

%Function PLOT may be used to view the nonlinearity responses of various models.
%plot(mx7, mx12, mx13)

%Estimation of Hammerstein-Wiener Model (Both Input and Output Nonlinearities)

%Indicate both input and output nonlinearities for a Hammerstein-Wiener model. As in the case of Nonlinear ARX models, we can use a string (rather than object) to specify the nonlinearity estimator.

%mhw1 = nlarx(de, [1 5 3], wavenet);
%ws = warning('off','Ident:estimation:NparGTNsamp');
%mhw1 = nlhw(de, [4, 8*ones(1,4), 8*ones(1,4)],pwlinear, pwlinear);

%mhw1 = nlhw(de, [ones(1,4), ones(1,4), zeros(1,4)], 'pwlinear', 'pwlinear');
mhw2 = nlhw(de, [2*ones(1,4), 2*ones(1,4), 2*zeros(1,4)], 'pwlinear', 'pwlinear');
%mhw3 = nlhw(de, [3*ones(1,4), ones(1,4), zeros(1,4)], 'pwlinear', 'pwlinear');

%compare(dv, mhw2)
%compare(dv,mhw1, mhw2, mhw3)

%mhw4 = nlhw(de, [ones(1,4), ones(1,4), zeros(1,4)], 'unitgain', 'deadzone');
%mhw5 = nlhw(de, [2*ones(1,4), 2*ones(1,4), 2*zeros(1,4)], 'unitgain', 'deadzone')
%mhw6 = nlhw(de, [3*ones(1,4), ones(1,4), zeros(1,4)], 'unitgain', 'deadzone');

%compare(dv,mhw4, mhw5, mhw6)

%mhw7 = nlhw(de, [ones(1,4), ones(1,4), zeros(1,4)], 'saturation', 'deadzone');
%mhw8 = nlhw(de, [2*ones(1,4), 2*ones(1,4), 2*zeros(1,4)], 'saturation', 'deadzone')
%mhw9 = nlhw(de, [3*ones(1,4), ones(1,4), zeros(1,4)], 'saturation', 'deadzone');

%compare(dv,mhw2, mhw5, mhw8)

```
%compare(dv, mx9, mhw2)


%AIC = aic (mx9, mhw2)

%The limit values of the SATURATION estimators can be accessed. The short-hands

'u'='input', 'y'='output', and 'nl'='Nonlinearity' are available


%Data processing

%Load data containing all the parameters from mat files into your own variables.

load('Methane_data.mat');


%extract from the K_data.mat the time vector.
Time=parameters(2:end,1);

Time=3600*Time;                %the time vector values in hours.

%From the data loaded extract  production for temperatures and pH, mixing speed and pressure.

Temperature32=parameters(2:end,2);    %Extract the measurements for the Temperature at 32 degrees.
pH5=parameters(2:end,3);                %Extract the measurements for pH of 5.
ms60=parameters(2:end,4);            %Extract the measurements for ms 60.
pressure700=parameters(2:end,5);        %Extract the measurements for pressure 700 mbar

Temperature34=parameters(2:end,6);    %Extract the measurements for the Temperature at 34 degrees.
pH6=parameters(2:end,7);                %Extract the measurements for pH of 6
ms270=parameters(2:end,8);            %Extract the measurements for ms 300
pressure600=parameters(2:end,9);

Temperature36=parameters(2:end,10);
pH65=parameters(2:end,11);
ms300=parameters(2:end,12);
pressure500=parameters(2:end,13);


Temperature38=parameters(2:end,14);
pH7=parameters(2:end,15);
ms500=parameters(2:end,16);
pressure300=parameters(2:end,17);

Temperature40=parameters(2:end,18);
```

```
pH75=parameters(2:end,19);
ms600=parameters(2:end,20);
pressure200=parameters(2:end,21);

Temperature42=parameters(2:end,22);
pH8=parameters(2:end,23);
ms2000=parameters(2:end,24);
pressure100=parameters(2:end,25);
Temperature35=parameters(2:end,26);
Temperature37=parameters(2:end,27);
% Creation of an iddata object for each of the experiments.
%Experiment 1
InputExp1=ones(25,4);
InputExp1(1:end,1)=32.*InputExp1(1:end,1);      %Temperature (C)
InputExp1(1:end,2)=7.1.*InputExp1(1:end,2);     %pH
InputExp1(1:end,3)=250.*InputExp1(1:end,3);     %Mixing speed (rpm)
InputExp1(1:end,4)=0.7.*InputExp1(1:end,4);     %Pressure (bar)
OutputExp1=Temperature32;
SystemEperiment1=iddata(OutputExp1,InputExp1,3600);
SystemEperiment1.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment1.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment1.OutputName='';
SystemEperiment1.OutputUnit='mL';

%Experiment 2
InputExp2=ones(25,4);
InputExp2(1:end,1)=34.*InputExp2(1:end,1);      %Temperature (C)
InputExp2(1:end,2)=7.1.*InputExp2(1:end,2);     %pH
InputExp2(1:end,3)=250.*InputExp2(1:end,3);     %Mixing speed (rpm)
InputExp2(1:end,4)=0.7.*InputExp2(1:end,4);     %Pressure (bar)
OutputExp2=Temperature34;
SystemEperiment2=iddata(OutputExp2,InputExp2,3600);
SystemEperiment2.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment2.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment2.OutputName='';
SystemEperiment2.OutputUnit='mL';

%Experiment 3
InputExp3=ones(25,4);
InputExp3(1:end,1)=36.*InputExp3(1:end,1);      %Temperature (C)
InputExp3(1:end,2)=7.1.*InputExp3(1:end,2);     %pH
InputExp3(1:end,3)=250.*InputExp3(1:end,3);     %Mixing speed (rpm)
InputExp3(1:end,4)=0.7.*InputExp3(1:end,4);     %Pressure (bar)
OutputExp3=Temperature36;
SystemEperiment3=iddata(OutputExp3,InputExp3,3600);
SystemEperiment3.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment3.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment3.OutputName='';
SystemEperiment3.OutputUnit='mL';
```

```
%Experiment 4
InputExp4=ones(25,4);
InputExp4(1:end,1)=38.*InputExp4(1:end,1);      %Temperature (C)
InputExp4(1:end,2)=7.1.*InputExp4(1:end,2);     %pH
InputExp4(1:end,3)=250.*InputExp4(1:end,3);     %Mixing speed (rpm)
InputExp4(1:end,4)=0.7.*InputExp4(1:end,4);     %Pressure (bar)
OutputExp4=Temperature38;
SystemEperiment4=iddata(OutputExp4,InputExp4,3600);
SystemEperiment4.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment4.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment4.OutputName='';
SystemEperiment4.OutputUnit='mL';

%Experiment 5
InputExp5=ones(25,4);
InputExp5(1:end,1)=40.*InputExp5(1:end,1);      %Temperature (C)
InputExp5(1:end,2)=7.1.*InputExp5(1:end,2);     %pH
InputExp5(1:end,3)=250.*InputExp5(1:end,3);     %Mixing speed (rpm)
InputExp5(1:end,4)=0.7.*InputExp5(1:end,4);     %Pressure (bar)
OutputExp5=Temperature40;
SystemEperiment5=iddata(OutputExp5,InputExp5,3600);
SystemEperiment5.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment5.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment5.OutputName='';
SystemEperiment5.OutputUnit='mL';

%Experiment 6
InputExp6=ones(25,4);
InputExp6(1:end,1)=42.*InputExp6(1:end,1);      %Temperature (C)
InputExp6(1:end,2)=7.1.*InputExp6(1:end,2);     %pH
InputExp6(1:end,3)=250.*InputExp6(1:end,3);     %Mixing speed (rpm)
InputExp6(1:end,4)=0.7.*InputExp6(1:end,4);     %Pressure (bar)
OutputExp6=Temperature42;
SystemEperiment6=iddata(OutputExp6,InputExp6,3600);
SystemEperiment6.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment6.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment6.OutputName='';
SystemEperiment6.OutputUnit='mL';

%Experiment 7
InputExp7=ones(25,4);
InputExp7(1:end,1)=39.*InputExp7(1:end,1);      %Temperature (C)
InputExp7(1:end,2)=5.*InputExp7(1:end,2);     %pH
InputExp7(1:end,3)=250.*InputExp7(1:end,3);     %Mixing speed (rpm)
InputExp7(1:end,4)=0.7.*InputExp7(1:end,4);     %Pressure (bar)
OutputExp7=pH5;
SystemEperiment7=iddata(OutputExp7,InputExp7,3600);
SystemEperiment7.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment7.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment7.OutputName='';
```

```
SystemEperiment7.OutputUnit='mL';

%Experiment 8
InputExp8=ones(25,4);
InputExp8(1:end,1)=39.*InputExp8(1:end,1);      %Temperature (C)
InputExp8(1:end,2)=6.*InputExp8(1:end,2);     %pH
InputExp8(1:end,3)=250.*InputExp8(1:end,3);     %Mixing speed (rpm)
InputExp8(1:end,4)=0.7.*InputExp8(1:end,4);     %Pressure (bar)
OutputExp8=pH6;
SystemEperiment8=iddata(OutputExp8,InputExp8,3600);
SystemEperiment8.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment8.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment8.OutputName='';
SystemEperiment8.OutputUnit='mL';

%Experiment 9
InputExp9=ones(25,4);
InputExp9(1:end,1)=39.*InputExp9(1:end,1);      %Temperature (C)
InputExp9(1:end,2)=7.*InputExp9(1:end,2);     %pH
InputExp9(1:end,3)=250.*InputExp9(1:end,3);     %Mixing speed (rpm)
InputExp9(1:end,4)=0.7.*InputExp9(1:end,4);     %Pressure (bar)
OutputExp9=pH7;
SystemEperiment9=iddata(OutputExp9,InputExp9,3600);
SystemEperiment9.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment9.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment9.OutputName='';
SystemEperiment9.OutputUnit='mL';

%Experiment 10
InputExp10=ones(25,4);
InputExp10(1:end,1)=39.*InputExp10(1:end,1);      %Temperature (C)
InputExp10(1:end,2)=8.*InputExp10(1:end,2);     %pH
InputExp10(1:end,3)=250.*InputExp10(1:end,3);     %Mixing speed (rpm)
InputExp10(1:end,4)=0.7.*InputExp10(1:end,4);     %Pressure (bar)
OutputExp10=pH8;
SystemEperiment10=iddata(OutputExp10,InputExp10,3600);
SystemEperiment10.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment10.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment10.OutputName='';
SystemEperiment10.OutputUnit='mL';

%Experiment 11
InputExp11=ones(25,4);
InputExp11(1:end,1)=39.*InputExp11(1:end,1);      %Temperature (C)
InputExp11(1:end,2)=7.1.*InputExp11(1:end,2);     %pH
InputExp11(1:end,3)=60.*InputExp11(1:end,3);     %Mixing speed (rpm)
InputExp11(1:end,4)=0.7.*InputExp11(1:end,4);     %Pressure (bar)
OutputExp11=ms60;
SystemEperiment11=iddata(OutputExp11,InputExp11,3600);
SystemEperiment11.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
```

```
SystemEperiment11.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment11.OutputName='';
SystemEperiment11.OutputUnit='mL';

%Experiment 12
InputExp12=ones(25,4);
InputExp12(1:end,1)=39.*InputExp12(1:end,1);      %Temperature (C)
InputExp12(1:end,2)=7.1.*InputExp12(1:end,2);     %pH
InputExp12(1:end,3)=300.*InputExp12(1:end,3);     %Mixing speed (rpm)
InputExp12(1:end,4)=0.7.*InputExp12(1:end,4);     %Pressure (bar)
OutputExp12=ms300;
SystemEperiment12=iddata(OutputExp12,InputExp12,3600);
SystemEperiment12.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment12.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment12.OutputName='';
SystemEperiment12.OutputUnit='mL';

%Experiment 13
InputExp13=ones(25,4);
InputExp13(1:end,1)=39.*InputExp13(1:end,1);      %Temperature (C)
InputExp13(1:end,2)=7.1.*InputExp13(1:end,2);     %pH
InputExp13(1:end,3)=600.*InputExp13(1:end,3);     %Mixing speed (rpm)
InputExp13(1:end,4)=0.7.*InputExp13(1:end,4);     %Pressure (bar)
OutputExp13=ms600;
SystemEperiment13=iddata(OutputExp13,InputExp13,3600);
SystemEperiment13.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment13.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment13.OutputName='';
SystemEperiment13.OutputUnit='mL';

%Experiment 14
InputExp14=ones(25,4);
InputExp14(1:end,1)=39.*InputExp14(1:end,1);      %Temperature (C)
InputExp14(1:end,2)=7.1.*InputExp14(1:end,2);     %pH
InputExp14(1:end,3)=2000.*InputExp14(1:end,3);    %Mixing speed (rpm)
InputExp14(1:end,4)=0.7.*InputExp14(1:end,4);     %Pressure (bar)
OutputExp14=ms2000;
SystemEperiment14=iddata(OutputExp14,InputExp14,3600);
SystemEperiment14.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment14.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment14.OutputName='';
SystemEperiment14.OutputUnit='mL';

%Experiment 15
InputExp15=ones(25,4);
InputExp15(1:end,1)=39.*InputExp15(1:end,1);      %Temperature (C)
InputExp15(1:end,2)=7.1.*InputExp15(1:end,2);     %pH
InputExp15(1:end,3)=250.*InputExp15(1:end,3);     %Mixing speed (rpm)
InputExp15(1:end,4)=0.1.*InputExp15(1:end,4);     %Pressure (bar)
OutputExp15=pressure100;
```

```
SystemEperiment15=iddata(OutputExp15,InputExp15,3600);
SystemEperiment15.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment15.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment15.OutputName='';
SystemEperiment15.OutputUnit='mL';

%Experiment 16
InputExp16=ones(25,4);
InputExp16(1:end,1)=39.*InputExp16(1:end,1);      %Temperature (C)
InputExp16(1:end,2)=7.1.*InputExp16(1:end,2);     %pH
InputExp16(1:end,3)=250.*InputExp16(1:end,3);     %Mixing speed (rpm)
InputExp16(1:end,4)=0.3.*InputExp16(1:end,4);     %Pressure (bar)
OutputExp16=pressure300;
SystemEperiment16=iddata(OutputExp16,InputExp16,3600);
SystemEperiment16.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment16.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment16.OutputName='';
SystemEperiment16.OutputUnit='mL';

%Experiment 17
InputExp17=ones(25,4);
InputExp17(1:end,1)=39.*InputExp17(1:end,1);      %Temperature (C)
InputExp17(1:end,2)=7.1.*InputExp17(1:end,2);     %pH
InputExp17(1:end,3)=250.*InputExp17(1:end,3);     %Mixing speed (rpm)
InputExp17(1:end,4)=0.5.*InputExp17(1:end,4);     %Pressure (bar)
OutputExp17=pressure500;
SystemEperiment17=iddata(OutputExp17,InputExp17,3600);
SystemEperiment17.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment17.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment17.OutputName='';
SystemEperiment17.OutputUnit='mL';

%Experiment 18
InputExp18=ones(25,4);
InputExp18(1:end,1)=39.*InputExp18(1:end,1);      %Temperature (C)
InputExp18(1:end,2)=7.1.*InputExp18(1:end,2);     %pH
InputExp18(1:end,3)=250.*InputExp18(1:end,3);     %Mixing speed (rpm)
InputExp18(1:end,4)=0.7.*InputExp18(1:end,4);     %Pressure (bar)
OutputExp18=pressure700;
SystemEperiment18=iddata(OutputExp18,InputExp18,3600);
SystemEperiment18.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment18.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment18.OutputName='';
SystemEperiment18.OutputUnit='mL';

%Experiment 19
InputExp19=ones(25,4);
InputExp19(1:end,1)=39.*InputExp19(1:end,1);      %Temperature (C)
InputExp19(1:end,2)=7.1.*InputExp19(1:end,2);     %pH
InputExp19(1:end,3)=270.*InputExp19(1:end,3);     %Mixing speed (rpm)
```

```
InputExp19(1:end,4)=0.7.*InputExp19(1:end,4);     %Pressure (bar)
OutputExp19=ms270;
SystemEperiment19=iddata(OutputExp19,InputExp19,3600);
SystemEperiment19.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment19.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment19.OutputName='';
SystemEperiment19.OutputUnit='mL';

%Experiment 20
InputExp20=ones(25,4);
InputExp20(1:end,1)=39.*InputExp20(1:end,1);      %Temperature (C)
InputExp20(1:end,2)=7.1.*InputExp20(1:end,2);     %pH
InputExp20(1:end,3)=250.*InputExp20(1:end,3);     %Mixing speed (rpm)
InputExp20(1:end,4)=0.6.*InputExp20(1:end,4);     %Pressure (bar)
OutputExp20=pressure600;
SystemEperiment20=iddata(OutputExp20,InputExp20,3600);
SystemEperiment20.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment20.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment20.OutputName='';
SystemEperiment20.OutputUnit='mL';

%Experiment 21
InputExp21=ones(25,4);
InputExp21(1:end,1)=39.*InputExp21(1:end,1);      %Temperature (C)
InputExp21(1:end,2)=(6.5).*InputExp21(1:end,2);     %pH
InputExp21(1:end,3)=250.*InputExp21(1:end,3);     %Mixing speed (rpm)
InputExp21(1:end,4)=0.7.*InputExp21(1:end,4);     %Pressure (bar)
OutputExp21=pH65;
SystemEperiment21=iddata(OutputExp21,InputExp21,3600);
SystemEperiment21.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment21.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment21.OutputName='';
SystemEperiment21.OutputUnit='mL';

%Experiment 22
InputExp22=ones(25,4);
InputExp22(1:end,1)=39.*InputExp22(1:end,1);      %Temperature (C)
InputExp22(1:end,2)=(7.5).*InputExp22(1:end,2);     %pH
InputExp22(1:end,3)=250.*InputExp22(1:end,3);     %Mixing speed (rpm)
InputExp22(1:end,4)=0.7.*InputExp22(1:end,4);     %Pressure (bar)
OutputExp22=pH75;
SystemEperiment22=iddata(OutputExp22,InputExp22,3600);
SystemEperiment22.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment22.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment22.OutputName='';
SystemEperiment22.OutputUnit='mL';

%Experiment 23
InputExp23=ones(25,4);
InputExp23(1:end,1)=39.*InputExp23(1:end,1);      %Temperature (C)
```

```
InputExp23(1:end,2)=7.1.*InputExp23(1:end,2);     %pH
InputExp23(1:end,3)=500.*InputExp23(1:end,3);     %Mixing speed (rpm)
InputExp23(1:end,4)=0.7.*InputExp23(1:end,4);     %Pressure (bar)
OutputExp23=ms500;
SystemEperiment23=iddata(OutputExp23,InputExp23,3600);
SystemEperiment23.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment23.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment23.OutputName='';
SystemEperiment23.OutputUnit='mL';

%Experiment 24
InputExp24=ones(25,4);
InputExp24(1:end,1)=39.*InputExp24(1:end,1);      %Temperature (C)
InputExp24(1:end,2)=7.1.*InputExp24(1:end,2);     %pH
InputExp24(1:end,3)=250.*InputExp24(1:end,3);     %Mixing speed (rpm)
InputExp24(1:end,4)=0.2.*InputExp24(1:end,4);     %Pressure (bar)
OutputExp24=pressure200;
SystemEperiment24=iddata(OutputExp24,InputExp24,3600);
SystemEperiment24.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment24.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment24.OutputName='';
SystemEperiment24.OutputUnit='mL';


% Experiment 25
InputExp25=ones(25,4);
InputExp25(1:end,1)=35.*InputExp25(1:end,1);      %Temperature (C)
InputExp25(1:end,2)=7.1.*InputExp25(1:end,2);     %pH
InputExp25(1:end,3)=250.*InputExp25(1:end,3);     %Mixing speed (rpm)
InputExp25(1:end,4)=0.7.*InputExp25(1:end,4);     %Pressure (bar)
OutputExp25=Temperature35;
SystemEperiment25=iddata(OutputExp25,InputExp25,3600);
SystemEperiment25.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment25.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment25.OutputName='';
SystemEperiment25.OutputUnit='mL';

% Experiment 26
InputExp26=ones(25,4);
InputExp26(1:end,1)=37.*InputExp26(1:end,1);      %Temperature (C)
InputExp26(1:end,2)=7.1.*InputExp26(1:end,2);     %pH
InputExp26(1:end,3)=250.*InputExp26(1:end,3);     %Mixing speed (rpm)
InputExp26(1:end,4)=0.7.*InputExp26(1:end,4);     %Pressure (bar)
OutputExp26=Temperature37;
SystemEperiment26=iddata(OutputExp26,InputExp26,3600);
SystemEperiment26.InputName={'Temperature', 'pH', 'Mixing speed', 'Pressure'};
SystemEperiment26.inputUnit={'C', ' ', 'rpm', 'bar'};
SystemEperiment26.OutputName='';
SystemEperiment26.OutputUnit='mL';
```

```
%Using KI, K2, K3 as estimation data sets and K6 as validation data set
%merging all estimation iddata to a single iddata object;
%This is for all the measurements that will be considered for the model extraction.
Overall_iddata=merge(SystemEperiment1, SystemEperiment2, SystemEperiment3,
SystemEperiment8, SystemEperiment11, SystemEperiment12, SystemEperiment17,
SystemEperiment18, SystemEperiment19, SystemEperiment20, SystemEperiment21,
SystemEperiment25, SystemEperiment16, SystemEperiment22, SystemEperiment23,
SystemEperiment24, SystemEperiment26);

                %VALIDATION PROCESS

 %merging data to a single iddata object;
%This is for all the measurements that will be considered for the model extraction.
Overall_iddata_val=merge(SystemEperiment4,SystemEperiment5, SystemEperiment6,
SystemEperiment9, SystemEperiment10, SystemEperiment13, SystemEperiment14,
SystemEperiment15);
%Overall_iddata=merge(SystemEperiment1,SystemEperiment2, SystemEperiment3,
SystemEperiment7, SystemEperiment8, SystemEperiment11, SystemEperiment12,
SystemEperiment17, SystemEperiment18, SystemEperiment19, SystemEperiment20,
SystemEperiment21,SystemEperiment25, SystemEperiment4,SystemEperiment5,
SystemEperiment6, SystemEperiment9, SystemEperiment10, SystemEperiment13,
SystemEperiment14, SystemEperiment15, SystemEperiment16, SystemEperiment22,
SystemEperiment23, SystemEperiment24,SystemEperiment26);

%Estimation data and validation data have been entered and two iddata objects have been
created:
%Overall_iddata contains the experiment results for the model identification process.
%Overall_iddata_val contains the experiment results for the model validation.
ze= Overall_iddata;
zv=(Overall_iddata_val);


%NALRX MODEL


%ms1 = nlarx(ze, [ones(1,1), ones(1,4), ones(1,4)], wavenet)
%ms2 = nlarx(ze, [ones(1,1), ones(1,4), 2*ones(1,4)], wavenet)
%ms3 = nlarx(ze, [2*ones(1,1), ones(1,4), 2*ones(1,4)], wavenet);
%ms4 = nlarx(ze, [2*ones(1,1), 2*ones(1,4), 2*ones(1,4)], wavenet)
%ms5 = nlarx(ze, [3*ones(1,1), ones(1,4), ones(1,4)], wavenet)
%ms6 = nlarx(ze, [3*ones(1,1), 3*ones(1,4), ones(1,4)], wavenet)

%ompare(zv,ms1, ms2, ms3, ms4, ms5, ms6)



%The numbers of units (wavelets) of the two WAVENET estimators have been automatically
chosen by the estimation algorithm. These numbers are displayed below. Notice the
abbreviations 'nl'='Nonlinearity' and 'num'='NumberOfUnits'
%mx4.Nonlinearity(1).NumberOfUnits %using full property names
```

```
%nanbnk = [2*ones(1,1), 2*ones(1,4), 2*ones(1,4)];


%The numbers of units (wavelets) of the two WAVENET estimators have been automatically chosen by the estimation algorithm. These numbers are displayed below. Notice the abbreviations 'nl'='Nonlinearity' and 'num'='NumberOfUnits'
%mx4.Nonlinearity(1).NumberOfUnits %using full property names


%The number of units in the WAVENET estimators can be explicitly specified instead of being automatically chosen by the estimation algorithm:
%ms7 = nlarx(ze, nanbnk, [wavenet('num',9)])
%ms8 = nlarx(ze, nanbnk, [wavenet('num',11)])
%ms9 = nlarx(ze, nanbnk, [wavenet('num',12)])


%compare(zv,ms7, ms8, ms9)

%Nonlinear ARX Model - Trying Other Nonlinearity Estimators

%ms10 = nlarx(ze, nanbnk, treepartition);

%The SIGMOIDNET estimator can also be used. Estimation options such as maximum iterations (MaxIter) and iteration display can be specified using NLARXOPTIONS command.
%opt = nlarxOptions('Display','on');
%opt.SearchOption.MaxIter = 2;
%ms11 = nlarx(ze, nanbnk, sigmoidnet);

%compare(zv,ms7, ms10, ms11)


%Nonlinear ARX Model - Trying Other Nonlinearity Estimators

%ms2 = nlarx(ze, nanbnk, treepartition);

%The SIGMOIDNET estimator can also be used. Estimation options such as maximum iterations (MaxIter) and iteration display can be specified using NLARXOPTIONS command.
%opt = nlarxOptions('Display','on');
%opt.SearchOption.MaxIter = 2;
%ms3 = nlarx(ze, nanbnk, sigmoidnet);

%compare(zv, ms2, ms3)

%ms4 = nlarx(ze, nanbnk, sigmoidnet ('NumberOfUnits',5));

%compare(zv, ms3, ms4)
```

```
%Function PLOT may be used to view the nonlinearity responses of various models.
%plot(mx1, mx2, mx3)



%NLHW Model

%mhws1=nlhw(ze, [2*ones(1,4), 3*ones(1,4), zeros(1,4)], 'saturation', 'deadzone');
%compare(zv, ms1, mhws1)


%mhws1 = nlhw(ze, [ones(1,4), ones(1,4), zeros(1,4)], 'pwlinear', 'pwlinear');
mhws2 = nlhw(ze, [2*ones(1,4), 2*ones(1,4), 2*zeros(1,4)], 'pwlinear', 'pwlinear');
%mhws3 = nlhw(ze, [3*ones(1,4), ones(1,4), zeros(1,4)], 'pwlinear', 'pwlinear');


%compare(zv,mhws1, mhws2, mhws3)
%compare(zv,mhws2)
%mhws4 = nlhw(ze, [ones(1,4), ones(1,4), zeros(1,4)], 'unitgain', 'deadzone');
%mhws5 = nlhw(ze, [2*ones(1,4), 2*ones(1,4), 2*zeros(1,4)], 'unitgain', 'deadzone');
%mhws6 = nlhw(ze, [3*ones(1,4), ones(1,4), zeros(1,4)], 'unitgain', 'deadzone');


%compare(zv,mhws4, mhws5, mhws6)

%mhws7 = nlhw(ze, [ones(1,4), ones(1,4), zeros(1,4)], 'saturation', 'deadzone');
%mhws8 = nlhw(ze, [2*ones(1,4), 2*ones(1,4), 2*zeros(1,4)], 'saturation', 'deadzone');
%mhws9 = nlhw(ze, [3*ones(1,4), ones(1,4), zeros(1,4)], 'saturation', 'deadzone');


%compare(zv,mhws2, mhws5, mhws8)
%compare(zv,ms7,mhws2)


%AIC = aic (ms7, mhws2)
```

Appendix C: Published Conference Paper