

**DESIGN AND STARTUP OF A SEMI PILOT SCALE  
REACTOR FOR HIGH SOLIDS ANAEROBIC DIGESTION  
OF SEWAGE SLUDGE**



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It is certified that the contents and forms of the thesis entitled

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TO

## OUR BELOVED PARENTS

FOR THEIR CONSISTANT LOVE SUPPORT AND  
ENCOURAGEMENT

*By virtue of whose prayers, we have been able to  
attain this position and whose hands are always  
raised for prayers, for our well-being.*

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## LIST OF ABBREVIATIONS

<b>Abbreviation</b>	<b>Description</b>
AD	Anaerobic Digestion
C/N ratio	Carbon Nitrogen Ratio
COD	Chemical Oxygen Demand
g	Gram
kg	Kilogram
m <sup>3</sup> /kg	Meter Cube per Kilogram
mg/L	Milli gram per liter
OLR	Organic Loading Rate
HRT	Hydraulic Retention Time
TS	Total Solids
VS	Volatile Solids
VFA	Volatile Fatty Acids
ALK	Alkalinity
TS	Total Solids
VS	Volatile Solids
%	Percentage
d	Day
°C	Degree Celsius
L	Liter
m	Meter

## **Abstract**

The world's population is increasing and resulting into production of billions of tons of waste every year, including sludge and wastewater. Sludge produced in waste water treatment plants is wasted or applied directly on agricultural lands. The drawback of direct application of untreated sewage sludge on agricultural land is increase in pollutant load (including heavy metals, organic compounds and pathogens. Consumption of these commodities by human beings and animals can cause health hazards to them. High cost of mineral fertilizers and escalating trends in their prices results in application of sewage sludge to agricultural soils and is sustainable and economical. Recognizing the need to address these issues, we designed a semi pilot scale reactor for anaerobic digestion of high solids wastewater sludge due to its cost effective treatment and less space requirement. Many sludge treatment options are available but most of them require high energy and space requirements and hence we opted for anaerobic digestion of high solids sewage sludge due to its cost effectiveness and less space requirements. The total solids content of the sewage sludge was increased by designing a sand drying bed for dewatering purposes. For the purpose of high solids anaerobic sludge digestion (HASD), a semi-pilot single stage mesophilic reactor was designed. For the startup phase, the reactor was loaded with sludge and inoculum (cow dung) mixture of the ratio 1:1. Startup phase proceeded well as indicated by biogas production, the phase ended after 30 days. The semi continuous phase started with sludge feeding into the reactor; meanwhile the reactor parameters were monitored. The biogas production did not increase considerably during this phase; this was due to the fact that C/N ratio of sludge is very low. In order to bring the C/N into optimum range, a suitable mixture of sludge, paper waste and food waste having the ratios of 60%, 35% and 5% respectively was prepared and fed into the reactor. The adjusted C/N of mixture was 28, which resulted into the high production of biogas up to 1.3L/L.d. Hence we came to our conclusion that sludge alone can't be used as a substrate as it has low C/N ratio, in order to achieve high biogas production, sludge needs to be codigested along with other substrates during the semi-continuous phase.

# Chapter 1

## Introduction

### 1.1 Background of the Study

Pakistan, once a water-surplus country, is now a water deficit country due to the depleting ground water and surface water resources, prevailing droughts and shift of water from agriculture to domestic and industrial use. According to a study, it is recognized that water availability in 1996-97 was 1,299 m<sup>3</sup> per capita and has decreased to 1,100 m<sup>3</sup> per capita in 2006 and it is projected to recede to be less than 700 m<sup>3</sup> per capita by 2025. Total discharge of wastewater for 14 major cities of Pakistan, computed on the basis of 1998 population census is about  $1.83 \times 10^7$  m<sup>3</sup> h<sup>-1</sup> (FAO, 2002). Latest estimates reveal (PWSS, 2002 ) that total quantity of wastewater produced in Pakistan is 962,335 million gallons ( $4.369 \times 10^9$  m<sup>3</sup>/yr) including 674,009 million gallons ( $3.060 \times 10^9$  m<sup>3</sup>/yr; a figure of  $5.54 \times 10^9$  m<sup>3</sup>/yr for the year 2011) from municipal and 288,326 million gallons ( $1.309 \times 10^9$  m<sup>3</sup>/yr) from industrial use.

In order to get a clean effluent that can be reused for many purposes to avoid the usage of fresh water and which can in turn be safely discharged to watercourses, the municipal waste water typically undergoes three or four distinct stages of treatment in waste water treatment plants to remove harmful contaminants. Preliminary wastewater treatment removes coarse solids and grit through screens and other filtering devices. Primary wastewater treatment usually involves gravity sedimentation of screened wastewater to remove settled solids. Half of the solids suspended in wastewater are removed through primary treatment. The residual material from this process is a concentrated suspension called primary sludge, which will undergo further treatment to become bio solids. Secondary sludge separated in secondary clarifiers includes treated sewage sludge from secondary treatment bioreactors (Droste, 1997). Sludge accumulated in the facility during the primary and secondary sedimentation processes contains a high concentration of organic

material that must be stabilized in order to be used as a raw material for organic fertilizer.

The disposal of sewage sludge is a big issue in municipalities. Consequently the valuable energy stored in organic content of sludge is lost without utilization in energy cycle (Kelessidis et al., 2012). Open dumping of sludge or application of untreated sludge on agricultural lands and farms is very dangerous to human health and it has numerous effects on environment as well. Results revealed that some reported health-related symptoms were statistically significantly elevated among the exposed residents, including excessive secretion of tears, abdominal bloating, jaundice, skin ulcer, dehydration, weight loss, and general weakness (Udom et al., 2004). The frequency of reported occurrence of bronchitis, upper respiratory infection, and giardiasis were also statistically significantly elevated. The findings suggest an increased risk for certain respiratory, gastrointestinal, and other diseases among residents living near farm fields on which the use of bio solids was permitted weakness (Udom et al., 2004). To reduce these health effects or to minimize to them to acceptable levels treatment of untreated sewage sludge is necessary. There are currently many methods present for the treatment of sewage sludge but in developing countries including Pakistan, little work is being conducted on this very important research area (Winkler, 1993). Limited information is available on the physical treatment of sewage sludge. This issue can be tackled by adopting sustainable methods for the treatment of organic sludge as the sludge can be used as a source of energy as well as nutrients. Infact, composting and anaerobic decomposition of organic sludge are the best options to be considered (Kosobucki et al., 2000). As in case of anaerobic decomposition energy in the form of biogas is produced. Anaerobic digestion involves a considerable reduction in the organic load, thanks to the biological reactions, and the pollutant load of the digested sludge. It is thus, a complete de-pollution. A correctly controlled anaerobic digestion leads to very high rates of purification. Moreover anaerobic digestion is becoming a popular method to produce biogas which is a huge source of renewable energy (Nijaguna, 2002). Biogas is increasingly becoming an attractive source of energy all over the world (Lise, 2008). Instead of fossil fuel, using biogas to meet our energy requirement, we can reduce greenhouse gas emissions in the atmosphere and can

resolve the problem of global warming. By developing strategies on a national level, which encourages the use of renewable energy sources like biogas, we can reduce the dependency on fossil fuels and can attain energy self-sufficiency, particularly in rural areas. The main benefit that biogas technology provides is its ability to convert waste to energy. Thus, by using this technology, we can somehow deal with the problem of sludge management in an effective manner (Holm-Nielsen et al., 2009).

A huge amount of domestic wastewater sludge is generated in the wastewater treatment plants operating in the world. The sludge has very high content of organic waste which has got a greater potential of energy production. High Solids Anaerobic Digestion often referred as dry anaerobic digestion is the type of anaerobic digestion where the feedstock used in the digester has a high content of solids. It differs from the customary wet digester that has liquid feedstock and the materials are mixed with moving parts. We used semi-batch style approach where the loaded material remains stationary during the anaerobic digestion. The major advantage of dry anaerobic digester is the ease in recovery of energy from almost every kind of waste. The anaerobic conditions and the moderate heat provide ideal conditions for the anaerobic microbes to breakdown the waste for the production of biogas. The high solids anaerobic digestion produces high yields of biogas and it also has a very low cost of operation both in cases of small and large volume digester. By using the dry anaerobic digestion technique, a very large amount of space and volume is saved as the moisture content is very low (Bendixen, 1999; Nielsen and Petersen, 2000).

## **1.2 Problem Statement**

This study is based on the design and establishment of a pilot scale reactor for anaerobic digestion of high solids sewage sludge and its optimization of different operational parameters: operating temperatures, operating pH inside the reactor, ammonia, volatile fatty acids.

## **1.3 Objectives of the Study**

- Dewatering of low solids sewage sludge
- Design and establishment of pilot scale reactor for dry anaerobic digestion.
- High solids (dry) anaerobic digestion of sewage sludge

#### **1.4 Scope of the Study**

- The study was conducted at semi-pilot scale.
- Sewage sludge from MBR treatment plant of NUST H-12 campus was used as substrate.
- Cow dung from a farm was utilized as an anaerobic inoculum in this study.
- Size of digester was decided to be of 50L and was made up of stainless steel.
- Reactor designed for carrying out the study was a single stage anaerobic digester.
- Mesophilic conditions i.e. temperature of 35–40° C was maintained inside the reactor.

## **Literature Review**

In this chapter some basic facts and defined parameters of biogas are discussed. The main objective of this chapter is to provide some basic information which is helpful for the interpretation of results in the coming chapters.

### **2.1 Background**

One of the major problems that we are facing today is lack proper solid waste management and disposal. The population rise is resulting in increased production of billions of tons of waste, including sludge and wastewater. The sewage sludge that is produced in waste water treatment plants is wasted or applied directly to agricultural lands.

Direct application of raw sludge to agricultural land results in many problems some of which include increased pollutant load on agricultural lands, health hazards to humans and animals, contamination of ground water and direct methane emissions into the air.

Shooting prices of mineral fertilizers is trending these days. This results in forcing farmers to opt for less expensive options of fertilizers or soil enhancements. Sewage sludge application on land is being practiced widely now. This proves to be a sustainable and an economical option. Stabilized sewage sludge proves to be a desirable option for farmers as compared to costly fertilizers that are available in the market.

Many sludge stabilization methods and processes are proposed so far which result in inactivation of potential pathogens prior to their disposal on land or in water bodies (Timothy et al., 1993).

### **2.1 Origin of the Sludge**

Sludge is generated during the process of waste water treatment. The treatment process involves the separation of undesirable substances from water. The first step of screening involves the removal of larger particles from wastewater. Other treatment may be biological or chemical. Sludge is formed when the larger particles clump together during suitable separation methods. (Casey, 1997) The sludge that

is separated during the treatment methods (mechanical, biological and chemical) is referred to as raw sludge, which requires various further treatments.

The wastewater sludge used in the study was recovered from NUST membrane bioreactor reactor treatment plant. It was waste activated sludge from biological treatment of wastewater.

## **2.2 Sludge Management and Treatment**

Several Sludge management techniques are proposed so far, some of which include digestion, incineration and direct disposal.

### **2.2.1 Direct Land Disposal**

The raw sewage sludge application has effects on the heavy metal content in soils and grasses (Wang, et al., 2008).

### **2.2.2 Landfill Disposal**

Expenditure for development of landfill areas is getting higher due to the decrease of space in highly populated countries. So, disposal of sewage sludge by landfill is to be phased out (Ødegaard et al., 2002).

### **2.2.3 Incineration**

Incineration costs energy for dewatering and normally needs input of external fuel. Also, the disposal of ash and the treatment of exhaust gas are expensive (Ahring, 2003).

### **2.2.4 Digestion**

Multiple techniques of sludge management are suggested, some of these include direct disposal (Albrecht R. Bresters, 1997), incineration (Luts et al., 2000) and digestion including both aerobic (Gregor D. Zupančič, 2008) and anaerobic. (Dubrovskis et al., 2010)

## **2.3 Total Solids of Sludge**

### **2.3.1 Low Solids Anaerobic Digestion**

Low solids sludge comprises of high water content and low concentrations of solids. The low solids digestion process involve larger volume of reactor. Operation of the anaerobic digestion -process at low solids increases the level of process water



and thereby the size and capital costs for the digester system. (Rivard and Christopher, 1995)

### **2.3.2 High Solids Anaerobic Digestion**

Higher the total solids content in sludge, more will be the biogas production per unit volume of the reactor. (David Bolzonella et al., 2005) The application of high-solids technology may offer several advantages over conventional low-solids digester technology.

### **2.4 Anaerobic Digestion Process**

So in the light of above discussion, anaerobic digestion is universal and sustainable amongst all sludge disposal and treatment methods. As this method can stabilize the organic materials by the reduction of the number of pathogens (Bendixen, 1999; Nielsen and Petersen, 2000), energy recovery and capability of recycling the plant nutrients back to the soil. Hence the method complies with the present day sludge management policies and is worth of further study.

The sludge has two main components; liquid and solids. The liquid phase of course contains water but also dissolved substances. Dry solids are the matter that remains as residue after evaporation and drying

In anaerobic digestion process biogas is produced in a process in which organic matter is breakdown in micro-organism in the absence of oxygen (Hessami et al., 1996). The chemical reaction that takes place is as under (Vesilind et al., 2002).



There are multiple sources of Biogas production like MSW, sewage sludge, crop residues, animal manure, etc (Igoni et al., 2008). The composition and characteristics of biogas are given in Table 2.1 (FAO, 1996; Deublein, 2008; Majid, 2006).

Table 2.1 Composition of Biogas

<b>Gas</b>	<b>Percentage</b>
Methane (CH <sub>4</sub> )	50 - 70%
Carbon dioxide (CO <sub>2</sub> )	30 - 50%
Nitrogen (N <sub>2</sub> )	1%
Hydrogen (H <sub>2</sub> )	0.1 - 0.5%

Carbon monoxide (CO)	0.1%
H <sub>2</sub> S	Traces

Table 2.2 Characteristics of biogas

Characteristics	Value
Density	1-2 kg/ m <sup>3</sup>
Calorific value	20 MJ/ m <sup>3</sup>

In an anaerobic process, biodegradable organic matter, both soluble and particulate are converted to methane and carbon dioxide. Anaerobic digestion of solid waste and / or waste water sludge has long been used to stabilize organic wastes prior to final disposal of the organic wastes. However, due to the involvement of the complex microbial ecosystem and sensible operational stability, it has continued to be the subject of research and new process development (Grady, 2011). Moreover, in our increasingly energy conscious society, generation of valuable by-product i.e. methane along with the waste treatment is very significant.

Anaerobic digestion is used extensively for the stabilization of biodegradable particulate organic matter. Apart from this, destruction of pathogens is important when bio-solids are used. The measure of percentage of VS reduction can be used as a parameter to estimate the performance of the digesters. It is estimated that 80 to 90% of the influent biodegradable particulate organic matter will be converted to methane when an SRT of 15 to 20 days is provided. (Grady, 2011).

This corresponds to destruction of about 60% of the VS contained in primary solids and 30 to 50% of the VS contained in waste activated sludge. (Grady, 2011).

There are several advantages of anaerobic waste treatments over aerobic treatment systems. Some are listed below. (Henze, 2008).

- A reduction in excess sludge production up to 90%.
- Up to 90% reduction in space requirement when using expanded sludge bed systems.

- High applicable COD loading rates reaching 20-35 kg COD per m<sup>3</sup> of reactor per day, requiring smaller reactor volumes.
- No use of fossil fuels for treatment, saving about 1 kWh/kg COD removed, depending on aeration efficiency.
- Improved sludge de-waterability
- No or very little use of chemicals
- Plain technology with high treatment efficiencies
- Generation of a potentially valuable by-product (methane) which is nearly 13.5 MJ CH<sub>4</sub> energy/ kg COD removed.
- Excess sludge has a market value.
- No oxygen is required.

Disadvantages of the process are (Tchobanoglous, G, *et al.*: 2003)

- May require alkalinity addition.
- Longer start-up time to develop necessary biomass stock.
- May require further treatment with an aerobic treatment process to meet discharge requirements.
- Biological nitrogen and phosphorus removal is not possible.
- Much more sensitive to the adverse effects of lower temperatures on the reaction rate.
- May be more susceptible to upsets due to toxic substances.
- Potential for production of odors and corrosive gases.

## **2.5 Dewatering**

Dewatering means that the volume of the sludge is greatly reduced by separating water. Raw sludge contains high amounts of water, usually more than 95% by weight (Casey, 1997). Different dewatering processes are drying beds, centrifuging, filter belt and filter press.

### **2.5.1 Centrifuge**

Centrifuge can be used for sludge dewatering but is an energy intensive technique.

### **2.5.2 Sand Beds**

Sand beds are cost effective devices which work under the effect of gravity while the sludge is recovered in the form of a cake above the head space of the filter.

## **2.6 Reactor Types**

Massey and Pohland (1978), Ghosh and Klass (1978), and Cohen et al. (1980) have all demonstrated improved process performance by TPDS systems, which optimize environmental conditions for each phase when compared with single-phase processes, in which both classes of organisms are forced to operate in a common suboptimal environment.

### **2.6.1 Two Phase, Two Stage**

A two stage reactor is easy to be maintained by controlling the parameters of each component. It consistently outperformed the single-stage systems. (Azbar and Speece, 2001)

### **2.6.2 Single Stage**

A single stage reactor was constructed for the purpose of anaerobic digestion as it requires comparatively less cost.

## **2.7 Operating Conditions**

There are multiple functioning parameters that effect the biogas production. Important parameters that really affect the biogas generation are pre-treatment, temperature, pH, rate of organic load, agitation, particle size, retention time etc. Sudden changes in these parameters can harmfully affect the biogas generation (Yadvika et al., 2004).

### **2.7.1 Temperature**

Temperature within the digester is one of the most important factors that extremely affect the process of biogas production. Naturally methane is formed at three temperature ranges that are psychrophlic (<25 °C), mesophilic (25–45 °C), and thermophilic (50–70 °C) (Yadvika et al., 2004).

The operating temperature plays a significant role in all the biological processes including anaerobic treatment process. Generally, anaerobic reactors are operated in the mesophilic temperature range i.e. 30 to 40 °C or thermophilic range i.e. 50 to 60 °C. These two regions represent the optima for growth of the methanogens. On the

other hand, it is possible to grow methanogens at lower temperatures by providing longer SRTs to compensate for the lower maximum specific growth rates. Different species of bacteria are involved in the anaerobic process. So, temperature effect is important in anaerobic systems because of the interacting populations. For example, different species of bacteria respond to changes in temperature in qualitatively similar but quantitatively dissimilar ways. Operating temperature affects both hydrolytic and acidogenic reactions in addition to the methanogens. The effect of temperature on methanogenesis is the primary concern for wastewater consisting largely of simple, readily biodegradable organic matter.

#### **2.7.1.1 Thermophilic**

Anaerobic microorganisms are more energetic in mesophilic and thermophilic temperature ranges (Yadvika et al., 2004). Higher temperature range supports the higher methane yield and higher degradation of organic matter which is helpful in decreasing the volume required for any particular substrate. Anaerobic digestion at higher temperatures kills harmful microorganisms thus supports better sanitation. As degradation is high at thermophilic temperature range so it is very sensitive to any change like ammonia inhibition (Yadvika et al., 2004).

#### **2.7.1.2 Mesophilic**

The operational and maintenance cost for thermophilic process are higher as compared to mesophilic process due to their heat requirements (Demetriades, 2008). Whereas mesophilic digestion requires higher retention time around 30 to 40 days as compared to thermophilic process which needs only 15 to 25 days. Also gas production is delayed in mesophilic range than in thermophilic (Avfall, 2009).

#### **2.7.2 pH**

As pH is a measure of acidity present in substrates so it is very important for the growth of microbes during anaerobic digestion. The pH range between 6.0 –8.0 within the digester is the optimum pH for better performance of microorganisms (Yadvika et al., 2004). So microbes and their enzymes can best survive in the above mentioned range and any change in pH value which is either above or below this range can severely inhibit the process of anaerobic digestion (Yadvika et al., 2004). During the process of anaerobic degradation of organic matter sometimes a situation arises in which pH

is adversely affected by multiple reasons like high values of VFA (Volatile Fatty Acid), CO<sub>2</sub>, acetic acid, and ammonia. Any pH change due to these factors negatively affects bacterial activity and can restrain the digestion process (Nijaguna, 2002; Yadvika et al., 2004).

### **2.7.3 Carbon to Nitrogen (C/N) ratio**

For effective functioning of biogas plant, influent substrate must contain carbon to nitrogen ratio in the preferred range because balance in nutrient composition can affect bacterial growth and its activity (Nijaguna, 2002). The important nutrients for anaerobic microorganisms are carbon and nitrogen. For the anaerobic digestion, the value of carbon is always 20-30 times greater than nitrogen. Therefore, for best working of microbes C/N ratio should be 20-30:1 with the major portion of carbon which can be easily degradable. Any deviation in this ratio results in low efficiency of process. Mixing variety of different substrates altogether in a single digestion process improves the nutrient balance and fulfill the requirements of missing nutrients and results in high biogas production (Nijaguna, 2002). Due to this reason cow dung is usually mixed with other organic waste like industrial and household wastes to optimize the digestion process and increase the production of biogas (IEA, 2005, Nijaguna, 2002). Cow manure has its C/N ratio around 16 – 25 (Nijaguna, 2002).

### **2.7.4 Solids Retention Time**

Solids retention time (SRT) is a fundamental parameter which controls the types of microorganisms that can grow in the process and the extent to which reactions will occur. SRT equals to the HRT in flow through systems such as anaerobic digesters. SRT is increased relative to the HRT in some systems by recycling solids back to the system. Generally, SRT of pilot scale anaerobic treatment systems range from 30 to 40 days but it can range up to more than 100 days depending upon the system.

### **2.7.5 Organic loading rate**

Organic loading rate (OLR) refers to the amount of substrate daily fed to the digester and mostly it is stated in kilogram volatile solids per meter cube per day (kg VS/m<sup>3</sup>/day). Gas produced per day in digester highly depends on its OLR (Yadvika et al., 2004).

### **2.7.6 Ammonia**

For anaerobic processes, ammonia concentrations between 50 and 200 mg/l as N are generally within the stimulatory range. However, ammonia is inhibitory at higher concentrations and toxic if the concentration is high enough. Ammonia is a weak base and dissociates in water.

### **2.7.7 VFA**

A study was conducted to determine whether differences in the levels of volatile fatty acids (VFAs) in anaerobic digester plants could result in variations in the indigenous methanogenic communities. (Ingrid et al., 2014)

VFA concentrations above 2000 mg/l can be inhibitory to methanogens. Volatile acids are weak acids that are largely dissociated at neutral pH. (Kayhanian, 1994)

### **2.7.8 Alkalinity**

Digestion stability depends on the buffering capacity of the digester contents. Higher ALK values indicate a greater capacity for resisting pH changes. ALK value in an anaerobic digester can range between 1500 and 5000 mg/L. (Marchaim and Krause 1991).

### **2.7.9 Inoculum to Substrate Ratio**

Batch digestion experiments have been mentioned in Literature review to have been conducted at inoculums/substrate ratio of three different ratios (ISRs) 0.3, 0.5 and 1. The biogas production increased as the (ISRs) value decreased. (Fathya et al., 2014)

## **2.8 Modes of Operation**

Mainly two modes of operations are used to operate digesters. The two most commonly used techniques are digestion under batch and continuous mode. Both types are explained below.

### **2.8.1 Batch digestion**

Digesters operating under batch mode are once fed with feedstock, inoculum to start digestion and in rare cases with some chemical to buffer pH changes. Then the digester is sealed and allowed to start the process of digestion (Nijaguna, 2002). In batch reactors material is completely filled once for digestion and after the completion of process the whole filled material is removed from reactor. The production of gas

slowly builds up in digester which after certain period of time reaches its peak and then production starts declining, forming a bell shape curve (Nijaguna, 2002). Though this technique supports easy handling of material, there is great difference in gas production both ways quality wise and quantity wise. The batch digestion also supports the high amount of material degradation if the digestion time is increased (Demetriades, 2008).

### **2.8.2 Continuous digestion**

In continuous digesters material is added and removed daily (Demetriades, 2008). During the process the newly fed material pushes out the equal amount of old fed material from the digester whose retention time is almost completed, thus maintaining the constant digester volume. Continuous type digesters provides the constant material feeding which then supports the consistency and stable biogas production in comparison to batch type digesters (Demetriades, 2008).

Hence the focal point of all the extensive literature review came down to the research topic anaerobic digestion of high solids sewage sludge under mesophilic conditions.

## **2.9 Methane Production Mechanism in anaerobic Digestion**

Three groups of bacteria viz. acidogens, acetogens and methanogens are involved in the biological anaerobic process and complex interactions of each species of bacteria are involved for the success of process. The process is generally considered to be four successive stages biological processes; i) hydrolysis, ii) acidogenesis iii) acetogenesis, and iv) methanogenesis involving waste conversion and stabilization. (Jarvis, 2004). The end products are principally methane (CH<sub>4</sub>), Carbon dioxide (CO<sub>2</sub>), and stable organic residues. These processes are further discussed below.



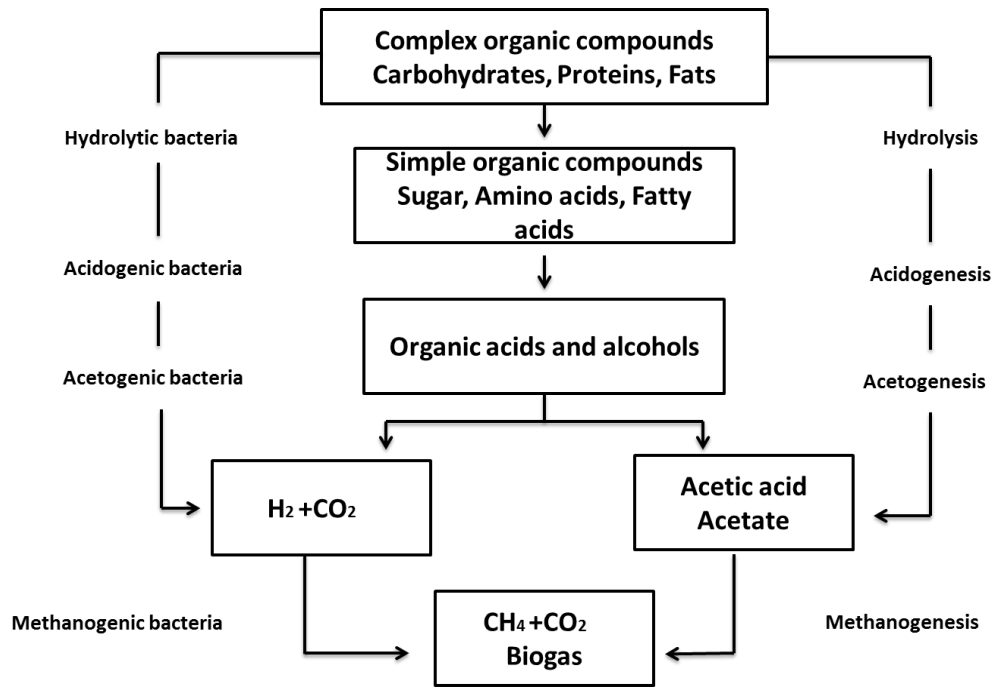


Figure 2.1. Mechanism of Anaerobic Digestion Process

### 2.9.1 Disintegration

Disintegration is the initial step involved in an anaerobic process. In this step, the anaerobic degradation of complex polymers and particulates (i.e. colloidal 10 - 1000 nm or larger particles > 1000 nm) takes place mainly by physical shearing and dissolution while some extracellular enzymes may also be involved (Kommedal, 2011). Composite particulate organic materials are disintegrated into its constituent products; carbohydrates, proteins, lipids and inerts.

### 2.9.2 Hydrolysis

The step of hydrolysis comprises of enzyme-facilitated change of insoluble organic compounds that has high molecular mass, i.e., fats, carbohydrate, lipids, and proteins etc., into soluble organic components such as, fatty acids, monosaccharide, amino acids and variety of other simple organic compounds (Yadvika et al., 2004). The large molecules that are not soluble comprises several small molecules linked with each other in a tight chemical bond and therefore must be broken down prior to enter into the cell of bacteria. Many different facultative and anaerobic bacteria carry out the process of hydrolysis (Yadvika et al., 2004). Acidogenesis is the most rapid conversion step in the anaerobic food chain, and the growth rate of fermenters.

### **2.9.3 Acetogenesis**

After acidogenesis process the short chain fatty acids (SCFA), more degraded by a variety of facultative anaerobes in different processes of fermentation.  $H_2$ ,  $CO_2$ , organic nitrogen compounds, organic sulphur compounds, alcohols and other organic acids are the products of fermentation process (Gerardi, 2003). This phenomenon is known as acetogenesis. Here at this stage production of acetic acid is the most important as it is the major organic acid used as a feed for methane-forming bacteria (Gerardi, 2003).

At this stage, those products that were formed other than acetic acid in the process of fermentation in acidogenic phase are further transformed to acetic acid,  $CO_2$  and  $H_2$  in variety of anaerobic oxidation reactions facilitated by acetogenic bacteria. (Jarvis, 2004).

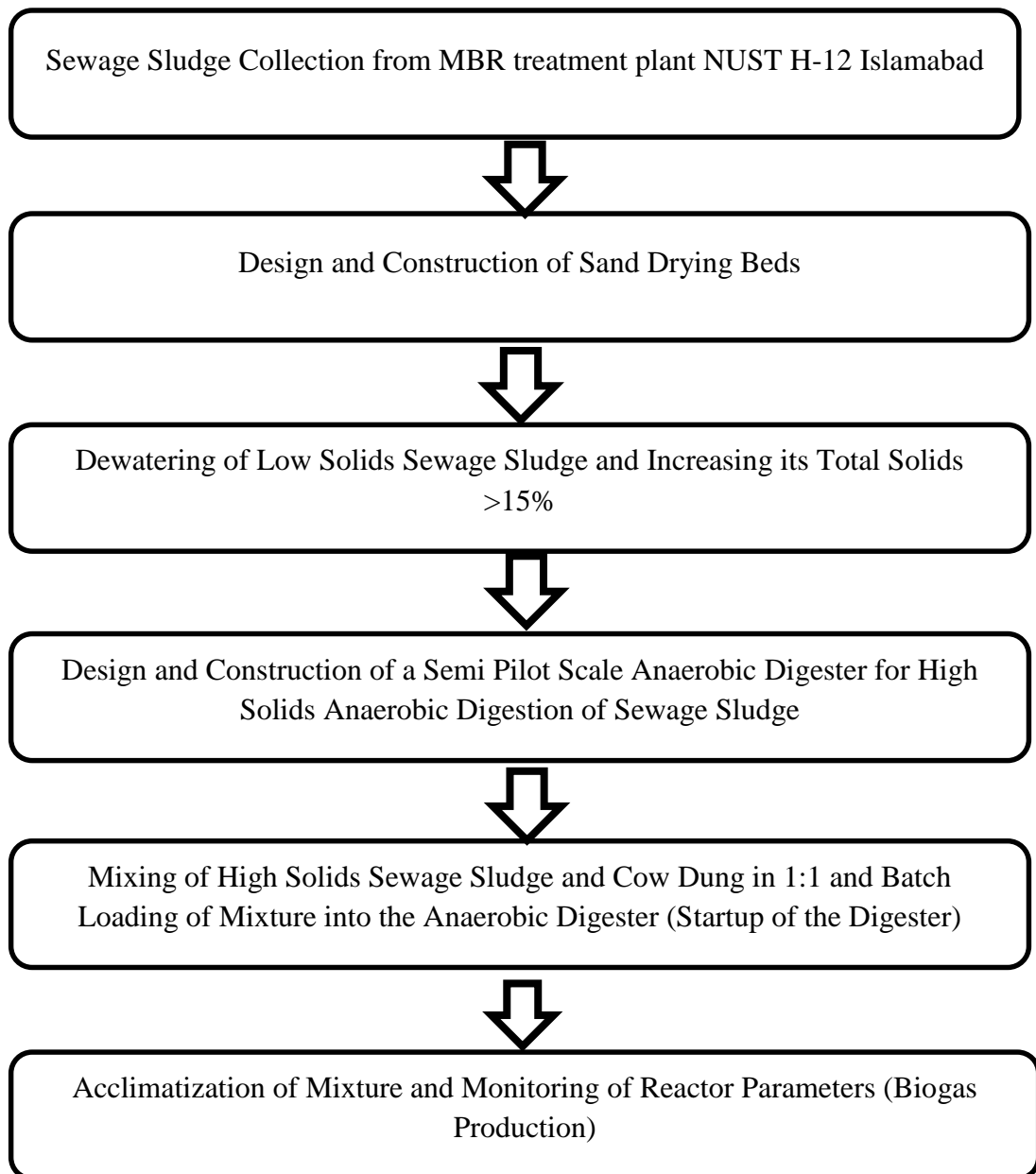
### **2.9.4 Methanogenesis**

Methanogenesis is the final stage in the overall anaerobic conversion of organic matter to methane and carbon dioxide. In this step, the products formed by acetogens are utilized by methanogens which reduce carbon dioxide using hydrogen to form  $CH_4$ . It is only in this stage, influent COD is converted to a gaseous form. (Henze et al., 2008). Methanogens exploit only certain specific substrates such as acetate, methylamines, methanol, formate, and  $H_2/CO_2$  or CO. Compounds like organic nitrogen; alcohols etc that are left over and cannot be converted by methanogens are collected in digestate (Gerardi, 2003).

### Materials and Methods

In this chapter, Methodology for anaerobic digester setup for high solids anaerobic digestion of sewage sludge, its operational parameters and other analysis of substrate, inoculum, digestate and biogas will be discussed.

Adopted methodology for experimental work is given below in figure 3.1



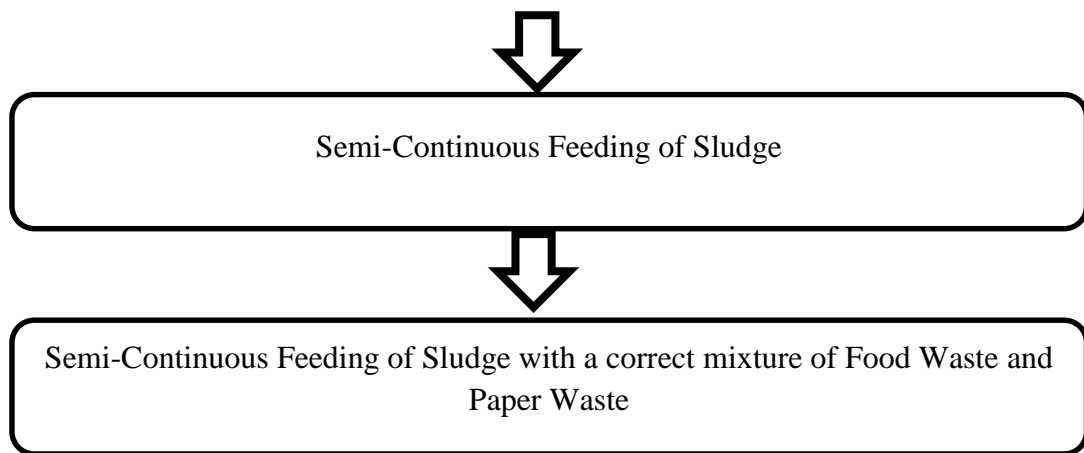


Figure 3.1. Methodology of experimental work carried out during the operation

### **3.1 Collection of Sewage Sludge and Cow Dung**

The sludge used in this experiment was collected from MBR treatment plant of NUST H-12 Campus, Islamabad. The fate of this sludge is open dumping in Pakistan though it still has high energy potential. Fresh cow dung was collected from a dairy farm. Sewage sludge was used as a substrate and cow dung was used as an inoculum source. After the collection, the waste was weighed using an electronic weighing scale. The total weight of cow dung used for the batch digestion was 16.5 kg and that of sludge used was 19 kg and for batch digestion. Cow dung and sludge were then mixed properly at I:S (inoculum to substrate ratio on weight VS) of 1.516/2.069 and loaded into the reactor for the batch mode.

### **3.2 Design and Construction of Sand Drying Beds**

Sand drying beds were made to decant the low solids sewage sludge to increase its total solids up to or greater than 15%. Three sand filters were constructed having total volume of 20 L including a head space of 5 L each. 5L of each was filled with coarse particles at the bottom, 5L with gravel in the middle whose size was smaller than coarse particles and 5 liter was filled with sand at the top. Above the sand, 5L head space was left for the dewatering of sludge.

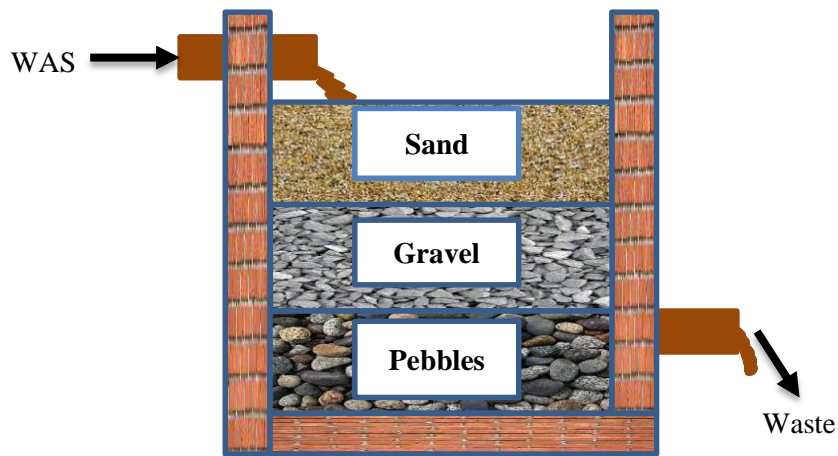


Figure 3.2.Sand filter constructed for dewatering purposes

### 3.3 Dewatering of Low Solids Sewage Sludge

Low solids sewage sludge was poured into the provided head space and allowed to decant for a day or two. Water from the sludge was filtered out and solid sludge in the form of a cake was then collected from the head space. Increased total solids sludge i.e. >15% was obtained.



Figure 3.3.High Solids Sewage Sludge

### 3.4 Design and Construction of Anaerobic Digester/Reactor

Stainless steel was used for the construction of anaerobic reactor in order to provide uniform heating amongst its contents. Another advantage of using stainless steel was to avoid corrosion and rusting by avoiding the reaction of its contents with the reactor body.

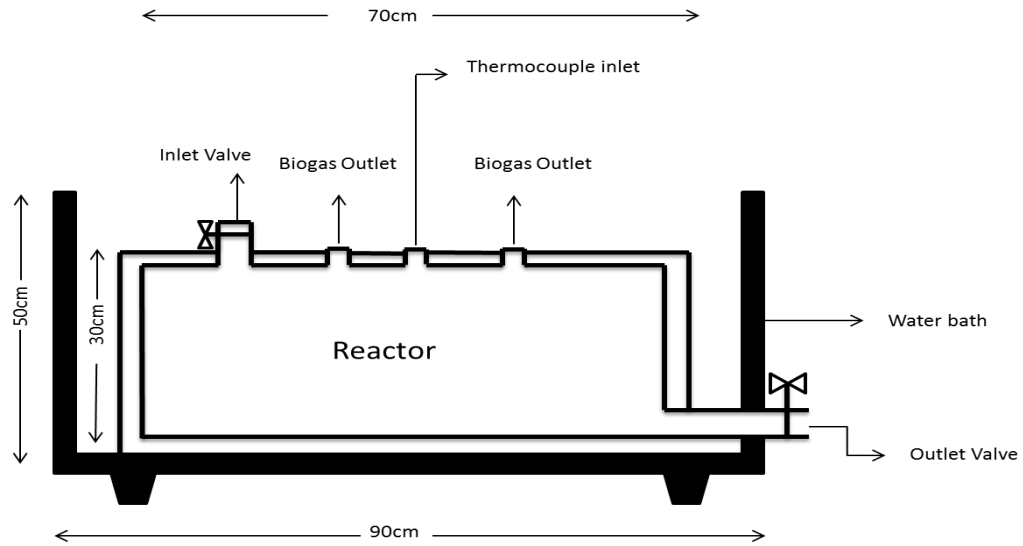


Figure 3.4. Engineering design of an anaerobic semi-pilot scale digestion reactor

### 3.4.1 Reactor Dimensions

The 50L reactor was designed in such a way that it was cylindrical in shape with an attached water bath in order to facilitate heating via immersion heater. The radius of the reactor 'R' was actually the height of the reactor as it was attached in a lateral position to the waterbath. Radius of reactor was 30cm while the appropriate length was calculated by using the equation as under:

$$V = \pi r^2 l$$

$$V = 50 \text{ L}$$

$$r = 30 \text{ cm}$$

$$\text{Hence } L = 70 \text{ cm}$$

### 3.4.2 Water Bath

The total length of assembly including the waterbath was 90cm. The waterbath was rectangular in shape having a height of 50cm. The waterbath was supplied with a temperature control system comprising of an immersion heater, temperature controller and a thermocouple whose probe was fixed on an outlet of the reactor.

### 3.4.3 Anaerobic Design

The reactor was designed in such a way that highly anaerobic conditions prevailed within it. The lid of the tank was tightly closed and sealed properly using a gas kit and silicones to avoid any loss due to leakage.

### 3.4.4 Reactor Inlet

Inlet was made in a vertical position in order to facilitate feeding of the reactor. A ball valve was installed above the inlet so as to avoid air from entering the reactor. The inlet diameter was kept to be 0.08m in order to ensure free flow of material within the body of the reactor during the feeding process.

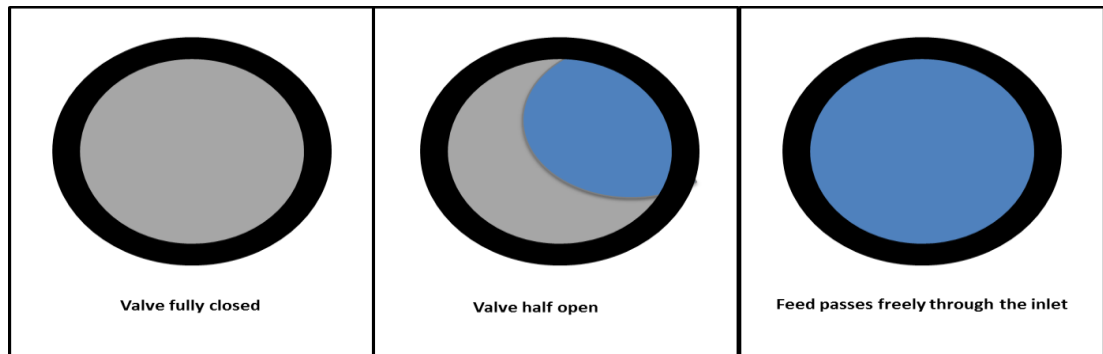


Figure 3.5. Top view of reactor inlet

### 3.4.5 Gas Outlets

The digester was provided with two gas outlets in order to collect the biogas produced. One of the gas outlets was made functional by connecting it with a biogas meter while the other one was sealed with a cork and was non-operational. The non-operational valve was left as a backup in case the operational valve becomes clogged with the material within the reactor.

A thermocouple inserting valve was also made for the maintenance of the temperature within the reactor. The thermocouple was connected to the display and relay of a temperature controller which was in turn connected to an immersion rod for temperature maintenance within the reactor.

### 3.4.6 Reactor Outlet

Reactor outlet diameter was kept to be 0.08m and anaerobic conditions were maintained as the contents were fully filled up to the outlet. The digestate for analysis was collected each day through the outlet during semi-continuous phase.

### 3.5 Mixing of High Solids Sewage Sludge and Cow Dung

High solids sewage sludge having T.S >15% was mixed with cow dung having T.S >28% in the ratio I:S (inoculum to substrate ratio on weight VS) of 1.516/2.069. The total weight of cow dung used for the batch digestion was 16.5 kg and that of sludge used was 19 kg. Sewage sludge and cow dung were mixed properly and a homogenized mixture was made.

### 3.6 Startup of the Anaerobic Digester

Startup of the digester was done by batch loading of this homogenized mixture into the anaerobic digester manually. Initial parameters including total solids, volatile solids, total carbon and pH of substrate, inoculum and mixture were measured before the startup.

Following table shows the initial characteristics of substrate and Inoculum.

Table 3.1 Characteristics of substrate and inoculum

<b>Parameters</b>	<b>Dried Sludge</b>	<b>Cow Dung</b>	<b>Mixture of sludge and cow dung</b>
<b>Total Solids</b>			
<b>T.S (%)</b>	15.01	28.0946	24.07
<b>Volatile Solids</b>			
<b>V.S (% of T.S)</b>	52.668	58.26	49.26



<b>pH</b>	11.1	8.95	9.03
<b>Moisture Content</b> <b>M.C (%)</b>	84.99	71.90	75.93
<b>Carbon Content</b> <b>C (%)</b>	29.26	32.36	27.77

### 3.7 Acclimatization and Monitoring of Reactor

After the batch loading of substrate-inoculum mixture, the mixture was allowed to acclimatize for 30 days. Reactor parameters i.e. temperature, pH, T.S, V.S, VFA, alkalinity, VFA/Alk ratio, Ammonia, were continuously monitored during this time span. Discussed below are the above mentioned parameters which were monitored and maintained during the experiment.

#### 3.7.1 Temperature control

Temperature effect can be classified under different categories; psychrophilic (0–20 °C), mesophilic (20–42 °C) and thermophilic (42–75 °C) which is shown in figure. Thermophilic anaerobic digestion has additional benefits as compared to mesophilic digestion such as high degree of waste stabilization, greater destruction of viral and bacterial pathogens, and improved post-treatment sludge dewatering (Lo et al., 1985, cited by Casey). However, due to the high heating energy requirement, operation in thermophilic range is not generally practical.

For temperature control, the digester was put in the water bath which was filled with water; a 1000 W electric immersion rod with thermostat was used to maintain the temperature to 35° C. A relay and a temperature sensor (thermocouple) were used to maintain the temperature inside the digester at 35° C.

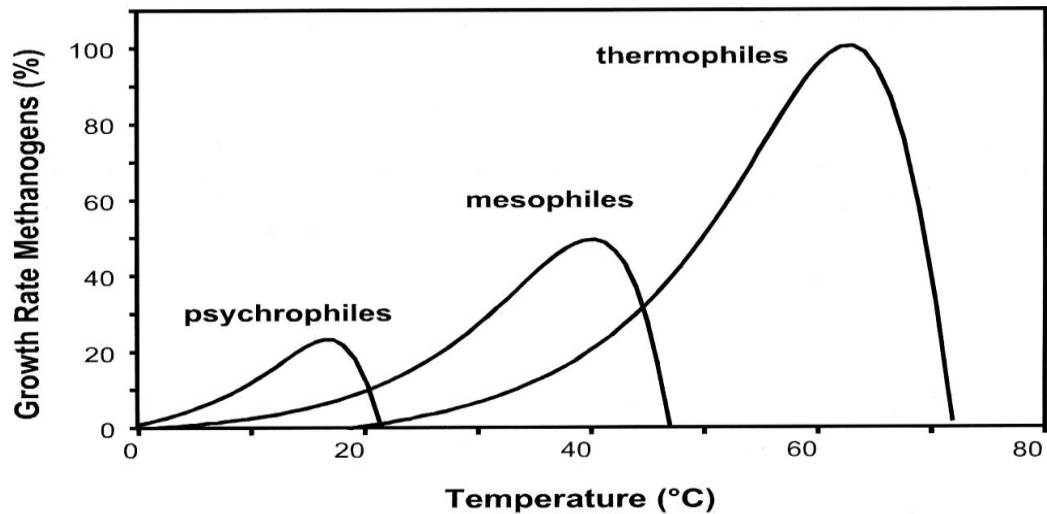


Figure 3.6. Effect of Temperature on Microorganisms

### 3.7.2 pH Control

pH inside the reactor was maintained between the range of 7.2 and 8.6 by testing the pH of digestate in the laboratory on daily basis.

### 3.7.3 Nutrients

Nutrients are necessary components for biomass build up in an anaerobic process like all other biochemical operations. On the other hand, nutrient requirements are less in the anaerobic processes than aerobic processes due to lower biomass yields in such processes. The C: N: P requirement ratio for a typical activated sludge process is 100:5:1 on a mass basis.

### 3.7.4 Inhibitory and toxic materials

In an anaerobic process, different groups of microorganisms are involved. The activity or inactivity of one group of bacteria affects the activity of other group. This makes anaerobic processes sensitive to inhibition by chemicals present in the wastewater or substances produced as process intermediates. Inhibitory materials are those materials which cause an adverse shift in the microbial population or inhibition to bacterial growth. A decrease of the steady-state rate of methane gas production and accumulation of organic acids can be taken as an indicator of inhibition (Kroeker et al., 1979). The maximum specific growth rate of microorganisms is reduced by inhibition which results in increment in the SRT of a biochemical operation to

maintain the same effluent quality prior to the inhibition. Nevertheless, if the inhibitor concentration increases sufficiently, toxic effects can be seen. It can cause total process failure due to the death of microorganisms. Generally, inhibition precedes toxicity as the concentration of compound is increased. Several inorganic materials like light metal cations, ammonia, sulphide etc. can also cause an inhibitory response except the organic materials.

#### **3.7.4.1 Ammonia-Nitrogen**

Ammonia-N is an essential nutrient which stimulates bacterial growth at low concentrations. The anaerobic degradation of wastes with high protein content releases nitrogen either in the form of ammonium ion ( $\text{NH}_4^+$ ), or dissolved free ammonia ( $\text{NH}_3$ ) depending upon the pH of the system. Ammonia combines with carbon dioxide and water to form ammonium bicarbonate which acts as natural pH buffer. For anaerobic processes, ammonia concentrations between 50 and 200 mg/l as N are generally within the stimulatory range. However, ammonia is inhibitory at higher concentrations and toxic if the concentration is high enough. Ammonia is a weak base and dissociates in water as



There are several mechanisms proposed for ammonia inhibition such as a change in the intracellular pH, increase of maintenance energy requirement, and inhibition of a specific enzyme reaction. Ammonium concentrations as high as 7000 to 9000 mg/l as N have been successfully treated without a toxic response with an acclimated culture, although concentrations as low as 1500 mg/l as N can be toxic.

Ammonia inhibition can be more severe to the methanogens among the four types of anaerobic microorganisms, affecting their growth (Kayhanian, 1994).

Ammonia is determined twice a week in the lab by following Kjeldahl distillation and titration method.

#### **3.7.5 Alkalinity**

ALK is the buffering capacity of water to neutralize acids. ALK is a measure of carbonates, bicarbonates, hydroxides, and, occasionally, borates, silicates, and phosphates. It is expressed in milligrams of equivalent calcium carbonate per liter. The methane formers (methanogens) in anaerobic digestion are affected by small pH changes, while the acid producers can function satisfactorily across a wide range of

pH. Digestion stability depends on the buffering capacity of the digester contents. Higher ALK values indicate a greater capacity for resisting pH changes. ALK value in an anaerobic digester can range between 1500 and 5000 mg/L.

### **3.7.6 Volatile Fatty Acids**

Volatile fatty acids (VFA) accumulation is the major cause of pH drops in anaerobic reactors with insufficient alkalinity. VFA concentrations above 2000 mg/l can be inhibitory to methanogens. Volatile acids are weak acids that are largely dissociated at neutral pH. As long as the pH remains within the normal range for the growth of methanogens (7.2-8.6), inhibition caused by VFAs is not significant since high concentration of VFAs is tolerated. When pH gets lower than this range, pH exerts considerable impacts which will be compounded by any inhibition by non-ionized VFAs.

VFA is measured in the laboratory was measured by titration method twice a week.

### **3.7.7 Total Solids Determination**

Total solids are the measure of total dissolved solids (TDS) and total suspended solids (TSS) in water. It is generally measured in mg/L.

T.S is measured in the laboratory by oven drying method twice a week.

### **3.7.8 Volatile Solid**

Volatile solids are those solids in water or other liquids that are lost on ignition of dry solids at 1020°F (550°C). It is a water quality measure obtained from the loss on ignition of total suspended solids. Volatile solids were measured twice a week in lab by using muffle furnace.

### **3.7.9 Biogas Volume Measurement**

As the anaerobic reactor proceeds biogas is generated. That generated biogas was measured by a biogas meter. A pipe from the digester biogas outlet was attached to the biogas meter. This biogas meters measured the units if biogas produced and those units were then converted into volume of biogas by making proper gas expansion calculations according to the ambient temperature measured per day.

Following table illustrates the expansion of gas at a given temperature.

Table 3.2 Biogas Expansion According to Ambient Temperature

Ambient Temperature	1 biogas unit is equal to
10°C	13 ml
15°C	14 ml
20°C	15 ml
25°C	16 ml
30°C	17 ml

With every 5°C increase in ambient temperature there is 1ml increase in biogas expansion.

### 3.7.10 Methane Measurement

Methane content in the biogas was determined 4-5 times during the whole span of experiment. Methane was measured by the CO<sub>2</sub> absorption method. CO<sub>2</sub> filtration bottle was used for this purpose. In this bottle, 5M (Molar) solution of NaOH was prepared to filter CO<sub>2</sub> from biogas and to get methane (Esposito *et al.*, 2012). As the major constituent of biogas are CH<sub>4</sub> and CO<sub>2</sub>, so only CO<sub>2</sub> and H<sub>2</sub>S were removed. Almost 90% of CO<sub>2</sub> and H<sub>2</sub>S can be removed by passing the gas through aqueous solution containing NaOH (Tippayawong, 2010). According to this test, methane measured was 65-70% of the total biogas produced.

### 3.7.11 Carbon to Nitrogen ratio

The estimated values for carbon to nitrogen ratio in substrate used during the experiment were derived from extensive literature review. No experiment was conducted. The values of carbon and nitrogen that were obtained by ultimate analysis in literature were used in calculations for estimating C/N ratio in experiment. For all the three substrates, the C/N ratio is thought to be appropriate for anaerobic digestion. Carbon content of high solids sewage sludge was measured to be 29.26% and that of cow dung was 32.36%. The optimum C/N ratio for anaerobic digestion process is 28-30. We adjusted C/N ratio at 28 as derived from the literature review.

### **3.8 Semi-Continuous Feeding of Sludge**

After the initiation of batch mode, biogas generation increased with time. The rate declined after a month which was a clear indication that the organic matter present in substrate was consumed. Then the semi continuous phase started with sludge feeding as a substrate.

#### **3.8.1 Solids Retention Time**

Hydraulic Retention Time (HRT) is the average time that substrate stays in the digester for digestion. HRT is related to volume of the digester and substrate volume fed per day. For batch experiment, HRT was 30 days under mesophilic conditions. After every 24 hours sludge was fed into the digester.

#### **3.8.2 Organic Loading Rate**

As the total capacity of the digesters used for batch experiment was 50L, digester was batch loaded only once at the start of the experiment.

For the continuous digestion, the digester was fed after every 24 hours with the substrate i.e. 200g of Sludge. 1.4 kg of digestate was recovered daily from the digester. 0.2 kg of this digestate was kept for parameters analysis while 1.2 kg was mixed with 0.2kg of fresh feed of mass flow within and out of the digester was maintained.

### **3.9 Semi-Continuous Feeding of Sludge with Food and Paper Waste**

Mere feeding of dried (high solids) sludge into the reactor resulted in very limited production of biogas, which soon declined. This indicated that sludge alone is not a suitable substrate and in order to cater this problem, a suitable mixture of substrate was fed into the reactor in the place of mere sludge feeding. Sludge was then mixed with food and paper waste to adjust the conditions inside the reactor. Proper calculations were made to take the correct amount of sludge, food waste and paper waste.

Following table shows the characteristics of substrate with food waste and paper waste.

Table 3.3. Characteristics of sludge, paper, food waste and mixture

<b>Substance</b>	<b>Percentage in mixture (%)</b>	<b>Moisture Content (%)</b>	<b>Carbon Content (%)</b>	<b>Nitrogen Content (%)</b>	<b>C/N Ratio</b>	<b>Total Solids</b>
<b>Sludge</b>	60	80	40	6	6.67	20
<b>Paper</b>	35	5	40	0.1	400	95
<b>Food Waste</b>	5	83	48	2.65	18.11	17
<b>Mixture</b>	-	54	42	1.5	28	46 (reduce to 20)

## Results and Discussions

### 4.1 Introduction

In this chapter the result obtained during the experimentation and reactor parameters optimization at different phases will be discussed. The data is shown in the form of tables and graphs followed by detailed discussions.

### 4.2 Daily Biogas Production

After loading the reactor for start-up, biogas production started. It gradually increased with time and reached to a maximum of 38 L/d on day 23. Then it started to reduce slowly and reached to a value of 9 L/d on day 30. At start, microorganisms started to acclimatize with the reactor conditions, therefore biogas production was less. With passage of time, microbes got acclimatized to the reactor and due to presence of sufficient food for them, biogas production increased. After attaining the maximum biogas production level on day 23, it started to reduce, which is due to the fact that the organic matter present within the reactor was utilized during the stabilization/acclimatization process. Similar trend of batch mode start-up of reactor for dry anaerobic digestion of sludge has been reported by Jih-Gaw et al., 1997.

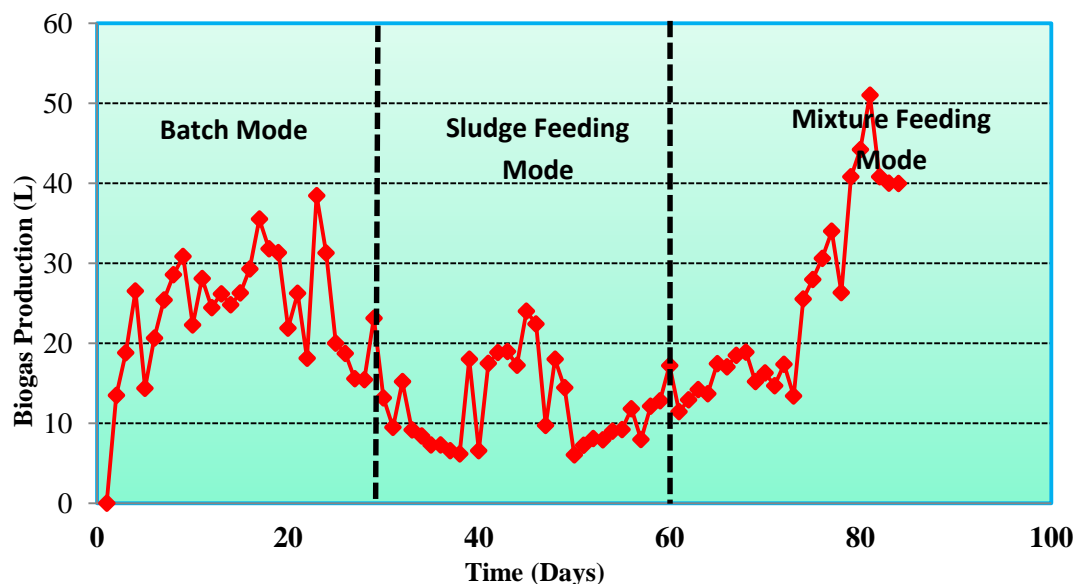


Figure 4.1. Daily Biogas Production



During the sludge feeding phase, biogas production increased gradually from 10L/d to the maximum production of 25L/d on the 45<sup>th</sup> day. It then reduced to 11L/d on 61<sup>st</sup> day. As the sludge feeding was started, the increased biogas production was observed in response to sludge digestion or stabilization within the reactor. The biogas production reduced after 45<sup>th</sup> day because daily sludge feeding resulted in inhibition of stabilization process as the sludge already has a low C/N ratio and a high pH (>9.5), while according to (JIUNN, 1997) optimum pH range for dry anaerobic digestion is between 6.6-8.2.

In response to the low biogas production as a result of sludge feeding, the feeding of a correct mixture was started on 62<sup>nd</sup> day after startup. This resulted into high volumes of daily biogas production. The biogas production reached up to the maximum value of 52L/d at 80<sup>th</sup> day. The increased biogas production was due to the fact that the mixture C/N ratio was optimized to be 28. Similar trend of biogas production by codigestion has been reported by Xiaojiao et al., 2014.

### **4.3 Biogas Production Rate**

The biogas production rate per unit volume of reactor shows the same pattern as the graph of biogas production volume.

After the startup of reactor, the biogas production rate per unit volume of reactor escalated initially and started receding at the end of acclimatization phase as the substrate was consumed up within the reactor body. During the post batch loading phase, the biogas production increased from 0.35L to about 1L per litre volume of reactor per day. Which then declined as the organic matter within substrate was consumed up. The same trend was seen in study of Jia Lina, 2011, biogas Volume for anaerobic digestion lied in the range 1- 2.17 L/(L·day) during batch phase.

As mentioned earlier, the biogas volume and hence the biogas production rate during the sludge feeding phase was very considerable. The biogas production rate lied in the range of 0.2-0.6L per litre of reactor volume each day. Samson et al., 1982 stated almost same results.

The mixture feeding was started and biogas production rate increased from 0.45L/L.d to 1.3L/L.d on 81st day. This increased biogas production rate is because the new

substrate is more suitable for anaerobic digestion as it has adjusted C/N ratio. The rate complied with the limits mentioned in literature, Jia Lina, 2011 mentioned that the daily biogas production lies in limits of 1-2.17 L/(L•day).

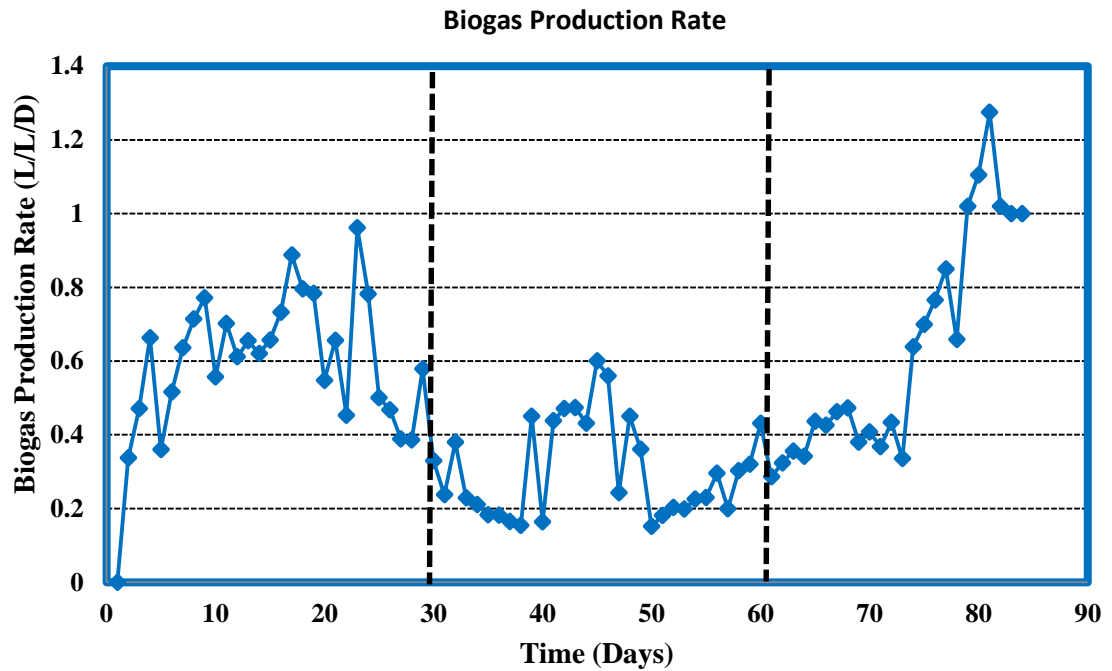


Figure 4.2. Biogas Production Rate

#### 4.4 pH Variation

pH was continuously monitored during the batch mode by testing the pH of representative sample of digestate collected from the reactor outlet. Initially the pH declined from 9 to 6.8 on 6th day until 15th day. After the 15th day, the pH started to increase and reached to a maximum value of 9.1. Initially the pH of the system declined which was an indication of fatty acids production and the forward proceeding of the reactor. Soon, the pH increased due to the fact that the pH of sludge is naturally high and partly because ammonia inhibition started and the process ceased to proceed further. Similar trends were represented in the study of Jiunn-Jyi, 1997 who quoted in his study that the optimum pH range for anaerobic process lies in the range 6.6-8.2.

The sludge feeding phase started and resulted in a very gradual and insignificant decrease in pH from 8.9 and reached to the lowest value of 7.95 on 60th day. The pH decreased very slowly due to the fact that the pH of sludge is naturally high which was being introduced into the reactor each day and partially because ammonia

inhibition started and the process ceased to proceed further as already discussed by Jiunn-Jyi, 1997.

During the mixture feeding phase, the pH declined and approached towards the optimum range (Jiunn-Jyi, 1997). The pH declined from 8.2 to 7.32 in just five days. It then increased gradually up to 8.2 but remained within the optimum range. The abrupt decrease in pH initially was due to the fact that the mixture was a suitable substrate having adjusted C/N. Decline in pH indicated that the mixture got rapidly converted to fatty acids.

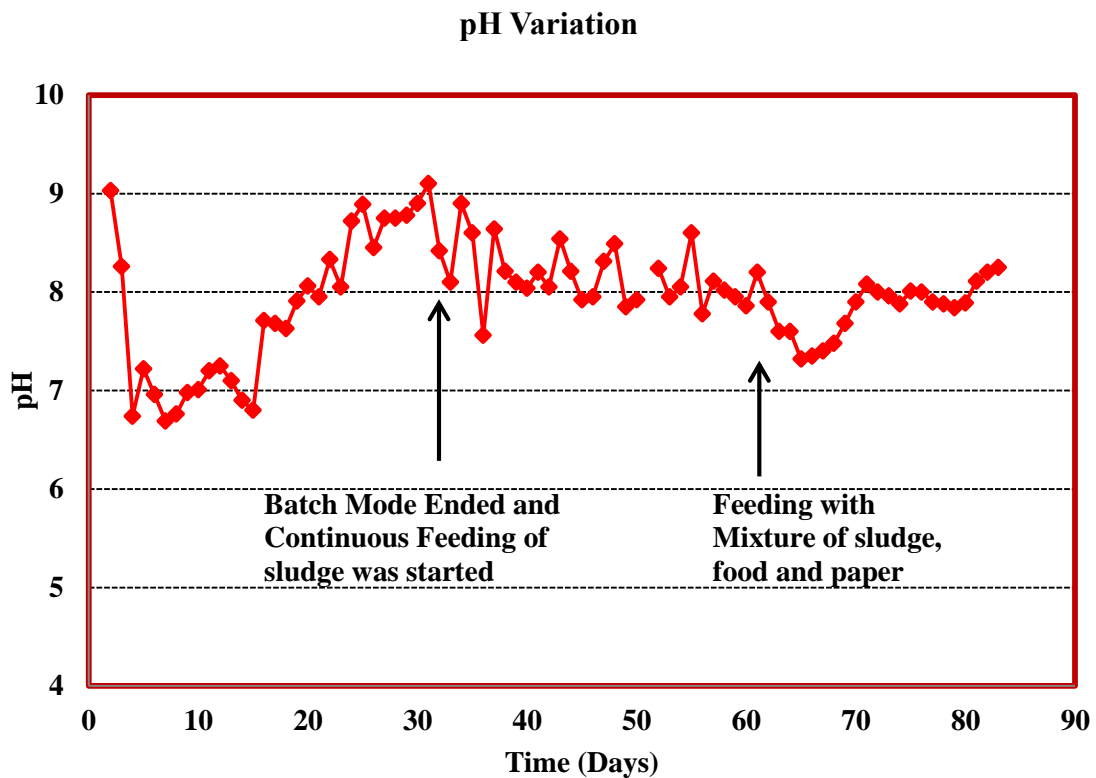


Figure 4.3. Daily pH Variation

#### 4.5 VFA/Alkalinity

The VFA/ Alkalinity ratio at the beginning of the process was 0.39 which soon increased up to 0.4. The fatty acids were being produced at a fast rate while these were continually being converted to methane by methanogens. The ratio stayed within the optimum range of pH had similar trends as the study of Zeshan et al., 2012 where VFA/Alk optimum range was mentioned to be 0.1-0.35.

During the sludge feeding phase, the ratio increased from 0.45 to 0.67 on 50th day, 0.5 on 60th day. The increased ratio indicated that the fatty acids were not completely being converted into methane. The ratio did not lie within optimum ratio. (Zeshan et al., 2012)

During the mixture feeding phase, the ratio declined from 0.67 (day 70) to 0.22 (day 73) in three days. This then increased to 0.3 until the 80th day. The pH and C/N of the mixture were optimum and hence the process flow remained smooth and the ratio was also observed within the limits. This was similar to the conditions of the study of Zeshan et al., 2012.

### VFA, Alkalinity and VFA/Alk Ratio Analysis

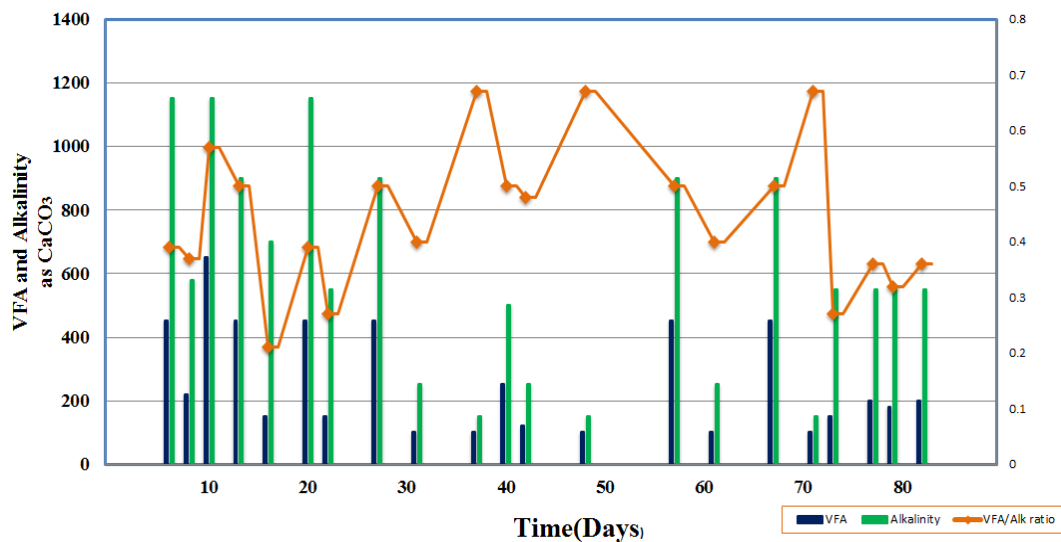


Figure 4.4. VFA, Alkalinity and VFA/Alk Ratio Analysis

### 4.6 Ammonia-Nitrogen

During the sludge feeding mode, initially the ammonia-nitrogen was 100mg/L after batch loading; this gradually increased to 125mg/L after a week, this further increased to a maximum value of 450mg/L. The increase in the value is due to the fact that sludge has high nitrogen contents. The same was studied by Angelidaki, 1993 in his research.

The ammonia nitrogen dropped from 450mg/L to 193.7mg/L within two weeks. The reason behind abrupt decrease in ammonia nitrogen was because the mixture had adjusted C/N ratio hence the value of ammonia nitrogen due to mere sludge feeding was soon reduced after mixture feeding.

El Hadj et al., 2009 mentioned that 50% inhibition of biomethane production occurs at level of 215 and 468 mg NH<sub>3</sub>-N/L under mesophilic and thermophilic conditions.

### Ammonia- Nitrogen Results

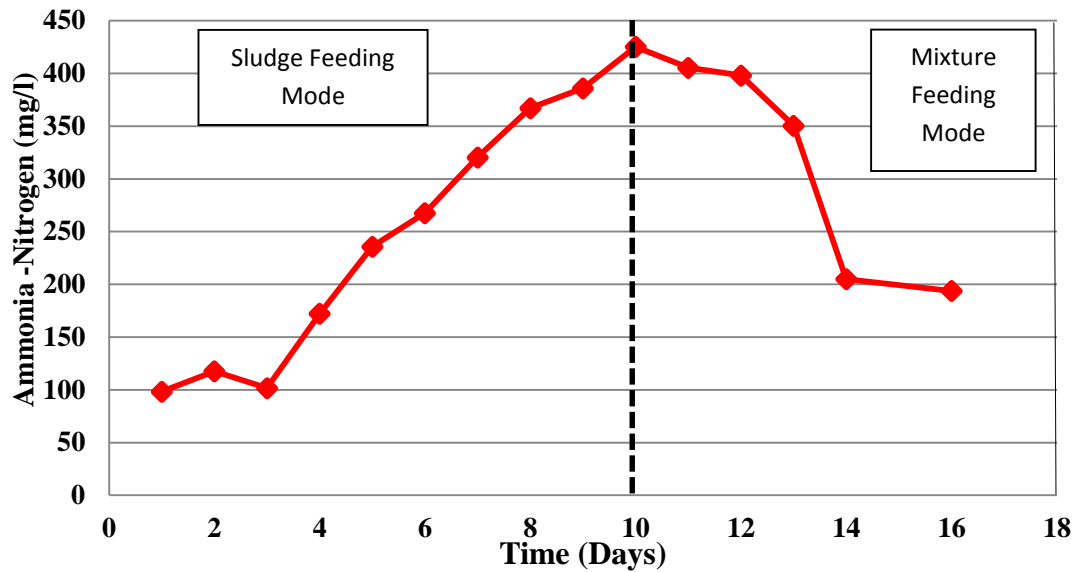


Figure 4.5.Variation of Ammonia-nitrogen during HSAD

#### 4.7 Total Solids

During the post batch phase, the total solids decreased up to 15% in 2 weeks, as the digestion of the substrate occurred in response to hydrolysis process. While it remained almost stable at the end of acclimatization phase. Lise et al., 2008 claimed that the anaerobic digestion reduces the amount of final sludge solids for disposal whilst destroying most of the pathogens present in the sludge and limiting odour problems associated with residual putrescible matter.

At the beginning of sludge feeding, TS of digestate receded at a very low rate and lied around 15% for three weeks which soon increased gradually to 20% and biogas production almost ceased to produce. This was due to the fact that pH and C/N of sludge was not adjusted; the hydrolysis did not occur completely and hence TS reduction was gradual (Lise et al., 2008). The increase of TS up to 23% was also because mere sludge was not a suitable substrate.

During the mixture feeding phase, the TS receded up to 20% in a week. In response to hydrolysis, as mixture was the better substrate to be fed. Similar trends were seen in the study of P Sosnowski et al., 2003 representing codigestion of sewage sludge.

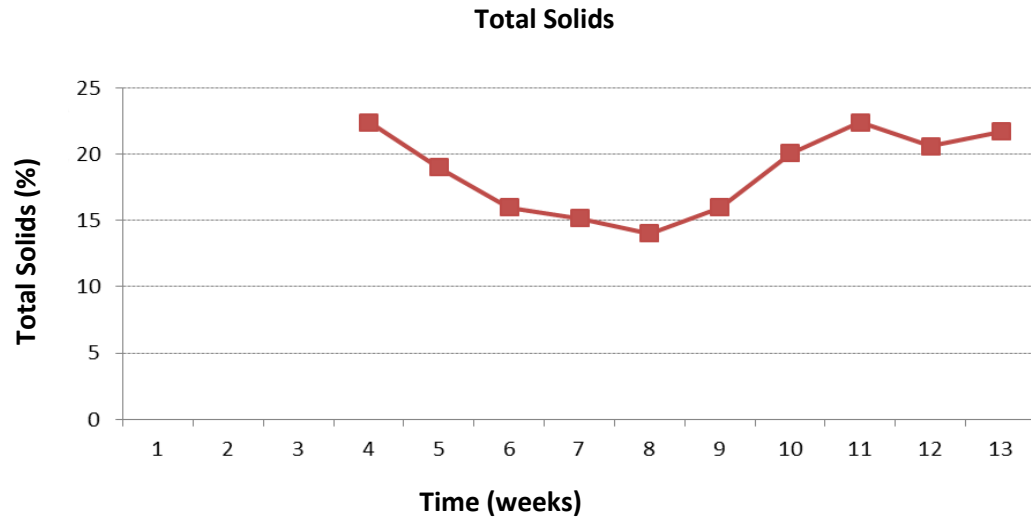


Figure 4.6. Variation of Total Solids during HSAD

#### 4.8 Biogas Percentage

The methane produced contained approximately 70% methane and 30% carbon dioxide and trace gases. The percentage was in accordance with the study of Lise, 2008 claiming that biogas contains 60–70% volume of methane, CH<sub>4</sub>.

Biogas composition was tested by using CO<sub>2</sub> absorption method in 5M NaOH solution. The test was performed 4-5 times and the pie chart depicts the average of each component within the biogas.

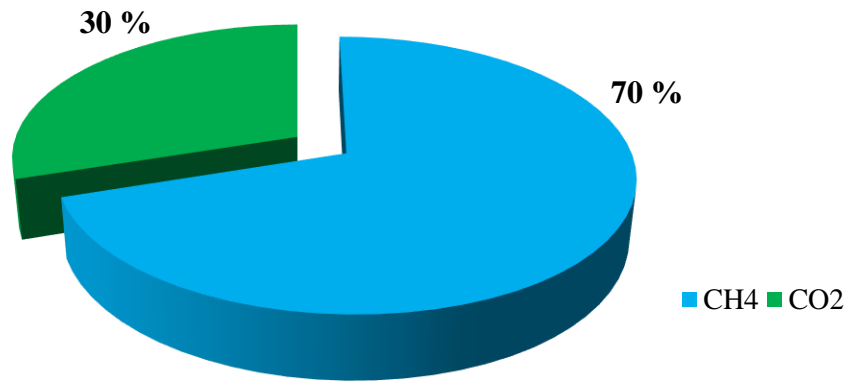


Figure 4.7. Biogas composition

### Conclusion and Recommendations

#### 5.1 Conclusions

Following conclusions were drawn from the study:

- Smooth startup (batch mode) of the reactor was observed as indicated by biogas production at I:S (inoculum to substrate ratio on weight VS) of 1.516/2.069.
- During batch phase, biogas production rate of around 0.9-1 L/L/d was observed i.e. specific biogas for the same phase was around 117639 L/kg.VS.
- During sludge feeding phase, biogas production rate of up to 1.3 L/L/d was observed.
- Considerable methane percentage in line with literature was observed. (65-70% methane)
- Dry Digestion of sludge alone cannot be progressed so, co-digestion of sludge is therefore required under high solids conditions.
- During the semi-continuous feeding phase, biogas yield declined due to increment in pH, high N content and low C/N ratio of sludge.
- The problem of declined biogas production was solved by using correct mixture of sludge, food waste and paper waste.
- T.S of Digestate decreased as anaerobic digestion proceeded.

#### 5.2 Recommendations

##### 5.2.1 Recommendations for field application

The following recommendation is made for field application:

- Dry digestion cannot be done alone, so we need to make a balanced mixture of sludge with other substrate such as food and paper is recommended.

##### 5.2.2 Recommendations for future study

The following recommendation is made for future study:

- Codigestion of sludge with other substrates under high solids conditions.



- Biotechnological method should be explored to digest sludge alone under dry conditions by introducing organic Nitrogen utilizing bacteria or bacteria which can reduce ammonia inhibition.
- Storage, purification and usage of biogas must be done.

## References

- Ahring, B.K. (2003). Perspectives for anaerobic digestion. In Advances in biochemical engineering/biotechnology. Biomethanation I. Edited by B.K. Ahring. Springer. Berlin, 1-30.
- Albrecht R. Bresters, I. C. (1997). Sludge Treatment and Disposal. Denmark, Folkmann Design & Promotion.
- Angelidaki, I., & Ahring, B. K. (1993). Thermophilic anaerobic digestion of livestock waste: the effect of ammonia. Environmental Biotechnology, Volume 38, Issue 4, 560-564.
- Avfall, S. (2009). Swedish waste management.
- Azbar, N., & Speece. (2001). Two-Phase, Two-Stage, and Single-Stage Anaerobic Process Comparison. Journal of Environmental Engineering, Volume 127, Issue 3, 240-248.
- Batstone, D.J., Keller, J., Angelidaki, I., Kalyuzhnyi, S.V., Pavlostathis, S.G., Rozzi, A., Sanders, W.T.M., Siegrist, H. and Vavilin, V.A. (2002). The IWA Anaerobic Digestion Model. Science and Technology Report, Volume 45, Issue 10, 65-73.
- Bendixen, H.J. (1999). Hygienic safety: results of scientific investigations in Denmark (sanitation requirements in Danish Biogas Plants). In Proceedings of the IEA workshop: "Hygienic and environmental aspects of anaerobic digestion: legislation and experiences in Europe." pp. 27-47. Universität Hohenheim, Stuttgart, Germany.
- Casey, Thomas Joseph, and T. J. Casey. (1997). Unit Treatment Processes in Water and Wastewater Engineering. Chichester: John Wiley & Sons Ltd. West Sussex, England: Wiley.
- Bolzonella, David, Paolo Pavan, Paolo Battistoni, and Franco Cecchi. (2005). Mesophilic Anaerobic Digestion of waste. Process Biochemistry, Volume 40, Issues 3–4, 1453-1460.
- Cohen, A., A. M. Breure, J. G. Van Andel, and A. Van Deursen. (1980). Influence of phase separation on the anaerobic digestion of glucose—I

maximum COD-turnover rate during continuous operation. *Water research*, Volume 14, Issue 10, 1439-1448.

- Demetriades, P. (2008). Thermal pre-treatment of cellulose rich biomass for biogas production. Doctoral Thesis. Uppsala SLU.
- Deublein, Dieter, and Angelika Steinhauser. (2011) *Biogas from waste and renewable resources: an introduction*. John Wiley & Sons.
- Droste, Ronald L. (1997). *Theory and practice of water and wastewater treatment*. John Wiley & Sons Incorporated.
- Dubrovskis, Vilis, Imants Plume, Vladimirs Kotelenecs, and Eduards Zabarovskis. (2010). Anaerobic digestion of sewage sludge. In *Engineering for Rural Development. Proceedings of the 9th International Scientific Conference*, Jelgava, Latvia, 27-28 May, 2010. Latvia University of Agriculture, Faculty of Engineering, Institute of Mechanics, 216-219.
- El Hadj, T. Benabdallah, S. Astals, A. Gali, S. Mace, and J. Mata-Alvarez. (2009). Ammonia influence in anaerobic digestion of OFMSW. *Water Science and Technology*, Volume 59, Issue 6 1153-1158.
- Esposito, Giovanni, Luigi Frunzo, Flavia Liotta, Antonio Panico, and Francesco Pirozzi. (2012). Bio-methane potential tests to measure the biogas production from the digestion and co-digestion of complex organic substrates. *Environmental Engineering Journal*, Volume 7, 1-81.
- Fathya, Salem, Kouki Assia, and Mokaddem Hamza. (2014). Influence of Inoculums/Substrate Ratios (ISRs) on the Mesophilic Anaerobic Digestion of Slaughterhouse Waste in Batch Mode: Process Stability and Biogas Production. *Energy Procedia*, Volume 50, 57-63.
- Food and agriculture organization of the united nations Rome, 2002 (FAO) Available at <http://www.fao.org/docrep/004/y6000e/y6000e00.htm> (Accessed at: 20.06.2016)
- Franke-Whittle, Ingrid H., Andreas Walter, Christian Ebner, and Heribert Insam. (2014). Investigation into the effect of high concentrations of volatile fatty acids in anaerobic digestion on methanogenic communities. *Waste Management*, Volume 34, Issue 11, 2080–2089.

- Gerardi, Michael H. (2003). *The microbiology of anaerobic Digesters*. Waste water Series. USA: John Wiley and Sons, Inc.
- Ghosh, Sambhunath, and Donald L. Klass. (1978). Two Phase Anaerobic Digestion. Proc. Biochem. U.S. Patent No. 4,022,665. Washington, DC: U.S. Patent and Trademark Office.
- Grady, C.P.L., G.T. Daigger, and H.C. Lim, *Biological wastewater treatment*. 2011, London: IWA Publishing. XXIX, 991 s.
- Henze, M. (2008). *Biological Wastewater Treatment*. London: 511s.
- Hessami, Mir-Akbar, Sky Christensen, and Robert Gani. (1996). Anaerobic digestion of household organic waste to produce biogas. *Renewable energy* Volume 9, Issue 1, 954-957.
- Holm-Nielsen, Jens Bo, Theodorita Al Seadi, and Piotr Oleskowicz-Popiel. (2009). The future of anaerobic digestion and biogas utilization. *Bioresource technology*, Volume 100, Issue 22, 5478-5484.
- Igoni, A. Hilkih, M. J. Ayotamuno, C. L. Eze, S. O. T. Ogaji, and S. D. Probert. (2008) Designs of anaerobic digesters for producing biogas from municipal solid-waste. *Applied energy*, Volume 85, Issue 6, 430-438.
- Jarvis. (2004). *BIOGAS: Renewable energy from organic waste*. Stockholm: Swedish Association.
- Kayhanian, Masoud. (1994). Performance of a high-solids anaerobic digestion process under various ammonia concentrations. *Journal of Chemical Technology and Biotechnology*, Volume 59, Issue 4, 349-352.
- Kelessidis, Alexandros, and Athanasios S. Stasinakis. (2012). Comparative study of the methods used for treatment and final disposal of sewage sludge in European countries. *Waste management*, Volume 32, Issue 6, 1186-1195.
- Kommedal, R., *Lecture notes on MET 160 Environmental Microbiology*. 2010, University of Stavanger.
- Kosobucki, P., A. Chmarzynski, and B. Buszewski. (2000) Sewage sludge composting. *Polish Journal of Environmental Studies*, Volume 9, Issue 4, 243-248.

- Kroeker, E. J., D. D. Schulte, A. B. Sparling, and H. M. Lapp. (1979). Anaerobic treatment process stability. *Journal (Water Pollution Control Federation)*. 718-727.
- Lay, Jiunn-Jyi, Yu-You Li, and Tatsuya Noike. (1997). Influences of pH and moisture content on the methane production in high-solids sludge digestion. *Water Research*, Volume 31, Issue 6, 1518-1524.
- Lin, Jia, Jiane Zuo, Lili Gan, Peng Li, Fenglin Liu, Kaijun Wang, Lei Chen, and Hainan Gan.. (2011). Effects of mixture ratio on anaerobic co-digestion with fruit and vegetable waste and food waste of China. *Journal of Environmental Sciences*, Volume 23, Issue 8, 1403–1408.
- Lin, Jih-Gaw, Cheng-Nan Chang, and Shou-Chung Chang, C.-N. C.-C. (1997). Enhancement of anaerobic digestion of waste activated sludge by alkaline solubilization. *Bioresource Technology*, Volume 62, 85-90.
- Lise Appels, J. B. (2008). Principles and potential of the anaerobic digestion of waste activated sludge. *Energy and Combustion Science*, Volume 34, Issue 6, 755-781.
- Luts, D., K. Devoldere, B. Laethem, W. Bartholomeeusen, and P. Ockier. (2000). Co-incineration of dried sewage sludge in coal-fired power plants: a case study. *Water science and technology*, Volume 42, Issue 9, 259-268.
- Majid, H. (2006). Biogas Plant, a source of clean fuel and bio-fertilizer. Internal Report of PCRET.  
(Accessed at: 20.06.2016)
- Marchaim, U. (1991) *Biol. Wastes* (in press).
- Massey, Michael L., and Frederick G. Pohland. (1978). Phase separation of anaerobic stabilization by kinetic controls. *Water Pollution Control*, Volume 50, Issue. 9, 2204–2222.
- National water sector profile Volume 5 October 2002.
- Nijaguna, B. (2002). *Biogas Technology*. 6<sup>th</sup> ed. New Delhi: New age international (P) ltd. publishers.
- Ødegaard, Hallvard, Bjarne Paulsrud, and Ingemar Karlsson. (2002). Wastewater sludge as a resource: disposal strategies and corresponding treatment technologies aimed at sustainable handling of wastewater sludge. *Water Science and Technology*, Volume 46, Issue10, 295-303.

- P. Aarne Vesilind, W. A. (2002). Solid Waste Engineering. Mishawaka: CL-Engineering.
- Pakistan Water Sector Strategy (PWSS). 2002. National water sector profile, Volume 5, October 2002, Ministry of Water and Power, Office of the Chief Engineering Advisor. Available at <http://waterinfo.net.pk/cms/pdf/vol5.pdf>
- Rivard, Christopher J. (1995). Intermediate-Scale High-Solids Anaerobic Digestion System Operational Development Anaerobic Digestion Innovative Technologies Group, 2-12.
- Samson, R. and Leduy. (1982). Biogas production from anaerobic digestion of *Spirulina maxima* algal biomass. Biotechnology and Bioengineering, Volume 24, Issue 8, 1919–1924.
- Sosnowski, P., A. Wiczorek, and S. Ledakowicz. (2003). Anaerobic co-digestion of sewage sludge and organic fraction of municipal solid wastes. Advances in Environmental Research, Volume 7, Issue 3, 609–616.
- Sreekrishnan, T.R., Kohli, S. and Rana, V. (2004). Enhancement of biogas production from solid substrates using different techniques. Bioresource Technology, Enhancement of biogas production from solid substrates using different techniques, Volume 95, Issue 1, 1-10.
- Straub, Timothy M., Ian L. Pepper, and Charles P. Gerba. (1993). Hazards from Pathogenic Microorganisms in Land-Disposed Sewage Sludge. Reviews of Environmental Contamination and Toxicology, 55-91.
- Tchobanoglous, G. F. (2003). Wastewater Engineering. Boston.
- Tippayawong, N. and Thanompongchart, P., (2010). Biogas quality upgrade by simultaneous removal of CO<sub>2</sub> and H<sub>2</sub>S in a packed column reactor. Energy, Volume 35, Issue 12, 1-5
- Udom, B. E., J. S. C. Mbagwu, J. K. Adesodun, and N. N. Agbim. (2004). Distributions of zinc, copper, cadmium and lead in a tropical ultisol after long-term disposal of sewage sludge. Environment International, Volume 30, Issue 4, 467-470.
- Wang, X., Lu, X., Li, F. and Yang, G. (2014). Effects of Temperature and Carbon-Nitrogen (C/N) Ratio on the Performance of Anaerobic Co-Digestion

of Dairy Manure, Chicken Manure and Rice Straw: Focusing on Ammonia Inhibition. PLoS one Volume 9, Issue 5.

- Wang, X., Chen, T., Ge, Y. and Jia, Y. (2008). Studies on land application of sewage sludge and its limiting factors. *Journal of Hazardous Materials*, Volume 160, Issues 2–3, 554-558.
- Winkler, M. (1993). *Sewage sludge treatments. Chemistry and Industry.* London. United Kingdom.
- Yadavika, Santosh, T. R. Sreekrishnan, S. Kohli, and V. Rana. (2004). Enhancement of biogas production from solid substrates using difference techniques—a review. *Bioresource Technology*.
- Zeshan, Karrthikeyan, O.P. and Visvanathan. (2012). Effect of C/N ratio and ammonia-N accumulation in a pilot-scale thermophilic dry anaerobic digester. *Bioresource Technology*, Volume 113, 294–302.
- Zupančič, Gregor D., and Milenko Roš. (2008). Aerobic and two-stage anaerobic–aerobic sludge digestion with pure oxygen and air aeration. *Bioresource Technology*, Volume 99, Issue 1, 100-109.