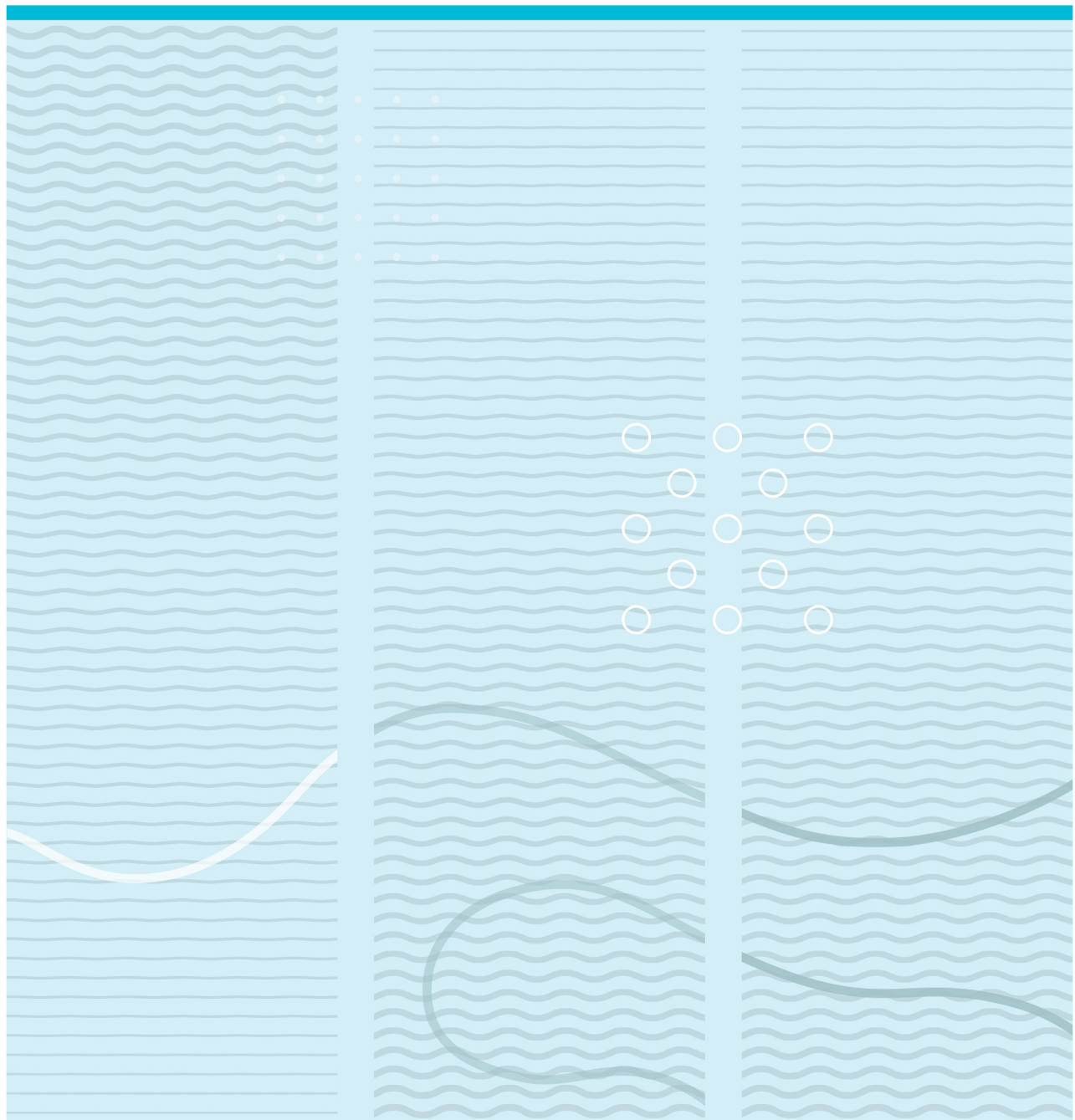


Nirmal Ghimire

# Investigation of Bioprocess for Industrial Wastewater Treatment



University College of Southeast Norway  
Faculty of Technology

<http://www.usn.no>

© 2016 <Nirmal Ghimire>

This thesis is worth 30 study points

# Abstract

A pilot scale Hybrid Vertical Anaerobic Biofilm (HyVAB<sup>®</sup>) reactor was applied to the treatment of waste oil refinery wastewater. The reactor comprised a bottom anaerobic digestion stage operated as an Up-flow Anaerobic Sludge Blanket (UASB) and a moving bed biofilm stage (during washing mode) on top operated as a Continuous Flow Intermittent Cleaning (CFIC<sup>®</sup>). The reactor was operated continuously for 90 days, with Organic Loading Rate (OLR) increasing from the lowest 3 kg COD/m<sup>3</sup>.d to the highest 33.1 kg COD/m<sup>3</sup>.d. Oil wastewater was heated to 35°C before pumping through the bottom of the reactor. Average Chemical Oxygen Demand (COD) concentration of the feed was 10 g/L.

The results showed that the HyVAB<sup>®</sup> reactor had good performance in terms of COD removal and biogas generation. Highest sCOD removal efficiency of 98.5% and tCOD removal efficiency of 95.4% was achieved at 12 hours Hydraulic Retention Time (HRT) in AD, and highest OLR of 18.7 kg COD/m<sup>3</sup>.d. Most of the COD removal took place in AD stage. Around 86% COD in oil wastewater was transformed into biogas in which methane content was 80-90%. Biogas formed was collected at anaerobic and aerobic interface via a three phase separator. Volatile Fatty Acid (VFA) accumulation was observed during the initial and later operation stage affecting COD removal, biogas production and total suspended solids in effluent. Suspended solid production measured in the effluent was approximately 0.16 kg VSS/kg COD removed. Meanwhile, sludge loading rate (SLR) increased from 0.1 kg COD/kg VS.d at the initial phase to 0.55 kg COD/kg VS.d at 71<sup>st</sup> day. Also, specific methanogenic activity (SMA) increased from 0.074 kg COD-CH<sub>4</sub>/ kg VS.d to 0.428 kg COD-CH<sub>4</sub>/ kg VS.d at 71<sup>st</sup> day.

On the whole, the compact HyVAB<sup>®</sup> exhibited good stability in terms of acidity and alkalinity. Replacing CFIC<sup>®</sup> with HyVAB<sup>®</sup> can save significant amount of money on aeration up to \$ 5.35 per day per m<sup>3</sup> of treated wastewater. HyVAB<sup>®</sup> reactor can be an attractive alternative while treating high strength wastewater at relatively lower operational cost.

# Contents

Abstract . . . . .	3
Preface . . . . .	6
List of Figures . . . . .	7
List of Tables . . . . .	9
1 Introduction . . . . .	10
1.1 Background . . . . .	10
1.2 Industrial Wastewater . . . . .	10
1.3 Treatment Method . . . . .	10
1.4 Objectives . . . . .	12
1.5 Structure of the thesis . . . . .	13
2 Literature Review . . . . .	14
2.1 Anaerobic Digestion . . . . .	14
2.1.1 Process Fundamentals . . . . .	15
2.1.2 UASB . . . . .	19
2.1.3 Granules . . . . .	21
2.1.4 Biogas . . . . .	22
2.2 Aerobic Digestion . . . . .	22
2.2.1 Biofilm . . . . .	23
2.2.2 Continuous Flow Intermittent Cleaning (CFIC®) . . . . .	23
2.3 Economic Analysis . . . . .	24
3 Methods and Materials . . . . .	26
3.1 Wastewater preparation . . . . .	26
3.2 Reactor Design and Setup . . . . .	26
3.2.1 Pilot Scale Reactor . . . . .	26
3.2.2 Laboratory Scale Reactor . . . . .	29
3.3 Assays . . . . .	33
4 Results . . . . .	35
4.1 Pilot Scale Reactor . . . . .	35
4.1.1 Start-up . . . . .	35
4.1.2 Reactor performance . . . . .	36
4.1.3 Biogas production . . . . .	44
4.1.4 Development of granules . . . . .	47
4.1.5 Aerobic biofilm stage . . . . .	52
4.1.6 Solids removal . . . . .	53
4.2 Laboratory Scale Reactor . . . . .	55
4.2.1 Reactor Performance . . . . .	55

	4.2.2	Aerobic biofilm stage . . . . .	57
	4.2.3	Solids removal . . . . .	58
4.3		Economic Analysis . . . . .	59
	4.3.1	Case I. Aeration cost based on aerobic complete mix biological treatment . . . . .	59
	4.3.2	Case II. Aeration cost based on real data from Bamble reactor	60
	4.3.3	Case III. Aeration cost after anaerobic digestion in AD stage of HyVAB® . . . . .	60
	4.3.4	Methane production from anaerobic digestion prior aeration .	61
	4.3.5	Asset value of 1 m <sup>3</sup> of biogas . . . . .	63
5		Discussions . . . . .	64
	5.1	Pilot scale reactor . . . . .	64
	5.1.1	Start-up of the reactor . . . . .	64
	5.1.2	Reactor performance . . . . .	64
	5.1.3	Biogas production . . . . .	64
	5.1.4	Development of granules . . . . .	65
	5.1.5	VFA buildup and consumption . . . . .	66
	5.1.6	Solids removal . . . . .	67
	5.1.7	Economics . . . . .	67
	5.1.8	Recycle pump . . . . .	67
	5.2	Laboratory scale reactor . . . . .	67
	5.2.1	Reactor performance . . . . .	67
	5.2.2	VFA buildup and consumption . . . . .	68
	5.2.3	Solids removal . . . . .	68
	5.3	Future Work . . . . .	68
	5.3.1	High purity oxygen digestion . . . . .	68
	5.3.2	Quantification of settled CFIC® biofilm in AD . . . . .	68
	5.3.3	Determination of integrity coefficient . . . . .	69
	5.3.4	Recovery from overloaded conditions . . . . .	69
	5.3.5	Effects of recirculation . . . . .	69
	5.3.6	Recommended pumps for recirculation . . . . .	69
6		Conclusion . . . . .	70

# Preface

This thesis is the final work of my Master study at University College of Southeast Norway (USN).

I wish to thank my supervisor Prof. Rune Bakke for motivating me to take this research project. His advice, guidance, encouragement and invaluable suggestions always pushed me to perform better. I thoroughly enjoyed each and every meeting I had with him. He not only taught me technical skills but also many life hacking skills which I'll be using in the future for sure.

I like to extend my gratitude to my co-supervisor Wang Shuai, PhD for his continuous support throughout the research. This thesis would not have been possible without him driving me to the industry to collect samples. Talks about movies, series and sometimes politics while driving to the industry made our travel short and interesting. I am grateful towards him for helping me by running GCs for VFA and biogas composition.

My project partner Gang Xin, PhD from Biowater Technology AS can't be forgotten. His suggestions were valuable and gave me deeper insight to the problems. I am impressed by his contagious enthusiasm.

I must thank Eshetu Janka Wakjera, PhD for helping me during the start-up phase of the reactor. I am grateful to him for the trust he has shown towards me.

I also like to thank Amund Heggholmen for providing me with the  $\LaTeX$  template for the thesis and Tonje Warholm Thomassen for helping me collect samples.

I am also grateful to staffs of NSO, Bamble for their cheerful cooperation. They were always welcoming and eager to help which made my work much easier.

Porsgrunn, June 3, 2016  
Nirmal Ghimire

# List of Figures

1-1	Types of combined anaerobic-aerobic sytem (Chan et al., 2009) . . . . .	12
2-1	The key process stages of anaerobic digestion (Abdelgadir et al., 2014) . . . . .	15
2-2	Schematic diagram of UASB reactor . . . . .	20
2-3	The CFIC <sup>®</sup> during a) normal operation, and during b) the cleaning cycle (Rusten et al., 2011) . . . . .	23
3-1	Processing diagram in NSO (del Mar Batista Seguí, 2014) . . . . .	26
3-2	Mature BWTS <sup>®</sup> biofilm carriers . . . . .	27
3-3	Three phase separator installed in pilot scale HyVAB <sup>®</sup> bioreactor at NSO . . . . .	27
3-4	Experimental setup of pilot scale HyVAB <sup>®</sup> bioreactor at NSO . . . . .	29
3-5	Experimental setup of lab scale HyVAB <sup>®</sup> bioreactor at HSN (Wang et al., 2015a). . . . .	30
4-1	COD removal with increase of OLR . . . . .	35
4-2	HRT vs OLR . . . . .	36
4-3	Overall performance of HyVAB <sup>®</sup> reactor . . . . .	37
4-4	Overall performance of AD in HyVAB <sup>®</sup> reactor . . . . .	37
4-5	tCOD removal in AD and CFIC <sup>®</sup> of HyVAB <sup>®</sup> . . . . .	38
4-6	sCOD removal in AD and CFIC <sup>®</sup> of HyVAB <sup>®</sup> . . . . .	39
4-7	COD removal efficiency along with influent and effluent COD . . . . .	39
4-8	COD removal efficiency along with influent and effluent COD . . . . .	40
4-9	Concentration of VFAs in effluent over time . . . . .	40
4-10	Concentration of VFAs in recycle over time . . . . .	41
4-11	Concentration of VFAs in AD3 over time . . . . .	41
4-12	Concentration of VFAs in Feed over time . . . . .	42
4-13	Biogas production with VFA concentration . . . . .	42
4-14	VFA concentration of feed and different stages . . . . .	43
4-15	Relation of ratio of VFA and tCOD along and Biogas production . . . . .	43
4-16	Overall performance of HyVAB <sup>®</sup> reactor with biogas production . . . . .	44
4-17	Biogas Production towards the end with instalment of biogas meter . . . . .	45
4-18	Biogas production with decreased HRT over time . . . . .	45
4-19	Methane yield with respect to COD removal . . . . .	46
4-20	Methane yield towards the end with instalment of biogas meter . . . . .	46
4-21	Biogas composition of HyVAB <sup>®</sup> reactor . . . . .	47
4-22	Development of granules in AD1 over time . . . . .	48
4-23	Development of granules in AD2 over time . . . . .	48
4-24	Spatial development of granules over time . . . . .	49
4-25	Development of granules in 18 <sup>th</sup> day . . . . .	49
4-26	Development of granules in 71 <sup>st</sup> day . . . . .	50

4-27 Development of granules along the height in 18 <sup>th</sup> day . . . . .	50
4-28 Development of granules along the height in 71 <sup>st</sup> day . . . . .	51
4-29 Sludge Loading of the reactor over the time . . . . .	51
4-30 Specific methanogenic activity (SMA) of the reactor over the time . . . . .	52
4-31 Sludge Loading of the reactor over the time . . . . .	52
4-32 Dissolved oxygen over the increase of OLR . . . . .	53
4-33 Effluent solids concentration over the increase of OLR . . . . .	53
4-34 TSS concentration in AD and recycle over time . . . . .	54
4-35 TSS concentration in AD and recycle over time . . . . .	54
4-36 HyVAB <sup>®</sup> performance at varying OLR and HRT . . . . .	55
4-37 tCOD Removal in AD and CFIC <sup>®</sup> stage of HyVAB <sup>®</sup> . . . . .	56
4-38 sCOD Removal in AD and CFIC <sup>®</sup> stage of HyVAB <sup>®</sup> . . . . .	56
4-39 Fluctuation of pH in different stages . . . . .	57
4-40 Biomass production calculated from effluent VSS . . . . .	57
4-41 Dissolved oxygen concentration in CIFIC <sup>®</sup> over time . . . . .	58
4-42 TSS and VSS effluent from HyVAB <sup>®</sup> at varying OLR . . . . .	58



# List of Tables

2-1	Advantages and disadvantages of UASB reactor (Latif et al., 2011),(Lim and Kim, 2014), (Chong et al., 2012), (Abbasi and Abbasi, 2012) . . . . .	21
3-1	Characteristics of wastewater used as HyVAB <sup>®</sup> feed at NSO (Bamble) (after chemical addition) . . . . .	26
3-2	Characteristics of biofilm carriers used for CFIC <sup>®</sup> stage of bioreactors . . . . .	27
3-3	Sample locations and analysis of HyVAB <sup>®</sup> reactors . . . . .	31
3-5	Design of pilot and laboratory scale HyVAB <sup>®</sup> bioreactor . . . . .	31
4-1	Assumed values to calculate aeration cost for complete mix biological treatment	59
4-2	Assumed values to calculate aeration cost after AD stage of HyVAB <sup>®</sup> reactor. .	61
4-3	Assumed values to calculate methane production prior aeration. . . . .	61
4-4	Assumed values to calculate methane production prior aeration. . . . .	62

# 1 Introduction

## 1.1 Background

The world faces environmental crisis due to population growth, industrialisation and urbanisation which increase the volume of wastewater. In 2003, 1.6 million people died of unsafe water and sanitation (WHO, 2015). So, treatment of wastewater is necessary before disposal in order to reduce environmental contamination and health hazards. New trends in wastewater treatment have been developed over years to combat against these problems in an effective and economic way.

A large quantity of wastewater is released as domestic sewage and effluent from food and food processing, dairy, distillery, tannery, oil, pulp and paper, and biotechnological industries. Effluents may contain pathogens, toxic chemicals and organic chemicals (Drechsel and Evans, 2010). The effects of unmanaged wastewater includes direct impact on biological diversity of the aquatic ecosystems which will ultimately disrupt the system supporting our fundamental integrity (Latif et al., 2011).

## 1.2 Industrial Wastewater

Industrial wastewater is very complex in the sense that each industry has its own specific constituents and pollution range is very wide in terms of types and concentrations (Asadi et al., 2012). Flora and fauna of the wastewater receiving water bodies are adversely affected if not treated properly (Botalova and Schwarzbauer, 2011). Oil refinery wastewater is one of the major industrial wastewater. Diya'uddeen et al. (2011) reports that oil will account for 32% of the world's energy supply by 2030 which will increase the oil demand rise to 107 mbpd over next two decades increasing more oil refinery waste which are difficult to treat (Xianling et al., 2005). This wastewater is characterised by high concentration of aliphatic and aromatic petroleum hydrocarbons which affects plants and aquatic life of surface and ground water sources (El-Naas et al., 2009). Untreated petroleum industry wastewater is similar to municipal wastewater in terms of pollutants and in addition also contains oil and grease, various hydrocarbons, phenolics, sulfides, and metals which makes the discharge a matter of concern because of potential toxicity of these constituents (Knight et al., 1999). Industrial oil refinery wastewater has been usually treated biologically as a well-established method to reduce its effects (Jou and Huang, 2003).

## 1.3 Treatment Method

In many environmentally sensitive and water-scarce areas, wastewater treatment and reuse has emerged as the most practical solution to combat water scarcity. Recent developments of high efficient treatment units have made the reuse of treated wastewater easier and economical. Stringent international regulations has acted as a catalyst for the development of advanced treatment facilities. High effluent quality, low maintenance and space requirement and operational costs are the most desired aspects of a competitive facility.

Anaerobic, anoxic and aerobic biological wastewater treatment systems are usually adapted in combination to enhance the removal of organics and nutrients and integrating them in a

single reactor with high biomass content is the most economic and effective method (Asadi et al., 2012). Treatment of low strength wastewater (biodegradable COD <1000 mg/L) is suitable by aerobic system while anaerobic system is recommended for high strength wastewater (biodegradable COD >4000 mg/L) (Chan et al., 2009). However, anaerobic digestion can also be used for low strength wastewater.

Aerobic or anaerobic treatment alone may not produce required effluent quality while treating high organic strength industrial wastewater (Chan et al., 2009). Chong et al. (2012) has listed many post-treatment of Upflow Anaerobic Sludge Blanket (UASB) effluent which are required for stabilisation or effluent polishing to comply with stringent effluent standards. These include: UASB-activated sludge (AS), UASB-sequencing-batch reactor (SBR), UASB-biofilter (BF), UASB-downflow hanging sponge (DHS), UASB-stabilising pond (SP), UASB-rotating-biological contactor (RBC), UASB-constructed wetland (CW), UASB-dissolved-air-flotation (DAF) etc. The use of anaerobic-aerobic treatment reduces the operation cost by factor of eight when compared to aerobic treatment alone (Vera et al., 1999) and also results in high organic matter removal, low aerobic sludge production and no pH correction (Chan et al., 2009). Benefits of the anaerobic-aerobic process are listed below: (Cervantes et al., 2006),(Frostell, 1983).

- Increased treatment efficiency: Anaerobic effluent is polished by aerobic post-treatment resulting in very high treatment efficiency and the aerobic treatment also smooths out fluctuation in the quality of the anaerobic effluent
- Low sludge volume: The cost of sludge disposal is low as excess aerobic sludge is digested in the anaerobic stage which produces a minimum stabilised total sludge. Gas yield is also increased.
- Reduced energy consumption: Anaerobic pretreatment also works as influent stabilisation tank which reduces diurnal variations of oxygen demand reducing aeration. Biogas as renewable energy is also generated.
- Volatile organics are degraded in anaerobic zone removing the possibility of volatilisation in the aerobic treatment

Figure 1–1 shows the three main types of combined anaerobic-aerobic system currently in use, with distinctions made between the different approaches used to obtain an anaerobic-aerobic reactor system. Four types of integrated anaerobic-aerobic bioreactor are (i) integrated bioreactors with physical separation of anaerobic-aerobic zone, (ii) integrated bioreactors without physical separation of anaerobic-aerobic zone, (iii) Sequencing Batch Reactors (SBR) based on temporal separation of the anaerobic and aerobic phase, and (iv) combined anaerobic-aerobic culture system based on the principle of limited oxygen diffusion in microbial biofilms (Chan et al., 2009).

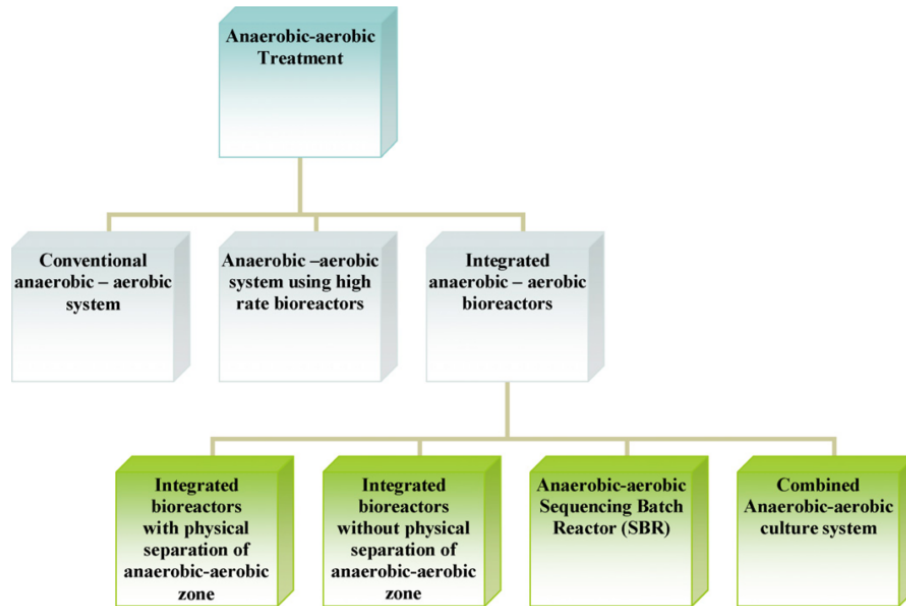


Figure 1–1: Types of combined anaerobic-aerobic system (Chan et al., 2009)

In recent years, researchers are working in development of combined aerobic-anaerobic process in a single reactor. It has been reported that combined aerobic-anaerobic degradation pathways in a single reactor can enhance overall degradation efficiency of the system (Chan et al., 2009). A high-rate Hybrid Vertical Anaerobic Biofilm (HyVAB<sup>®</sup>), belonging to type i, has been used successfully in laboratory and pilot scale for treating high-strength oil refinery (del Mar Batista Seguí, 2014)(Wang et al., 2015a). HyVAB<sup>®</sup> is a hybrid setup with aerated biofilm carrier chamber vertically above an anaerobic sludge chamber with basic design criteria as aerobic biofilm reactor and anaerobic sludge bioreactor (Phattaranawik and Leiknes, 2010). This stacked configuration reduces space requirements, provide lower capital cost and gives higher COD removal rate. The biogas produced in anaerobic zone is channeled from the intermediate height ensures that the methane produced is not oxidised in the aerobic zone (Chan et al., 2009). Bubbled air is used to aerate the upper chamber containing biofilm carriers which is separated from anaerobic chamber by roof like structure called as three phase separator. It helps in preventing oxygen diffusion into the anaerobic chamber and controlling the direction of excess sludge floc settlement (Phattaranawik and Leiknes, 2010). Most of the organic waste is stabilised at anaerobic chamber producing biogas and remaining unconsumed organic waste is consumed by aerobic biofilm anabolism and metabolism (Wang et al., 2015a).

## 1.4 Objectives

This thesis basically evaluates the performance of newly developed HyVAB<sup>®</sup> bioreactor on high strength (~10000 mgCOD/L) oil refinery wastewater. The bioreactor is of hybrid type consisting of an anaerobic stage at the bottom and an aerobic stage at the top operated as CFIC<sup>®</sup> in washing mode. Laboratory scale bioreactor is installed at HSN and has been running since March 2015 while pilot scale bioreactor is installed at NSO and is running since February 2016 (inception of the thesis). This thesis considers close evaluation of pilot scale bioreactor from the

startup while evaluation of laboratory scale is based on established facility. The following points are mainly considered as objectives. The detailed task description is shown in the Appendix.

- Analysis of laboratory and pilot scale HyVAB<sup>®</sup> reactor including experimental planning, bioreactor operation, sampling and analysis of samples
- Establish mass and energy balances for the bioreactors from the experimental results
- Evaluation of process performance
- Propose for industrial implementation
- Economic analysis of HyVAB<sup>®</sup> reactor compared to aerobic process

## **1.5 Structure of the thesis**

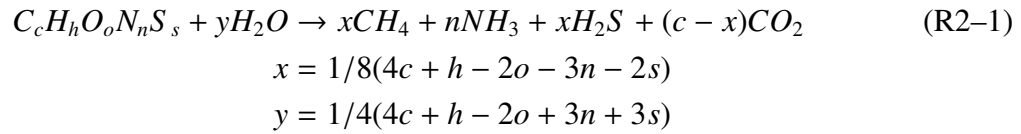
This chapter gives general introduction about the industrial wastewater and treatment approaches. It lists the objective of this thesis and gives brief introduction about the high-rate Hybrid Vertical Anaerobic Biofilm (HyVAB<sup>®</sup>). Chapter 2 gives literature study of different processes involved in biological treatment including aerobic and anaerobic processes. It also briefly explains about the inhibition and enhancement factors of digestion processes. Chapter 3 mainly deals with the methods applied during this experiment including the general introduction about the reactor design and startup. Results are presented in Chapter 4. Chapter 5 interprets and discusses the results obtained to draw conclusions and provide recommendations. Chapter 6 gives conclusions.

## 2 Literature Review

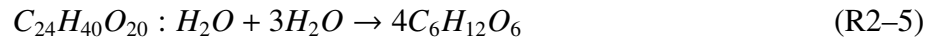
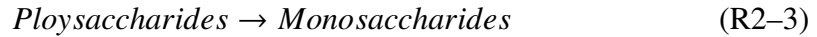
### 2.1 Anaerobic Digestion

Anaerobic digestion is implemented for its ability to reduce Chemical Oxygen Demand (COD) and Biochemical Oxygen Demand (BOD) from waste streams of agriculture, food and wastewater sludge and producing renewable energy (Chen et al., 2008). It is governed by critical operating parameters and reactor design such as continuity, operating temperature, reactor design and solid content (Li et al., 2011). Hydrolysis of complex organic substrates such as proteins, fat, and lipid takes place in the anaerobic digestion followed by fermentation to acetate, formate, hydrogen and carbon dioxide which are converted to methane by methanogenesis organisms (Gujer and Zehnder, 1983).

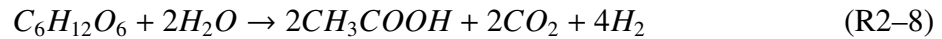
Reaction R2-1 shows transformation of feed organic solids in to biogas, which is a mixture of CH<sub>4</sub>, CO<sub>2</sub> and traces of other gases, in an anaerobic condition (Tezel et al., 2011)



Anaerobic digestion consists of three basic steps as shown in Figure 2-1. In the first hydrolysis process, particulate material is converted to soluble compounds for organisms to hydrolyse to simple monomers while some industrial wastewater might lack this step (Tchobanoglous et al., 2003). Reaction R2-3 shows an example of hydrolysis reaction where a polysaccharide is broken down into glucose (Clark et al., 2009), (Kayhanian, 1995). Hydrolysis reactions are as follows:



In fermentation (also called acidogenesis), amino acids, sugars, and some fatty acids are degraded to acetate, hydrogen and CO<sub>2</sub> which are precursors of methane formation; in methanogenesis, methanogens are involved in methane production. Reactions R2-6, R2-7 and R2-8 show how glucose is converted to ethanol, acetate, and propionate respectively (Kangle et al., 2012).



Aceticlastic methanogens converts acetate into methane and CO<sub>2</sub> as in Reaction R2–9 (Kangle et al., 2012) while hydrogen utilising methanogens use hydrogen as the electron donor and CO<sub>2</sub> as electron acceptor to produce methane (Tchobanoglous et al., 2003) as shown in Reaction R2–10 (Kangle et al., 2012).



Anaerobic process is preferred over aerobic processes for the treatment of high organic carbon concentrated wastewater because of less sludge production and methane produced can be used for heat or electricity generation (Escudié et al., 2011). Low energy consumption, macro/micro nutrients demand and space requirement are other benefits of anaerobic digestion (Lim and Kim, 2014). Ye et al. (2011) has demonstrated anaerobic digestion to be the most useful technology in treating high strength organic wastewater.

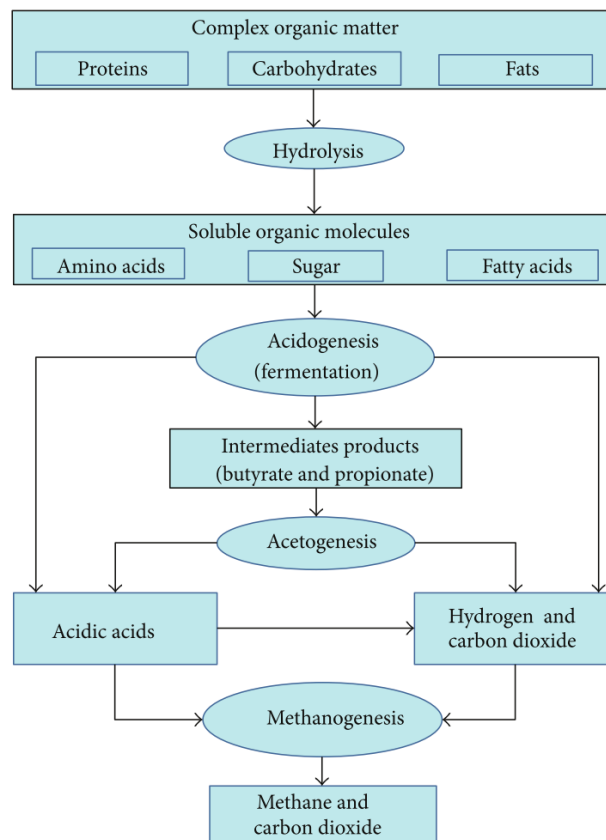


Figure 2–1: The key process stages of anaerobic digestion (Abdelgadir et al., 2014)

### 2.1.1 Process Fundamentals

pH and alkalinity, temperature, availability of nutrients, presence of inhibitory substances and retention times are important parameters that affect the rates of the different steps of the diges-

tion process (Appels et al., 2008)(Tchobanoglous et al., 2003).

**pH:** Optimal pH range is different for each group of microorganism. An effective conversion rate for sensitive methanogenic organisms is in pH range 6-7 (Daisy and Kamaraj, 2012). pH decreases during the initial period of digestion as large amounts of organic acids are produced. When the concentration of ammonia increases with the digestion of nitrogen containing substances at the later stage of digestion, pH increases (Abbasi et al., 2012). pH remains 7.2 to 8.2 when methane gas production stabilises (Verma, 2002). pH drop in the reactor and increase in CO<sub>2</sub> concentration in biogas indicates a disturbance in the digestion process (Abbasi et al., 2012).

**Alkalinity:** It is very important to maintain proper alkalinity in anaerobic digestion to reduce the large pH fluctuations during initial and growth period (Singh et al., 1999). At low buffering capacity, even small increase in VFAs concentration can reduce pH which adversely affect the methanogens (Singh et al., 1999). Extra cost of maintaining alkalinity concentration of 2000 to 3000 mg/L as CaCO<sub>3</sub>, by adding lime, soda ash, sodium bicarbonate, or magnesium hydroxide, is the most negative factor of anaerobic treatment compared to aerobic treatment (Tchobanoglous et al., 2003). Addition of a moderate amount of NaHCO<sub>3</sub>(≈0.84-1.68 g/l) can overcome the problem in the reactor when the population of acid-utilising bacteria is not in pace with the growth of acid forming bacteria (Singh et al., 1999). The relationship between pH and alkalinity is determined by bicarbonate chemistry as shown in Reaction 2-1(Tchobanoglous et al., 2003).

$$K_{a1} = \frac{[ \text{HCO}_3^- ] [ \text{H}^+ ]}{[ \text{H}_2\text{CO}_3 ]} \quad (2-1)$$

where,  $K_{a1}$  = first acid dissociation constant, which is a function of ionic strength and temperature

Once the carbonic acid concentration is known as shown in reaction 2-2, the bicarbonate (HCO<sub>3</sub><sup>-</sup>) alkalinity needed to maintain the required pH is estimated.

$$x_g = \frac{P_T}{H} p_g \quad (2-2)$$

where,

- $x_g$ =mole fraction of gas in water, mole gas/mole water
- $P_T$ =total pressure, usually 1.0 atm
- $H$ =Henry's law constant
- $p_g$ = mole fraction of gas in air, mole gas/mole of air



**Temperature:** Temperature not only influences the metabolic activities of microbial population but also has impact in gas transfer rates and settling characteristics of biological solids (Tchobanoglous et al., 2003). It has profound influence on the growth rate and metabolism of micro-organisms influencing overall population dynamics in the anaerobic reactor; however, stable operating temperature should be maintained to avoid negative effect on bacteria, especially methanogens (Appels et al., 2008). Thermophilic anaerobic digestion (55°C) is generally more efficient than the mesophilic (37°C) but thermophilic process needs extra energy input and difficult control mechanism (Eliyan, 2007). Thermophilic anaerobic digestion, contrary to mesophilic, has additional benefits including a high degree of waste stabilisation, thorough destruction of viral and bacterial pathogens and helps in improvement of post treatment sludge dewatering (Lo et al., 1985). Also, rates of hydrolysis drop sharply with the temperature (Sayed et al., 1984).

**Solids and Hydraulic Retention Times:** HRT and SRT are important parameters to be considered while designing a bioreactor to allow significant destruction of particulate matter. SRT is the average time of biomass held in the digester while HRT is the average time the liquid held in the digestion process. SRT is total sludge present in reactor (kg) divided by sludge withdrawn per day (kg/d). SRT is a fundamental design and operating parameter for all anaerobic processes. In anaerobic reactors, SRT has to be in excess of HRT to maintain higher biomass densities (SRT >>HRT). Higher SRT gives high rate anaerobic treatment and provide greater resistance to any inhibitory substances in the influent. Usually values of greater than 20 days are needed for effective treatment process and higher values for lower temperature (Tchobanoglous et al., 2003). Hydrolysis, fermentation, and methanogenesis are directly related to SRT (or HRT) as increase or decrease of SRT can increase or decrease in the extent of each reaction (Tchobanoglous et al., 2003). Najafpour et al. (2006) applied constant HRT of 1.5 days during a start-up of hybrid anaerobic reactor inoculated with a granular sludge and obtained OLR of 23 kg COD/m<sup>3</sup>.d after 26 days. HRT is the ratio of volume of digester to influent flow rate:

$$HRT[d] = \frac{\text{Volume of the reactor [m}^3\text{]}}{\text{Influent flow rate [m}^3\text{/d]}} = \frac{V}{Q} \quad (2-3)$$

where, HRT is hydraulic retention time (d), V is the volume of the reactor (m<sup>3</sup>), Q is the influent flow rate (m<sup>3</sup>/d)

**Organic Loading Rate (OLR):** Liquid flow rate and influent COD concentration are taken into account in OLR which is defined as the mass of pollutant introduced in a unit volume of the reactor per unit time. According to claim of several authors, the treatment efficiency of complex wastewater increases with increase in OLR up to a certain limit (Abdelgadir et al., 2014). A further increase can lead to operational problems like sludge bed floatation, excessive foaming in the gas-liquid-solid (GLS) separator and accumulation of undigested ingredients (Abdelgadir et al., 2014). OLR is increased progressively and continuously during start-up with careful monitoring to avoid overloading of the system which could inhibit methanogens and hamper the start-up process (Escudié et al., 2011). The OLR can be controlled by changing the influent COD concentration and by changing the flow rate.

$$OLR = \frac{(Q \times COD)}{V} \quad (2-4)$$

where, OLR is organic loading rate (kgCOD/m<sup>3</sup>.d), Q is flow rate (m<sup>3</sup>/d), COD is chemical oxygen demand(kg COD/m<sup>3</sup>), and V is reactor volume (m<sup>3</sup>).

The above reaction can be simplified as:

$$OLR = \frac{COD}{HRT} \quad (2-5)$$

**Food-to-Organisms Ratio (F/M) or Sludge loading rate and Specific methanogenic activity (SMA):** F/M or sludge loading rate is a ratio of influent COD concentration and microbial mass and is defined as the mass of substrate loaded to a unit mass of microbial mass per unit time. SMA is defined as the fraction of organic load biodegraded in a unit mass of sludge. Following relationships are used to determine the above parameters:

$$\frac{F}{M} = \frac{Q \times COD_{in}}{V \times VSS} \quad (2-6)$$

$$SMA = \frac{Q \times (COD_{in} - COD_{out})}{V \times VSS} \quad (2-7)$$

where, Q is the influent flow rate (L/day), V is the volume of the reactor (L) and VSS is the sludge concentration in the reactor.

**Inhibition on Anaerobic Digestion:** Process sensitivity, failure prone, odor problems, long start-up period, low methane yield and post treatment for effluent discharge are some disadvantages to be considered but injection of enough inoculum can overcome these problems (Lim and Kim, 2014), (Abdelgadir et al., 2014). Ammonia, sulfide, light metal ions, and heavy metals, and organics are the most common inhibitors of anaerobic processes (Chen et al., 2008) which are either present in the influent itself or are generated during the digestion (Appels et al., 2008). Anaerobic digestion process is also inhibited by high ammonia concentration (Hansen et al., 1998) and methanogens are the least tolerant among the four types of anaerobic microorganisms and most likely stop the growth due to ammonia inhibition (Kayhanian, 1994). Most of industrial wastewaters contain sulfate and it is reduced to sulphide by sulfate reduction bacteria (SRB) (O’Flaherty et al., 1998)(Koster et al., 1986). SRBs compete with methane producing bacteria (MPB) (or archaea) for utilisation of hydrogen and acetate leading to lower methane production and SRBs convert to sulphide which is toxic to both MRBs(or archaea) and SRBs at higher concentration (Zhou and Fang, 1998). Light metal ions present in the influent of anaerobic digester are required in moderate concentration for microbial growth but higher concentration can cause severe inhibition or toxicity (Soto et al., 1993). Although minimal information is found about aluminium inhibition in literatures, both acetogenic and methanogenic microorganisms were found to be inhibited by addition of Al(OH)<sub>3</sub> (Shayegan et al., 2005).

**Nutrients requirements** Anaerobic process requires macronutrients to sustain growth and perform biochemical transformations in microbial treatment (Singh et al., 1999). The quantity of biological nutrients, nitrogen and phosphorus is directly proportional to their growth (McCarty, 1964). Availability of adequate quantities of nitrogen, phosphorus, micronutrients, and water is required for an organic substrate to degrade anaerobically and produce methane-rich gas (Singh et al., 1999),(Takashima et al., 2011). Singh et al. (1999) has concluded on various

formulations of nutrients and trace metals used in different studies on UASB reactors that the feed medium must contain essential nutrients like N, P, Mg, Ca, K and yeast extract as well as trace elements like Fe, Al, Zn, Ni, Co, Mo, Cu, B, Se, Resazurine, and ethylene diamine tetra acetic acid (EDTA) to cultivate heterogenous anaerobic cultures in UASB reactors. Micronutrients are known for their role as biochemical cofactors for the methane producing Archaea (Bhattacharya et al., 1995). Calcium is known to be essential for the growth of certain strains of methanogens (Murray and Zinder, 1985). Supplement of N,P and K prevents floatation of granules as well as retards the effects of shock loading. Common nitrogen and phosphorus sources are  $\text{NH}_4\text{Cl}$  and  $\text{NaH}_2\text{PO}_4$  respectively in the ratio of COD/N/P equal to 400/7/1 (Cresson et al., 2007),(Cresson et al., 2006). González et al. (1998) found that upon stopping addition of nutrients from 80<sup>th</sup> day, the process performance was as it was without any adverse affect on sludge granulation and showed a good acclimatisation of the micro-organisms and the process as well stable. However, the characteristics of the feed was similar throughout the experiment.

### **2.1.2 UASB**

The anaerobic sludge blanket (UASB) was developed in the late 1970s in the Netherlands (Lettinga, 1996). Around 80% of the world's anaerobic wastewater treatment is assumed to be based on UASB technology (Abbasi and Abbasi, 2012). A schematic diagram is shown in Figure 2–2. Biological reactions take place throughout the highly active sludge bed and blanket zone where soluble organic matters are converted to biogas (Chong et al., 2012). Biogas is trapped by the three phase separator and is collected to use as renewable energy. Three phase separator (or Gas Solids Separator (GSS)) is installed to avoid washout of active granules or bacterial flocs which are settled back in the sludge bed. Active granules have made it able to treat wastewater of higher strength as well as municipal wastewater (Lim and Kim, 2014). Feed pumped vertically upward from the bottom expands the sludge bed allowing microflora to act upon the wastewater. The core factors which determine the success of UASB reactors are quality of granular sludge (to withstand shock loads) and contact of sludge and wastewater (Abbasi and Abbasi, 2012; Rajeshwari et al., 2000). Hydraulic retention time (HRT) and Solid retention time (SRT) are uncoupled to maintain high biomass retention in high rate systems like UASB (Rajeshwari et al., 2000). Although slow start up is a disadvantage of UASB, it can be countered by enhancing granulation process by adding mature granular sludge, adding certain natural and synthetic polymers or external additives (Gujer and Zehnder, 1983). Advantages and disadvantages of UASB are listed in Table 2–1. Apart from these advantages, effective removal of Chemical Oxygen Demand (COD) by granules or flocculent sludge without the need of a support material is an attraction of UASB (Chong et al., 2012).

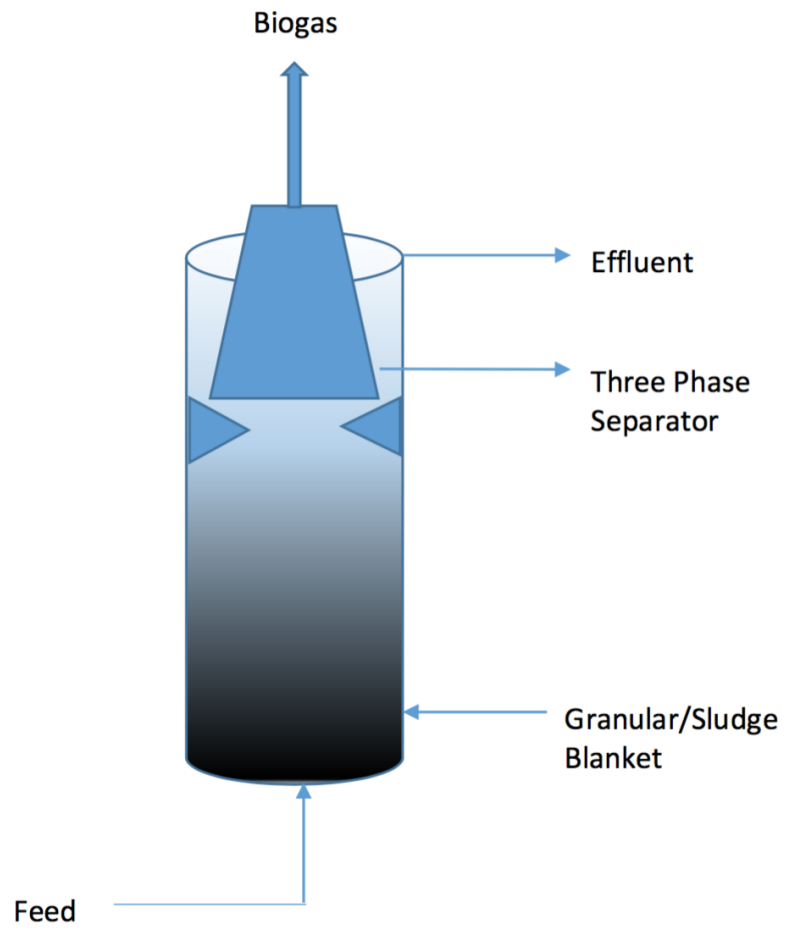


Figure 2-2: Schematic diagram of UASB reactor

Table 2–1: Advantages and disadvantages of UASB reactor (Latif et al., 2011),(Lim and Kim, 2014), (Chong et al., 2012), (Abbasi and Abbasi, 2012)

Advantages	Disadvantages
Good removal efficiency can be achieved even at high loading rates and low temperature	Pathogens removal is partial, except helminthes eggs which are effectively captured in sludge bed. Incomplete nutrient removal, so post treatment is often required
Relatively simple construction and operation and locally produced construction material, plant components, spare parts with low maintenance is possible	Longer startup before steady state operation due to slowly growing methanogenic organisms if no seeding
Anaerobic treatment can be applied on very large to very small scale	Hydrogen Sulphide can cause nuisance if the influent containing high sulphur is not handled properly
Low cost due to high organic loading and small area	Post treatment is usually required to meet the stringent regulations on organic matter (OM), nutrients and pathogens
Energy consumption is low. Also methane produced can be used as a source of energy	15–35°C should be maintained for colder climates
Less sludge production due to low yield. The sludge has good dewatering characteristics and is well stabilised for final disposal	Low performance at very low loading
Organic shock loads are handled effectively	
pH can be stabilised without addition of chemicals and low nutrients and chemical required especially in case of sewage	
Verified reactor performance and design	

### 2.1.3 Granules

Liu et al. (2002) points out ‘Anaerobic granule indeed can be regarded as the gathering together of cells to form fairly stable, contiguous, multicellular association under physiological conditions in a defined biological system’. Granulation process with a particular organic wastewater determines the functionality of UASB system as anaerobic granular sludge is the main component (Liu et al., 2002). Typical sludge granules in UASB reactors are in range of 0.4 to 3 mm (Boardman et al., 1995). Feed strength, reactor hydraulics, nutrients supplements and other factors can be well manipulated to transform sludge particles to granules which significantly

improves the overall performance of UASB by providing more active biofilms and allowing intimate substrate-microorganism contact which ultimately strengthen the reactor to withstand shock-loads (Abbasi and Abbasi, 2012). Granular sludges are rich in microbial communities of different species, dense and synchronisation among each individual species is required to degrade complex organic wastes (Liu et al., 2002). A common problem of spontaneous and sudden washout of the established granular sludge bed is encountered when there is change in wastewater composition which can be explained reasonably by cell-to-cell communication mechanism (Liu et al., 2002). This problem of washout is explained as disruption of spatially organised UASB granules associated bacteria upon change of composition of wastewater to best cope with the constraints imposed by the substrate fed and corresponding metabolic processes (Liu et al., 2002). Gujer and Zehnder (1983) have pointed out the factors that influence the mechanism of granule formation and impact of granules on treatment efficiency:

- Operational temperatures determine granule composition in a UASB as different species achieve optimum growth rates at different temperatures. Sudden increase of temperature can disintegrate granules.
- Alkalinity should be in optimum quantity to maintain the reactor pH and buffer VFA concentration fluctuations.
- High partial pressure of hydrogen and neutral pH enhance granulation. Optimum range of HRT and OLR should be maintained to keep the granules intact.
- Layer geometry of granules is determined by the substrate type and strength.
- Adequate concentrations of bioavailable nutrients and certain metals is essential for granulation. However, higher than essential concentrations can hamper the process.

#### **2.1.4 Biogas**

Biogas, produced by anaerobic digestion, is a clean and environmentally friendly fuel containing 55-65% of CH<sub>4</sub>, 30-40% of CO<sub>2</sub>, water vapour fractions, traces of H<sub>2</sub>S and H<sub>2</sub> and other contaminants like siloxanes (Appels et al., 2008). Methane gas has a Lower Heating Value (LHV) of 35800 kJ/m<sup>3</sup> while LHV of biogas (assuming 65% methane) is 22400 kJ/m<sup>3</sup> at standard temperature and pressure (20°C and 1 atm) (Tchobanoglous et al., 2003). It can be used for heating without any treatment at the site of production. However, removing of contaminants can increase its energy content making it transportable over larger distance after compression to use as renewable fuel. Removal of water moisture, H<sub>2</sub>S and trace gases is important to make use of biogas in engines. Gas formed in digester has a specific gravity of approximately 0,86 relative to air (Tchobanoglous et al., 2003). Gas production is one of many indicators of health of the digester, so it has to be monitored well. Depending on the volatile solids content of the feed and biological activity, gas production can fluctuate over time.

## **2.2 Aerobic Digestion**

Aerobic digestion is a process in which biodegradable COD is taken up by heterotrophic bacteria to mineralise a fraction to CO<sub>2</sub> and water, another fraction to store as intracellular bipolymers

for later use and rest for biomass synthesis to increase the amount of bacteria (Smitshuijzen et al., 2016). According to Tchobanoglous et al. (2003), under all operating conditions the oxygen residual should be maintained at 1 mg/L or above.

### 2.2.1 Biofilm

Biofilms are complex structures which consist of collection of microorganisms held together by extracellular polymer substances (EPS) and a balance between biomass formation and detachment rates influence the biofilm morphology (van Loosdrecht et al., 1995). Microbial cells grow using nutrients whose availability are influenced by the fluid flow as they are transported by diffusion and convection and influence the biomass formation along with EPS formation (Bottero et al., 2013). Nicoletta et al. (2000) concluded that the shear force on the biofilm primarily influenced the biofilm structure which depended on the reactor types. Hence, it is difficult to obtain dense and compact biofilm in aerobic systems.

### 2.2.2 Continuous Flow Intermittent Cleaning (CFIC®)

The Continuous Flow Intermittent Cleaning (CFIC®) biofilm reactor, patented by Biowater Technology with the help of external R&D institutions, contains highly packed biofilm carriers (typically 90-99% bulk volume fill) allowing little movement of carriers during normal operation. CFIC® is similar to Moving Bed Biofilm Reactor (MBBR) which has filling ratio of 40-60% (Ødegaard, 2006). Typical process has continuous inflow of the feed removing excess biomass from the carriers intermittently (Rusten et al., 2011). Carriers are designed as such to provide large surface to volume ratio so that more biomass grows on the carriers and reduce the frequency of cleaning cycle by increasing turbulence during cleaning cycle (Rusten et al., 2011). Figure 3–5 a shows the normal operation while Figure 3–5 b shows the operation during cleaning cycles. Rusten et al. (2011) verified that CFIC® process produced lower effluent sCOD and TSS concentration at higher biofilm surface loading and volumetric loading rates than MBBR process.

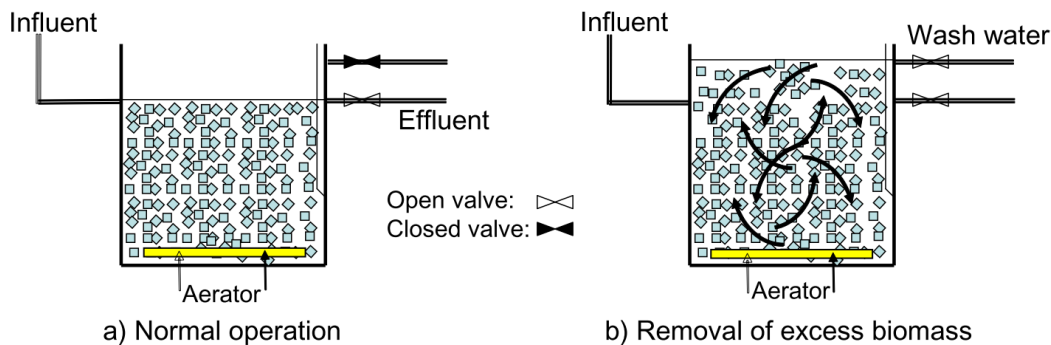


Figure 2–3: The CFIC® during a) normal operation, and during b) the cleaning cycle (Rusten et al., 2011)

Advantages of CFIC® compared to activated sludge and MBBR are: (Andersen, 2012)

- Higher oxygen transfer rate, due to a longer lifetime of the air bubble, leading to lower energy consumption.
- Can be integrated into existing WWTP to lower CAPEX by utilising existing infrastructure.
- TSS is reduced drastically even at higher loading rates.
- Integrating CFIC® leads to compact bioreactors due to optimised process and bio-carrier design.

## 2.3 Economic Analysis

Aeration cost accounts to 45-75% of plant energy costs making it the most energy-intensive operation in wastewater treatment (Rosso et al., 2008). Intensive works are being done to reduce the aeration cost by integration of anaerobic digestion to aerobic digestion. Anaerobic digestion prior aerobic digestion can save good amount of money depending upon the types of anaerobic reactor used. Aerobic digestion can be used to polish the effluent from anaerobic digester reducing significant operational cost. Vera et al. (1999) upon parametric sensitivity analysis showed that the total annual cost increases exponentially with the COD concentration, increases linearly with the energy cost and decreases with increased COD legal limit and is insensitive to oxygen cost; however, opting for oxygen over air for aeration makes aerobic degradation 27% cheaper. The oxygenation cost (US\$ day<sup>-1</sup>) can be found using Equation 2–8 (Vera et al., 1999):

$$C_{oxyg} = F_{O_2} \frac{\delta}{\eta_{oxyg}} \quad (2-8)$$

where,  $F_{O_2}$ =required oxygen flow in the aerobic reactor,  $\delta$ = oxygen cost(US\$ day<sup>-1</sup>),  $\eta_{oxyg}$ = oxygenation efficiency

The oxygen's mass flow is calculated as (Ramalho, 2012):

$F_{O_2} = F_{[O_2]fororganicmatteroxidation} + F_{[O_2]forendogenousrespiration}$   
and, according to material balances (Ramalho, 2012):

$$F_{O_2} = QS_0(1 - f_1)f_2(1 - 1.42Y_{X/S_2}) + 1.42\epsilon_2X_2V_{ae} \quad (2-9)$$

where, 1.42 is the amount of oxygen consumed by the cells for substrate oxidation per amount of cells produced by this process [kg O<sub>2</sub> (kg biomass)<sup>-1</sup>]. And,  $\epsilon_2$  approaches a value of 0.1 [kg O<sub>2</sub> (kg biomass)<sup>-1</sup> day<sup>-1</sup>],  $f_1$  is the substrate conversion factor in the anaerobic reactor,  $f_2$  is the substrate conversion factor in aerobic reactor,  $Y_{X/S_2}$  is the cellular yield in the aerobic reactor [kg biomass or VSS (kg COD)<sup>-1</sup>],  $S_0$  is substrate concentration at the anaerobic reactor inflow [kg COD m<sup>-3</sup>],  $Q$  is volumetric flow of the effluent to be treated [m<sup>3</sup> day<sup>-1</sup>],  $X_2$  is sludge concentration [kg m<sup>-3</sup>] and  $V_{ae}$  is volume of the aerobic reactor [m<sup>3</sup>] as given by Equation 2–10 (Vera et al., 1999).

$$V_{ae} = Q \frac{F_{OD2}\theta_c Y_{X/S_2}(S_1 - S_2)}{X_2(1 + b_2\theta_c)} \quad (2-10)$$

where,  $Q$  is volumetric flow of the wastewater to be treated [m<sup>3</sup> day<sup>-1</sup>],  $F_{OD2}$  is overdimension factor for aerobic reactor,  $\theta_c$  is sludge hydraulic retention time[day],  $Y_{X/S_2}$  is cellular yield



in the aerobic reactor [kg biomass or VSS (kg COD)<sup>-1</sup>],  $S_1$  is substrate concentration of the anaerobically treated effluent [kg COD m<sup>-3</sup>],  $S_2$  is substrate concentration of the aerobically treated effluent [kg COD m<sup>-3</sup>],  $X_2$  is sludge concentration [kg m<sup>-3</sup>], and  $b_2$  is specific decay rate in the aerobic reactor [day<sup>-1</sup>].

Using methane produced by the anaerobic degradation of the organic matter gives an economic benefit (US\$ day<sup>-1</sup>) as given by Equation 2–11 (Ramalho, 2012):

$$C_{CH_4} = \Omega_{comb} G_o Q f_1 S_0 (1 - 1.42 Y_{X/S_1}) \beta \quad (2-11)$$

where,  $\Omega_{comb}$  is methane's combustion heat [Jm<sup>-3</sup> STD CH<sub>4</sub>],  $G_o$  is methane's theoretical yield [m<sup>3</sup> STD CH<sub>4</sub> (kg COD)<sup>-1</sup>],  $Q$  is volumetric flow of the wastewater to be treated [m<sup>3</sup> day<sup>-1</sup>],  $f_1$  is the substrate conversion factor in the anaerobic reactor,  $S_0$  is substrate concentration at the anaerobic reactor inflow [kg COD m<sup>-3</sup>],  $Y_{X/S_2}$  is the cellular yield in the aerobic reactor [kg biomass or VSS (kg COD)<sup>-1</sup>] and  $\beta$  is power cost [US\$ J<sup>-1</sup>]

## 3 Methods and Materials

### 3.1 Wastewater preparation

The feed used in both the reactors was from Norsk Spesialolje (NSO) Bamble, Norway. The wastewater collected from manufacturing industry, auto repair shops, shipping and oil & offshore industry is cleaned using distillation process to separate oil from water of the used oil. The detailed process adopted in NSO is shown in Figure 3–1. The wastewater used as feed in these reactors comes from the distillation carried out during the cleaning process before going through any chemical or biological process. With an average COD of 10 g/L, it ranged from 6-16 g/L due to different sources. Its characteristics are given in Table 3–1 . NaOH and HNO<sub>3</sub> were used whenever necessary to maintain the pH in the range of 6.5 to 7.5. Sometimes, feed was diluted with tap water in the case of laboratory reactor. NSO injected nutrients in the feed (N and P) for stimulating organisms. Also, flower nutrients (containing mainly N, P and K and other trace metals) were added in the feeding tank to fulfil micro nutrients requirements before pumping in to the anaerobic stage as shown in Figure 3–4. NaHCO<sub>3</sub> was used as alkalinity (1500 mg/L as CaCO<sub>3</sub>) for maintaining neutral pH.

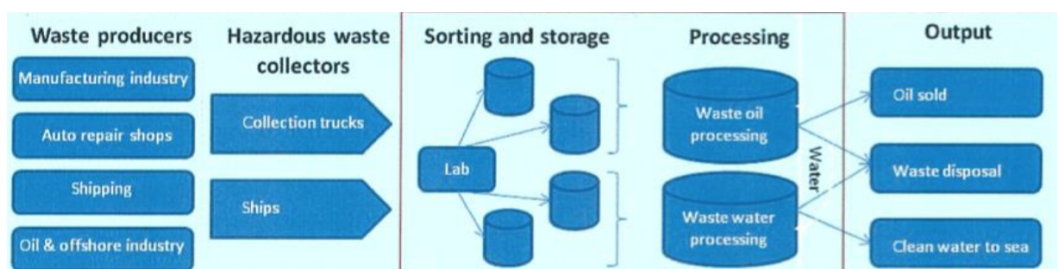


Figure 3–1: Processing diagram in NSO (del Mar Batista Seguí, 2014)

Table 3–1: Characteristics of wastewater used as HyVAB<sup>®</sup> feed at NSO (Bamble) (after chemical addition)

Parameters	Units	Average	Range
pH	-	7.3	8.7-5.6
COD	mg/L	9802	16080-6250
Conductivity	μS/cm	3976.67	2300-5520
Sulphate	mg/L	6	-
Sulphide	mg/L	0.034	-
Alkalinity	mg CaCO <sub>3</sub> /L	1059.25	937.5-1181

### 3.2 Reactor Design and Setup

#### 3.2.1 Pilot Scale Reactor

**Reactor Design:** The pilot scale HyVAB<sup>®</sup> reactor as shown in Figure 3–4 has been used to treat high strength oil wastewater since 18<sup>th</sup> February 2016 at NSO, Bamble, Norway. The

reactor is made up of glass tube (an upper part of AD was later changed to PVC due to leakage) having a cross sectional area of  $0.018 \text{ m}^2$ . Working volume of lower UASB section was 44 L while upper aerobic stage was 22 L with total height of 3.75 m. The aerobic biofilm stage, termed as CFIC<sup>®</sup>, is filled with mature BWTS<sup>®</sup> biofilm carriers (as shown in Figure 3–2) from NSO's aerobic reactor (carrier effective surface to volume ratio  $650 \text{ m}^2/\text{m}^3$ ). Aerobic chamber was filled with approximately 3280 numbers of carriers. Characteristics of biofilm carrier is given in Table 3–2. Aeration from the bottom of CFIC<sup>®</sup> stage helps in cultivating heterotrophic bacteria to consume COD from AD section.

Three phase separator used in biogas collection, retaining anaerobic granular sludge and facilitating detached aerobic sludge to settle in to the anaerobic stage is installed in between the two stages. It is shown in Figure 3–3.



Figure 3–2: Mature BWTS<sup>®</sup> biofilm carriers



Figure 3–3: Three phase separator installed in pilot scale HyVAB<sup>®</sup> bioreactor at NSO

Table 3–2: Characteristics of biofilm carriers used for CFIC<sup>®</sup> stage of bioreactors

	Pilot and Lab scale reactor
Type of biofilm carrier	BWTS <sup>®</sup>
Surface to volume ratio	$650 \text{ m}^2/\text{m}^3$
Length	14,5 mm
Height	18,5 mm
Width	7,3 mm
Number of cells per carrier element	400000

**Experimental Management:** The pilot scale reactor was operated continuously for 90 days at NSO. Feed in the feed tank (equalisation tank) was heated to  $35^\circ\text{C}$  using aquarium heater before pumping it to the AD section of the reactor. Internal circulation pump was installed to stir feed to avoid deposition of particles at the bottom of the tank and mix the feed with external

added buffer solution. Despite insulating pipes and reactor with polyflex pipe insulation and insulating carpet respectively, temperature at AD stage was only at around  $19\pm 3^{\circ}\text{C}$  due to heat losses. Temperature reached as much as  $27^{\circ}\text{C}$  with the onset of summer towards the end of the operation. Peristaltic pump was used for the feed pump. Feeding rate was maintained at 18.5 L/d in the beginning and later increased to 90.7 L/d . This increased OLR from 3 kg COD/m<sup>3</sup>.d to 33.1 kg COD/m<sup>3</sup>.d to facilitate adaptation of organisms to the feed substrates. Meanwhile, HRT of AD stage decreased from 57 hours to 12 hours. External recirculation from top to the bottom of AD was done using peristaltic pump at the rate of 80 L/hour (it was 90 L/hour for first 14 days and reduced due to blockage caused by high TSS concentration in the recycled liquid). The recirculation helped to expand sludge bed and enhance contact between the granular sludge and feed substrates. It also helped to dilute the high concentration of feed COD and other inhibitory chemicals. This gave an up flow velocity of 4.4 m/h. Aeration was supplied at a rate of  $600\pm 100$  which gave DO level  $3\pm 1$  mg/L. The aerobic biofilm was run in washing mode throughout the experiment.

Granular sludge, with relative size of 2 mm from an industrial wastewater treatment facility in Netherland was applied as inoculum. Approximately 20 L of the sludge was seeded in the beginning and approximately 5 L after 14 days. The initial total solids (TS) content of the inoculum seeded at first was 125.5 g/L with volatile solids (VS) content of 64.6 g/L. Inoculum seeded later was not as good as the previously seeded. Initial TS and VS may be assumed as the same as of day 18: 77.9 g/L and 56.6 g/L respectively.

Liquid samples were collected from different sampling points thrice a week (daily during weekdays at the beginning for 22 days). COD (total and soluble), pH, VFA, TSS and VSS were determined regularly while ammonium, total nitrogen (TN), total phosphorus (TP), alkalinity, biogas composition were measured sometimes to monitor the digester conditions. Table 3–3 shows sampling locations and types of analysis performed.

Biogas flow rate was monitored by collecting gas volume in a gas bag thrice a week for certain time interval. Digital biogas meter was installed at the later stages of the operation.

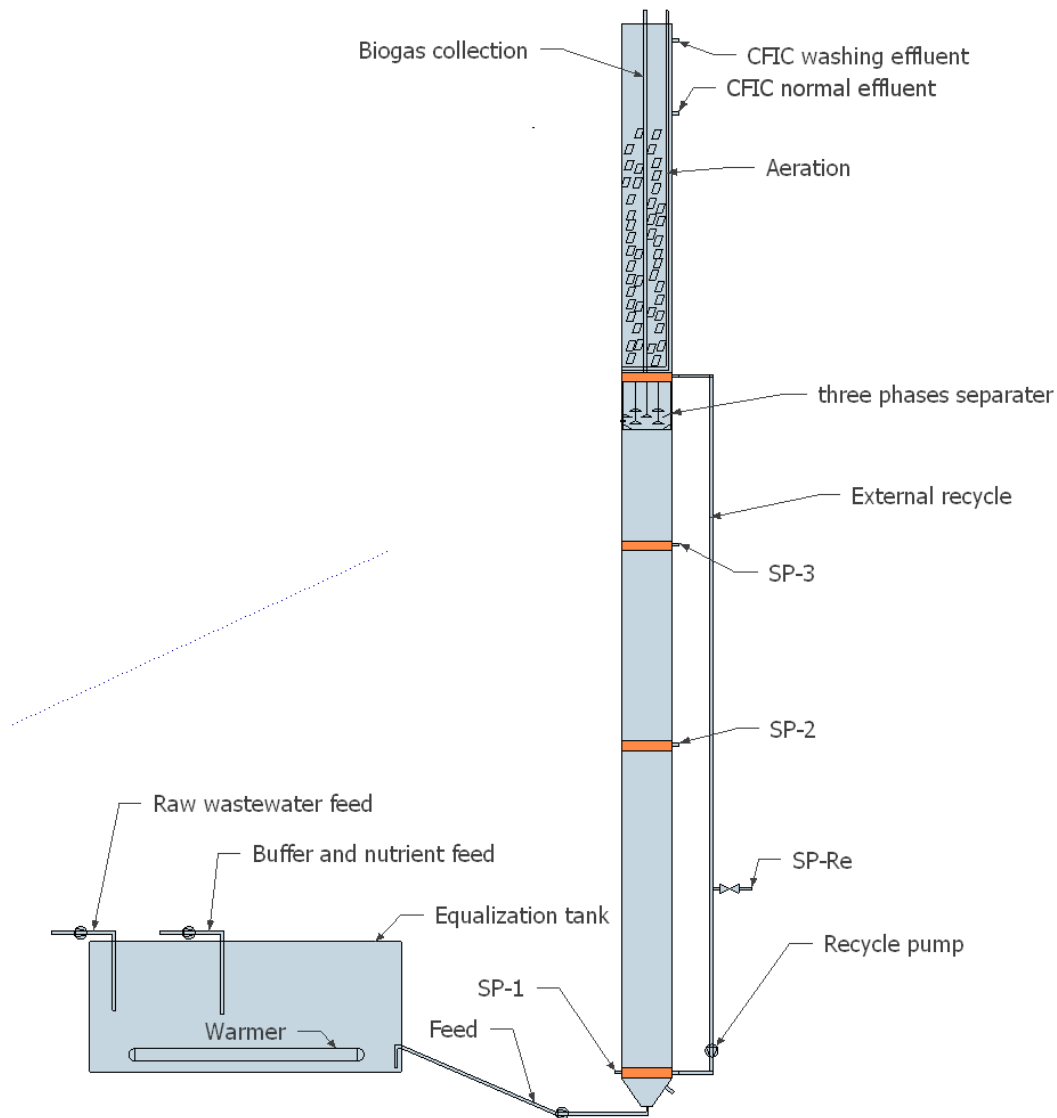


Figure 3–4: Experimental setup of pilot scale HyVAB<sup>®</sup> bioreactor at NSO showing different stages of the reactor along with equalisation tank <sup>a</sup>.SP refers to sampling point. SP1 is AD1, SP2 is AD2, SP3 is AD3 and SP-Re is recycle.

<sup>a</sup>Printed with permission from Wang Shuai

### 3.2.2 Laboratory Scale Reactor

**Reactor Design:** The laboratory scale reactor with cross sectional area of 0.016 m<sup>2</sup> is made up of acrylic tube. The bottom 9 L AD stage and the top 4.5 L CFIC<sup>®</sup> stage are constituted in this hybrid reactor. The two stages are separated by a three phase separator to separate gas from the liquid and solid phases. Wastewater was continuously fed from the feed tank to the bottom of the anaerobic compartment using peristaltic pump. Liquid recycling from the top to the bottom of the reactor is done every 30 minutes for 1 minutes by peristaltic pump. The aerobic biofilm stage, termed as Continuous Flow Intermittent Cleaning (CFIC<sup>®</sup>), is filled with plastic bio-carriers. The CFIC<sup>®</sup> stage is operated in normal and washing mode. The normal mode CFIC<sup>®</sup>

stage is filled with 95% BWTS<sup>®</sup> ( number of carriers is 1141) biofilm carriers (carrier effective surface to volume ratio 650 m<sup>2</sup>/m<sup>3</sup>). During washing mode, the carriers filling ratio reduces to 75% due to elevated liquid level. Excess biofilm and sludge is washed during bio-carriers washing which is conducted according to pre-determined washing frequency. Air supplied at the bottom of the aerobic compartment cultivates heterotrophic bacteria which oxidises the remaining COD after the AD stage. Sketch of the reactor is shown in Figure 3–5.

**Experimental Management:** The laboratory scale reactor was running for 10 months before being considered for this thesis work. The reactor was operated continuously at a temperature range of 19±3°C with organic loading rate (OLR) being increased gradually from 2 to 15 kg COD/m<sup>3</sup>.d by increasing the feed flow from 8 to 19 L/day, for microorganisms to adapt to the feed. However, during the study for this thesis, OLR varied between 2.2 to 10.73 kg COD/m<sup>3</sup>.d due to varied concentration of feed COD with the Hydraulic Retention Time (HRT) of 27 hours (feed flow of 7.9 L/day). The CFIC<sup>®</sup> stage was 95% v/v filled with bio-carriers with surface to volume ratio of 650 m<sup>2</sup>/m<sup>3</sup> with surface area of 4.2 m<sup>2</sup> in the reactor. Aeration was maintained at 600±200 L/h during the test which gave Dissolved Oxygen (DO) level in the range of 0.2 to 6.51 mg/L.

Liquid samples were collected from the two stages once or twice a week. COD (total and soluble), pH, VFA, TSS and VSS were determined regularly while ammonium, total nitrogen (TN), total phosphorus (TP), alkalinity, biogas composition were measured sometimes to monitor the digester conditions. Table 3–3 shows sampling locations and types of analysis.

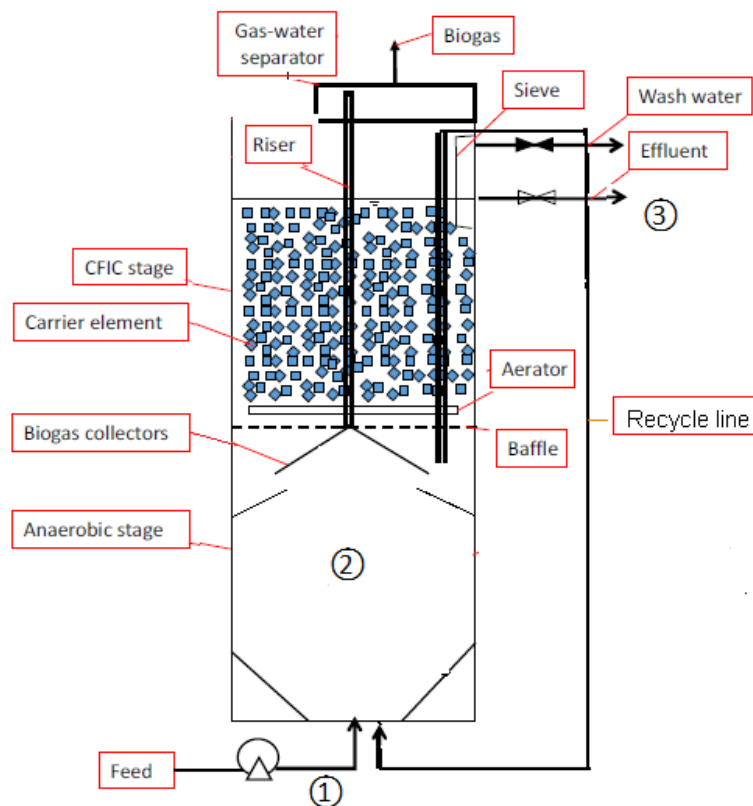


Figure 3–5: Experimental setup of lab scale HyVAB<sup>®</sup> bioreactor at HSN (Wang et al., 2015a).

Table 3–3: Sample locations and analysis of HyVAB® reactors

Sampling Points <sup>1</sup>							
Measurements	Feed tank	CFIC®	Recycle	Anaerobic top	Anaerobic middle	Anaerobic bottom	Biogas
pH	✓×	✓×	✓	✓×	✓	✓	
Temperature	✓×	✓×	✓	✓×	✓	✓	
DO		✓×					
TS					✓	✓	
VS					✓	✓	
TSS		✓×	✓	✓×			
VSS		✓×	✓	✓×			
tCOD	✓×	✓×	✓	✓×			
sCOD		✓×	✓	✓×			
VFA	✓×	✓×	✓	✓×			
Gas Composition							✓×

Table 3–5: Design of pilot and laboratory scale HyVAB® bioreactor

Parameters	Units	Formula	Pilot Values	Lab Values
Design flow (hour)	m <sup>3</sup> /h		0,0033	0,00033
Design flow (day)	m <sup>3</sup> /d	Design flow(hour)*24	0,079	0,00792
Design COD concentration	mg/L		10000	10000
Design feed COD loading (hour)	kg/h	Design flow(hour)*Design COD conc./1000	0,03	0,0033
Design feed COD loading (day)	kg/d	Design feed COD loading (hour)*24	0,79	0,079
Design temperature	°C		20-30	20-30
Design TSS	mg/L		200	200
<b>Anaerobic stage</b>				
Working volume	m <sup>3</sup>	Water depth*Cross-sectional area	0,044	0,009
Water depth	m		2,5	0,57
Reactor diameter	m		0,15	0,142

<sup>1</sup>✓=Pilot scale reactor; ×=laboratory scale reactor

Cross-sectional area	m <sup>2</sup>	3,14*Reactor diameter <sup>2</sup> /4	0,018	0,016
Recirculation flow rate	m <sup>3</sup> /h		0,1	0,016
Design upflow velocity	m/h	(Design flow(hour)+Recirculation flow rate)/Cross-sectional area	5,8	1
Hydraulic retention time	h	Working volume/Design flow(day)*24	13,4	27,27
Design volumetric COD loading	kg/m <sup>3</sup> /d	Design total COD loading/Working Volume	17,9	8,8
Expected COD removal efficiency	%		60	70
<b>CFIC<sup>®</sup> stage</b>				
Working volume	m <sup>3</sup>	Water depth*Cross-sectional area	0,022	0,0045
Water depth-washing	m		1,250	0,284
Water depth-normal	m	Water depth-washing*Filling rate-washing/Filling rate-normal	1	0,238
Cross-sectional area	m <sup>2</sup>		0,018	0,016
Type of media			BWT15	BWT15
Filling rate-washing	%		72	75,3
Filling rate-normal	%		90	90
Amount of carries needed	m <sup>3</sup>	Filling rate-washing*Working volume/100	0,0159	0,0034
Total protected surface area	m <sup>2</sup>	Amount of carriers needed*828	0,0159	2,20
Design volumetric COD loading	kg/d/m <sup>3</sup>	Design feed COD loading (day)*(1-Expected COD removal efficiency/100)/Working volume	14,3	5,2



Design biofilm COD loading	g/d/m <sup>2</sup>	(1-Expected COD removal efficiency/100)*Design feed COD loading (day)/Total protected surface area*1000	24,1	10,77
Hydraulic retention time-aerobic	h	Working volume/Design flow	6,7	13,6
CFIC <sup>®</sup> washing up-flow speed(with internal recirculation)	m/h	Design upflow velocity*Cross-sectional area/CFIC <sup>®</sup> washing crossing area	327,9	57,14
<b>Anaerobic stage</b>				
Expected max biogas production	m <sup>3</sup> /d	Design feed COD loading (day)*0,8*0,35/0,67	0,33	0,033
Expected max methane production	m <sup>3</sup> /d	Expected max biogas production*0,67	0,22	0,022
Expected average biogas production	m <sup>3</sup> /d	Design feed COD loading (day)*0,65*0,35/0,67	0,27	0,027
Expected average methane production	m <sup>3</sup> /d	Expected average biogas production*0,67	0,18	0,018

### 3.3 Assays

Gas chromatography was used to measure VFA concentrations and biogas composition. Gas chromatograph (HP 6890 serial C) with a flame ionisation detector and a capillary column (DB-FFAP 30 m long and 0,25  $\mu$ m film) was used to analyse VFAs. Hydrogen and air were the detector gases with helium as the carrier gas at flow velocity of 24 mL/min. The injector and the detector temperatures were set to 200°C and 250°C respectively while the oven started at 80°C, hold for a minute, and reached to 180°C at a rate of 30°C/min then to 230°C at a rate of 100°C/min.

SRI gas chromatography, model 8610C was used to determine biogas composition. Carrier gas was Helium. The oven temperature was kept constant at 83°C.

TS, VS, TSS and VSS (filtered with 1,5  $\mu$ m pore size glass filter) were determined based on the standard methods (APHA 1995). To determine TSS, 3-20ml of sample was vacuum filtered through 1,5 $\mu$ m glass microfibres filters (VWR European Cat No 516-0875) which were dried at 105°C for at least 2 hours after rinsing with distilled water and cooled in desiccator. The residue retained on the filter was dried at 105°C for at least 2 hours and cooled before weighing. Then the sample was kept at 550°C in muffle furnace for 15 minutes and cooled before weighing for VSS. TS of granular sludge was determined by keeping volume of sample

in porcelain basin(washed, heated at 105°C and desiccated) overnight at 105°C and desiccated before weighing. The basin was transferred to muffle furnace at 550°C for 30 minutes and desiccated before weighing VS.

The Alkalinity was determined by potentiometric titration to end-point pH (APHA 1999). Complexometric titration was used to determine calcium ion concentration. Commercial kits (HACH LANGE) were used to determine total nitrogen(TN), total phosphorus(TP),tCOD, sCOD (filtered right after sampling with 0,45  $\mu\text{m}$  pore size glass filter).

pH and temperature were measured using VWR pH110 (shown in appendix) and dissolved oxygen(DO) was measured using WTW Oxi 3315 (shown in appendix)

All the samplings on the reactor were done when recycle pump was running.

# 4 Results

## 4.1 Pilot Scale Reactor

### 4.1.1 Start-up

A successful operation of HyVAB<sup>®</sup> reactor was achieved with short start-up time with OLR increased (by increasing the feed flow from 18.5 to 90.7 L/d) from values around 3 kg COD/m<sup>3</sup>.d to around 33.1 kg COD/m<sup>3</sup>.d with few abrupt increases due to high COD influent in the feed. In the mean time HRT decreased from 57 hours to merely 12 hours at AD stage as shown in Figure 4–2. Figure 4–1 shows the tCOD removal at different OLR. tCOD removal was around 80% at the beginning even at low OLR of 3 kg COD/m<sup>3</sup>.d but reached above 90% consistently even at higher OLR of 20 kg COD/m<sup>3</sup>.d. It is noteworthy that, sludge loading rate (SLR) increased from 0.103 to 0.55 kg COD/kg VS.d and specific methanogenic activity (SMA) increased from 0.08 to 0.17 kg COD-CH<sub>4</sub>/kg VS. d at the end of day 71. It is clear from the graph that tCOD removal decreased with increase of OLR above 20 kg COD/m<sup>3</sup>.d implying that the system was under stress. But the reactor bounced back with OLR reducing below 20 kg COD/m<sup>3</sup>.d. Few drops of tCOD removal efficiency corresponding to increase in OLR can be seen in the graph, but the reactor recovered shortly with system adapting to the condition until the load was too high to handle towards the end. Ultimately, reactor could not recover from stress caused by high OLR of 33.1 kg COD/m<sup>3</sup>.d and removal efficiency decreased to less than 40% indicating a complete system failure. Also, granules disintegrated giving high effluent TSS concentration.

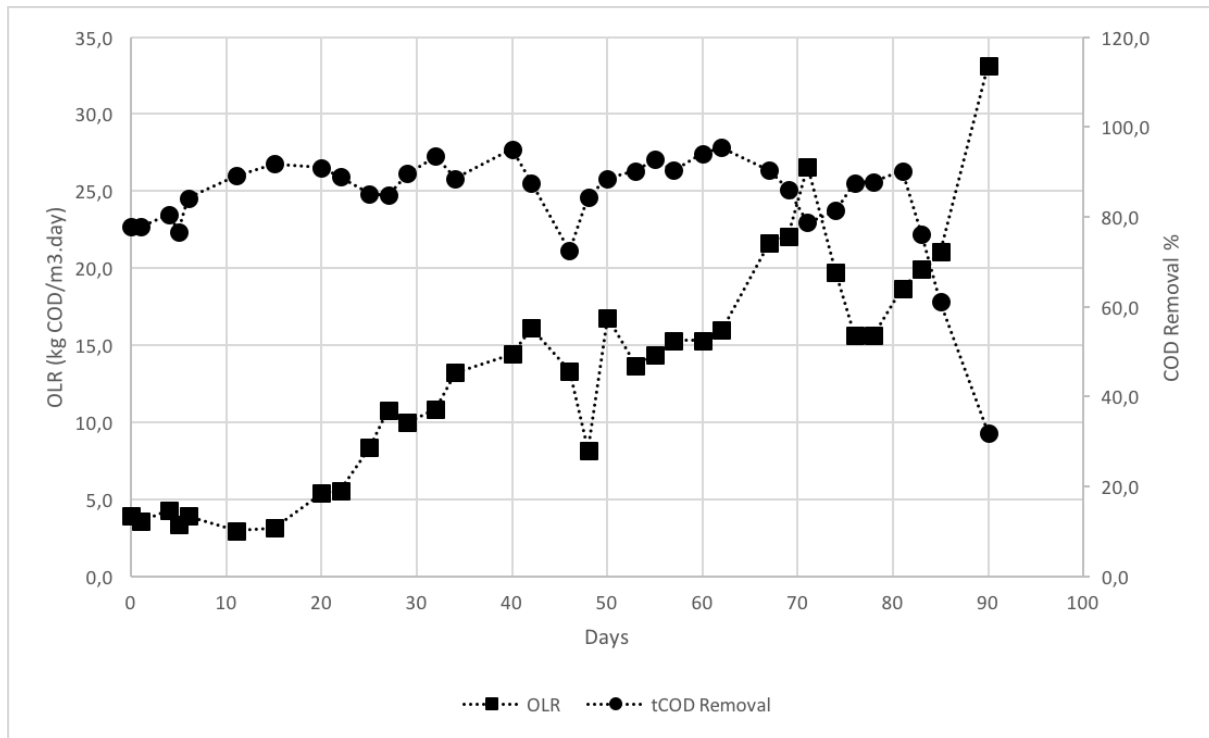


Figure 4–1: COD removal with increase of OLR

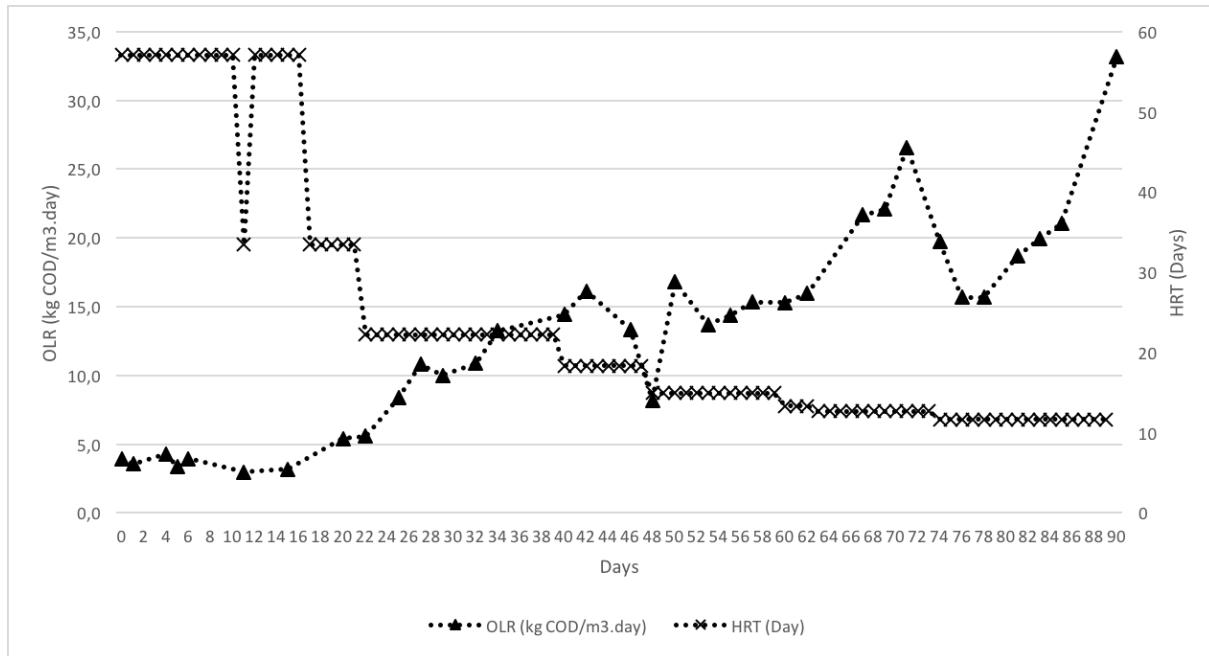


Figure 4–2: HRT vs OLR

#### 4.1.2 Reactor performance

A successful operation of HyVAB<sup>®</sup> reactor was achieved with sCOD removal efficiency consistently above 90% and tCOD removal consistently above 85% even at high OLR around 20 kg COD/m<sup>3</sup>.d. Figure 4–3 shows the COD removal efficiency with increasing OLR and decreasing HRT. Temperature of CFIC<sup>®</sup> also increased from around 20°C to 25°C towards the end of the operation due to onset of warmer weather and insulation of the reactor. The graph clearly shows the startup time taken by the reactor for acclimatisation of microorganisms to the new feed type (granules were from industrial wastewater treatment plant). Removal efficiencies were low even at lower OLR of 3 kg COD/m<sup>3</sup>.d but increased later when microorganisms acclimatised with the new environment. Some drops in efficiencies were seen with the increased OLR but the reactor bounced back until OLR was over 20 kg COD/m<sup>3</sup>.d. Towards the end, it is visible that tCOD removal efficiency decreased drastically to 30% when reactor was operated at 33.1 kg COD/m<sup>3</sup>.d for few days. Also, decrease in sCOD removal efficiency was clear with values reducing as low as 60%.

Highest tCOD removal of 95.4% was achieved at HRT of 13 hours and OLR of 16 kg COD/m<sup>3</sup>.d. Najafpour et al. (2006) treated palm oil mill effluent (POME) at a removal efficiency of 85% at an OLR of 23.15 kg COD/m<sup>3</sup>.d (with startup time of 26 days) which was a marked improvement over POME treatment using a UASB reactor by Borja and Banks (1994) in which 90% removal efficiency was achieved at a much lower OLR of 1.27 kg COD/m<sup>3</sup>.d (after 30 days of startup time).

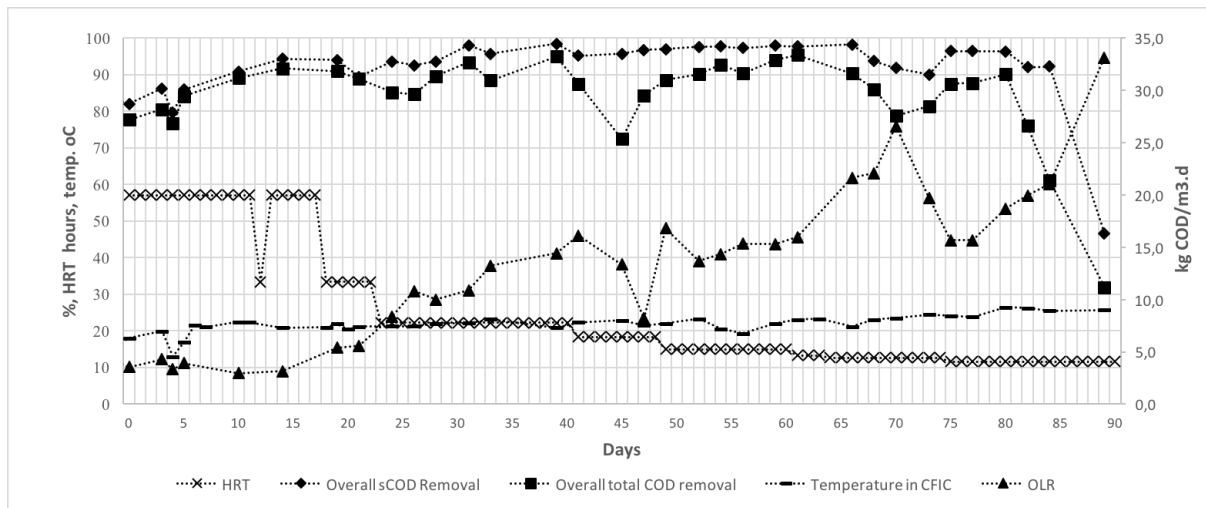


Figure 4-3: Overall performance of HyVAB<sup>®</sup> reactor

Figure 4-4 shows the overall performance of AD stage of HyVAB<sup>®</sup> reactor with increasing OLR. sCOD removal was normal as in overall reactor performance with efficiency increasing gradually and decreasing at the end when OLR was above 20 kg COD/m<sup>3</sup>.d. tCOD removal showed some anomalies when new granules were seeded in the reactor after day 14. Negative removal efficiency as shown in the graph is due to the fact that the newly seeded granules contained lots of small light particles which were being washed out increasing tCOD values in the AD3 and recycle (values were greater than the COD of influent feed). When washing of particles was finished, COD removal increased continuously to more than 80%. At overloading, disintegrated particles started being recycled increasing tCOD value to more than 20000 g/L (the graph excluded this value)

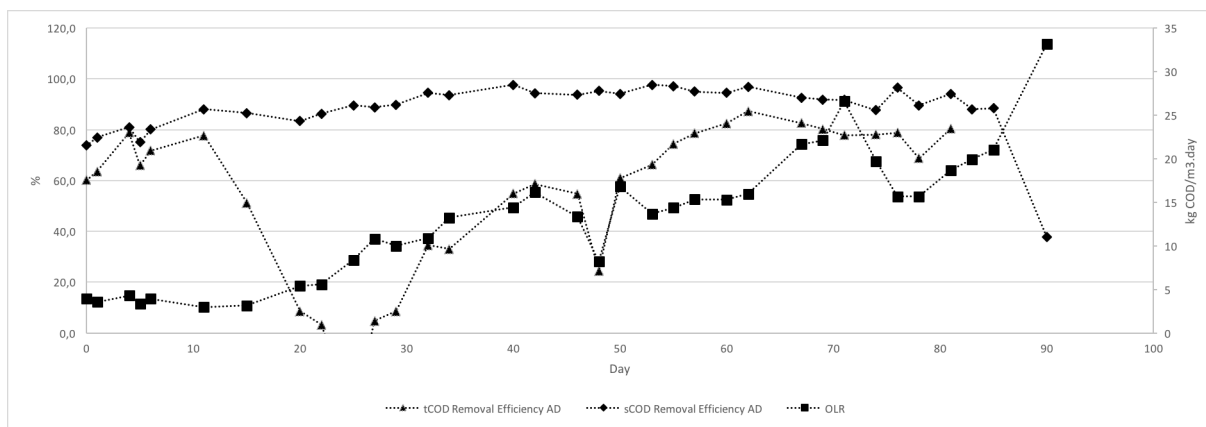


Figure 4-4: Overall performance of AD in HyVAB<sup>®</sup> reactor

Comparative tCOD removal efficiency in AD and CFIC<sup>®</sup> of HyVAB<sup>®</sup> reactor with increasing OLR is shown in Figure 4-5. The graph fails to explain COD removal phenomena well in this case. It shows that CFIC<sup>®</sup> removed the remaining COD (to give effluent quality) from the AD stage (when small light particles were being washed from day 10 to 50). However, it doesn't remove all the COD as shown in the graph. The particles that were being washed were being recycled back to the AD and small particles couldn't reach to the CFIC<sup>®</sup> stage due to

reduced vertical velocity above the recycling point. It means that most of the particles were being recycled in the AD stage. This caused the tCOD of the effluent to be low and not because of the degradation that happened in the CFIC<sup>®</sup> stage. As the washing of small particles was finished (indicated by low effluent TSS concentration and clearer recycling liquid), COD remaining from AD stage was digested in CFIC<sup>®</sup> as seen in the later days of operation. This graph doesn't include the values when OLR was above 20 kg COD/m<sup>3</sup>.d for few days (and disintegrated particles were being recycled as before) as removal efficiency was negative for AD part and above 100% for CFIC<sup>®</sup> part which was misleading. tCOD removal reached up to around 80% and up to 20% in AD stage and CFIC<sup>®</sup> stage respectively. Low efficiency of AD means higher COD load in CFIC<sup>®</sup>. During day 80, when tCOD removal efficiency was low in AD, CFIC<sup>®</sup> removed around 20% of tCOD (after washing away all the particles).

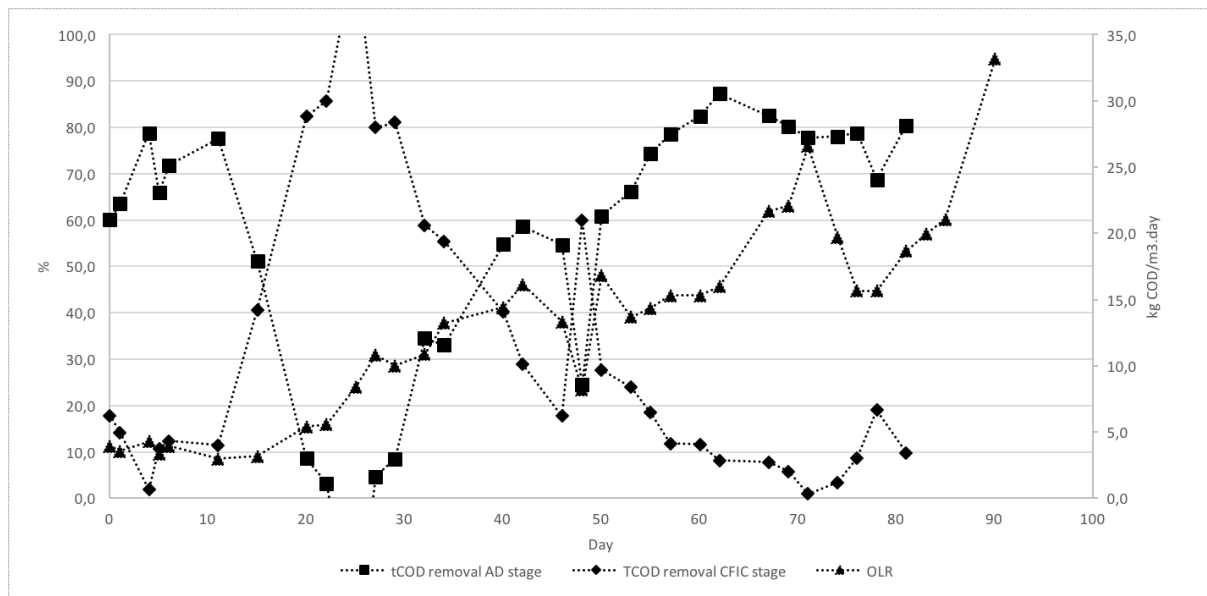


Figure 4-5: tCOD removal in AD and CFIC<sup>®</sup> of HyVAB<sup>®</sup>

sCOD removal in both the AD stage and CFIC<sup>®</sup> stage is shown in Figure 4-6. The removal efficiency increased with number of operational days for some time and remained stable before started decreasing with overloaded condition. The performance of AD in sCOD removal was consistent even at increasing OLR with efficiency reaching well above 95% at stable condition. This put less stress on CFIC<sup>®</sup> stage and sCOD removal requirement was close to 0. But whenever necessary, CFIC<sup>®</sup> complemented well with AD to remove remaining sCOD from the AD stage as shown by peaks in the graph. At the end, sCOD removal efficiency in AD stage dropped below 40%.

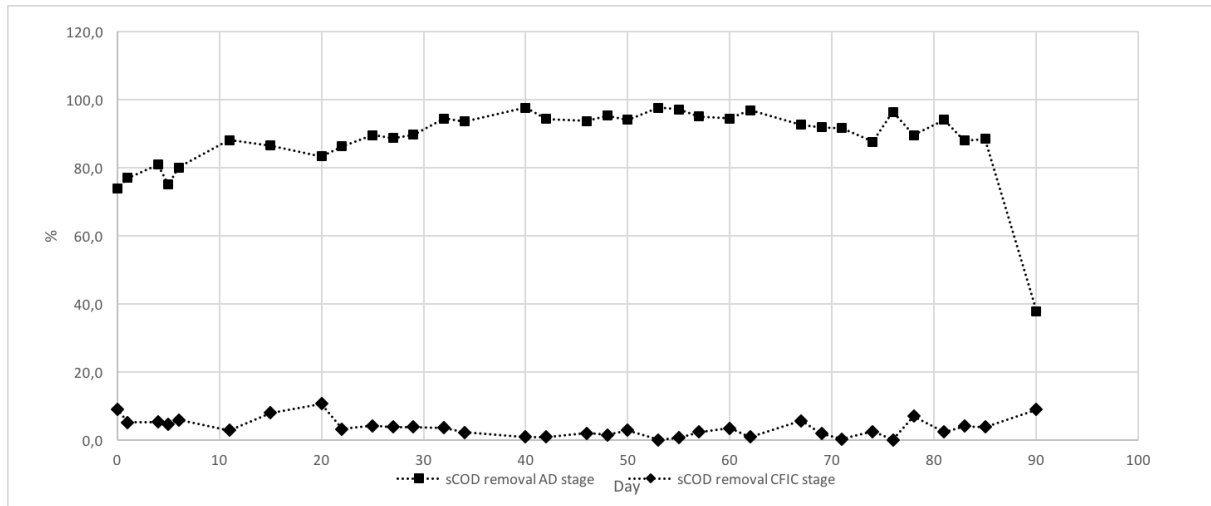


Figure 4–6: sCOD removal in AD and CFIC® of HyVAB®

Figure 4–7 shows the effluent COD at different OLR and removal efficiency. It is seen from the graph that effluent COD at the beginning was a bit higher than at the stable conditions. Some higher effluent peaks are seen corresponding to change to higher OLR. But the system adapted to the new condition well and effluent COD was reduced as in stable condition. tCOD value increased drastically at the end when OLR was above critical value of 20 kg COD/m<sup>3</sup>.d.

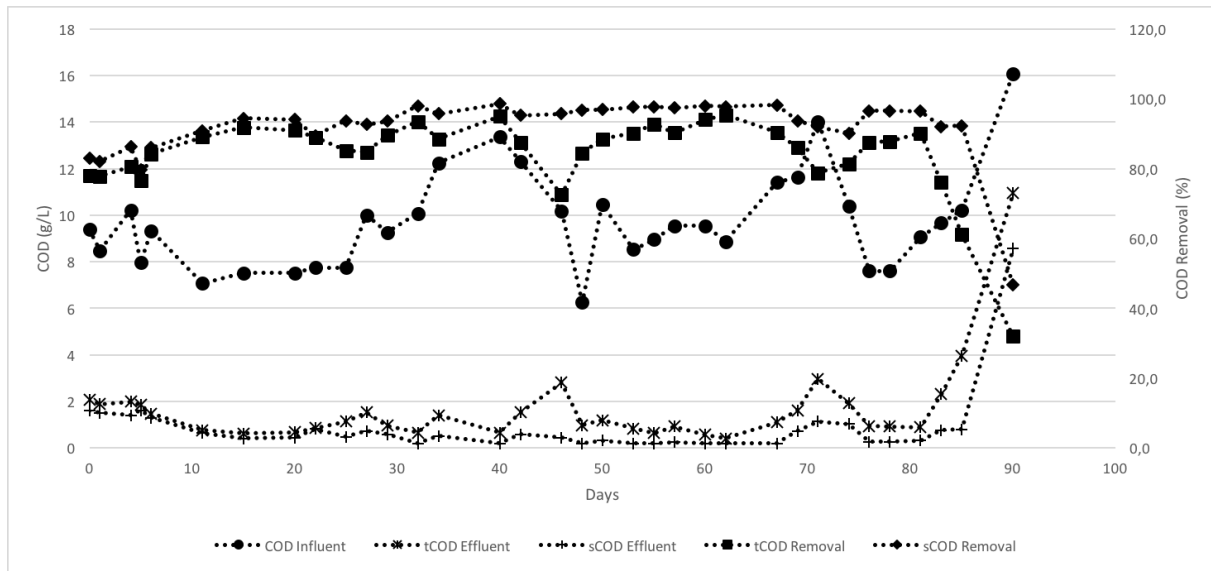


Figure 4–7: COD removal efficiency along with influent and effluent COD

Figure 4–8 shows the variation of pH at different stages of the reactor. NaHCO<sub>3</sub> was pumped to feed tank as buffer solution to maintain pH around 7 to make a balance in the process of hydrolysis, acidification of the organic matter and methane formation (Chan et al., 2012). Feed pH was not stable due to different types of waste from different sources used in the process. CFIC® pH was mostly above 8 indicating good health of the reactor. Buffer solution helped to maintain the pH of all anaerobic stages at around 7. During the overloaded condition, pH dropped noticeably in CFIC®, AD3 and recycle.

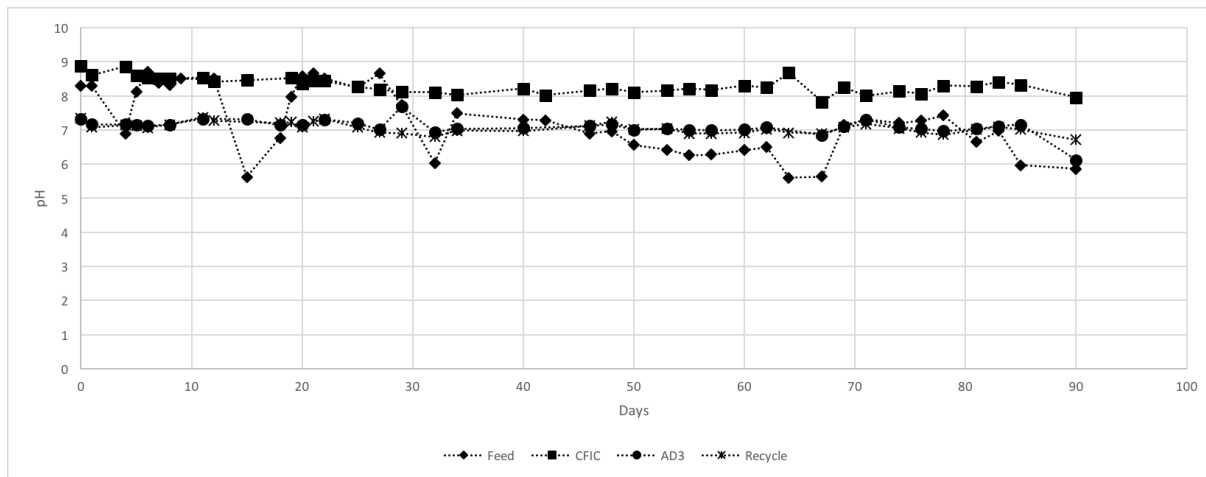


Figure 4–8: COD removal efficiency along with influent and effluent COD

Figure 4–9 shows the effluent VFA concentration vs the time of the reactor operation. It is seen from the graph that both the acetic and propionic acid concentration decreased with time. At day 20, both acids had concentration close to 0 mg COD/L from the initial concentration of around 200 mg COD/L. With the increase of OLR, some spikes of concentration were seen but went back to close to null with time indicating well adaptation of microorganisms. During the initial condition, removal efficiency was low but with microorganisms adapting well to the feed, efficiency increased indicating stable condition. It is worthy to be noted that even at OLR as high as 19 kg COD/m<sup>3</sup>.d, VFA was not seen in higher concentration with propionic and acetic acid concentration well below 20 mg COD/L. At the end of the operation, when the system was fully overloaded, the concentration of both the acids along with total acid went drastically up. This ensured that the reactor was overloaded.

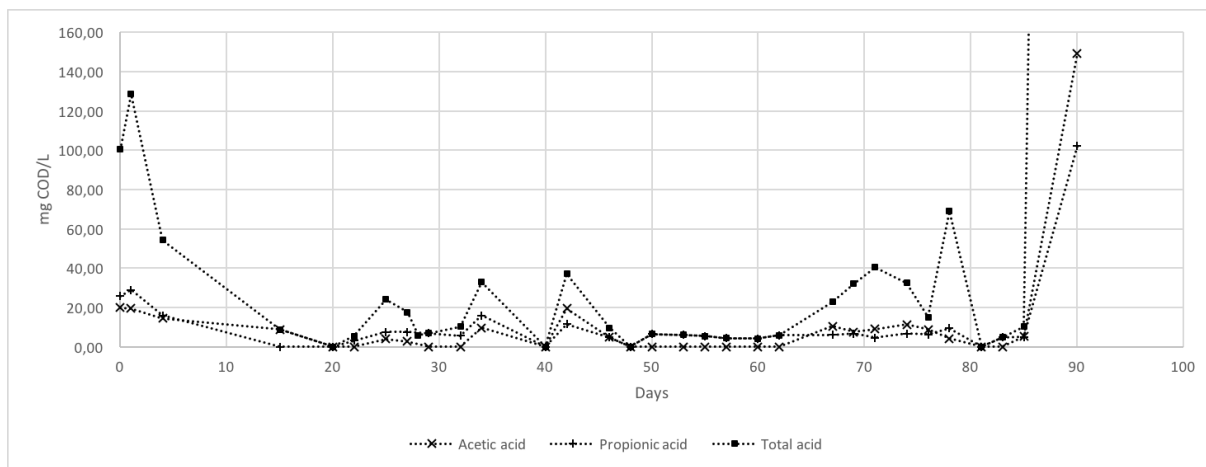


Figure 4–9: Concentration of VFAs in effluent over time

Figure 4–10 shows VFA concentration in recycle vs time of the reactor operation. Similar trend was seen as in Figure 4–9. Concentrations of total acids, acetic acid and propionic acid were higher at the startup but concentration decreased with time. At the end when the reactor was overloaded, concentration of total acid sky rocketed decreasing pH of the reactor. The spikes were indication of the onset of overloading condition.



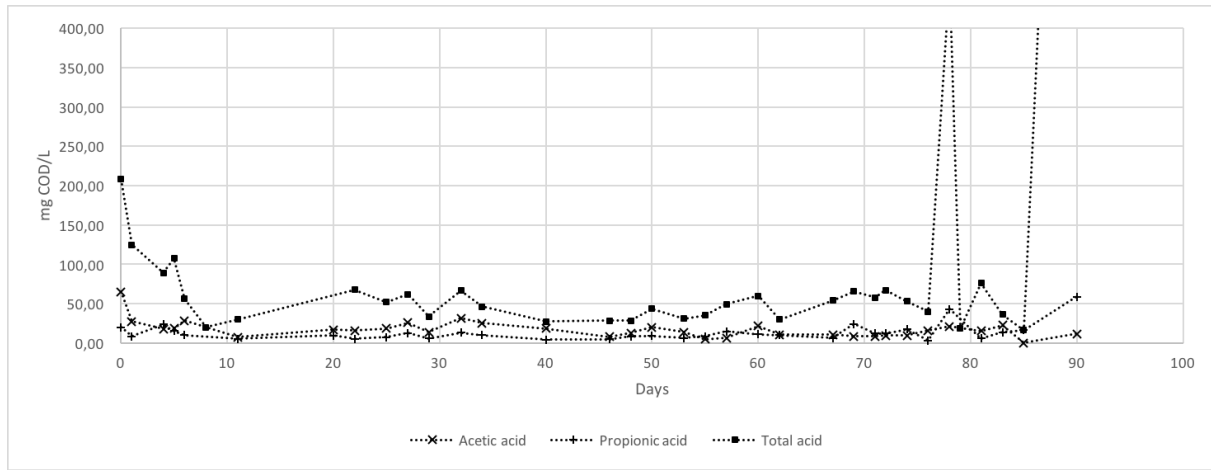


Figure 4–10: Concentration of VFAs in recycle over time

Figure 4–11 shows VFA buildup in AD3 section. Trend is similar to that of recycle section with initial concentration being high and decreasing gradually over time with few higher concentration spikes during OLR increase. At the overloading condition, increased VFA concentration was seen indicating system failure. This even reduced the pH of the reactor.

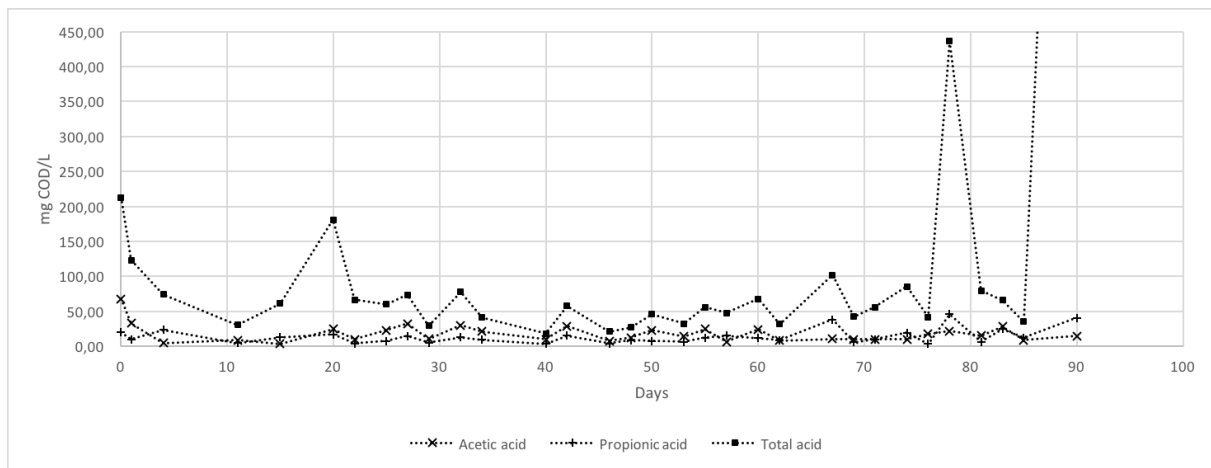


Figure 4–11: Concentration of VFAs in AD3 over time

Figure 4–12 shows the VFA concentration of feed. As seen from the graph, total acid was fluctuating over time due to different sources of feed. For some reason, acetic acid and propionic acid were in more detectable concentration till day 46 but later reduced down to negligible concentration.

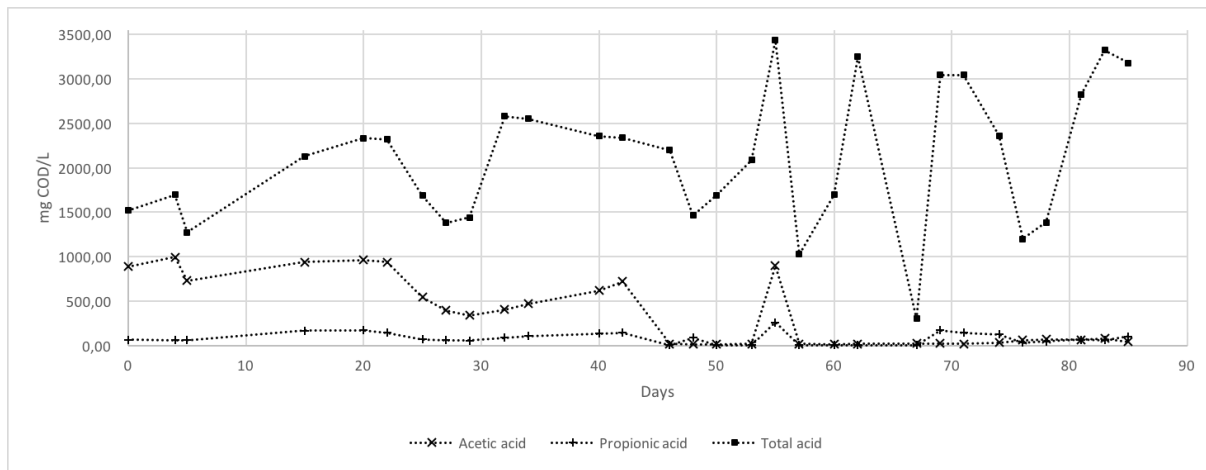


Figure 4–12: Concentration of VFAs in Feed over time

As seen from the Figure 4–13, total VFA consumption in the reactor started from the beginning of the operation. The effluent VFA concentration was in negligible concentration compared to feed VFA. But still higher effluent VFA concentration could be noticed at the beginning and towards the end. Biogas production was low even at considerable amount of VFA during the initial stage but when the reactor was stable, amount of methane COD produced was increased with almost the same concentration of VFA. Increased OLR and well acclimatisation of microorganism to the feed contributed in the greater biogas production.

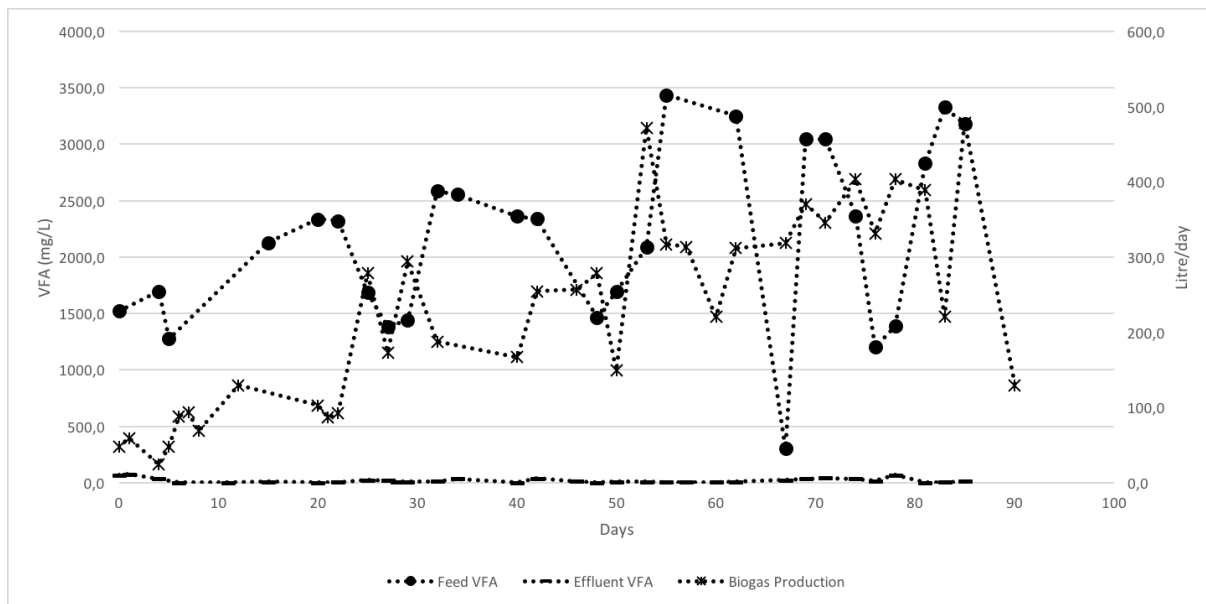


Figure 4–13: Biogas production with VFA concentration

Figure 4–14 shows VFA concentration at different stages of the reactor. It is seen that VFA concentration was higher during the startup phase at all the stages but reduced to negligible amount along with the operational days. At the end, VFA concentration spiked drastically indicating system failure.

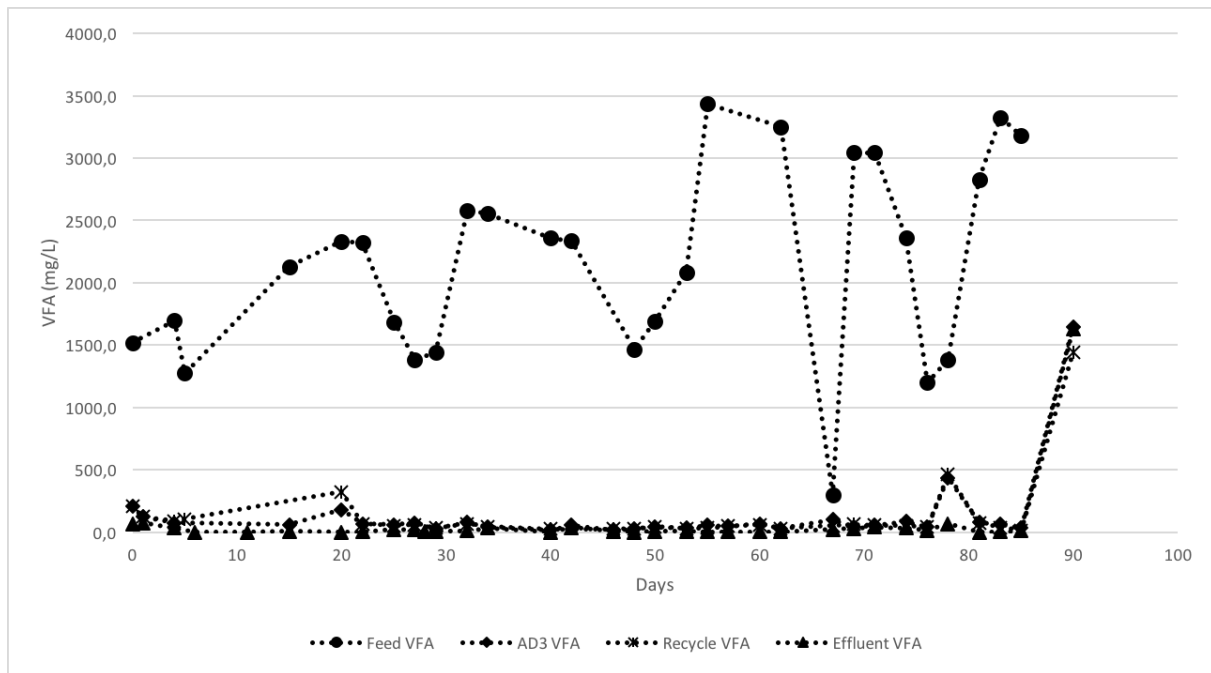


Figure 4-14: VFA concentration of feed and different stages

Relationship between ratio of VFA and tCOD and biogas production is shown in Figure 4-15. At the initial stage during startup, relation was not clear but as the reactor became stable, there was a close relationship. Although it is not clear from the graph, increase in ratio of VFA and tCOD increased the biogas production. However, continuous data recording is required to come up with concrete conclusion.

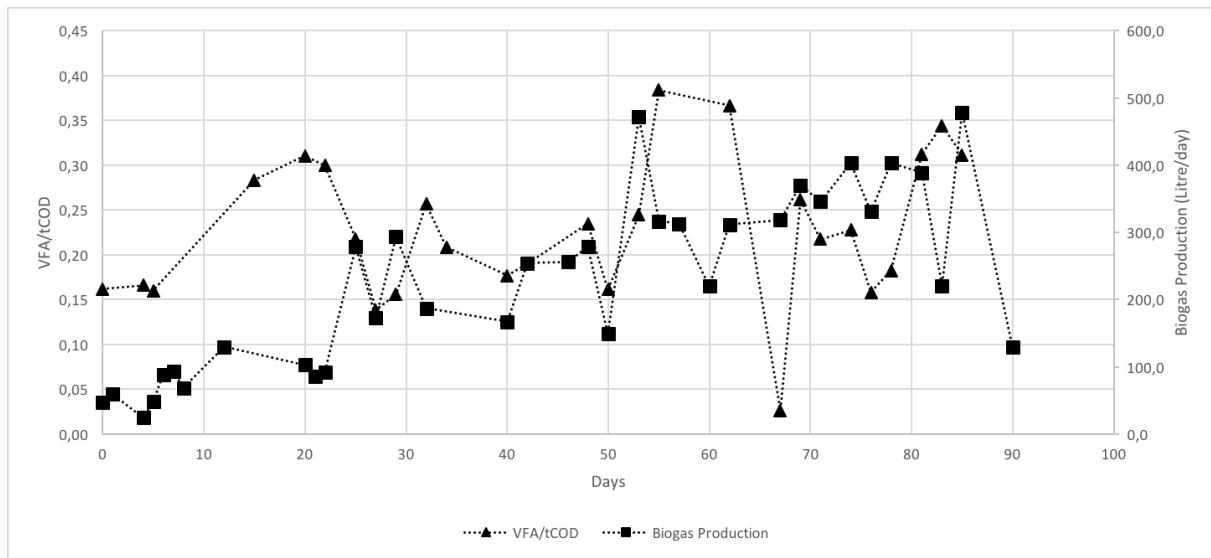


Figure 4-15: Relation of ratio of VFA and tCOD along and Biogas production

### 4.1.3 Biogas production

Biogas was monitored daily to determine the flow rate and gas composition by collecting in a gas bag for 2-5 minutes depending on the gas flow. The gas produced was collected from the anaerobic stage through three phase separator. Some dissolved methane and carbon dioxide will flow from AD stage to CIFC stage and the solubility depends upon temperature and pressure according to Henry's law. At 25°C and pressure of 1.03 atm, about 28 ml methane is dissolved in one litre of liquid solution (Wang et al., 2015b). So, 2% methane is lost to the aerobic stage where it will be consumed (Segers, 1998). CO<sub>2</sub> transferred to aerobic stage will be in larger portion due to its higher solubility in water.

Figure 4–16 shows overall biogas production with increased OLR along with COD removal rate and HRT. The biogas production was increasing with increase of OLR up to OLR of 20 kg COD/m<sup>3</sup>.d before decreasing sharply when the system was overloaded. The graph doesn't represent the true biogas production as the values were single point data for each day. However, Figure 4–17 shows the exact amount of biogas production as these values were taken from the continuously running digital biogas meter.

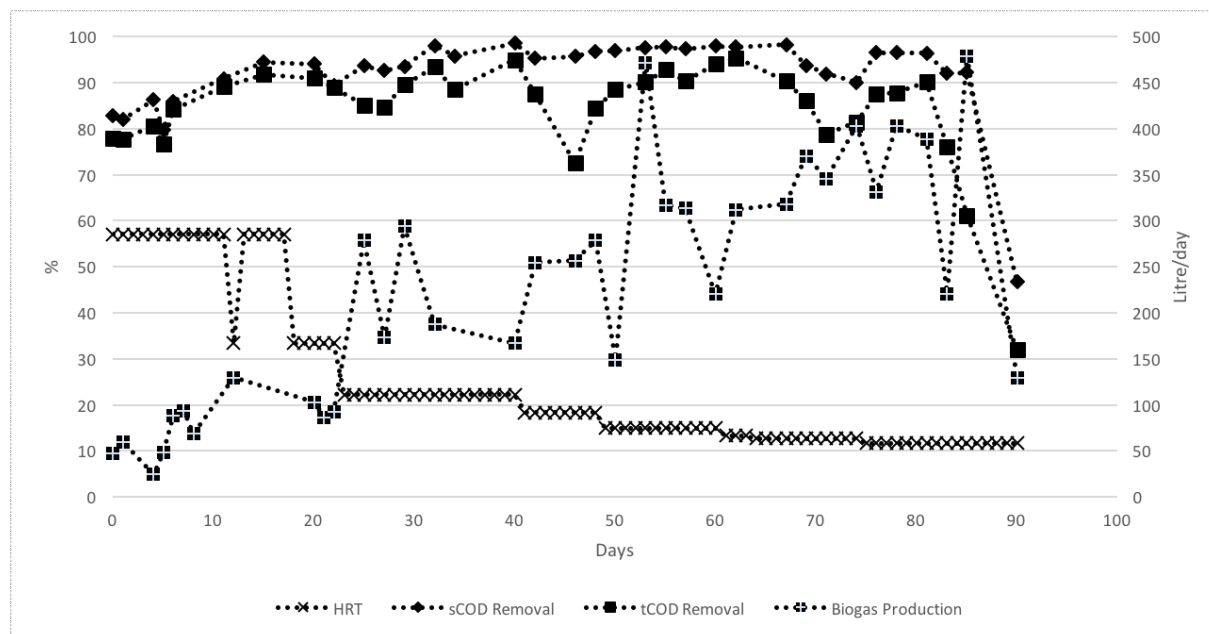


Figure 4–16: Overall performance of HyVAB® reactor with biogas production

Figure 4–17 shows the biogas production over the operational days at later stage. It is clear from the graph that during the stable operating condition, biogas production was around 350-400 L/day. It decreased to around 300 L/day with increased OLR. It is expected that biogas production at the initial stage was also low than at the stable condition.

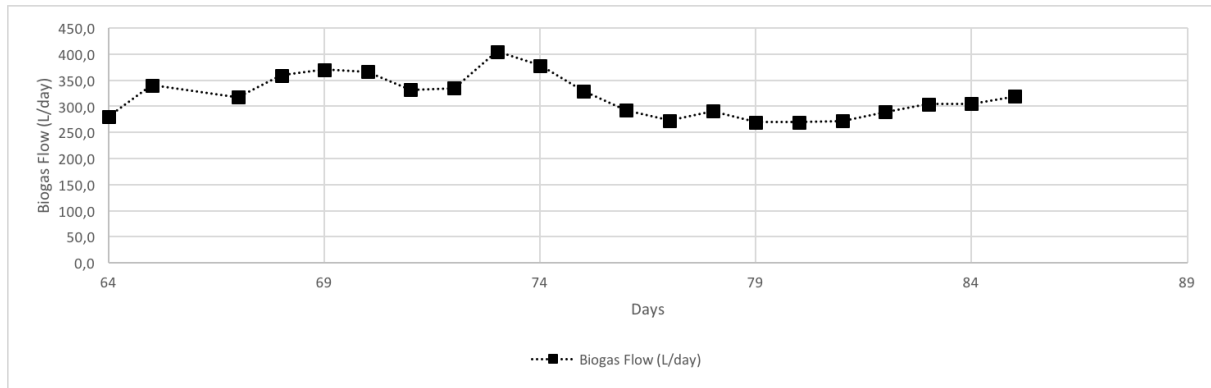


Figure 4–17: Biogas Production towards the end with instalment of biogas meter

Figure 4–18 shows the biogas production with change in HRT. It is seen that the biogas production increased linearly ( $R^2=0.6122$ ) with the decreased HRT (or increased OLR) until the system suffered from excessive load and biogas production dropped drastically. It should be noted that this graph is plotted from single data point and doesn't completely represent the true scenario. However, a trend can be seen.

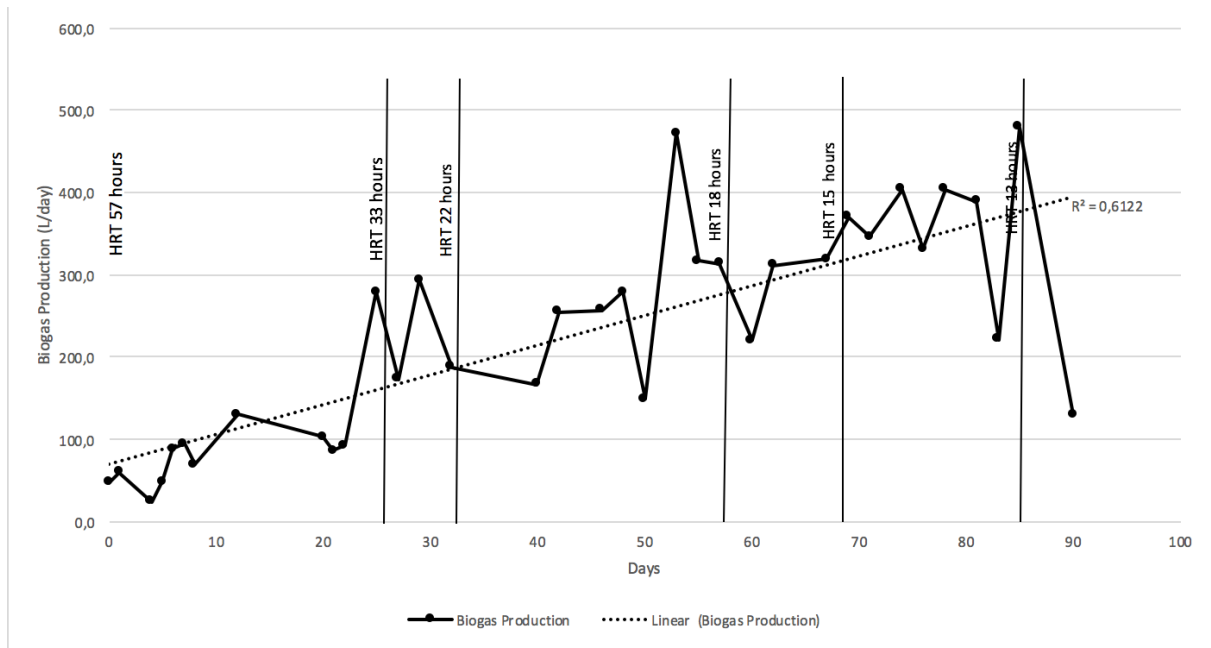


Figure 4–18: Biogas production with decreased HRT over time

Figure 4–19 shows variation of methane yield with COD removal rate. As seen from the graph, methane yield is way over the theoretical value of 0.35 for the most of the time which may be due to contribution from degrading aerobic sludge from CFIC<sup>®</sup> stage. Also, the measurements were single point data for each day and may not represent the overall scenario. Methane yield was expected to remain around the value of 0.35 at stable condition and decrease with the overloading of the system.

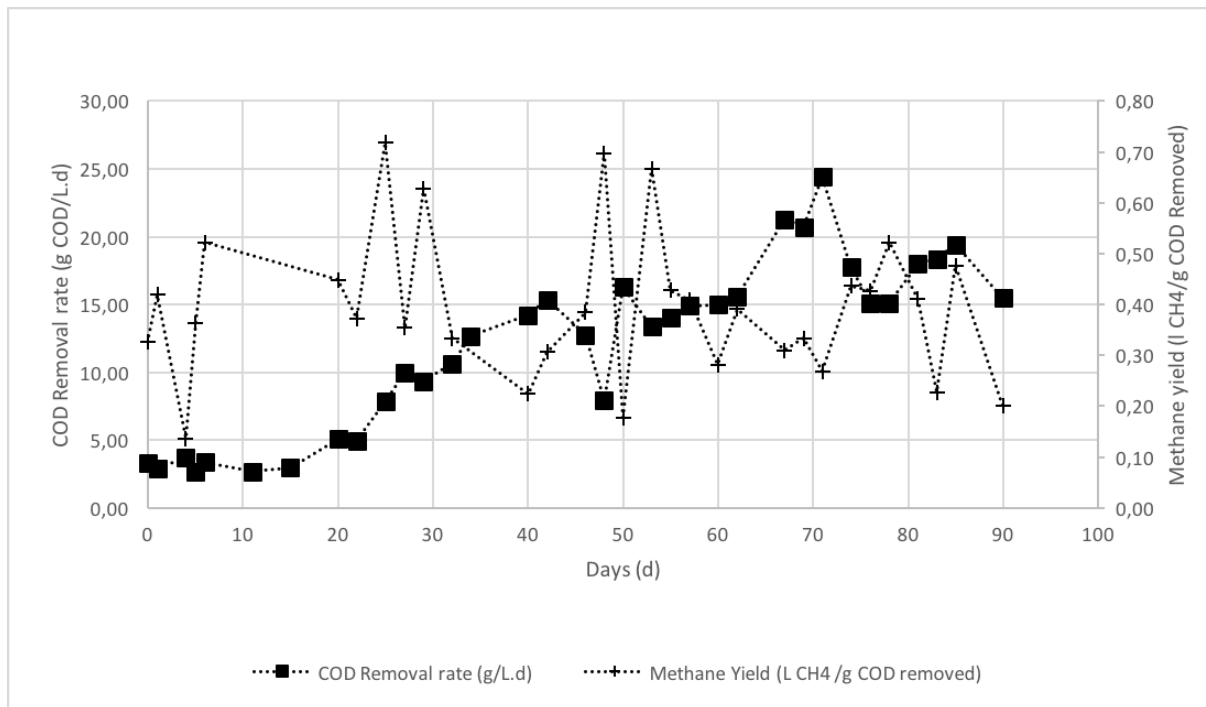


Figure 4-19: Methane yield with respect to COD removal

Figure 4-20 shows the trend of methane yield at the later stage of operation with digital biogas meter installed. It gives the representative value in this case as data were monitored continuously. As seen from the graph, methane yield value was around 0.35 (the theoretical value at STP) during the stable conditions. But the value decreased to around 0.3 when the system was overloaded.

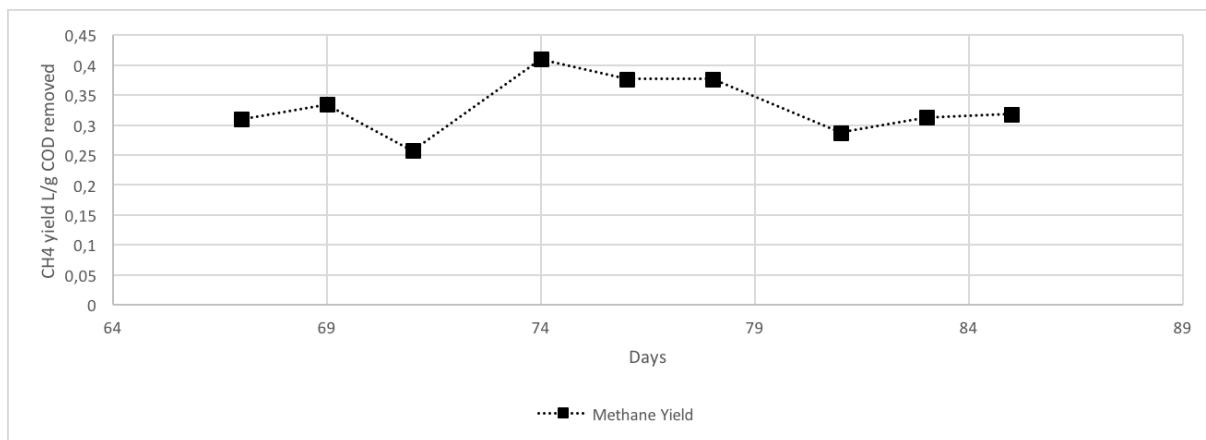


Figure 4-20: Methane yield towards the end with instalment of biogas meter

The biogas was found to be rich in methane with concentration of 80-90%; the balance being carbon dioxide as shown in Figure 4-21. It can be seen in the graph that methane concentration was found to decrease with increase of OLR although not drastically. At lowest OLR, methane concentration was close to 90% while at the end it dropped to just below 80% which may be due to increase in reactor temperature. Obviously, CO<sub>2</sub> concentration increased with the decrease

of CH<sub>4</sub> concentration. Najafpour et al. (2006) also found similar trend while treating POME at higher OLR producing 6.23 L CH<sub>4</sub>/L day.

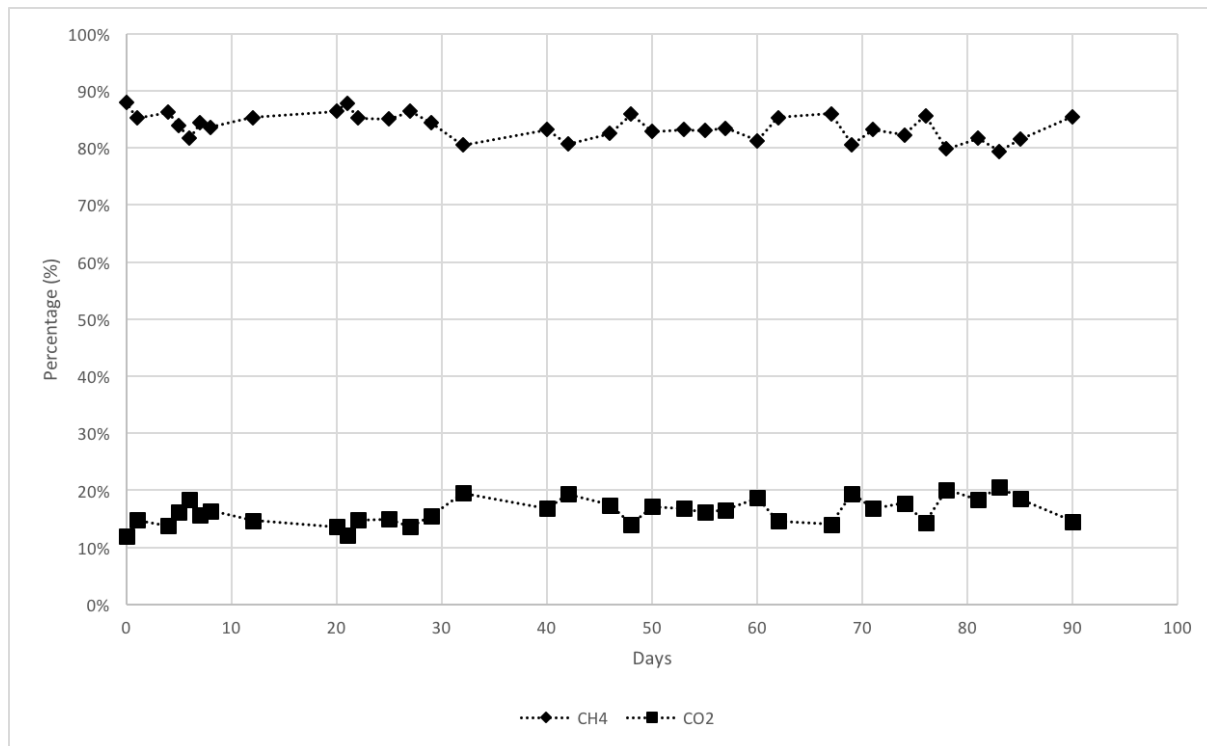


Figure 4-21: Biogas composition of HyVAB<sup>®</sup> reactor

#### 4.1.4 Development of granules

Sludge samples were taken at least once a month to monitor the development of granules. Figure 4-22 shows the development of granules at AD1 (sampling point is at 300 mm from the base of the reactor) over the time. This part contains the most dense granules. TS and VS increased for some time before decreasing and started to increase later. TS and VS increased to 141.4 g/L and 69.8 g/L respectively before reducing to 92.9 g/L and 68.6 g/L respectively from the initial value of 125.5 g/L and 64.6 g/L respectively. The decrease in TS and VS may be as a result of washing away loosely packed fine particles from the granules with the increase of OLR and mixing with low quality granules added later with time because of recycling. But both the TS and VS increased at the later stage to 123 g/L and 86.5 g/L respectively.

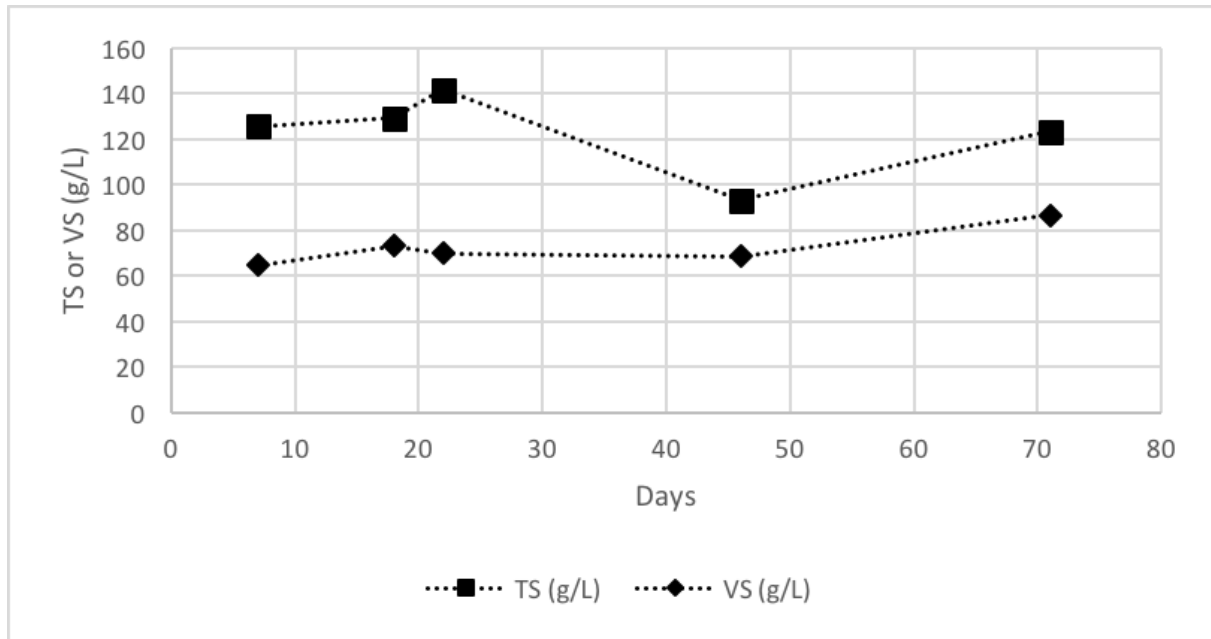


Figure 4-22: Development of granules in AD1 over time

Figure 4-23 for development of granules at AD2 (sampling point is at the height of 800 mm from the base) also shows the similar trend as in AD1. TS and VS decreased for some time before increasing and is expected to increase for some time. TS and VS decreased to 48.2 g/L and 31.8 g/L respectively before increasing to 88.4 g/L to 65.5 g/L respectively.

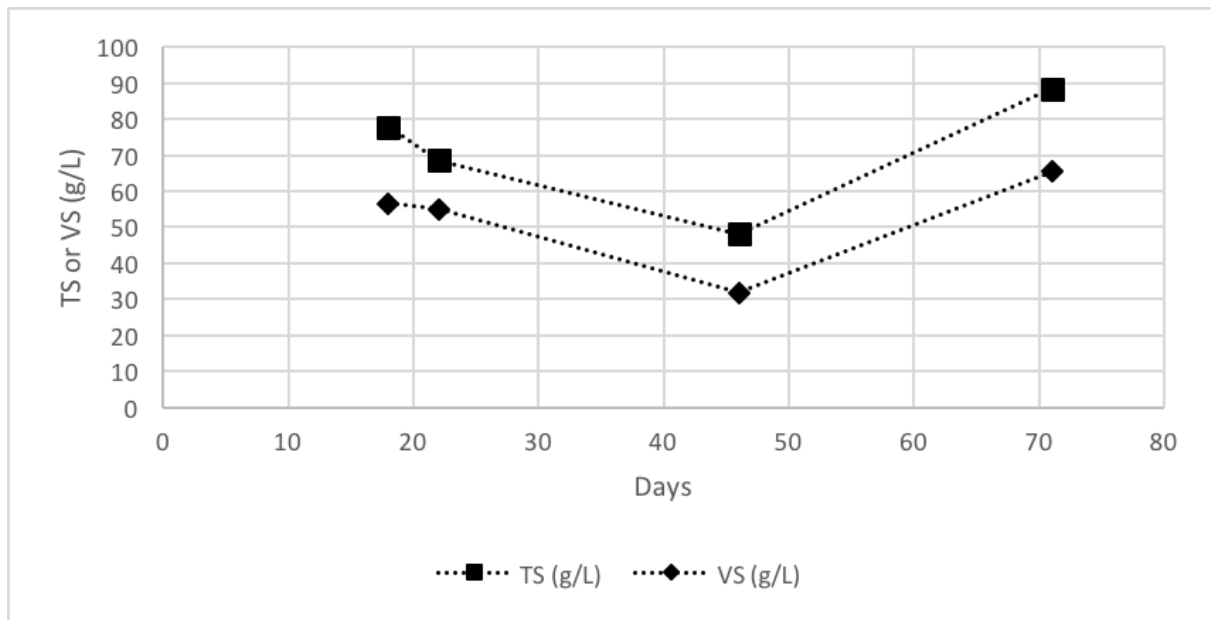


Figure 4-23: Development of granules in AD2 over time

Figure 4-24 shows the comparative development of granules at sampling points over time. Solids in 71<sup>st</sup> day are higher than in day 18 at all sampling points except TS at AD1. Figure 4-25 and Figure 4-26 shows solids content in day 18 and 71 respectively.



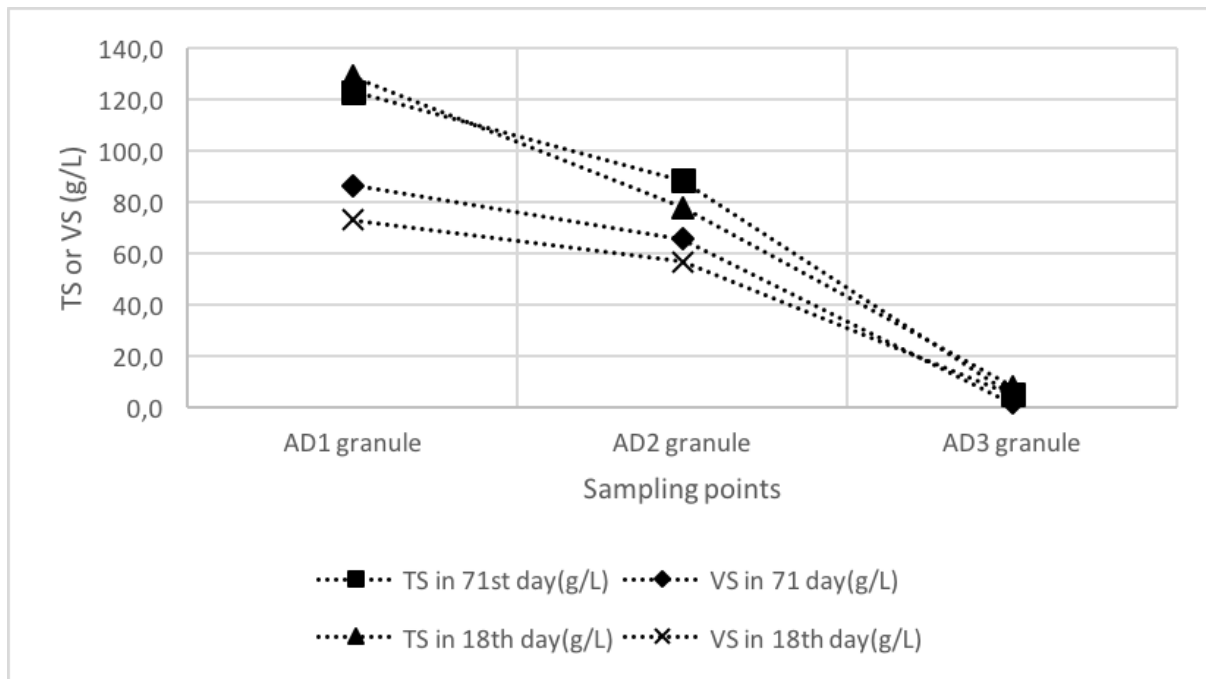


Figure 4-24: Development of granules over time

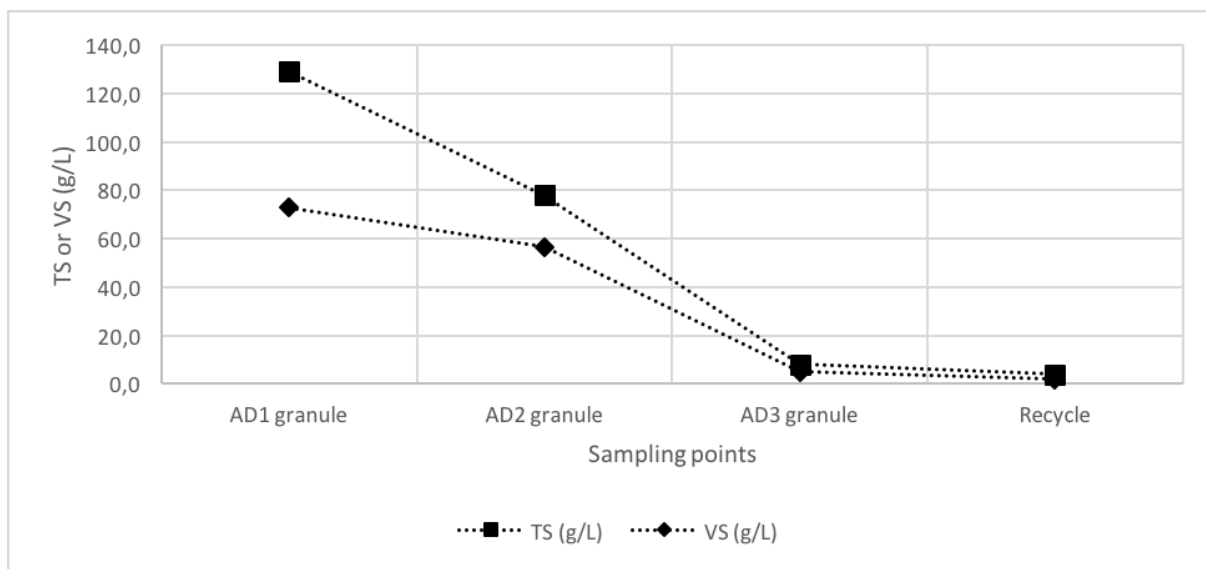


Figure 4-25: Development of granules in 18<sup>th</sup> day

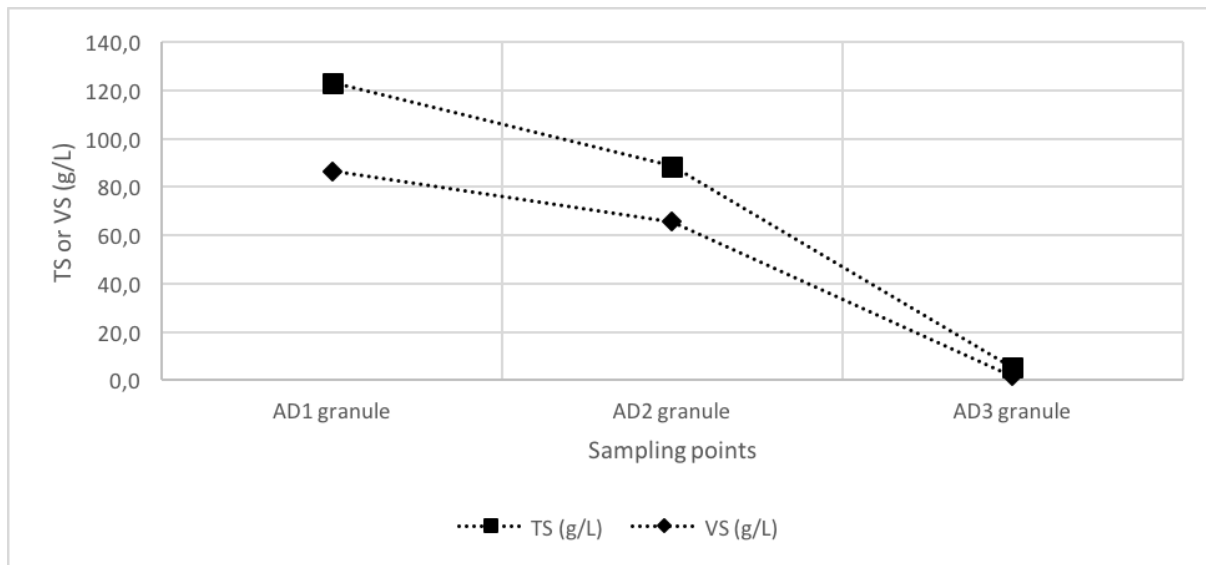


Figure 4-26: Development of granules in 71<sup>st</sup> day

A profile of sludge solid concentration in different height of the reactor in day 18 and 71 is shown in Figure 4-27 and Figure 4-28 respectively. There is gradient of solids concentration over the height of the reactor with dense sludge being at the bottom and light at the top. Change in solids concentration over time can be seen distinctly in the figures. VS of the bottom sludge increased from 72.9 g/L to 86.5 g/L while VS of the middle sludge increased from 56.6 g/L to 65.5 g/L. Lower concentration of solids in the top part at the latter day indicates the washing away of smaller particles with time.

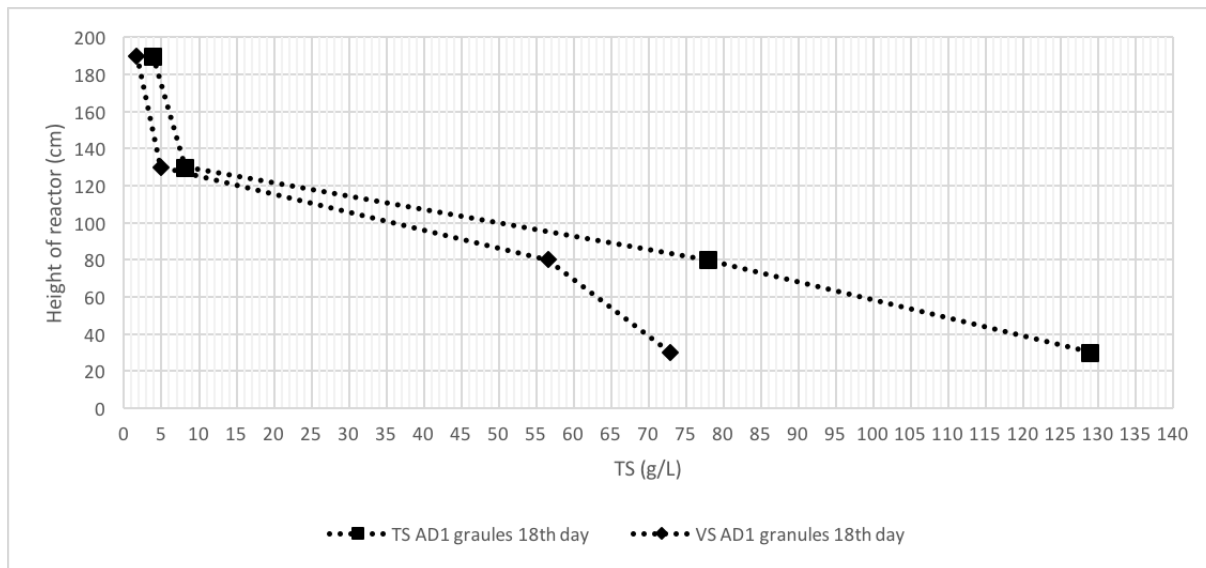


Figure 4-27: Development of granules along the height in 18<sup>th</sup> day

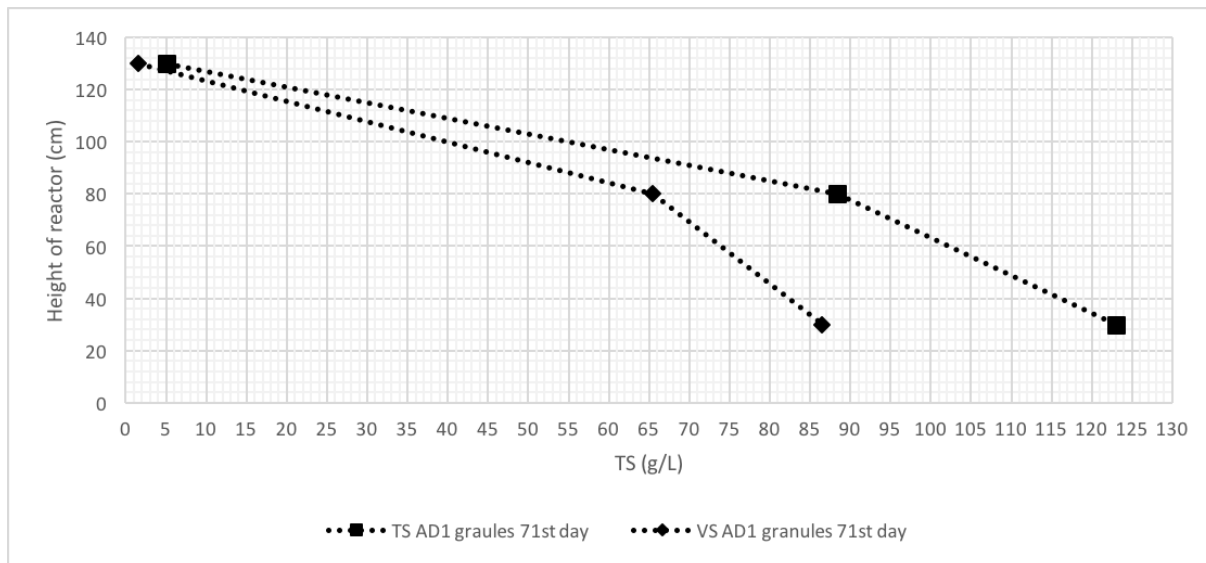


Figure 4-28: Development of granules along the height in 71<sup>st</sup> day

As 20 + 5 litres of seed sludge were inoculated into the reactor, the corresponding sludge loading rate was 0.1 kg COD/kg VS.d. Weighted average of all the three sampling points was considered while determining the VS weight. Figure 4-29 shows the gradual development of sludge loading rate. At the end of 71 days, it reached to 0.55 kg COD/kg VS.d. Borja and Banks (1994) has showed that a sludge loading rate of around 0.6-0.9 kg COD/kg VS.d is the best to ensure favourable conditions for biomass growth.

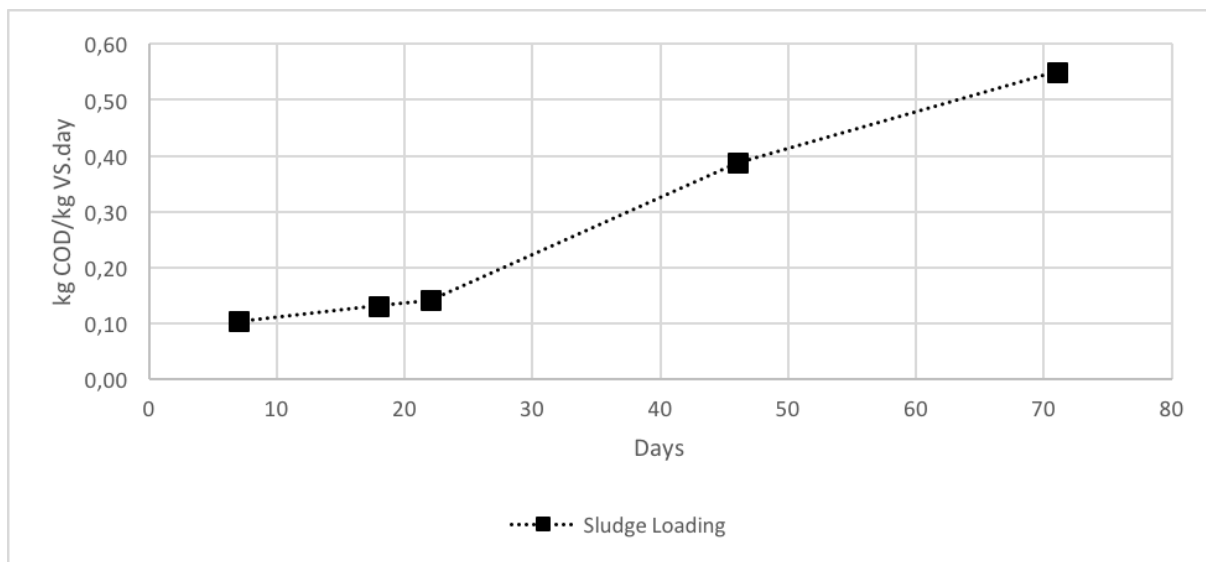


Figure 4-29: Sludge Loading of the reactor over the time

Specific methanogenic activity (SMA) is expressed to denote metabolic activities of granules and is considered as an important characteristic of granular sludge. It was measured at different operational days. It is seen from the Figure 4-30 that SMA decreased for some time and then increased linearly. Initial value was 0.074 kg COD-CH<sub>4</sub>/kg VS.d, decreased to the

least value of 0.05 kg COD-CH<sub>4</sub>/kg VS.d before increasing the highest value of 0.428 kg COD-CH<sub>4</sub>/kg VS.d at the end of day 71.

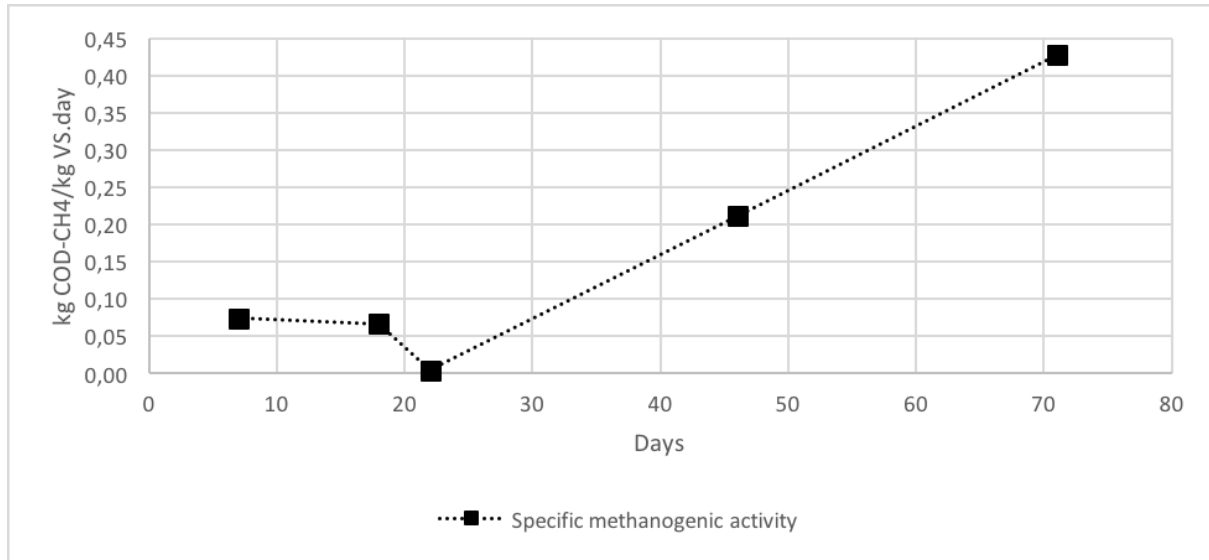


Figure 4-30: Specific methanogenic activity (SMA) of the reactor over the time

#### 4.1.5 Aerobic biofilm stage

The biomass yield was found to be around 0.04 g VSS/g COD removed on average. It is clear from the Figure 4-31 that the biomass yield increased overtime with increasing OLR. Towards the end, when OLR was overloaded, the biomass increased well over average value. It is due to washing out of granular sludge in AD.

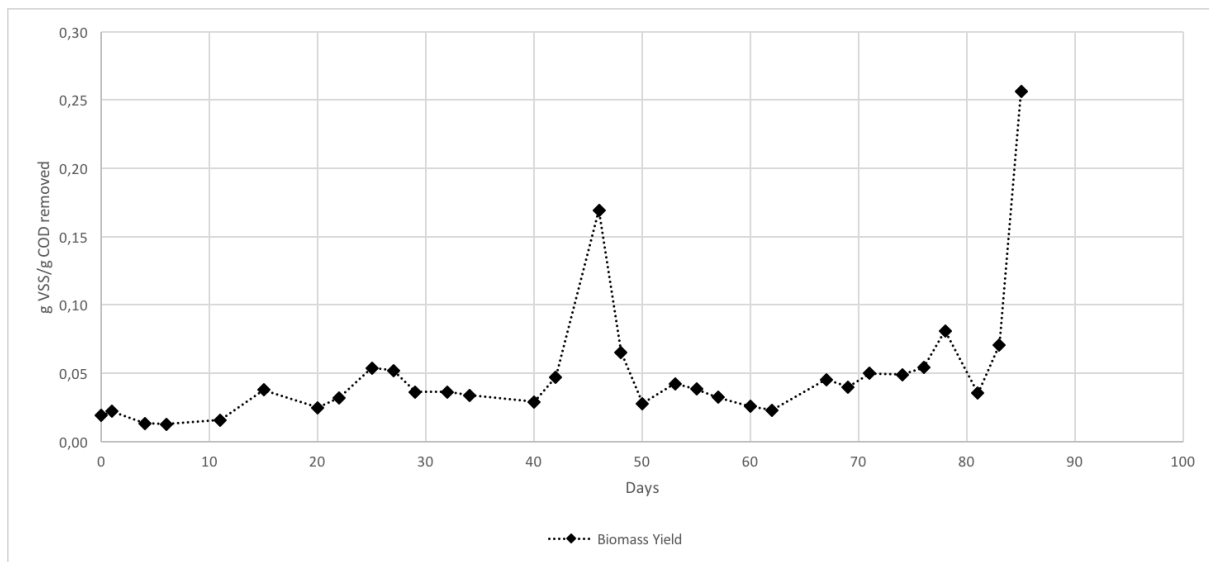


Figure 4-31: Sludge Loading of the reactor over the time

Change in DO concentration along with OLR change is shown in Figure 4-32. As seen from the graph, DO was very high at the beginning to the level of saturation. Then, aeration was

decreased to 400 L/h from 500 L/h in day 4 to maintain at around 5 mg/L. With the increasing OLR, DO dropped to below 1 mg/L indicating higher COD load in CFIC® stage. Then aeration was increased to 600 L/h in day 71 to increase DO concentration to facilitate oxidation. At the end of the operation, despite increased aeration, DO reduced below 1 mg/L indicating higher organic load in CFIC® and high biofilm activity.

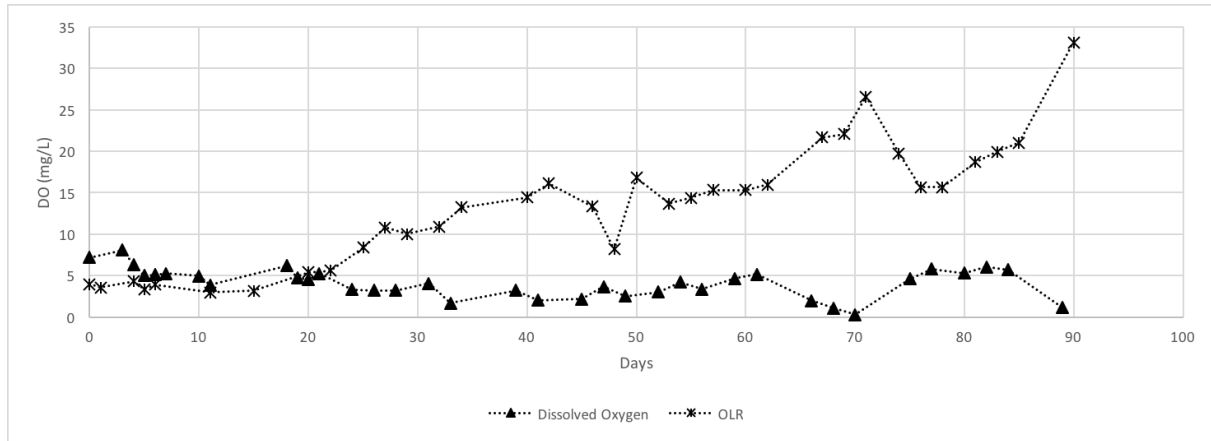


Figure 4-32: Dissolved oxygen over the increase of OLR

#### 4.1.6 Solids removal

Total suspended solids in the effluent were monitored to observe the performance of HyVAB® reactor. It can be seen from the Figure 4-33 that TSS increased slightly with increase in OLR. Suspended solids concentration was less than 100 mg/L at OLR of 3-4 kg COD/m³.d. When OLR was increased to above 5 kg COD/m³.d, suspended solids concentration increased above 300 mg/L. When OLR was above 15 kg COD/m³.d, suspended solids concentration increased above 500 mg/L. During the overloaded condition, the concentration was much higher due to disintegration of granules indicating the failure of the reactor.

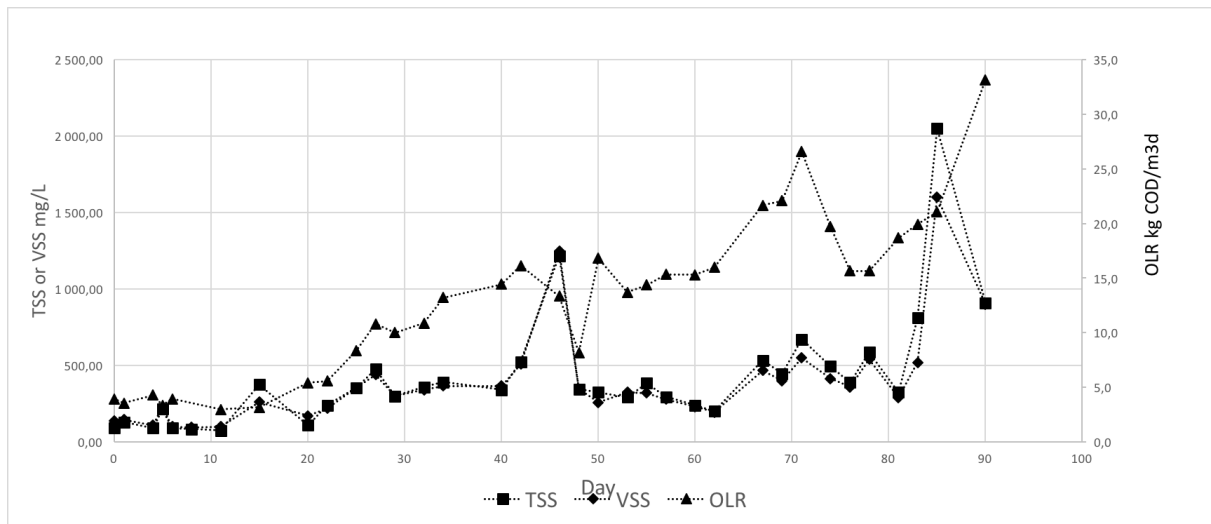


Figure 4-33: Effluent solids concentration over the increase of OLR

Figure 4–34 shows suspended solids concentration in AD3 and recycle. The concentration of SS was fairly low at the beginning of the start-up indicating less particles being recycled. But the concentration started increasing rapidly with the addition of new poor granules as the light floating particles and smaller particles were recycling. The TSS concentration increased to above 8000 mg/L during the stable operation from the initial value of less than 1000 mg/L. But with the time, SS concentration decreased back to the initial concentration giving well settled dense granules. It is to be noted that the both tCOD and sCOD removal efficiency was not affected much during the whole operational period within the design OLR. At the end when OLR was much higher, SS concentration sky rocketed indicating disturbances in the sludge bed because of overloaded condition. Figure 4–35 shows similar trend with VSS concentration.

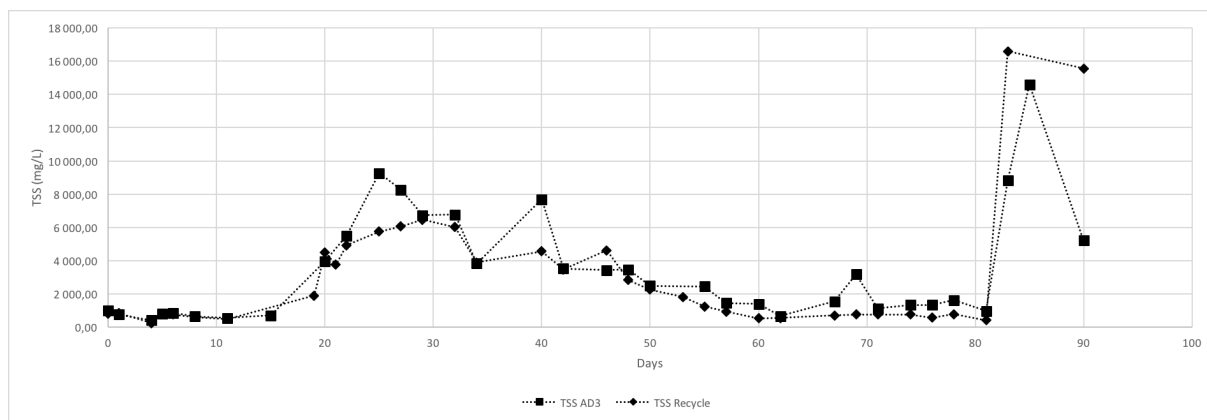


Figure 4–34: TSS concentration in AD and recycle over time

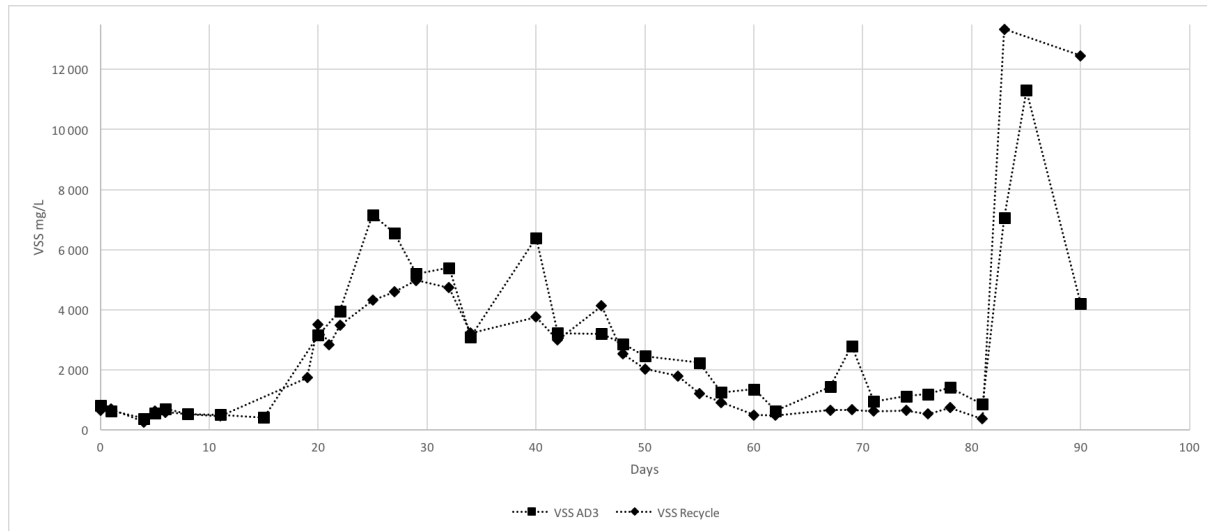


Figure 4–35: TSS concentration in AD and recycle over time

## 4.2 Laboratory Scale Reactor

### 4.2.1 Reactor Performance

General HyVAB<sup>®</sup> performance can be seen in Figure 4–36. The overall sCOD removal within the design OLR was around 60 to 70% and even around 80% for some days. But as OLR increased over the design value, removal efficiency decreased to around 30-40%. High OLR was observed from day 25 to 50 and 100 to 130. tCOD removal also showed similar trend. The efficiency in this case ranged between 50-60% and sometimes to around 70% below design OLR and decreased to around 30% at higher OLR. HRT was kept constant throughout the experiment. Variation in OLR was due to varying feed COD.

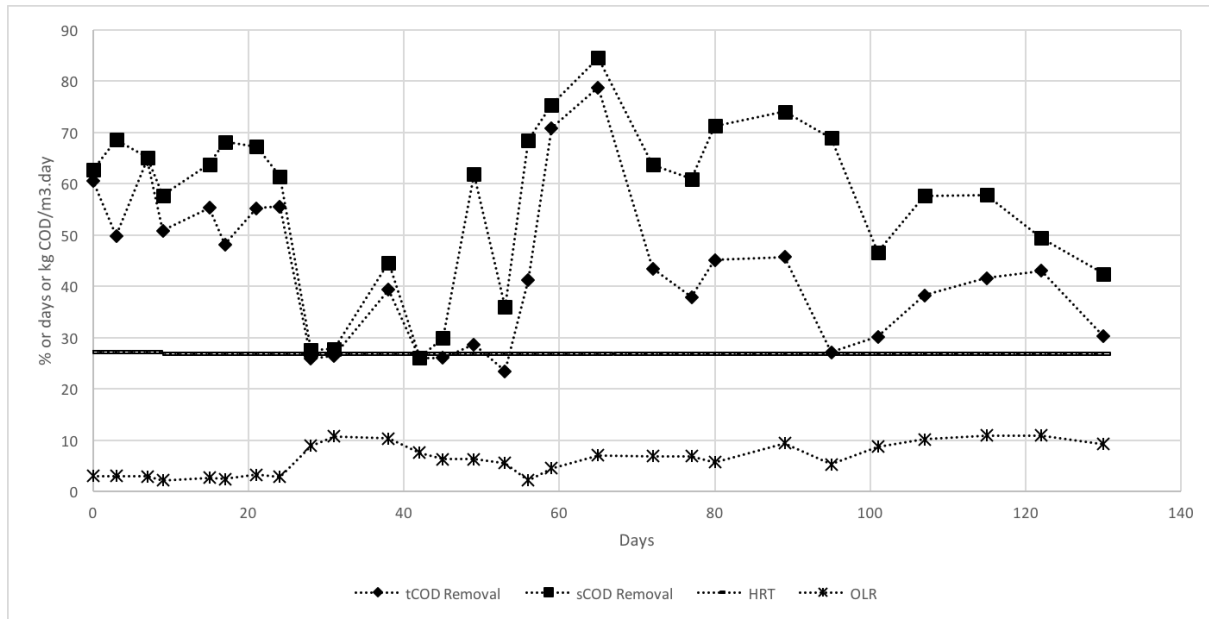


Figure 4–36: HyVAB<sup>®</sup> performance at varying OLR and HRT

Figure 4–37 shows tCOD removal in AD and CIFC<sup>®</sup> stage which shows similar trend as in overall COD removal. At OLR higher than the design load, tCOD removal efficiency in both stages decreased below 40%. It tried to recover with decreased OLR after day 55 but went past below 40% again with increased OLR after day 90. Negative efficiency in AD stage is may be due to biomass washout.

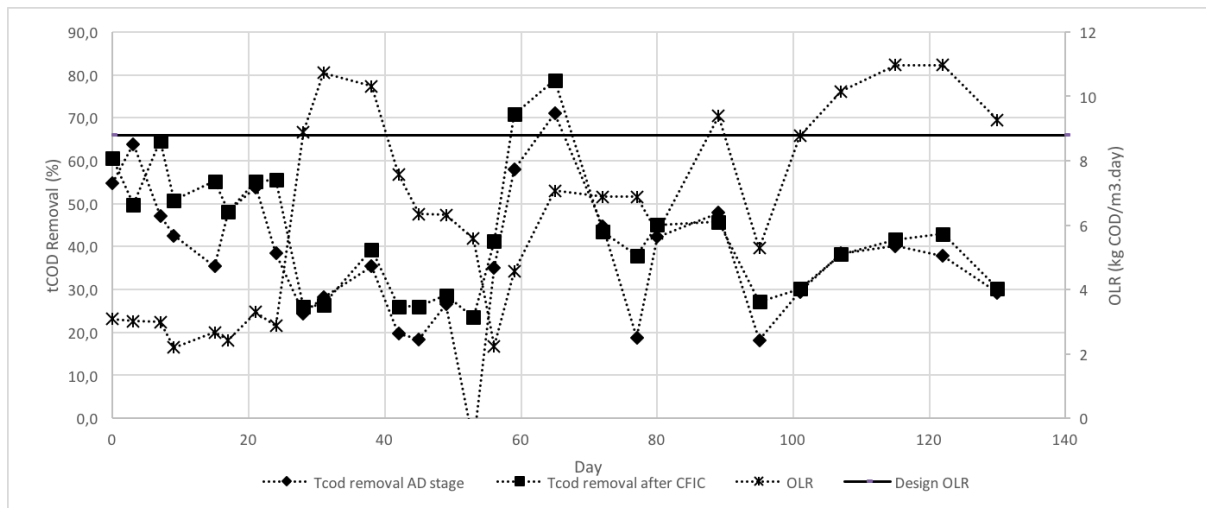


Figure 4-37: tCOD Removal in AD and CFIC<sup>®</sup> stage of HyVAB<sup>®</sup>

sCOD removal trend is similar to the tCOD and overall COD removal cases as shown in Figure 4-38. sCOD removal decreased with increased OLR which also reduced Dissolved Oxygen (DO) in CFIC<sup>®</sup>. DO reduced to less than 0.5 mg/L towards the end of the experiment at higher OLR. sCOD removal efficiency is severely affected at this period. Less COD removal in AD stage caused the higher consumption of dissolved oxygen affecting COD removal efficiency in CFIC<sup>®</sup> stage also due to overloading condition.

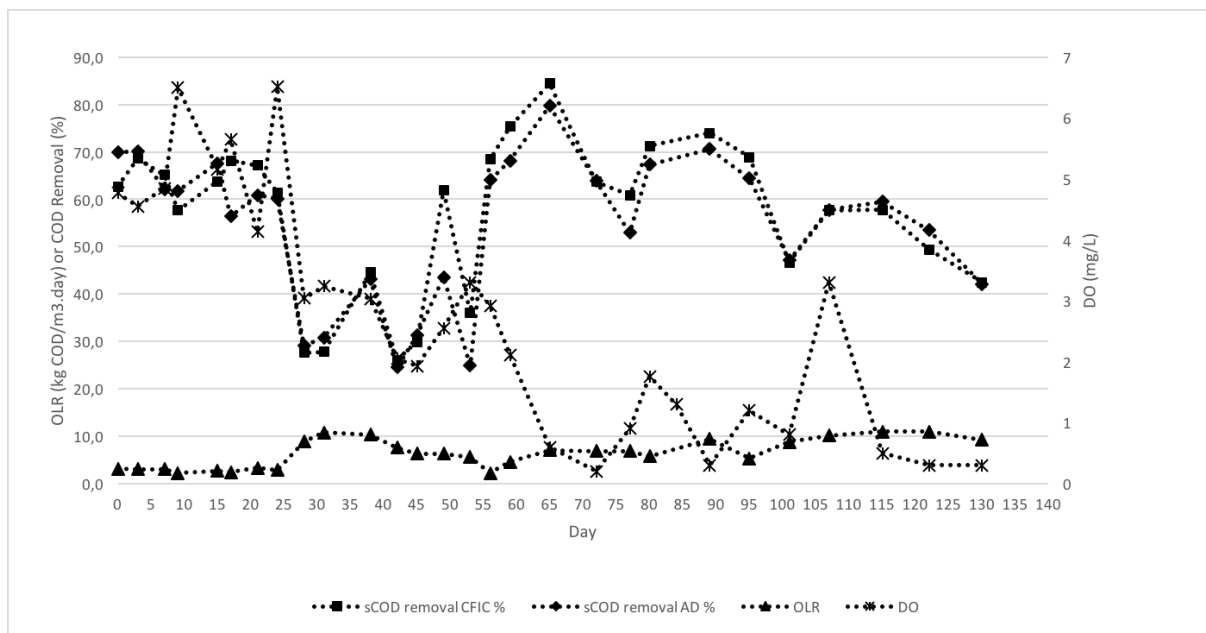


Figure 4-38: COD Removal in AD and CFIC<sup>®</sup> stage of HyVAB<sup>®</sup>

During the 130 days of operation, CFIC's pH was over 8 most of the time. During the period when OLR was high (From day 25 to 50 and day 100 to 130), CFIC's pH was between 6 and 7 despite the feed pH being in the range of 6 and 7. AD's pH also dropped to around 6.5 during the high OLR period. (Figure 4-39)



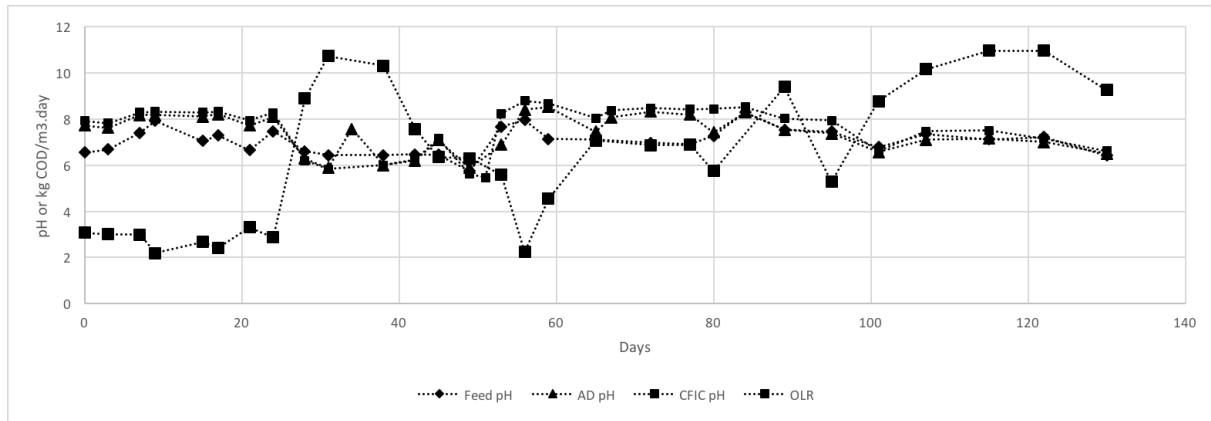


Figure 4–39: Fluctuation of pH in different stages

#### 4.2.2 Aerobic biofilm stage

Total and Volatile suspended solids (TSS and VSS) from the CFIC<sup>®</sup> effluent were monitored and were on average found to be 462 and 432 mg/L respectively. The biomass yield of HyVAB<sup>®</sup> was on average found to be 0.160 g VSS/ g COD removed (Figure 4–40).

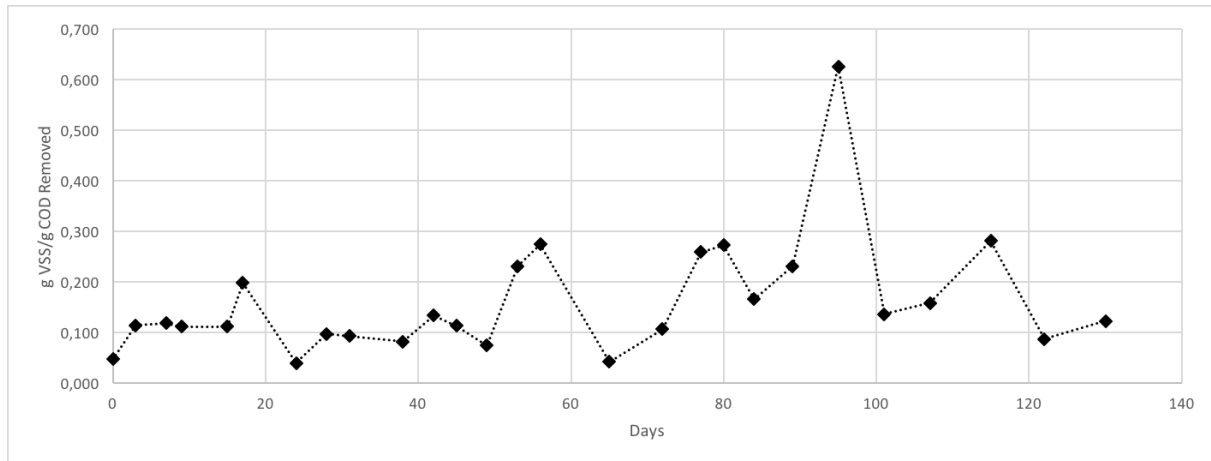


Figure 4–40: Biomass production calculated from effluent VSS

Figure 4–41 shows depletion of dissolved oxygen in high organic loading rate. When OLR increased over the design load after day 20, DO started decreasing. At around days 50 and 100, DO showed some improvement with onset of decreased OLR but went below 1 mg/L when OLR started rising again. The dissolved oxygen concentration can explain lower COD removal efficiency in CFIC<sup>®</sup> stage.

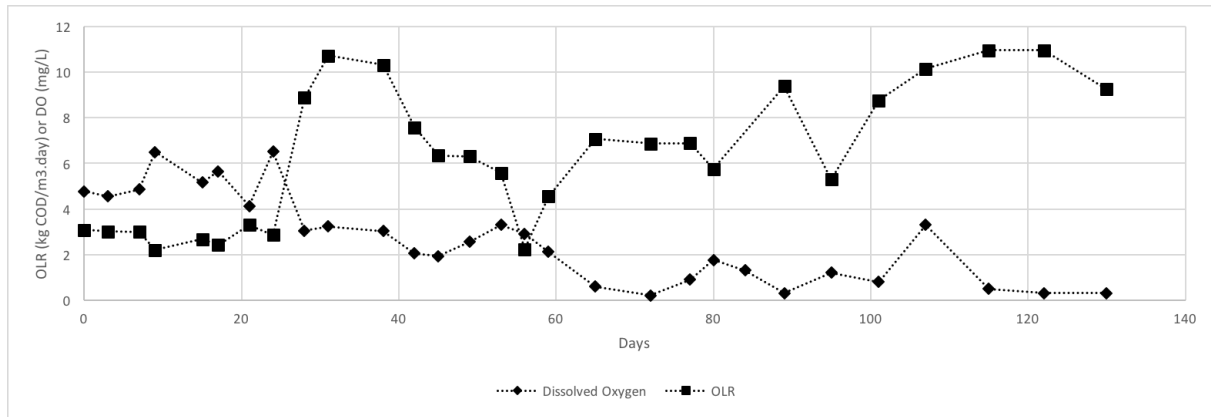


Figure 4–41: Dissolved oxygen concentration in CFIC<sup>®</sup> over time

### 4.2.3 Solids removal

Figure 4–42 shows the suspended solids concentration at two different stages of the reactor at varying OLR. We can see that when OLR was higher than design load, SS increased in both stages with AD stage having the highest value. SS in CFIC<sup>®</sup> effluent was much less compared to AD stage at all the time. When the reactor was run for more than 10 days at higher OLR, SS concentration was almost the same in both the stages.

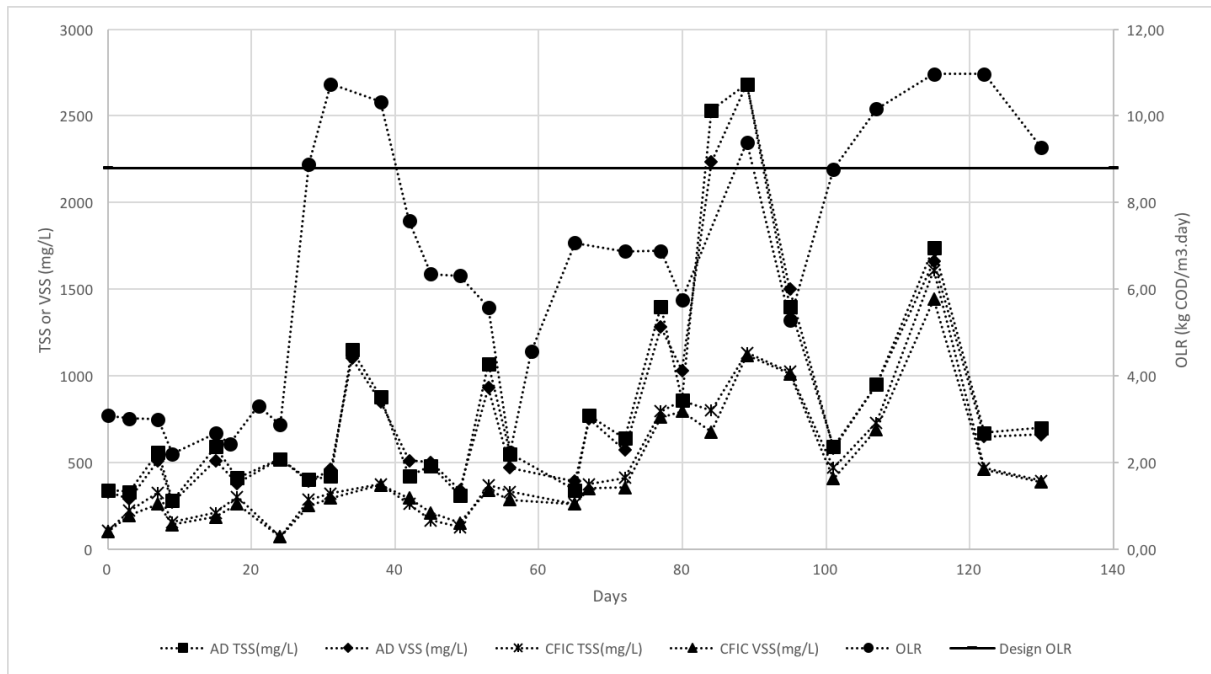


Figure 4–42: TSS and VSS effluent from HyVAB<sup>®</sup> at varying OLR

## 4.3 Economic Analysis

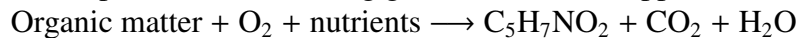
### 4.3.1 Case I. Aeration cost based on aerobic complete mix biological treatment

In case I, an aerobic complete mix biological process without recycle is considered to calculate the aeration cost. Assumed values are given in Table 4–1.

Table 4–1: Assumed values to calculate aeration cost for complete mix biological treatment

Flow rate of wastewater	120 m <sup>3</sup> /day
Influent COD	10 kg COD/m <sup>3</sup>
Effluent COD	0.5 kg COD/m <sup>3</sup>
VSS effluent	0.2 kg VSS/m <sup>3</sup>

Assumption of the following general reaction is applicable: (Tchobanoglous et al., 2003)



The kg VSS/d produced	$0.2 \text{ kg VSS/m}^3 \times 120 \text{ m}^3/\text{day}$	24 kg VSS/d
The kg COD removed	$(10-0.5) \text{ kg COD/m}^3 \times 120 \text{ m}^3/\text{day}$	1140 kg COD/d
Observation yield	$Y_{obs} = \frac{24 \text{ kg VSS/day}}{1176 \text{ kg COD/day}}$	0.021 kg VSS/kg COD removed

COD mass balance around the reaction is given as:

Accumulation = inflow - outflow + conversion

$0 = \text{COD}_{in} - \text{COD}_{out} - \text{oxygen used (expressed as COD)}$

Oxygen used =  $\text{COD}_{in} - \text{COD}_{out}$

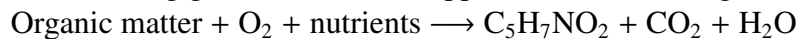
Also,  $\text{COD}_{out} = \text{COD}_{out} + \text{biomass COD}_{out}$

$\text{COD}_{in}$	$10 \text{ kg COD/m}^3 \times 120 \text{ m}^3/\text{day}$	1200 kg COD/d
$\text{COD}_{out}$	$0.5 \text{ kg COD/m}^3 \times 120 \text{ m}^3/\text{day}$	60 kg COD/d
Biomass $\text{COD}_{out}$	$1.42 \text{ kg COD/kg VSS} \times 24 \text{ kg VSS/d}$	34.08 kg COD/d
Total $\text{COD}_{out}$	$24 \text{ kg COD/d} + 34.08 \text{ kg COD/d}$	94.08 kg COD/d
Oxygen used	$(1200 - 94.08) \text{ kg COD/d} = 1105.92 \text{ kg COD/d}$	1106 kg O <sub>2</sub> /d
Oxygen/COD	$1106 \text{ kg O}_2/\text{d} / 1140 \text{ kg COD/d}$	0.97 kg O <sub>2</sub> /kg COD
$\delta$		1 \$ /kg
$\eta$		0.9
Cost of aeration	$(1106 \text{ kg O}_2/\text{d} \times 1 \text{ $ /kg}) / 0.9$	<u>1229 \$ /day</u>

It is found that it costs around 1229 \$/day to treat wastewater with COD concentration of 10 kg/m<sup>3</sup> and the flow of 120 m<sup>3</sup>/day in an aerobic complete mix biological treatment without recycle.

### 4.3.2 Case II. Aeration cost based on real data from Bamble reactor

In case II, real data were used from Bamble CFIC<sup>®</sup> reactor to calculation the cost. Values for flow rate, influent and effluent COD and VSS effluent were used as in Case I. Assumption of the following general reaction is applicable: (Tchobanoglous et al., 2003)



The kg VSS/d produced	$0.41 \times 10 \text{ kg COD/m}^3 \times 120 \text{ m}^3/\text{day}$	492 kg VSS/d
The kg COD removed	$(10-0.5) \text{ kg COD/m}^3 \times 120 \text{ m}^3/\text{day}$	1140 kg COD/d
Observation yield		0.41 kg VSS/kg COD removed

COD mass balance around the reaction is given as:

Accumulation = inflow - outflow + conversion

$0 = \text{COD}_{in} - \text{COD}_{out} - \text{oxygen used (expressed as COD)}$

Oxygen used =  $\text{COD}_{in} - \text{COD}_{out}$

Also,  $\text{COD}_{out} = \text{COD}_{out} + \text{biomass COD}_{out}$

$\text{COD}_{in}$	$10 \text{ kg COD/m}^3 \times 120 \text{ m}^3/\text{day}$	1200 kg COD/d
$\text{COD}_{out}$	$0.5 \text{ kg COD/m}^3 \times 120 \text{ m}^3/\text{day}$	60 kg COD/d
Biomass $\text{COD}_{out}$	$1.42 \text{ kg COD/kg VSS} \times 492 \text{ kg VSS/d}$	698.64 kg COD/d
Total $\text{COD}_{out}$	$60 \text{ kg COD/d} + 698.64 \text{ kg COD/d}$	758.64 kg COD/d
Oxygen used	$(1200 - 758.64) \text{ kg COD/d} = 441.36 \text{ kg COD/d}$	441.36 kg O <sub>2</sub> /d
Oxygen/COD	$1106 \text{ kg O}_2/\text{d} / 1140 \text{ kg COD/d}$	0.97 kg O <sub>2</sub> /kg COD
$\delta$		1 \$ /kg
$\eta$		0.9
Cost of aeration	$(441.36 \text{ kg O}_2/\text{d} \times 1 \text{ $ /kg})/0.9$	<u>490.4 \$ /day</u>

Based on the real data from the CFIC reactor at NSO, Bamble<sup>®</sup>, it is found that it costs approximately 490.4 \$/day which is comparatively less than the cost obtained while treating the same amount of wastewater with same concentration of COD in an aerobic complete mix biological treatment without recycle.

### 4.3.3 Case III. Aeration cost after anaerobic digestion in AD stage of HyVAB<sup>®</sup>

Values in Table 4-2 were assumed to calculate aeration cost after anaerobic digestion in AD stage of HyVAB<sup>®</sup> reactor.

Table 4–2: Assumed values to calculate aeration cost after AD stage of HyVAB<sup>®</sup> reactor.

Flow rate of wastewater	120 m <sup>3</sup> /day
Influent COD	10 kg COD/m <sup>3</sup>
Effluent COD	0.5 kg COD/m <sup>3</sup>
VSS effluent	0.2 kg VSS/m <sup>3</sup>
f <sub>1</sub>	0.9
f <sub>2</sub>	0.1
Y <sub>X/S2</sub>	0.3 kg VSS/kg COD
ε	0.1
X <sub>2</sub>	3 kg/m <sup>3</sup>
F <sub>OD2</sub>	4 (Benefield and Randall, 1981)
θ	10 (for activated sludge)
b <sub>2</sub>	0.2 1/day
δ	1 \$ /kg
η	0.9

Following values were calculated based on the above assumptions.

V <sub>aer</sub>	$\frac{(120 \times 4 \times 10 \times 0.3 \times 10 \times (1 - 0.9) \times 0.1)}{3 \times (1 + 0.2 \times 10)}$	16 m <sup>3</sup>
F <sub>O2</sub>	$(120 \times 10 \times (1-0.9) \times 0.1 \times (1-1.42 \times 0.3)) + (1.42 \times 0.1 \times 3 \times 16)$	13.7 kg O <sub>2</sub> /day
Cost of aeration	$(13.7 \text{ O}_2/\text{d} \times 1 \text{ \$ /kg})/0.9$	<u>15.23 \$ /day</u>

Based on the assumptions listed in Table 4–2 it is found that treating wastewater anaerobically in AD stage of HyVAB<sup>®</sup> reactor before aeration could reduce the cost of aeration down to 15.23 \$ /day. It is 475.2 \$ /day less than the cost of aeration in CFIC reactor at NSO, Bamble.

#### 4.3.4 Methane production from anaerobic digestion prior aeration

Ramalho (2012) calculated total methane produced as follows. Assumptions are listed in Table 4–3

Table 4–3: Assumed values to calculate methane production prior aeration.

Flow rate of wastewater	120 m <sup>3</sup> /day
Influent COD	10 kg COD/m <sup>3</sup>
f <sub>1</sub>	90%
Y <sub>X/S2</sub>	0.19 kg VSS/kg COD
β	0.061 \$/(kW.h)
Ω <sub>comb</sub>	35770 kJ/(m <sup>3</sup> STD CH <sub>4</sub> /kg COD)
G <sub>o</sub>	0.35 m <sup>3</sup> STD CH <sub>4</sub> /kg COD

Following values were calculated based on the above assumptions.

Cost benefit (methane production)	$\frac{(35770 \times 0.35 \times 120 \times 0.9 \times 10 \times (1 - 1.42 \times 0.19) \times 0.061)}{3600}$	<u>167.29 \$ /day</u>
Energy produced	$35770 \times 0.35 \times 120 \times 0.9 \times 10 \times (1-1.42 \times 0.19)$ kJ/day	<u>9873 MJ/day</u>
Total methane produced	$0.35 \times 120 \times 0.9 \times 10 \times (1-1.42 \times 0.19)$ kJ/day	<u>276 m<sup>3</sup> STD CH<sub>4</sub>/ day</u>

From the above assumptions, it is found that value worth 167.29 \$ /day was gained in terms of methane. The equivalent energy produced is 9873 MJ/day.

Tchobanoglous et al. (2003) calculated total methane produced as follows. Assumptions are listed in Table 4-4.

Table 4-4: Assumed values to calculate methane production prior aeration.

Methane production at 35°C	0.4 m <sup>3</sup> /kg COD)
Density at 35°C	0.635 kg/m <sup>3</sup>
Content of gas	80%
Energy content	50.1 kJ/g

Following values were calculated based on the above assumptions:

Methane density at 30°C	$0.6346 \times (273.15+35)/(273.15+30)$	0.645 kg/m <sup>3</sup>
Methane production at 30°C	$0.4 \times (273.15+30)/(273.15+35)$	0.394 m <sup>3</sup> /kg COD
COD removed	$(10-0.5)$ kg COD/m <sup>3</sup> $\times$ 120 m <sup>3</sup> /day	1140 kg COD/d
Amount of CH <sub>4</sub> produced/day	$1140$ kg COD/d $\times$ 0.394 m <sup>3</sup> /kg COD	<u>448.6 m<sup>3</sup>/day</u>
Total gas produced	$448.6$ m <sup>3</sup> /day / 80%	<u>560.8 m<sup>3</sup>/day</u>

It is found from the above assumptions that the methane produced is 448.6 m<sup>3</sup>/day which is higher than 306 m<sup>3</sup>/day (276 m<sup>3</sup> STD CH<sub>4</sub>/ day) obtained at assumptions by Ramalho (2012)(shown above).

Savings based on aerobic complete mix biological treatment:

=(Aeration cost based on aerobic complete mix biological treatment-Aeration cost after anaerobic digestion)+Methane production from anaerobic digestion prior aeration (lowest value is used among two assumptions shown above)

$$= (1229-15.23) \$ /kg + 167.3 \$ /day = \underline{\underline{1380.9 \$ /day}}$$

Savings based on real data from Bamble reactor:

= (Aeration cost based on aerobic complete mix biological treatment - Aeration cost after anaerobic digestion) + Methane production from anaerobic digestion prior aeration lowest value is used among two assumptions shown above)

$$= (490.4 - 15.23) \$ / \text{kg} + 167.3 \$ / \text{day} = \underline{\underline{642.5 \$ / \text{day}}}$$

#### 4.3.5 Asset value of 1 m<sup>3</sup> of biogas

Fuels (Murphy et al., 2004)	
Energy value of petrol	32.23 MJ/l
Energy value of diesel	40.7 MJ/l
Energy value of CH <sub>4</sub> -enriched biogas (95%CH <sub>4</sub> )	$(0.95 \times 37.78 \text{ MJ/Nm}^3) = 35.9 \text{ MJ/Nm}^3$

Efficiencies (Murphy et al., 2004):		
Petrol run Volvo V70 bi-fuel	9.8 km/l	0.3 km/MJ
CH <sub>4</sub> -enriched biogas run V70 bi-fuel	9.6 km/Nm <sup>3</sup>	0.267 km/MJ
Diesel run Volvo S60	13.17 km/l	0.32 km/MJ
CH <sub>4</sub> -enriched biogas run Volvo S60 bi-fuel	10 km/Nm <sup>3</sup>	0.29 km/MJ

Asset value as petrol substitute

Petrol \$1.46/l in Norway = \$ 0.046/MJ

Biogas has 90% efficiency of petrol = \$ 0.04/MJ

1 m<sup>3</sup> CH<sub>4</sub>-enriched biogas = \$ 1.46 equates to \$ 1.2 m<sup>3</sup> biogas @ 80% CH<sub>4</sub> (30.22 MJ/Nm<sup>3</sup>)

Therefore, Asset value of 1 m<sup>3</sup> biogas = \$ 0.9 allowing for 25% tax

Asset value as diesel substitute

Petrol \$1.34/l in Norway = \$ 0.033/MJ

Biogas has 90% efficiency of petrol = \$ 0.03/MJ

1 m<sup>3</sup> CH<sub>4</sub>-enriched biogas = \$ 1.064 equates to \$ 0.89 m<sup>3</sup> biogas @ 80% CH<sub>4</sub> (30.22 MJ/Nm<sup>3</sup>)

Therefore, Asset value of 1 m<sup>3</sup> biogas = \$ 0.668 allowing for 25% tax

# 5 Discussions

## 5.1 Pilot scale reactor

### 5.1.1 Start-up of the reactor

The start-up should be monitored well for smooth operation of anaerobic bioreactor. Factors like wastewater characteristics, acclimatisation of seed sludge, pH, nutrient, presence of toxic compounds, loading rate, up-flow velocity, hydraulic retention time, liquid mixing and reactor design affect the sludge growth and make the start-up of UASB reactors more complicated (Rizvi et al., 2013). Sludge granulation is an important factor in UASB process. The startup procedure is extremely important for active sludge development with both high specific activity and settleability (Borja and Banks, 1994). Addition of mature granules as inoculum avoided the problems regarding start-up in our case. Excellent COD removal rate (more than 90%) at a short period time of 15 days is the result of inoculation of mature granules and good hydraulic contact between the substrate and the sludge due to recirculation. tCOD removal efficiency decrease from day 40 for few days and day 68 for few days was back to track due to self-regulation capability inherent in the biological system which makes it possible for the microbial consortium to acclimate itself to increased OLR (Chan et al., 2012).

### 5.1.2 Reactor performance

The high rate of COD removal efficiency even at high OLR may also be attributed to high ratio of effluent recycle (Najafpour et al., 2006). Mohan et al. (2007) observed high COD removal efficiency and biogas yield by the introduction of recirculation to the treatment of hypersaline composite complex chemical wastewater. Chan et al. (2012) attributed this enhancement to the improvement in mass transfer between the substrate present in the bulk liquid and the attached biofilm. The concentration gradient (substrate inhibition) of substrate and reaction by-products (VFA) resulted in mixed flow condition due to hydrodynamic behaviour of recirculation mode (Mohan et al., 2007). It was found that during the start-up of a hybrid system consisting of an UASB and anaerobic fixed filter (AFF), recirculation ratios (ratio of recirculation flow to feed flow) of 19 to 28 caused decline in COD removal efficiency but 8-19 did not have any effect on it (Zhang et al., 2009). Mutual collisions caused the breakdown of the sludge granules as suggested by these results. HyVAB<sup>®</sup> reactor had no problem even at the highest recirculation ratio of 104 at the beginning. As the reactor approached its maximum operational capacity, COD removal efficiency started decreasing after reaching the maximum value. It was a sign of insufficient microbial accumulation in the reactor to handle the higher organic load (Strydom et al., 1995) and decrease in contact time of wastewater with sludge granules utilising less organic matter (Farajzadehha et al., 2012). Long SRT also could effectively increase efficiency of hydrolysis and subsequent digestion of organic matter (Farajzadehha et al., 2012).

### 5.1.3 Biogas production

Biogas was collected from the very first day of the operation of the reactor. Although, the single point data doesn't give the true scenario, graph obtained from the continuous monitoring towards the end of operation revealed that the gas collection was stable until the critical organic



loading rate of 20 kg COD/m<sup>3</sup>.d and started decreasing with the increased OLR. Chan et al. (2012) and Kiely et al. (1997) found that methane content and methane yield increased proportionally with OLR, as the increasing available organic matter is converted to biomass. But such phenomena was not observed in our case in terms of methane content. Such trend could not be observed in terms of methane yield as continuous data were taken only during the end of the operation. The methane yield achieved was close to the theoretical value of 0.35 L CH<sub>4</sub> (STP)/g COD removed at OLR of around 20 kg COD/m<sup>3</sup>.d but started decreasing beyond that loading rate. The biogas collected was rich in methane (80-90%). It can be burnt directly or compressed for other use with less purification cost.

#### 5.1.4 Development of granules

Distinct sludge regions were observed along the height of the reactor with highest sludge concentration being in the bottom. The sludge concentration decreased along the height of the reactor from bottom to top. When loading rates increased beyond the design load, the distinct interface between granular sludge and sludge blanket vanished indicating poor settleability of the granules. This was also substantiated by the fact that the amount of granular sludges in the collected sample from AD3 was also decreased. Rather, thick slurry was collected with tCOD concentration over 10 g/L. Recycling pump was also blocked due to high solids concentration from the attrition of granules. The sludge washout could also be a result of the increased gas production at higher loading rates and relatively high amount of suspended solids in influent solution although most of the insoluble poorly biodegradable matters are entrapped in the sludge bed (Sayed et al., 1984). This caused in reduction of total sludge concentrations in the reactor which reduced COD removal efficiency and biogas production.

Sludge loading rate was increasing over the operational days. (Ghangrekar et al., 2005) recommended the SLR value of 0.1 - 0.25 kg COD/kg VSS.d at OLR of 2 - 4.5 kg COD/m<sup>3</sup>.d during start up which was the exact case in this study. This has helped the reactor to achieve high COD removal because there was detrimental effect on granules formation at very high loading rates during start-up and granulation probably due to vigorous mixing conditions produced by higher biogas generation (Ghangrekar et al., 2005). Under sludge loading rates higher than 0.3 kg COD/kg VSS.d, the volatile fatty acid (VFA) concentration in the reactor was observed to be greater than 300 mg/L as acetic acid, favouring *Methanosarcina* sp. over *Methanothrix* species (Ghangrekar et al., 2005). Although both of them have tendency to grow as granules, the *Methanothrix* granules is better when compared to *Methanosarcina* (Vanderhaegen et al., 1992). Also, granules developed at higher VFA concentration are fragile in nature making them more susceptible to wash out (Ghangrekar et al., 2005).

Specific methanogenic activity (SMA) of the reactor increased with the increase in sludge. The SMA also increased with the increase in size of the granules (Jijai et al., 2014). The activity of the sludge increased from the low specific methanogenic activity of 0.074 kg CH<sub>4</sub>-COD/kg VSS.d at the start of the experiment to 0.428 kg CH<sub>4</sub>-COD/kg VSS.d at day 71. Even at low SMA, COD removal efficiency was higher.

The sludge retention was fairly satisfactory till the design OLR except for some days after addition of new poor granules. It is required to minimise the sludge washout from the UASB reactor as around 67% of the excess sludge disappear from the reactor either as suspended solids or in the form of increasing scum layers (Sayed et al., 1984). Because of hybrid effect,

sludge washout was not seen in the effluent although it could be seen in recycling point. It was confirmed by measured recycle COD higher than the feed COD. Above recycling point, vertical velocity decreased allowing particles to either settle down or recycle back to AD stage. It was confirmed by determining TSS concentration of the sample from the point just above the recycling point. TSS concentration in higher point was around 7-18% less than the recycling point. At the end of the operation when OLR was too high, suspended solids were very high in the effluent indicating heavy washout of granules generating much smaller particles which could reach to the effluent. However, the wash out of some sludge is necessary to avoid excess sludge accumulation.

### **5.1.5 VFA buildup and consumption**

VFA was always less than 500 mg/L as acetic acid indicating a good start-up (Shivayogimath and Ramanujam, 1999). Some increased VFA concentrations were seen while increasing OLR stressing and inhibiting the biomass. But such condition lasted only for few days before returning back to normal and COD removal efficiency reached up to the normal value. This result is typical during start-up, where the response of acid-producing fraction of the microbial consortium is always more rapid than that of methanogens to step-wise increases in feed loading (Borja and Banks, 1995). This can also be seen in the biogas production graph where biogas production is low during VFA accumulation and started to increase with the consumption of VFA. During the initial phase till day 20, gas production appeared to be repressed which may be attributed to temporary inhibition of the methanogens during acclimatisation to higher acidity level (Borja and Banks, 1995). Both the concentration of acetic acid and propionic acid increased with the increase in loading rate and then decreased back to normal concentration indicating well adaptation of microorganisms. Such trend was seen in all the three sampling points. However, at the end when the system was overloaded, VFA concentration reached 1630 mg/L stressing the biomass and decreasing its activity. Although, time frame was not enough to see how the reactor would act when OLR was reduced back to design load, it can be postulated that the reactor performance would recover easily. pH drop was visible in the effluent indicating VFA buildup.

It is interesting to notice a parallel increase in effluent COD and VFA concentrations as shown in Figure 4–9 and Figure 4–7. This result agrees with the results obtained by Zhang et al. (2008), Borja and Banks (1995) and Chan et al. (2012) which revealed that approximately two-thirds of anaerobic effluent COD was caused by VFA. This confirms the fact that a positive correlation existed between the effluent COD and VFA concentration.

Although Chan et al. (2012) found out that the pH demonstrated an increasing trend throughout the anaerobic compartment from bottom to top and conversely, the VFA demonstrated an opposite trend to pH, such case as not always true in our case. From the VFA analysis in day 0 and 1, it was found that lowest part AD1 had the highest concentration of VFA but AD3 had higher VFA concentration than at AD2. Even recycle had higher VFA concentration than AD2 and AD3 in day 2. Also, pH variation along the height was not always in decreasing order. This can be attributed to strong recirculation. It is postulated that in the first two steps of anaerobic digestion, hydrolysis and acidogenesis prevailed in the sludge bed, where complex organics in oil wastewater were first hydrolysed to enzymes, forming sugars, amino acids and fatty acids and acidogens further degrading them to form VFAs. Acidogens were probably predominant

at AD1 as they are fast grower and less sensitive to pH variation compared to acetogens and methanogens (Chan et al., 2012). Strong concentration decrease in VFA from AD1 to AD2 and above suggests that acetogenesis took place at these points degrading VFAs.

### **5.1.6 Solids removal**

Less effluent suspended solids is one of the major characteristics of HyVAB<sup>®</sup> reactor. At the stable condition, TSS concentration in influent was always below 500 mg/L. The concentration increased with the increase in loading rate. VSS/TSS ratio was 86% on average.

The biomass yield value is less compared to other reactors due to the combination of anaerobic and aerobic process in compact system. The low sludge yield is because of anaerobically digested COD which has much less sludge production compared to aerobically digested COD. Some of the detached biofilm biomass also settle down to anaerobic stage due to gravity and is consumed to contribute to the overall low sludge production. However, it still remains to be quantified.

### **5.1.7 Economics**

One of the most important factors to be kept in mind while designing a reactor is economics. Significant amount of money can be saved upon a suitable design. Economic analysis showed that HyVAB<sup>®</sup> reactor could save around 1380.86 \$/day on treating wastewater of 120 m<sup>3</sup>/day instead of treating by complete mix biological treatment. This saving includes 1213.57 \$/day from reduced cost and 167 \$/day from biogas production. Replacing CFIC<sup>®</sup> in NSO by HyVAB<sup>®</sup> reactor could save 642.46 \$/day including 475.17 \$/day from reduced aeration cost and 167 \$/day from biogas production. Asset value of 1 m<sup>3</sup> biogas as petrol substitute was found to be \$ 0.9 allowing for 25% tax with the real data biogas composition from HyVAB<sup>®</sup> reactor. Also, asset value of 1 m<sup>3</sup> biogas as diesel substitute was found to be \$ 0.6675 allowing for 25% tax.

### **5.1.8 Recycle pump**

Peristaltic pump used in recirculation was not a good option. It performed well when the recycling fluid was free of particles. But when the fluid was particle laden, the tube got blocked due to accumulation of particles. This even damaged the tubes due to attrition caused by the particles. Also, recycling granules got crushed due to peristaltic effect. It was the original choice because of low pumping speed (90 L/h) required for the recirculation although it was very expensive. The best alternative would be a pump with fairly low speed and based on the principle 'Whatever goes into the pump comes out of the pump'. It should also handle solid particles up to 7 mm and even bigger soft particles. Some recommended pumps are listed under Future work subsection below.

## **5.2 Laboratory scale reactor**

### **5.2.1 Reactor performance**

Laboratory scale reactor was used to emulate the pilot scale by operating in similar conditions. It helped to observe how the reactor would perform at different conditions like organic over-

loading, low and high pH of feed, lower alkalinity etc. COD removal efficiency was fluctuating with values reaching close to 80% for the both sCOD and tCOD during stable condition and dropping down to less than 30% in overloaded conditions. It is observed that the drop was due to overloaded organic condition which stressed the organisms. Upon reducing the load, the system reduced back to normal operation. COD removal efficiency was not good compared to lab scale reactor in stable condition may be because of oxygen diffusion into the anaerobic section.

### **5.2.2 VFA buildup and consumption**

pH fluctuation was seen clearly during the overloaded condition. VFA and especially acetate accumulation was observed during this condition lowering pH of all stages. This decreased the removal efficiency of AD section putting more organic load in the CFIC<sup>®</sup> stage. It could be verified by reduced DO concentration.

### **5.2.3 Solids removal**

Overloaded condition also affected the solids removal as discussed in pilot scale section. At stable conditions, TSS and VSS concentration was below 500 mg/L as in pilot scale reactor. VSS/TSS was around 90%. At higher loading, effluent TSS and VSS increased indicating stress in the granules. Biomass washout was not observed in this case unlike in pilot scale.

The biomass yield value is less in this case also which is due to anaerobically digested COD which has much less sludge production compared to aerobically digested COD. Detached biofilm biomass from CFIC<sup>®</sup> settled down to AD section which contributed to overall low sludge production.

## **5.3 Future Work**

### **5.3.1 High purity oxygen digestion**

It is a modification of the aerobic digestion process using pure oxygen instead of air. It is particularly applicable in cold weather climate as it is very less sensitive to change in ambient air temperatures because of high rate of biological activity and exothermal nature of the process (Tchobanoglous et al., 2003). Opting for oxygen over aeration makes aerobic degradation 27% cheaper. (Vera et al., 1999). Positive aspects of high purity oxygen process are better sludge settling and thickening, lower sludge yield, higher oxygen transfer efficiency and more stable operation while negative aspects are that the efficacy of the process is still unclear, mechanism is still dubious and aeration cost is high (Pérez-Elvira et al., 2006). Ball and Humenick (1972) found higher intrinsic metabolism rates by the organisms and lower treatment costs as compared to conventional aeration process because of reduced reactor volume as other advantages.

### **5.3.2 Quantification of settled CFIC<sup>®</sup> biofilm in AD**

Amount of biofilm that settle in the AD stage can be quantified as a future work. The study of types of microorganisms that grow in AD can also give an interesting result. These study can help in understanding the overall process of HyVAB to improve its performance even more.

### 5.3.3 Determination of integrity coefficient

Ghangrekar et al. (2005) had observed the relationship between the strength of granules developed, COD removal efficiency and applied loading rate during the start-up. It can also be observed how strength of granules affects the effluent solid concentration as a future work. This could help to make HyVAB<sup>®</sup> more desirable.

### 5.3.4 Recovery from overloaded conditions

It would be very interesting and useful to see how and at what time the reactor would recover from overloaded conditions. These findings would be very useful while treating complex type of wastewater. These informations help to decide if any monitoring device is needed in the reactor.

### 5.3.5 Effects of recirculation

It would be very interesting to see how different recycling ratios (ratio of recycle flow to feed flow) would affect in COD removal and biogas production.

### 5.3.6 Recommended pumps for recirculation

Some of the pumps that can be used in both the pilot and full scale plant are listed below:

**Abaque Peristaltic Hose Pumps** This pump handles toughest pumping needs from abrasive and aggressive fluids to sheer sensitive and viscous materials. Flow range is 15 L/h to 480 L/h and is suitable for industrial and municipal water/wastewater treatment. Hose option is also available depending upon different types to fluids to be pumped and operation temperature. This pump exactly suits for the pilot reactor and probably for full scale depending upon the si.

**Magnetic drive pump** These pumps are comparatively cheap and is available in different models depending upon the types of fluid. However, flow rate is very high making it unsuitable for the pilot plant. However, it can be an attractive option for full scale.

**Centrifugal pedestal pumps** Although not suitable for pilot scale because of high flow rate, this can be used in full scale because of its ability to handle abrasive liquid (specific models only). Lowest flow rate is 1 gallon per minute (approximately 227 L/h).

## 6 Conclusion

The overall performance of a new hybrid HyVAB<sup>®</sup> reactor was studied on laboratory scale and pilot scale. High strength oil wastewater was used as feed. The relatively good performance of the reactor was found to be attributed to several factors like good start-up techniques, use of mature granular sludge, good recirculation in anaerobic stage and integration of CFIC<sup>®</sup> as a polishing unit of UASB effluent. The following points were observed during the study of pilot scale reactor:

- The startup of a HyVAB<sup>®</sup> should be initiated with low OLR and increased step-wise after confirming good performance of the reactor with by the analysis of VFA concentration, biogas production, methane concentration in biogas and effluent COD concentration. Pilot reactor had better performance compared to laboratory reactor.
- Use of mature granules as inoculum could reduce the startup time drastically. It also helped to stabilise the reactor quickly upon increased OLR till the design load.
- The HyVAB<sup>®</sup> hybrid reactor is an efficient alternative to treat high strength oil wastewater with over 95% COD removal achievable. It could perform well up to the loading rate of 20 kg COD/m<sup>3</sup>.d but performance worsened drastically beyond this load.
- OLR of 18 kg COD/m<sup>3</sup>.d looked to be a good design load with some extra loading as buffer to counteract increased OLR due to variation in feed composition.
- VFA consumption was good during the stable operation of the reactor. VFA accumulation could be seen during the startup and the overloaded condition.
- The biomass yield was considerably low on average 0.16 kg VSS/ kg COD removed.
- Biogas rich in methane content (80-90%) with methane yield close to the theoretical value of 0.35 STP L CH<sub>4</sub>/ g COD removed (0.4 at our working temperature) could be produced besides treating the oil wastewater effectively.
- Total solids (TS), volatile solids (VS), specific methanogenic activity (SMA) and sludge loading rate (SLR) of the granules increased over operational days.
- The hybrid effect of HyVAB<sup>®</sup> can reduce the operational cost significantly compared to aerobic process only.
- Treating high strength oil wastewater within short period of time at reduced space utility and operational cost could be achieved by using high-rate integrated anaerobic-aerobic HyVAB<sup>®</sup> bioreactor.
- Replacing complete mix biological treatment and CFIC<sup>®</sup> with HyVAB<sup>®</sup> could save significant amount money up to \$ 11.5 and \$ 5.35 per day per m<sup>3</sup> of treated wastewater respectively.
- Hybrid HyVAB<sup>®</sup> design could be a very feasible alternative in the treatment of oil wastewater at higher COD and TSS removal efficiency and lower operational cost.

The following points were observed during the study of laboratory scale reactor:

- The reactor has good COD removal within design OLR of 8.7 kg COD/m<sup>3</sup>.d with highest sCOD and tCOD removal rate of 80% at HRT of 27.3 hours.
- OLR of 8.7 kg COD/m<sup>3</sup>.d looked to be the best design load with some buffer value to counteract variable feed composition.
- Anaerobic stage removed about half of the wastewater organic, mostly recovered as methane rich biogas(approx. 60%) by a collector at the anaerobic and aerobic interface.
- The effluent sludge production was approximately 0.16 g VSS/g COD removed.
- TSS and VSS were well below 500 mg/L with values increasing with increased OLR over design load.
- The stable and high waste removal efficiency pose HyVAB<sup>®</sup> as a lucrative and compact alternative for high strength wastewater.

# Bibliography

- Abbasi, T. and Abbasi, S. a. (2012). Formation and impact of granules in fostering clean energy production and wastewater treatment in upflow anaerobic sludge blanket (UASB) reactors. *Renewable and Sustainable Energy Reviews*, 16(3):1696–1708.
- Abbasi, T., Tauseef, S. M., and Abbasi, S. A. (2012). Anaerobic digestion for global warming control and energy generation - An overview. *Renewable and Sustainable Energy Reviews*, 16(5):3228–3242.
- Abdelgadir, A., Chen, X., Liu, J., Xie, X., Zhang, J., Zhang, K., Wang, H., and Liu, N. (2014). Characteristics, process parameters, and inner components of anaerobic bioreactors. *BioMed Research International*, 2014.
- Andersen, T. (2012). Biowater technology: Leading solutions for biological waste water treatment.
- Appels, L., Baeyens, J., Degrève, J., and Dewil, R. (2008). Principles and potential of the anaerobic digestion of waste-activated sludge. *Progress in energy and combustion science*, 34(6):755–781.
- Asadi, A., Zinatizadeh, A., and Isa, M. H. (2012). Performance of intermittently aerated up-flow sludge bed reactor and sequencing batch reactor treating industrial estate wastewater: A comparative study. *Bioresource Technology*, 123:495 – 506.
- Ball, J. E. and Humenick, M. J. (1972). High-purity oxygen in biological treatment of municipal wastewater. *Journal (Water Pollution Control Federation)*, pages 65–76.
- Benfield, L. D. and Randall, C. W. (1981). Biological process design for wastewater treatment. In *Prentice Hall Series in Environmental Sciences*. Prentice hall.
- Bhattacharya, S. K., Uberoi, V., Madura, R. L., and Haghghi-Podeh, M. R. (1995). Effect of cobalt on methanogenesis. *Environmental technology*, 16(3):271–278.
- Boardman, G. D., Tisinger, J. L., and Gallagher, D. L. (1995). Treatment of clam processing wastewaters by means of upflow anaerobic sludge blanket technology. *Water Research*, 29(6):1483–1490.
- Borja, R. and Banks, C. (1994). Anaerobic digestion of palm oil mill effluent using an up-flow anaerobic sludge blanket reactor. *Biomass and Bioenergy*, 6(5):381–389.



- Borja, R. and Banks, C. J. (1995). Comparison of an anaerobic filter and an anaerobic fluidized bed reactor treating palm oil mill effluent. *Process Biochemistry*, 30(6):511–521.
- Botalova, O. and Schwarzbauer, J. (2011). Geochemical characterization of organic pollutants in effluents discharged from various industrial sources to riverine systems. *Water, Air, & Soil Pollution*, 221(1):77–98.
- Bottero, S., Storck, T., Heimovaara, T. J., van Loosdrecht, M. C. M., Enzien, M. V., and Picioroanu, C. (2013). Biofilm development and the dynamics of preferential flow paths in porous media. *Biofouling*, 29(9):1069–86.
- Cervantes, F., Pavlostathis, S., and van Haandel, A. (2006). Advanced biological treatment processes for industrial wastewaters-principles & applications. *Water Intelligence Online*, 5:9781780402345.
- Chan, Y. J., Chong, M. F., and Law, C. L. (2012). An integrated anaerobic–aerobic bioreactor (iaab) for the treatment of palm oil mill effluent (pome): Start-up and steady state performance. *Process Biochemistry*, 47(3):485–495.
- Chan, Y. J., Chong, M. F., Law, C. L., and Hassell, D. (2009). A review on anaerobic–aerobic treatment of industrial and municipal wastewater. *Chemical Engineering Journal*, 155(1-2):1–18.
- Chen, Y., Cheng, J. J., and Creamer, K. S. (2008). Inhibition of anaerobic digestion process: A review. *Bioresource Technology*, 99(10):4044 – 4064.
- Chong, S., Sen, T. K., Kayaalp, A., and Ang, H. M. (2012). The performance enhancements of upflow anaerobic sludge blanket (UASB) reactors for domestic sludge treatment – A State-of-the-art review. *Water Research*, 46(11):3434–3470.
- Clark, D. P., Dunlap, P., Madigan, M., and Martinko, J. (2009). Brock biology of microorganisms.
- Cresson, R., Carrère, H., Delgenès, J. P., and Bernet, N. (2006). Biofilm formation during the start-up period of an anaerobic biofilm reactor-Impact of nutrient complementation. *Biochemical Engineering Journal*, 30(1):55–62.
- Cresson, R., Escudié, R., Carrère, H., Delgenès, J.-P., and Bernet, N. (2007). Influence of hydrodynamic conditions on the start-up of methanogenic inverse turbulent bed reactors. *Water research*, 41(3):603–612.
- Daisy, A. and Kamaraj, S. (2012). The impact and treatment of night soil in anaerobic digester: a review. *Journal of Microbial & Biochemical Technology*, 2011.
- del Mar Batista Seguí, M. (2014). Small pilot scale study for new wastewater treatment process: HyVAB. Master's thesis, Norwegian University of Life Sciences, Faculty of Environmental Science and Technology, Ås, Norway.

- Diya'uddeen, B. H., Daud, W. M. A. W., and Aziz, A. A. (2011). Treatment technologies for petroleum refinery effluents: a review. *Process Safety and Environmental Protection*, 89(2):95–105.
- Drechsel, P. and Evans, A. E. (2010). Wastewater use in irrigated agriculture. *Irrigation and Drainage Systems*, 24(1-2):1–3.
- El-Naas, M. H., Al-Zuhair, S., Al-Lobaney, A., and Makhlof, S. (2009). Assessment of electrocoagulation for the treatment of petroleum refinery wastewater. *Journal of environmental management*, 91(1):180–185.
- Eliyan, C. (2007). Anaerobic digestion of municipal solid waste in thermophilic continuous operation. Master's thesis, Asian Institute of Technology.
- Escudié, R., Cresson, R., Delgenès, J.-P., and Bernet, N. (2011). Control of start-up and operation of anaerobic biofilm reactors: An overview of 15 years of research. *Water Research*, 45(1):1–10.
- Farajzadehha, S., Mirbagheri, S., Farajzadehha, S., and Shayegan, J. (2012). Lab scale study of hrt and olr optimization in uasb reactor for pretreating fortified wastewater in various operational temperatures. *APCBEE Procedia*, 1:90–95.
- Frostell, B. (1983). Anaerobic-aerobic biological treatment of starch industry waste waters. *Starch-Stärke*, 35(6):185–189.
- Ghangrekar, M., Asolekar, S., and Joshi, S. (2005). Characteristics of sludge developed under different loading conditions during {UASB} reactor start-up and granulation. *Water Research*, 39(6):1123 – 1133.
- González, J. S., Rivera, A., Borja, R., and Sánchez, E. (1998). Influence of organic volumetric loading rate, nutrient balance and alkalinity: COD ratio on the anaerobic sludge granulation of an UASB reactor treating sugar cane molasses. *International Biodeterioration and Biodegradation*, 41(2):127–131.
- Gujer, W. and Zehnder, A. J. B. (1983). Conversion processes in anaerobic digestion. *Water Science and Technology*, 15(8-9):127–167.
- Hansen, K. H., Angelidaki, I., and Ahring, B. K. (1998). Anaerobic digestion of swine manure: inhibition by ammonia. *Water research*, 32(1):5–12.
- Jijai, S., Srisuwan, G., O-thong, S., Ismail, N., and Siripatana, C. (2014). Specific methanogenic activities (sma) and biogas production of different granules size and substrate. In *The 1st Environmental and Natural Resources International Conference*, The Sukosol hotel, Bangkok, Thailand.
- Jou, C.-J. G. and Huang, G.-C. (2003). A pilot study for oil refinery wastewater treatment using a fixed-film bioreactor. *Advances in Environmental Research*, 7(2):463–469.
- Kangle, K., Kore, S., Kore, V., Kulkarni, G., et al. (2012). Recent trends in anaerobic codigestion: a review. *Universal Journal of Environmental Research and Technology*, 2(4):210–219.

- Kayhanian, M. (1994). Performance of a high-solids anaerobic digestion process under various ammonia concentrations. *Journal of Chemical Technology and Biotechnology*, 59(4):349–352. cited By 71.
- Kayhanian, M. (1995). Biodegradability of the organic fraction of municipal solid waste in a high-solids anaerobic digester. *Waste Management & Research*, 13(2):123–136.
- Kiely, G., Tayfur, G., Dolan, C., and Tanji, K. (1997). Physical and mathematical modelling of anaerobic digestion of organic wastes. *Water Research*, 31(3):534–540.
- Knight, R. L., Kadlec, R. H., and Ohlendorf, H. M. (1999). The use of treatment wetlands for petroleum industry effluents. *Environmental science & technology*, 33(7):973–980.
- Koster, I., Rinzema, A., De Vegt, A., and Lettinga, G. (1986). Sulfide inhibition of the methanogenic activity of granular sludge at various ph-levels. *Water Research*, 20(12):1561–1567.
- Latif, M. A., Ghufuran, R., Wahid, Z. A., and Ahmad, A. (2011). Integrated application of upflow anaerobic sludge blanket reactor for the treatment of wastewaters. *Water Research*, 45(16):4683–4699.
- Lettinga, G. (1996). Sustainable integrated biological wastewater treatment. *Water Science and Technology*, 33(3):85 – 98.
- Li, Y., Park, S. Y., and Zhu, J. (2011). Solid-state anaerobic digestion for methane production from organic waste. *Renewable and Sustainable Energy Reviews*, 15(1):821 – 826.
- Lim, S. J. and Kim, T. H. (2014). Applicability and trends of anaerobic granular sludge treatment processes. *Biomass and Bioenergy*, 60:189–202.
- Liu, Y., Xu, H. L., Yang, S. F., and Tay, J. H. (2002). Mechanisms and models for anaerobic granulation in upflow anaerobic sludge blanket reactor. *Water Research*, 37(3):661–673.
- Lo, K., Liao, P., and March, A. (1985). Thermophilic anaerobic digestion of screened dairy manure. *Biomass*, 6(4):301–315.
- McCarty, P. L. (1964). Anaerobic waste treatment fundamentals. *Public works*, 95(9):107–112.
- Mohan, S. V., Babu, V. L., Bhaskar, Y. V., and Sarma, P. (2007). Influence of recirculation on the performance of anaerobic sequencing batch biofilm reactor (ansbbr) treating hypersaline composite chemical wastewater. *Bioresource technology*, 98(7):1373–1379.
- Murphy, J., McKeogh, E., and Kiely, G. (2004). Technical/economic/environmental analysis of biogas utilisation. *Applied Energy*, 77(4):407–427.
- Murray, P. A. and Zinder, S. H. (1985). Nutritional requirements of methanosarcina sp. strain tm-1. *Applied and environmental microbiology*, 50(1):49–55.
- Najafpour, G., Zinatizadeh, A., Mohamed, A., Isa, M. H., and Nasrollahzadeh, H. (2006). High-rate anaerobic digestion of palm oil mill effluent in an upflow anaerobic sludge-fixed film bioreactor. *Process Biochemistry*, 41(2):370 – 379.

- Nicolella, C., van Loosdrecht, M. C., and Heijnen, S. J. (2000). Particle-based biofilm reactor technology. *Trends in Biotechnology*, 18(7):312 – 320.
- Ødegaard, H. (2006). Innovations in wastewater treatment:–the moving bed biofilm process. *Water Science and Technology*, 53(9):17–33.
- O’Flaherty, V., Mahony, T., O’Kennedy, R., and Colleran, E. (1998). Effect of pH on growth kinetics and sulphide toxicity thresholds of a range of methanogenic, syntrophic and sulphate-reducing bacteria. *Process Biochemistry*, 33(5):555–569.
- Pérez-Elvira, S., Diez, P. N., and Fdz-Polanco, F. (2006). Sludge minimisation technologies. *Reviews in Environmental Science and Bio/Technology*, 5(4):375–398.
- Phattaranawik, J. and Leiknes, T. (2010). Study of Hybrid Vertical Anaerobic Sludge-Aerobic Biofilm Membrane Bioreactor for Wastewater Treatment. *Water Environment Research*, 82:273–280.
- Rajeshwari, K., Balakrishnan, M., Kansal, A., Lata, K., and Kishore, V. (2000). State-of-the-art of anaerobic digestion technology for industrial wastewater treatment. *Renewable and Sustainable Energy Reviews*, 4(2):135–156.
- Ramalho, R. (2012). *Introduction to wastewater treatment processes*. Elsevier.
- Rizvi, H., Ahmad, N., Abbas, F., Bukhari, I. H., Yasar, A., Ali, S., Yasmeen, T., and Riaz, M. (2013). Start-up of uasb reactors treating municipal wastewater and effect of temperature/sludge age and hydraulic retention time (hrt) on its performance. *Arabian Journal of Chemistry*.
- Rosso, D., Stenstrom, M. K., and Larson, L. E. (2008). Aeration of large-scale municipal wastewater treatment plants: state of the art. *Water Science & Technology*, 57(7).
- Rusten, B., Stang, P., Rogne, E., Siljudalen, J., and Marcolini, L. (2011). Development of a Compact, Cost Effective, and Energy Efficient Biofilm Reactor for Wastewater Treatment and Effluent Reuse. *Proceedings of the Water Environment Federation*, 2011(11):5222–5235.
- Sayed, S., de Zeeuw, W., and Lettinga, G. (1984). Anaerobic treatment of slaughterhouse waste using a flocculant sludge uasb reactor. *Agricultural wastes*, 11(3):197–226.
- Segers, R. (1998). Methane production and methane consumption: a review of processes underlying wetland methane fluxes. *Biogeochemistry*, 41(1):23–51.
- Shayegan, J., Ghavipankeh, F., and Mirjafari, P. (2005). The effect of influent {COD} and upward flow velocity on the behaviour of sulphate-reducing bacteria. *Process Biochemistry*, 40(7):2305 – 2310.
- Shivayogimath, C. and Ramanujam, T. (1999). Treatment of distillery spentwash by hybrid uasb reactor. *Bioprocess Engineering*, 21(3):255–259.
- Singh, R., Kumar, S., and Ojha, C. (1999). Nutrient requirement for uasb process: a review. *Biochemical Engineering Journal*, 3(1):35–54.

- Smitshuijzen, J., Pérez, J., Duin, O., and Loosdrecht, M. C. (2016). A simple model to describe the performance of highly-loaded aerobic {COD} removal reactors. *Biochemical Engineering Journal*, pages –.
- Soto, M., Méndez, R., and Lema, J. (1993). Methanogenic and non-methanogenic activity tests. theoretical basis and experimental set up. *Water Research*, 27(8):1361–1376.
- Strydom, J., Mostert, J., and Britz, T. (1995). Anaerobic treatment of a synthetic dairy effluent using a hybrid digester. *Water SA*, 21(2):125–130.
- Takashima, M., Shimada, K., and Speece, R. E. (2011). Minimum requirements for trace metals (iron, nickel, cobalt, and zinc) in thermophilic and mesophilic methane fermentation from glucose. *Water environment research*, 83(4):339–346.
- Tchobanoglous, G., Burton, F. L., Stensel, H. D., and Eddy, M. . (2003). *Wastewater Engineering: Treatment and Reuse*. McGraw-Hill Education.
- Tezel, U., Tandukar, M., and Pavlostathis, S. (2011). Anaerobic biotreatment of municipal sewage sludge. *in-Chief. Comprehensive biotechnology, 2nd ed. In: Agathos S, editor. Environmental biotechnology and safety. Amsterdam, The Netherlands: Elsevier*, 6.
- van Loosdrecht, M., Eikelboom, D., Gjaltema, A., Mulder, A., Tjihuis, L., and Heijnen, J. (1995). Biofilm structures. *Water Science and Technology*, 32(8):35 – 43. Biofilm Structure, Growth and Dynamics Selected Proceedings of the {IAWQ} International Conference and Workshop on Biofilm Structure, Growth and Dynamics.
- Vanderhaegen, B., Ysebaert, E., Favere, K., Van Wambeke, M., Peeters, T., Panic, V., Vandengangenbergh, V., and Verstraete, W. (1992). Acidogenesis in relation to in-reactor granule yield. *Water Science and Technology*, 25(7):21–30.
- Vera, M., Aspé, E., Martí, M., and Roeckel, M. (1999). Optimization of a sequential anaerobic-aerobic treatment of a saline fishing effluent. *Process Safety and Environmental Protection*, 77(5):275–290.
- Verma, S. (2002). Anaerobic digestion of biodegradable organics in municipal solid wastes. Master's thesis, Columbia University.
- Wang, S., Xin, G., Dinamarca, C., and Bakke, R. (2015a). Study of an anaerobic aerobic hybrid (HyVAB) reactor treating oil refinery wastewater. In *WEFTEC, Chicago*.
- Wang, S., Xin, G., Siljudalen, J. G., Dinamarca, C., and Bakke, R. (2015b). Study of HyVAB in treating high strength oil industry wastewater. Unpublished.
- WHO (2015). Drinking water: Fact sheets. Online.
- Xianling, L., Jianping, W., Qing, Y., and Xueming, Z. (2005). The pilot study for oil refinery wastewater treatment using a gas-liquid-solid three-phase flow airlift loop bioreactor. *Biochemical Engineering Journal*, 27(1):40–44.

- Ye, J., Mu, Y., Cheng, X., and Sun, D. (2011). Treatment of fresh leachate with high-strength organics and calcium from municipal solid waste incineration plant using UASB reactor. *Bioresource Technology*, 102(9):5498–5503.
- Zhang, Y., Li, Y., Lina, C., Xiuhua, L., Zhijian, M., and ZHANG, Z. (2008). Startup and operation of anaerobic egsb reactor treating palm oil mill effluent. *Journal of Environmental Sciences*, 20(6):658–663.
- Zhang, Y., Ma, Y., Quan, X., Jing, Y., and Dai, S. (2009). Rapid startup of a hybrid uasb-aff reactor using bi-circulation. *Chemical Engineering Journal*, 155(1):266–271.
- Zhou, G.-M. and Fang, H. (1998). Competition between methanogenesis and sulfidogenesis in anaerobic wastewater treatment. *Water Science and Technology*, 38(8-9 -9 pt 7):317–324. cited By 16.

# Appendices

Appendix 1: Project Abstract (not numbered)

Appendix 2: Project Task Description

Appendix 3: Lab Images

Appendix 4: Data

**MASTER'S THESIS, COURSE CODE FMH606**

Students: Nirmal Ghimire

Thesis title: Investigation of bioprocess for industrial wastewater treatment

Signatures: .....

Number of pages: 117

Keywords: Oil wastewater, HyVAB<sup>®</sup>, CFIC<sup>®</sup>, Anaerobic and Aerobic Digestion

Supervisor: Rune Bakke sign.: .....

2<sup>nd</sup> Supervisor: Wang Shuai sign.: .....

Censor: sign.: .....

External partner: Gang Xin (Biowater Technology AS) sign.: .....

Availability: Open

Archive approval (supervisor signature) sign.: ..... Date : .....

Abstract:

A pilot scale Hybrid Vertical Anaerobic Biofilm (HyVAB<sup>®</sup>) reactor was applied to the treatment of waste oil refinery wastewater. The reactor comprised a bottom anaerobic digestion stage operated as an Up-flow Anaerobic Sludge Blanket (UASB) and a moving bed biofilm stage (during washing mode) on top operated as a Continuous Flow Intermittent Cleaning (CFIC<sup>®</sup>). The reactor was operated continuously for 90 days, with Organic Loading Rate (OLR) increasing from the lowest 3 kg COD/m<sup>3</sup>.d to the highest 33.1 kg COD/m<sup>3</sup>.d. Oil wastewater was heated to 35°C before pumping through the bottom of the reactor. Average Chemical Oxygen Demand (COD) concentration of the feed was 10 g/L.

The results showed that the HyVAB<sup>®</sup> reactor had good performance in terms of COD removal and biogas generation. Highest sCOD removal efficiency of 98.5% and tCOD removal efficiency of 95.4% was achieved at 12 hours Hydraulic Retention Time (HRT) in AD, and highest OLR of 18.7 kg COD/m<sup>3</sup>.d. Most of the COD removal took place in AD stage. Around 86% COD in oil wastewater was transformed into biogas in which methane content was 80-90%. Biogas formed was collected at anaerobic and aerobic interface via a three phase separator. Volatile Fatty Acid (VFA) accumulation was observed during the initial and later operation stage affecting COD removal, biogas production and total suspended solids in effluent. Suspended solid production measured in the effluent was approximately 0.16 kg VSS/kg COD removed. Meanwhile, sludge loading rate (SLR) increased from 0.1 kg COD/kg VS.d at the initial phase to 0.55 kg COD/kg VS.d at 71<sup>st</sup> day. Also, specific methanogenic activity (SMA) increased from 0.074 kg COD-CH<sub>4</sub>/kg VS.d to 0.428 kg COD-CH<sub>4</sub>/kg VS.d at 71<sup>st</sup> day.

On the whole, the compact HyVAB<sup>®</sup> exhibited good stability in terms of acidity and alkalinity. Replacing CFIC<sup>®</sup> with HyVAB<sup>®</sup> can save significant amount of money on aeration up to \$ 5.35 per day per m<sup>3</sup> of treated wastewater. HyVAB<sup>®</sup> reactor can be an attractive alternative while treating high strength wastewater at relatively lower operational cost.

University College of Southeast Norway accepts no responsibility for results and conclusions presented in this report.



# Appendix 2: Project Task Description



Telemark University College  
Faculty of Technology

## FMH606 Master's Thesis

**Title:** Investigation of bioprocess for industrial wastewater treatment

**TUC supervisors:** Rune Bakke and Wang Shuai

**External partner:** Gang Xin, Biowater Technology AS, Tønsberg:  
<http://www.biowatertechnology.com/>

**Task description:** The master thesis project will include experimental planning, bioreactor operation, sampling and analysis of samples. Analysis of laboratory and pilot scale reactors will be applied. The experimental results will be used to establish mass and energy balances for the bioreactor(s) and evaluation of process performance.

**Task background:** A newly developed HyVAB<sup>®</sup> (Hybrid Vertical Anaerobic Biofilm) process has been tested on a high strength (~10,000 mgCOD/L) industrial wastewater in laboratory pilot scale since March 2013. The reactor comprises a bottom anaerobic digestion stage, operated as an UASB (up-flow anaerobic sludge blanket), and a fixed biofilm stage on top, which is operated as a CFIC<sup>®</sup> (continuous flow intermittent cleaning). High overall COD removal efficiencies (90-97%) are obtained, wherein 50-70% of the COD is removed in the anaerobic digestion stage. Low sludge yields are also observed. Cost analysis shows that the HyVAB<sup>®</sup> reactor can achieve 30% saving (OPEX) compared to aerobic treatment. An ongoing research project to further document and optimize the HyVAB<sup>®</sup> with TUC as the main research partner is the framework for this thesis project.

**Student category:**


EET or PT student.

**Practical arrangements:**

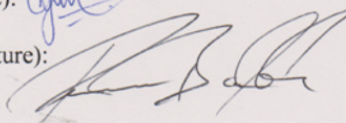
Work will be carried out at TUC and at an industrial site.

**Signatures:**

Student (date and signature):

 29.01.2016

Supervisor (date and signature):



Address: Kjølnes ring 56, NO-3918 Porsgrunn, Norway. Phone: 35 57 50 00. Fax: 35 55 75 47.

## Appendix 3: Lab images



Figure 1: Insulated reactor at NSO along with feed and recycle pump



Figure 2: Feed and recycle pump at NSO, Bamble



Figure 3: Equalisation tank along with buffer solution pumping setup



Figure 4: Dissolved Oxygen (DO) meter



Figure 5: pH meter



Figure 6: Spectroquant



Figure 7: CODDigester

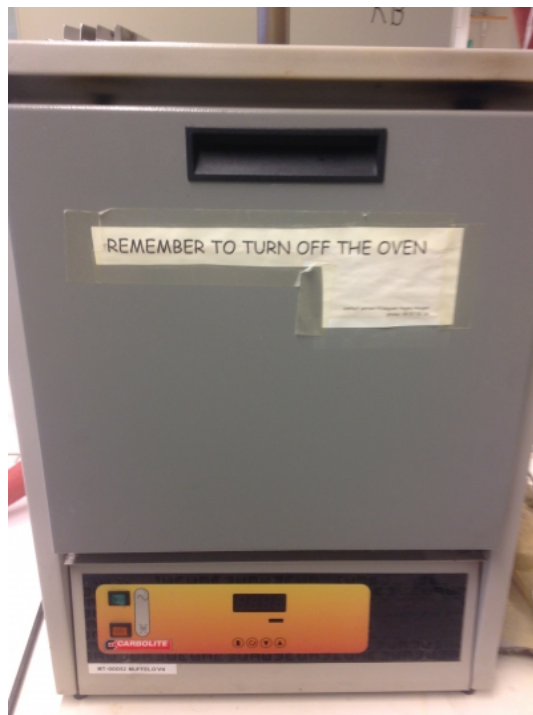


Figure 8: Blast Furnace



Figure 9: Dessicator



Figure 10: Oven

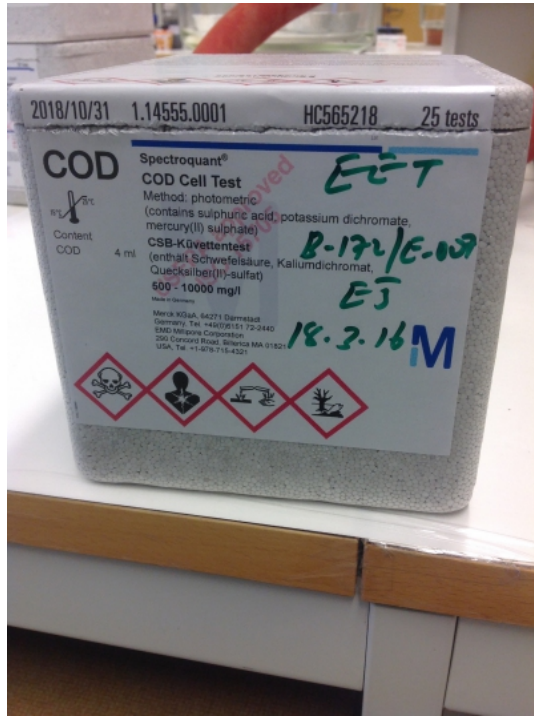


Figure 11: CODKit



Figure 12: Filtration unit

# Appendix 4: Data

Date	DAY	feed rate		Temp	pH	Total COD	COD Removed	Filt. VFAs	MFA/COD
		L/d	oC						
2016.02.18	0	18,5	16,9	8,29	9,39	3,27	1519,5	0,16	
2016.02.19	1	18,5	28,5	8,29	8,48	2,93			
2016.02.20	2	18,5							
2016.02.21	3	18,5							
2016.02.22	4	18,5	18	6,9	10,22	3,70	1695,1	0,17	
2016.02.23	5	18,5	29,2	8,13	7,96	2,67	1274,2	0,16	
2016.02.24	6	18,5	28,7	8,71	9,33	3,37			
2016.02.25	7	18,5	31,9	8,38					
2016.02.26	8	18,5	31,6	8,32					
2016.02.27	9	18,5		8,5					
2016.02.28	10	18,5							
2016.02.29	11	18,5	31,6	8,5	7,065	2,70			
2016.03.01	12	31,6	31,3	8,52					
2016.03.02	13	18,5							
2016.03.03	14	18,5							
2016.03.04	15	18,5	34,7	5,62	7,51	2,98	2128,7	0,28	
2016.03.05	16	18,5							
2016.03.06	17	18,5							
2016.03.07	18	31,6	34	6,76					
2016.03.08	19	31,6	29,2	7,98					
2016.03.09	20	31,6	34,7	8,58	7,52	5,08	2333,6	0,31	
2016.03.10	21	31,6	34,6	8,65					
2016.03.11	22	31,6	34,1	8,52	7,75	4,98	2321,6	0,30	
2016.03.12	23	47,52							
2016.03.13	24	47,52							
2016.03.14	25	47,52	34,2	8,24	7,745	7,84	1685,9	0,22	
2016.03.15	26	47,52							
2016.03.16	27	47,52	34,4	8,65	10	10,00	1382,8	0,14	
2016.03.17	28	47,52							
2016.03.18	29	47,52	32,6	7,73	9,255	9,35	1439,4	0,16	
2016.03.19	30	47,52							
2016.03.20	31	47,52							
2016.03.21	32	47,52	34,8	6,03	10,07	10,66	2582,4	0,26	
2016.03.22	33	47,52							
2016.03.23	34	47,52	35	7,49	12,25	12,67	2554,3	0,21	
2016.03.24	35	47,52							
2016.03.25	36	47,52							
2016.03.26	37	47,52							
2016.03.27	38	47,52							
2016.03.28	39	47,52							
2016.03.29	40	47,52	34,9	7,3	13,36	14,21	2359,2	0,18	
2016.03.30	41	57,6							
2016.03.31	42	57,6	34,9	7,28	12,32	15,36	2337,3	0,19	
2016.04.01	43	57,6							
2016.04.02	44	57,6							

Figure 13: Data for Feed of Pilot Reactor(1)



2016.04.03	45	57,6						
2016.04.04	46	57,6	35	6,89	10,19	12,76		
2016.04.05	47	57,6						
2016.04.06	48	57,6	34,7	6,96	6,25	7,92	1466,2	0,23
2016.04.07	49	70,6						
2016.04.08	50	70,6	33,9	6,56	10,47	16,29	1691,4	0,16
2016.04.09	51	70,6						
2016.04.10	52	70,6						
2016.04.11	53	70,6	33,3	6,42	8,53	13,37	2085,3	0,24
2016.04.12	54	70,6						
2016.04.13	55	70,6	35,6	6,26	8,95	14,04	3434,9	0,38
2016.04.14	56	70,6						
2016.04.15	57	70,6	35,1	6,28	9,55	14,91		
2016.04.16	58	70,6						
2016.04.17	59	70,6						
2016.04.18	60	70,6	34,8	6,41	9,54	14,99		
2016.04.19	61	79,2						
2016.04.20	62	79,2	33,2	6,49	8,87	15,61	3250,2	0,37
2016.04.21	63	79,2						
2016.04.22	64	83,5	34,8	5,6				
2016.04.23	65	83,5						
2016.04.24	66	83,5						
2016.04.25	67	83,5	32,8	5,63	11,41	21,27	302,4	0,03
2016.04.26	68	83,5						
2016.04.27	69	83,5	29,8	7,14	11,64	20,70	3043,5	0,26
2016.04.28	70	83,5						
2016.04.29	71	83,5	31,7	7,3	14,01	24,42	3044,8	0,22
2016.04.30	72	83,5						
2016.05.01	73	83,5						
2016.05.02	74	83,5	32,1	7,21	10,39	17,76	2363,1	0,23
2016.05.03	75	90,7						
2016.05.04	76	90,7	34,1	7,27	7,6	15,12	1200,6	0,16
2016.05.05	77	90,7						
2016.05.06	78	90,7	29,6	7,42	7,6	15,12	1386,0	0,18
2016.05.07	79	90,7						
2016.05.08	80	90,7						
2016.05.09	81	90,7	34,1	6,65	9,06	18,00	2828,9	0,31
2016.05.10	82	90,7						
2016.05.11	83	90,7	31,9	6,97	9,67	18,35	3326,6	0,34
2016.05.12	84	90,7						
2016.05.13	85	90,7	30,8	5,97	10,21	19,41	3179,8	0,31

Figure 14: Data for Feed of Pilot Reactor(2)

Date	Day	AD1					AD2					AD3					Recycle				
		pH	Temp.	Filt.	TCOD	Filt.	pH	Temp.	Filt.	TCOD	Filt.	pH	Temp.	Filt.	TCOD	Filt.	pH	Temp.	Filt.	TCOD	Filt.
		C	mgCOD/L	g/L	g/L	g/L	C	mgCOD/L	g/L	g/L	C	mgCOD/L	g/L	g/L	C	mgCOD/L	g/L	C	mgCOD/L	g/L	g/L
2016.02.18	0	7,44	13,9	979,81	6,225	4,915	7,39	17,4	164,94	3,795	2,335	7,32	18	212,55	3,745	2,46	7,35	17,5	208,01	3,365	2,365
2016.02.19	1	7,36	13,2	769,05	5,945	4,88	7,22	17,7	92,99	2,8	1,96	7,17	17,7	122,91	3,09	1,955	7,08	17,8	124,59	3,445	1,845
2016.02.20	2																				
2016.02.21	3																				
2016.02.22	4	7,19	16,9	77,87	2,03	1,7	7,25	19	85,81	2,2	1,77	7,17	19	73,94	2,17	1,95	7,14	18,5	89,4	2,38	1,82
2016.02.23	5	7,52	13,3	1025,32	5,875	5,335	7,2	17,7		2,515	1,99	7,14	19,6		2,71	1,985	7,13	18,8	107,48	2,645	2,065
2016.02.24	6	7,51	19,4		6,24	5,865	7,38	21,3		2,16	1,69	7,12	22,1		2,63	1,86	7,07	21,9		2,475	1,81
2016.02.25	7	7,3	17,8																		
2016.02.26	8	7,1	18,5				7,44	20,4				7,15	20,1				7,15	21,1			
2016.02.27	9																				
2016.02.28	10																				
2016.02.29	11	7,33	19,1		5,195	2,465	7,29	21,1		1,2	0,475	7,33	21		1,58	0,845	7,37	21,5		1,66	0,925
2016.03.01	12	7,33	18,1														7,27	22,1			
2016.03.02	13																				
2016.03.03	14																				
2016.03.04	15						7,3	19,3		2,2	1,81	7,32	20,2	61,0	3,67	1,015					
2016.03.05	16																				
2016.03.06	17																				
2016.03.07	18	7,12	19,4				7,05	20,6				7,14	20,2				7,21	20,1			
2016.03.08	19																7,23	21,1			
2016.03.09	20								461,01			7,15	22,4	180,7	6,875	1,25	7,09	22,6	324,27	6,39	1,175
2016.03.10	21																7,25	17,6			
2016.03.11	22	7,85	14,7				7,35	18				7,3	18,1	66,4	7,5	1,065	7,33	19	67,9	6,76	0,97
2016.03.12	23																				
2016.03.13	24																				
2016.03.14	25											7,2	19,5	60,3	10,24	0,81	7,08	19,7	52,1	8,56	0,865
2016.03.15	26																				
2016.03.16	27	7,07	18,5									7,01	19,9	73,8	9,53	1,125	6,93	20,2	61,9	8,62	1,095
2016.03.17	28																				
2016.03.18	29											7,68	20,5	29,3	8,47	0,95	6,91	21	33,4	8,66	0,91
2016.03.19	30																				
2016.03.20	31																				
2016.03.21	32											6,94	21,1	77,9	6,59	0,56	6,8	21,8	66,9	6,45	0,56
2016.03.22	33																				
2016.03.23	34	6,75	21,8				7,08	22,3				7,03	22,6	41,1	8,2	0,79	6,98	22,6	46,0	7,66	0,775
2016.03.24	35																				
2016.03.25	36																				
2016.03.26	37																				
2016.03.27	38																				
2016.03.28	39																				
2016.03.29	40											7,06	19,3	18,1	6,04	0,315	6,97	20	27,6	6,15	0,255
2016.03.30	41																				
2016.03.31	42													57,5	5,1	0,695			4,94		0,73
2016.04.01	43																				
2016.04.02	44																				

Figure 15: Data for AD of Pilot Reactor(1)



Date	Day											Biomass	Biomass Yield
		DO	pH	Temp.	Total COD	Filt. COD	Filt. VFAs	Filt. VFA/Scod	Filt. TN	Filt. TPO4-P	Carrier biomass	Biomass	Biomass Yield
		mg/L		C	g/L	g/L	mg/L	%	mg/L	mg/L	g TS/carrie	g TS/L	g VSS/g COD used
2016.02.18	0		8,87	16,3	2,08	1,61	66,89	4,2					0,02
2016.02.19	1	7,17	8,61	18	1,89	1,52	75,04	4,9					0,02
2016.02.20	2												
2016.02.21	3												
2016.02.22	4	8,05	8,86	19,9	1,99	1,41	35,43	2,5					0,01
2016.02.23	5	6,32	8,6	13	1,86	1,62							
2016.02.24	6	4,98	8,53	16,9	1,48	1,31	0	0,0					0,01
2016.02.25	7	5,07	8,5	21,5									
2016.02.26	8	5,25	8,5	21,1					26,6	0,35			
2016.02.27	9												
2016.02.28	10												
2016.02.29	11	4,95	8,54	22,5	0,77	0,645	0	0,0					0,02
2016.03.01	12	3,83	8,42	22,4									
2016.03.02	13												
2016.03.03	14												
2016.03.04	15		8,46	20,9	0,62	0,42	8,82	2,1					0,04
2016.03.05	16												
2016.03.06	17												
2016.03.07	18												
2016.03.08	19	6,2	8,53	21									
2016.03.09	20	4,76	8,36	21,9	0,68	0,448	0	0,0					0,02
2016.03.10	21	4,5	8,45	20,5									
2016.03.11	22	5,25	8,44	21,2	0,86	0,822	5,55	0,7					0,03
2016.03.12	23												
2016.03.13	24												
2016.03.14	25	3,33	8,27	21,3	1,15	0,49	24,19	4,9					0,05
2016.03.15	26												
2016.03.16	27	3,23	8,19	21,3	1,53	0,74	17,65	2,4					0,05
2016.03.17	28						5,96						
2016.03.18	29	3,22	8,12	22	0,965	0,595	6,92	1,2					0,04
2016.03.19	30												
2016.03.20	31												
2016.03.21	32	4,03	8,1	22,2	0,66	0,2	10,32	5,2					0,04
2016.03.22	33												
2016.03.23	34	1,67	8,03	23,2	1,41	0,52	32,85	6,3					0,03
2016.03.24	35												
2016.03.25	36												
2016.03.26	37												
2016.03.27	38												
2016.03.28	39												
2016.03.29	40	3,2	8,21	20,9	0,67	0,2	0,00	0,0					0,03
2016.03.30	41												
2016.03.31	42	2	8,02	22,4	1,54	0,585	37,08	6,3					0,05
2016.04.01	43												

Figure 17: Data for CFIC of Pilot Reactor(1)

2016.04.02	44								
2016.04.03	45								
2016.04.04	46	2,15	8,16	22,9	2,805	0,44	9,67	2,2	0,17
2016.04.05	47								
2016.04.06	48	3,66	8,21	21,5	0,975	0,2	0,00	0,0	0,07
2016.04.07	49								
2016.04.08	50	2,51	8,1	22	1,2	0,32	6,60	2,1	0,03
2016.04.09	51								
2016.04.10	52								
2016.04.11	53	2,99	8,16	23,3	0,84	0,2	5,99	3,0	0,04
2016.04.12	54								
2016.04.13	55	4,2	8,21	20,6	0,645	0,2	5,54	2,8	0,04
2016.04.14	56								
2016.04.15	57	3,35	8,17	19,3	0,925	0,255	4,43	1,7	0,03
2016.04.16	58								
2016.04.17	59								
2016.04.18	60	4,65	8,3	22	0,57	0,2	4,12	2,1	0,03
2016.04.19	61								
2016.04.20	62	5,15	8,24	23,1	0,41	0,2	5,92	3,0	0,02
2016.04.21	63								
2016.04.22	64		8,68	23,3					
2016.04.23	65								
2016.04.24	66								
2016.04.25	67	1,95	7,82	21,2	1,1015	0,2	22,94	11,5	0,05
2016.04.26	68								
2016.04.27	69	1,09	8,24	23,1	1,625	0,73	32,09	4,4	0,04
2016.04.28	70								
2016.04.29	71	0,3	8,01	23,5	2,975	1,14	37,21	3,3	0,05
2016.04.30	72								
2016.05.01	73								
2016.05.02	74		8,14	24,5	1,93	1,03	32,43	3,1	0,05
2016.05.03	75								
2016.05.04	76	4,6	8,06	24,1	0,95	0,265	15,10	5,7	0,05
2016.05.05	77								
2016.05.06	78	5,8	8,3	23,8	0,93	0,265	69,05	26,1	0,08
2016.05.07	79								
2016.05.08	80								
2016.05.09	81	5,3	8,28	26,5	0,89	0,33	0,00	0,0	0,04
2016.05.10	82								
2016.05.11	83	6,03	8,41	26,3	2,32	0,77	4,90	0,6	0,07
2016.05.12	84								
2016.05.13	85	5,7	8,32	25,5	3,97	0,795			0,26

Figure 18: Data for CFIC of Pilot Reactor(2)

date	day	feed					AD1				AD2											
		volume (ml) Blank (g)	105 C (g)	550 C (g)	TSS (mg/L) VSS (mg/L) vss/tss		volume (ml) Blank (g)	105 C (g)	550 C (g)	TSS (mg/L) VSS (mg/L) vss/tss		volume (ml) Blank (g)	105 C (g)	550 C (g)	TSS (mg/L) VSS (mg/L) vss/tss							
2016.02.18	0	20	0,151	0,155	0,1504	200	230	115														
2016.02.19	1								10	0,1539	0,1631	0,1564	920,00	670,00	72,8	10	0,1559	0,1658	0,1584	990,00	740,00	74,7
2016.02.20	2								10	0,1589	0,1649	0,1595	600,00	540,00	90,0	10	0,1625	0,1671	0,1628	460,00	430,00	93,5
2016.02.21	3																					
2016.02.22	4								10	0,1515	0,1546	0,1517	310,00	290,00	93,5	10	0,157	0,1617	0,1574	470,00	430,00	91,5
2016.02.23	5								10	0,1551	0,1598	0,1555	470,00	430,00	91,5	10	0,1504	0,1579	0,1517	750,00	620,00	82,7
2016.02.24	6								10	0,1574	0,1594	0,1569	200,00	250,00	125,0	10	0,1567	0,1596	0,157	280,00	260,00	89,7
2016.02.25	7																					
2016.02.26	8								10	0,1559	0,1632	0,1564	730,00	680,00	93,2	10	0,158	0,1599	0,1576	190,00	230,00	121,1
2016.02.27	9																					
2016.02.28	10																					
2016.02.29	11								10	0,1535	0,1735	0,1576	2000,00	1900,00	79,5	10	0,1574	0,1628	0,1578	540,00	500,00	92,6
2016.03.01	12																					
2016.03.02	13																					
2016.03.03	14																					
2016.03.04	15															10	0,1596	0,166	0,1616	640,00	440,00	68,8

Figure 19: Data for TSS and VSS of Pilot Reactor(1)

AD3						Recycle						CFC								
volume (ml)	Blank (g)	105 C (g)	550 C (g)	TSS (mg/L)	VSS (mg/L)	vss/tss	volume (ml)	Blank (g)	105 C (g)	550 C (g)	TSS (mg/L)	VSS (mg/L)	vss/tss	volume (ml)	Blank (g)	105 C (g)	550 C (g)	TSS (mg/L)	VSS (mg/L)	vss/tss
10	0.1546	0.1645	0.1563	990.00	820.00	82.8	10	0.1561	0.1692	0.1627	820.00	650.00	79.3	20	0.1544	0.1562	0.1534	90.00	140.00	155.6
10	0.1541	0.1618	0.1554	770.00	640.00	83.1	10	0.1571	0.1655	0.1585	840.00	700.00	83.3	20	0.1556	0.1582	0.1553	130.00	145.00	111.5
10	0.1563	0.1606	0.1567	430.00	390.00	90.7	10	0.1555	0.158	0.1553	250.00	270.00	108.0	20	0.1542	0.156	0.1538	90.00	110.00	122.2
10	0.1583	0.1663	0.1606	800.00	570.00	71.3	10	0.1571	0.1653	0.159	820.00	630.00	76.8	20	0.1583	0.1626	0.158	215.00	230.00	107.0
10	0.1519	0.1606	0.1535	870.00	710.00	81.6	10	0.1538	0.1614	0.1554	780.00	600.00	78.9	20	0.1488	0.1506	0.1486	90.00	100.00	111.1
10	0.1565	0.1631	0.1577	660.00	540.00	81.8	10	0.1575	0.1637	0.1584	820.00	530.00	85.5	20	0.155	0.1567	0.1548	85.00	95.00	111.8
10	0.1554	0.1609	0.1558	550.00	510.00	92.7	10	0.1568	0.1619	0.1572	510.00	470.00	92.7	20	0.1585	0.16	0.158	75.00	100.00	133.3
10	0.1568	0.164	0.1598	720.00	420.00	58.3								20	0.1575	0.165	0.1598	375.00	260.00	69.3
5	0.1589	0.1788	0.163	3 980.00	3 160.00	79.4	5	0.1584	0.1679	0.1592	1 900.00	1 740.00	91.6	20	0.1575	0.1597	0.1563	110.00	170.00	154.5
5	0.1543	0.1818	0.162	5 500.00	3 960.00	72.0	5	0.1601	0.1826	0.165	4 500.00	3 520.00	78.2	20	0.1528	0.1576	0.1532	240.00	220.00	91.7
5	0.1553	0.1966	0.1638	8 260.00	6 560.00	79.4	5	0.1613	0.1801	0.1659	3 760.00	2 840.00	75.5	20	0.157	0.1641	0.157	355.00	355.00	100.0
5	0.1576	0.1913	0.1653	6 740.00	5 200.00	77.2	5	0.1577	0.188	0.165	6 060.00	4 600.00	75.9	20	0.155	0.1645	0.1557	475.00	440.00	92.6
3	0.1493	0.1696	0.1534	6 766.67	5 400.00	79.8	5	0.155	0.1873	0.1624	6 460.00	4 980.00	77.1	20	0.156	0.162	0.156	300.00	300.00	100.0
3	0.1504	0.1619	0.1526	3 833.33	3 100.00	80.9	3	0.1574	0.1755	0.1613	6 033.33	4 733.33	78.5	12	0.1559	0.1602	0.1561	358.33	341.67	95.3
3	0.1565	0.1796	0.1604	7 700.00	6 400.00	83.1	3	0.1523	0.164	0.1543	3 900.00	3 233.33	82.9	12	0.1534	0.1581	0.1537	291.67	266.67	91.6
3	0.1532	0.1638	0.1541	3 533.33	3 233.33	91.5	3	0.1557	0.1694	0.1581	4 566.67	3 766.67	82.5	12	0.1544	0.1585	0.1541	341.67	366.67	107.3
3	0.1584	0.1687	0.1591	3 433.33	3 200.00	93.2	3	0.155	0.1654	0.1564	3 466.67	3 000.00	86.5	12	0.1548	0.1611	0.155	525.00	508.33	96.8
3	0.1554	0.1658	0.1572	3 466.67	2 866.67	82.7	3	0.1535	0.1674	0.155	4 633.33	4 133.33	89.2	6	0.1539	0.1612	0.1537	1 216.67	1 250.00	102.7
3	0.1565	0.164	0.1566	2 500.00	2 466.67	98.7	3	0.1566	0.1651	0.1575	2 833.33	2 533.33	89.4	9	0.1485	0.1516	0.1485	344.44	344.44	100.0
5	0.1507	0.163	0.1518	2 460.00	2 240.00	91.1	3	0.1544	0.1612	0.1551	2 266.67	2 033.33	89.7	12	0.1554	0.1593	0.1562	325.00	258.33	79.5
5	0.1478	0.1551	0.1488	1 460.00	1 280.00	86.3	3	0.1467	0.1522	0.1468	1 833.33	1 800.00	98.2	12	0.1497	0.1532	0.1493	291.67	325.00	111.4
5	0.1529	0.1599	0.1531	1 400.00	1 360.00	97.1	5	0.15	0.1562	0.1501	1 240.00	1 220.00	98.4	20	0.1451	0.1528	0.1464	385.00	320.00	83.1
10	0.1536	0.1604	0.154	880.00	640.00	94.1	5	0.1567	0.1614	0.1568	940.00	920.00	97.9	20	0.1525	0.1584	0.1528	295.00	280.00	94.9
10	0.1179	0.1335	0.1191	1 560.00	1 440.00	92.3	10	0.1581	0.1635	0.1585	540.00	500.00	92.6	20	0.1536	0.1584	0.1538	240.00	230.00	95.8
5	0.1176	0.1335	0.1195	3 180.00	2 800.00	88.1	10	0.1526	0.1581	0.1532	550.00	490.00	89.1	20	0.1572	0.1612	0.1573	200.00	195.00	97.5
10	0.1179	0.1292	0.1197	1 130.00	950.00	84.1	10	0.1203	0.1274	0.1208	710.00	660.00	93.0	15	0.1189	0.1269	0.1199	533.33	466.67	87.5
10	0.1132	0.1266	0.1153	1 340.00	1 130.00	84.3	10	0.1189	0.1265	0.1197	760.00	680.00	89.5	15	0.1186	0.1253	0.1193	446.67	400.00	89.6
10	0.1197	0.1331	0.1211	1 340.00	1 200.00	89.6	10	0.1201	0.1277	0.1214	760.00	630.00	82.9	10	0.1176	0.1243	0.1188	670.00	550.00	82.1
5	0.1167	0.1249	0.1178	1 640.00	1 420.00	86.6	10	0.1131	0.1208	0.1143	770.00	650.00	84.4	15	0.1142	0.1216	0.1154	493.33	413.33	83.8
10	0.1162	0.126	0.1174	980.00	860.00	87.8	15	0.1195	0.1282	0.1202	580.00	533.33	92.0	20	0.1188	0.1266	0.1194	390.00	360.00	92.3
5	0.1142	0.1583	0.123	8 820.00	7 060.00	80.0	10	0.1185	0.1263	0.1188	780.00	750.00	96.2	15	0.117	0.1258	0.1177	586.67	540.00	92.0
2	0.1121	0.1413	0.1187	14 600.00	11 300.00	77.4	15	0.1153	0.1216	0.116	420.00	373.33	88.9	20	0.1147	0.1212	0.1154	325.00	290.00	89.2
							3	0.1149	0.1647	0.1247	16 600.00	13 333.33	80.3	10	0.1134	0.1215	0.1163	810.00	520.00	64.2
														4	0.1163	0.1245	0.1181	2 050.00	1 600.00	78.0

Figure 20: Data for TSS and VSS of Pilot Reactor(2)

date	day	sample	volume (ml)	Blank(g)	105 C (g)	550 C (g)	TS(g/L)	VS(g/L)	height (m)	total TS Kg	total VS Kg SMA	SA(kg COD-CH4/kg VSS.day)	Sludge Loading	Weighted average T	
2016.02.25	7	granule 1	65	52,4	60,5	56,3	125,5	64,6	1	2,22	1,14	0,056	0,067	0,103	38,04
2016.03.07	18	AD1 granule	54	49,253	56,2142	52,2786	128,9	72,9	1,4	3,19	1,80	0,057	0,034	0,131	41,15
		AD2 granule	38	41,5271	44,4885	42,3395	77,9	56,6							
		AD3 granule	51	48,5264	48,9405	48,6885	8,1	4,9							
		Recycle	61	45,2444	45,4759	45,3696	3,8	1,7							
2016.03.11	22	AD1 granule	50	50,93	58	54,51	141,4	69,8	1,45	3,62	1,79	0,053	0,019	0,141	39,56
		AD2 granule	43	51,01	53,957	51,59	68,5	55,0	1,5	1,82	1,46				
2016.04.04	46	AD1 granule	40	51,2528	54,97	52,2242	92,9	68,6	1,5	2,46	1,82	0,169	0,086	0,39	34,44
		AD2 granule	40	45,242	47,17	45,9	48,2	31,8							
2016.04.29	71	AD1 granule	48	45,241	51,1468	46,995	123,0	86,5	1,5	3,26	2,29	0,162	0,164	0,55	48,34
		AD2 granule	63	45,245	50,8144	46,6883	88,4	65,5							
		AD3 granule	30	50,9156	51,0673	51,02	5,1	1,6							

Figure 21: Data for TS and VS of Pilot Reactor



Date	Day	CH4	CO2	flowrate mL/nflowrate L/d	CH4 g COD/d	CH4 yield l/g COD removed	
2016.02.18	0	0,88	0,12	33,00	47,52	100,48	0,33
2016.02.19	1	0,85	0,15	41,20	59,33	126,89	0,42
2016.02.20	2						
2016.02.21	3						
2016.02.22	4	0,86	0,14	16,88	24,30	51,44	0,14
2016.02.23	5	0,84	0,16	33,30	47,95	105,96	0,36
2016.02.24	6	0,82	0,18	61,40	88,42	180,36	0,52
2016.02.25	7	0,84	0,16	65,14	93,81	196,57	
2016.02.26	8	0,84	0,16	47,75	68,76	143,06	
2016.02.27	9						
2016.02.28	10						
2016.02.29	11						
2016.03.01	12	0,85	0,15	90,00	129,60	291,38	
2016.03.02	13						
2016.03.03	14						
2016.03.04	15						
2016.03.05	16						
2016.03.06	17						
2016.03.07	18						
2016.03.08	19						
2016.03.09	20	0,86	0,14	71,43	102,86	221,35	0,45
2016.03.10	21	0,88	0,12	60,00	86,40	199,97	
2016.03.11	22	0,85	0,15	64,20	92,45	196,99	0,37
2016.03.12	23						
2016.03.13	24						
2016.03.14	25	0,85	0,15	193,33	278,40	590,62	0,72

Figure 22: Data for Biogas Production of Pilot Reactor(1)

2016.03.15	26						
2016.03.16	27	0,86	0,14	120,00	172,80	393,55	0,35
2016.03.17	28						
2016.03.18	29	0,84	0,16	204,00	293,76	624,30	0,63
2016.03.19	30						
2016.03.20	31						
2016.03.21	32	0,81	0,19	130,00	187,20	368,35	0,33
2016.03.22	33						
2016.03.23	34						
2016.03.24	35						
2016.03.25	36						
2016.03.26	37						
2016.03.27	38						
2016.03.28	39						
2016.03.29	40	0,83	0,17	116,00	167,04	346,78	0,22
2016.03.30	41						
2016.03.31	42	0,81	0,19	176,67	254,40	511,52	0,31
2016.04.01	43						
2016.04.02	44						
2016.04.03	45						
2016.04.04	46	0,83	0,17	178,00	256,32	521,27	0,38
2016.04.05	47						
2016.04.06	48	0,86	0,14	193,33	278,40	588,10	0,70
2016.04.07	49						
2016.04.08	50	0,83	0,17	103,33	148,80	308,25	0,18
2016.04.09	51						
2016.04.10	52						
2016.04.11	53	0,83	0,17	327,50	471,60	974,54	0,67
2016.04.12	54						
2016.04.13	55	0,83	0,16	220,00	316,80	651,25	0,43

Figure 23: Data for Biogas Production of Pilot Reactor(2)

2016.04.14	56						
2016.04.15	57	0,83	0,17	217,50	313,20	639,95	0,41
2016.04.16	58						
2016.04.17	59						
2016.04.18	60	0,81	0,19	153,00	220,32	433,90	0,28
2016.04.19	61						
2016.04.20	62	0,85	0,15	216,50	311,76	660,24	0,39
2016.04.21	63						
2016.04.22	64						
2016.04.23	65						
2016.04.24	66						
2016.04.25	67	0,86	0,14	221,00	318,24	721,18	0,31
2016.04.26	68						
2016.04.27	69	0,81	0,19	257,00	370,08	739,58	0,33
2016.04.28	70						
2016.04.29	71	0,83	0,17	240,00	345,60	707,72	0,27
2016.04.30	72						
2016.05.01	73						
2016.05.02	74	0,82	0,18	280,00	403,20	810,03	0,44
2016.05.03	75						
2016.05.04	76	0,86	0,14	230,00	331,20	681,31	0,43
2016.05.05	77						
2016.05.06	78	0,80	0,20	280,00	403,20	797,97	0,52
2016.05.07	79						
2016.05.08	80						
2016.05.09	81	0,82	0,18	270,00	388,80	787,86	0,41
2016.05.10	82						
2016.05.11	83	0,79	0,21	153,33	220,80	436,69	0,23
2016.05.12	84						
2016.05.13	85	0,82	0,18	332,50	478,80	966,53	0,48

Figure 24: Data for Biogas Production of Pilot Reactor(3)

Date	Day	HRT h	OLR kg COD/m <sup>3</sup> d	AD		CFIC		AD + CFIC		COD feed		CH4 COD/Feed COD
				SCOD removal (%)	TCOD removal (%)	sCOD removal (%)	tCOD removal (%)	SCOD removal (%)	TCOD removal (%)	g/d	%	
18/02/16	0	57	3,9	73,8	60,1	9,1	17,8	82,9	77,9	173,7	0,58	
19/02/16	1	57	3,6	76,9	63,6	5,1	14,2	82,1	77,8	156,9	0,81	
20/02/16	2	57										
21/02/16	3	57										
22/02/16	4	57	4,3	80,9	78,8	5,3	1,8	86,2	80,6	189,1	0,27	
23/02/16	5	57	3,3	75,1	66,0	4,6	10,7	79,6	76,6	147,3	0,72	
24/02/16	6	57	3,9	80,1	71,8	5,9	12,4	86,0	84,2	172,6	1,04	
25/02/16	7	57										
26/02/16	8	57										
27/02/16	9	57										
28/02/16	10	57										
29/02/16	11	57	3,0	88,0	77,6	2,8	11,5	90,9	89,1	130,7		
01/03/16	12	33										
02/03/16	13	57										
03/03/16	14	57										
04/03/16	15	57	3,2	86,5	51,1	7,9	40,6	94,4	91,7	138,9		
05/03/16	16	57										
06/03/16	17	57										
07/03/16	18	33										
08/03/16	19	33										
09/03/16	20	33	5,4	83,4	8,6	10,7	82,4	94,0	91,0	237,6	0,93	
10/03/16	21	33										
11/03/16	22	33	5,6	86,3	3,2	3,1	85,7	89,4	88,9	244,9	0,80	
12/03/16	23	22										
13/03/16	24	22										
14/03/16	25	22	8,4	89,5	-32,2	4,1	117,4	93,7	85,2	368,0	1,60	
15/03/16	26	22										
16/03/16	27	22	10,8	88,8	4,7	3,9	80,0	92,6	84,7	475,2	0,83	
17/03/16	28	22										
18/03/16	29	22	10,0	89,7	8,5	3,8	81,1	93,6	89,6	439,8	1,42	
19/03/16	30	22										
20/03/16	31	22										
21/03/16	32	22	10,9	94,4	34,6	3,6	58,9	98,0	93,4	478,5	0,77	
22/03/16	33	22										
23/03/16	34	22	13,2	93,6	33,1	2,2	55,4	95,8	88,5	582,1		
24/03/16	35	22										
25/03/16	36	22										
26/03/16	37	22										
27/03/16	38	22										
28/03/16	39	22										
29/03/16	40	22	14,4	97,6	54,8	0,9	40,2	98,5	95,0	634,9	0,55	
30/03/16	41	18										
31/03/16	42	18	16,1	94,4	58,6	0,9	28,9	95,3	87,5	709,6	0,72	
01/04/16	43	18										

Figure 25: Data for Mass Balance of Pilot Reactor(1)

02/04/16	44	18									
03/04/16	45	18									
04/04/16	46	18	13,3	93,7	54,7	2,0	17,8	95,7	72,5	586,9	0,89
05/04/16	47	18									
06/04/16	48	18	8,2	95,3	24,5	1,5	59,9	96,8	84,4	360,0	1,63
07/04/16	49	15									
08/04/16	50	15	16,8	94,1	60,9	2,9	27,7	96,9	88,5	739,2	0,42
09/04/16	51	15									
10/04/16	52	15									
11/04/16	53	15	13,7	97,7	66,2	0,0	24,0	97,7	90,2	602,2	1,62
12/04/16	54	15									
13/04/16	55	15	14,4	97,2	74,4	0,6	18,4	97,8	92,8	631,9	1,03
14/04/16	56	15									
15/04/16	57	15	15,3	95,0	78,5	2,3	11,8	97,3	90,3	674,2	0,95
16/04/16	58	15									
17/04/16	59	15									
18/04/16	60	15	15,3	94,4	82,4	3,5	11,6	97,9	94,0	673,5	0,64
19/04/16	61	13									
20/04/16	62	13	16,0	96,9	87,3	0,8	8,1	97,7	95,4	702,5	0,94
21/04/16	63	13									
22/04/16	64	13									
23/04/16	65	13									
24/04/16	66	13									
25/04/16	67	13	21,7	92,6	82,6	5,7	7,8	98,2	90,3	952,7	0,76
26/04/16	68	13									
27/04/16	69	13	22,1	91,8	80,2	1,9	5,8	93,7	86,0	971,9	0,76
28/04/16	70	13									
29/04/16	71	13	26,6	91,6	77,8	0,2	1,0	91,9	78,8	1169,8	0,60
30/04/16	72	13									
01/05/16	73	13									
02/05/16	74	13	19,7	87,6	78,0	2,5	3,4	90,1	81,4	867,6	0,93
03/05/16	75	12									
04/05/16	76	12	15,7	96,4	78,8	0,1	8,7	96,5	87,5	689,3	0,99
05/05/16	77	12									
06/05/16	78	12	15,7	89,5	68,8	7,0	19,0	96,5	87,8	689,3	1,16
07/05/16	79	12									
08/05/16	80	12									
09/05/16	81	12	18,7	94,0	80,4	2,3	9,8	96,4	90,2	821,7	0,96
10/05/16	82	12									
11/05/16	83	12	19,9	88,0	-29,1	4,0	105,1	92,0	76,0	877,1	0,50
12/05/16	84	12									
13/05/16	85	12	21,0	88,4	100,0	3,8	-38,9	92,2	61,1	926,0	1,04
14/05/16	86	12									
15/05/16	87	12									
16/05/16	88	12									
17/05/16	89	12									
18/05/16	90	12	33,1	37,8	-21,1	8,9	53,1	46,7	32,0	1458,5	0,18
19/05/16	91	17									

Figure 26: Data for Mass Balance of Pilot Reactor(2)

Date	DAY	Flow rate L/d	Temp	pH	Total COD
			oC		g/L
2015.12.28	0	7,9	19,7	6,56	3,51
2015.12.29	1	7,9			
2015.12.30	2	7,9			
2015.12.31	3	7,9	22,3	6,69	3,435
2016.01.01	4	7,9			
2016.01.02	5	7,9			
2016.01.03	6	7,9			
2016.01.04	7	7,9	23,6	7,41	3,41
2016.01.05	8	7,9			
2016.01.06	9	7,9	25	7,93	2,47
2016.01.07	10	7,9			
2016.01.08	11	7,9			
2016.01.09	12	7,9			
2016.01.10	13	7,9			
2016.01.11	14	7,9			
2016.01.12	15	7,9	21,9	7,05	3,005
2016.01.13	16	7,9			
2016.01.14	17	7,9	19,5	7,29	2,73
2016.01.15	18	7,9			
2016.01.16	19	7,9			
2016.01.17	20	7,9			
2016.01.18	21	7,9	17,3	6,65	3,715
2016.01.19	22	7,9			
2016.01.20	23	7,9			
2016.01.21	24	7,9	19,1	7,47	3,225
2016.01.22	25	7,9			
2016.01.23	26	7,9			
2016.01.24	27	7,9			
2016.01.25	28	7,9	22,4	6,61	10
2016.01.26	29	7,9			
2016.01.27	30	7,9			
2016.01.28	31	7,9	20,7	6,43	12,07
2016.01.29	32	No Feeding			
2016.01.30	33	No Feeding			
2016.01.31	34	No Feeding			
2016.02.01	35	7,9			
2016.02.02	36	7,9			
2016.02.03	37	7,9			
2016.02.04	38	7,9	21,2	6,43	11,61
2016.02.05	39	7,9			
2016.02.06	40	7,9			
2016.02.07	41	7,9			
2016.02.08	42	7,9	21,1	6,47	8,52

Figure 27: Data for feed of Lab Reactor(1)

2016.02.09	43	7,9			
2016.02.10	44	7,9			
2016.02.11	45	7,9	21,6	6,46	7,14
2016.02.12	46	7,9			
2016.02.13	47	7,9			
2016.02.14	48	7,9			
2016.02.15	49	7,9	21,5	5,75	7,1
2016.02.16	50	7,9			
2016.02.17	51	7,9			
2016.02.18	52	7,9			
2016.02.19	53	7,9	22,7	7,67	6,28
2016.02.20	54	7,9			
2016.02.21	55	7,9			
2016.02.22	56	7,9	21,8	7,96	2,52
2016.02.23	57	7,9			
2016.02.24	58	7,9			
2016.02.25	59	7,9	21,9	7,14	5,125
2016.02.26	60	7,9			
2016.02.27	61	7,9			
2016.02.28	62	7,9			
2016.02.29	63	7,9			
2016.03.01	64	7,9			
2016.03.02	65	7,9	24,1	7,11	7,95
2016.03.03	66	7,9			
2016.03.04	67	7,9			
2016.03.05	68	7,9			
2016.03.06	69	7,9			
2016.03.07	70	7,9			
2016.03.08	71	7,9			
2016.03.09	72	7,9	22,4	6,98	7,735
2016.03.10	73	7,9			
2016.03.11	74	7,9			
2016.03.12	75	7,9			
2016.03.13	76	7,9			
2016.03.14	77	7,9	23,8	6,91	7,745
2016.03.15	78	7,9			
2016.03.16	79	7,9			
2016.03.17	80	7,9	22,1	7,28	6,475
2016.03.18	81	7,9			
2016.03.19	82	7,9			
2016.03.20	83	7,9			
2016.03.21	84	7,9	20,6	8,29	7,81
2016.03.22	85	7,9			
2016.03.23	86	7,9			
2016.03.24	87	7,9			

Figure 28: Data for feed of Lab Reactor(1)

2016.03.25	88	7,9			
2016.03.26	89	7,9	21	7,54	10,57
2016.03.27	90	7,9			
2016.03.28	91	7,9			
2016.03.29	92	7,9			
2016.03.30	93	7,9			
2016.03.31	94	7,9			
2016.04.01	95	7,9	22,5	7,45	5,95
2016.04.02	96	7,9			
2016.04.03	97	7,9			
2016.04.04	98	7,9			
2016.04.05	99	7,9			
2016.04.06	100	7,9			
2016.04.07	101	7,9	21,8	6,79	9,87
2016.04.08	102	7,9			
2016.04.09	103	7,9			
2016.04.10	104	7,9			
2016.04.11	105	7,9			
2016.04.12	106	7,9			
2016.04.13	107	7,9	25,5	7,36	11,43
2016.04.14	108	7,9			
2016.04.15	109	7,9			
2016.04.16	110	7,9			
2016.04.17	111	7,9			
2016.04.18	112	7,9			
2016.04.19	113	7,9			
2016.04.20	114	7,9			
2016.04.21	115	7,9	22,9	7,13	12,34
2016.04.22	116	7,9			
2016.04.23	117	7,9			
2016.04.24	118	7,9			
2016.04.25	119	7,9			
2016.04.26	120	7,9			
2016.04.27	121	7,9			
2016.04.28	122	7,9	23	7,22	12,34
2016.04.29	123	7,9			
2016.04.30	124	7,9			
2016.05.01	125	7,9			
2016.05.02	126	7,9			
2016.05.03	127	7,9			
2016.05.04	128	7,9			
2016.05.05	129	7,9			
2016.05.06	130	7,9	24,4	6,42	10,42

Figure 29: Data for feed of Lab Reactor(1)

Date	Days	AD		Filt.			
		pH	temp.	TCOD g/L	COD g/L	TSS mg/L	VSS mg/L
2015.12.28	0	7,72	19,5	1,585	1,055	340	330
2015.12.29	1						
2015.12.30	2						
2015.12.31	3	7,63	21,8	1,24	1,03	330	290
2016.01.01	4						
2016.01.02	5						
2016.01.03	6						
2016.01.04	7	8,17	21,3	1,8	1,29	560	510
2016.01.05	8						
2016.01.06	9	8,18	19,3	1,42	0,945	410	380
2016.01.07	10						
2016.01.08	11						
2016.01.09	12						
2016.01.10	13						
2016.01.11	14						
2016.01.12	15	8,11	19,3	1,94	0,975	590	510
2016.01.13	16						
2016.01.14	17	8,21	19	1,415	1,19	410	380
2016.01.15	18						
2016.01.16	19						
2016.01.17	20						
2016.01.18	21	7,72	16,3	1,715	1,455		
2016.01.19	22						
2016.01.20	23						
2016.01.21	24	8,09	19,2	1,985	1,285	520	520
2016.01.22	25						
2016.01.23	26						
2016.01.24	27						
2016.01.25	28	6,29	20,5	7,555	7,09	400	390
2016.01.26	29						
2016.01.27	30						
2016.01.28	31	5,88	20,6	8,675	8,355	420	460
2016.01.29	32						
2016.01.30	33						
2016.01.31	34	7,58	21,2	3,195	2,035	1150	1100
2016.02.01	35						
2016.02.02	36						
2016.02.03	37						
2016.02.04	38	6	20,3	7,495	6,61	880	850
2016.02.05	39						
2016.02.06	40						
2016.02.07	41						
2016.02.08	42	6,2	19,5	6,84	6,43	420	510
2016.02.09	43						

Figure 30: Data for AD of Lab Reactor(1)



2016.02.10	44						
2016.02.11	45	7,09	20,1	5,83	4,91	480	500
2016.02.12	46						
2016.02.13	47						
2016.02.14	48						
2016.02.15	49	5,88	20,2	5,215	4,005	310	340
2016.02.16	50						
2016.02.17	51						
2016.02.18	52						
2016.02.19	53	6,89	21,8	6,65	4,715	1070	930
2016.02.20	54						
2016.02.21	55						
2016.02.22	56	8,41	21,2	1,635	0,905	550	470
2016.02.23	57						
2016.02.24	58						
2016.02.25	59	8,52	20,8	2,155	1,635	NA	NA
2016.02.26	60						
2016.02.27	61						
2016.02.28	62						
2016.02.29	63						
2016.03.01	64						
2016.03.02	65	7,45	21,2	2,3	1,615	340	390
2016.03.03	66						
2016.03.04	67	8,08	20,9	4,98	2,73	770	750
2016.03.05	68						
2016.03.06	69						
2016.03.07	70						
2016.03.08	71						
2016.03.09	72	8,32	21,3	4,28	2,795	640	570
2016.03.10	73						
2016.03.11	74						
2016.03.12	75						
2016.03.13	76						
2016.03.14	77	8,2	21,2	6,29	3,645	1400	1280
2016.03.15	78						
2016.03.16	79						
2016.03.17	80	7,43	20,8	3,74	2,115	860	1030
2016.03.18	81						
2016.03.19	82						
2016.03.20	83						
2016.03.21	84	8,29	20,6	4,495	2,93	2533	2233
2016.03.22	85						
2016.03.23	86						
2016.03.24	87						
2016.03.25	88						
2016.03.26	89	7,54	21	5,505	3,105	2683	2683
2016.03.27	90						

Figure 31: Data for AD of Lab Reactor(2)

2016.03.28	91						
2016.03.29	92						
2016.03.30	93						
2016.03.31	94						
2016.04.01	95	7,38	20,2	4,87	2,12	1400	1500
2016.04.02	96						
2016.04.03	97						
2016.04.04	98						
2016.04.05	99						
2016.04.06	100						
2016.04.07	101	6,57	20,7	6,965	5,225	590	580
2016.04.08	102						
2016.04.09	103						
2016.04.10	104						
2016.04.11	105						
2016.04.12	106						
2016.04.13	107	7,11	22,9	7,035	4,83	950	950
2016.04.14	108						
2016.04.15	109						
2016.04.16	110						
2016.04.17	111						
2016.04.18	112						
2016.04.19	113						
2016.04.20	114						
2016.04.21	115	7,16	20,7	7,39	4,995	1740	1660
2016.04.22	116						
2016.04.23	117						
2016.04.24	118						
2016.04.25	119						
2016.04.26	120						
2016.04.27	121						
2016.04.28	122	7,01	20,9	7,675	5,735	670	650
2016.04.29	123						
2016.04.30	124						
2016.05.01	125						
2016.05.02	126						
2016.05.03	127						
2016.05.04	128						
2016.05.05	129						
2016.05.06	130	6,5	23,2	7,37	6,035	700	660

Figure 32: Data for AD of Lab Reactor(3)

Day	Date	DO mg/L	pH	Total Temp. C	Filt. COD g/L	COD g/L	TSS mg/L	VSS mg/L	VSS/TSS %
0	2015.12.28	4,77	7,92	19,3	1,385	1,31	105	100	0,95
1	2015.12.29								
2	2015.12.30								
3	2015.12.31	4,55	7,83	20,7	1,725	1,08	220	195	0,89
4	2016.01.01								
5	2016.01.02								
6	2016.01.03								
7	2016.01.04	4,85	8,3	20,2	1,205	1,19	325	260	0,80
8	2016.01.05								
9	2016.01.06	6,5	8,33	25	1,215	1,045	155	140	0,90
10	2016.01.07								
11	2016.01.08								
12	2016.01.09								
13	2016.01.10								
14	2016.01.11								
15	2016.01.12	5,16	8,3	18,3	1,345	1,09	210	185	0,88
16	2016.01.13								
17	2016.01.14	5,65	8,33	18	1,415	0,87	300	260	0,87
18	2016.01.15								
19	2016.01.16								
20	2016.01.17								
21	2016.01.18	4,13	7,95	15,5	1,665	1,22	NA	NA	
22	2016.01.19								
23	2016.01.20								
24	2016.01.21	6,51	8,28	17,7	1,435	1,245	70	70	1,00
25	2016.01.22								
26	2016.01.23								
27	2016.01.24								
28	2016.01.25	3,04	6,2	19,1	7,41	7,24	285	250	0,88
29	2016.01.26								
30	2016.01.27								
31	2016.01.28	3,24	5,84	19,9	8,875	8,71	320	295	0,92
32	2016.01.29								
33	2016.01.30								
34	2016.01.31								
35	2016.02.01								
36	2016.02.02								
37	2016.02.03								
38	2016.02.04	3,03	6	19,8	7,045	6,435	375	370	0,99
39	2016.02.05								
40	2016.02.06								
41	2016.02.07								
42	2016.02.08	2,05	6,25	19,5	6,3	6,295	260	295	1,13
43	2016.02.09								

Figure 33: Data for CFIC® of Lab Reactor(1)

44	2016.02.10								
45	2016.02.11	1,93	7,16	19,5	5,28	5,005	165	210	1,27
46	2016.02.12								
47	2016.02.13								
48	2016.02.14								
49	2016.02.15	2,55	5,62	19,4	5,065	2,71	125	150	1,20
50	2016.02.16								
51	2016.02.17		5,46						
52	2016.02.18								
53	2016.02.19	3,3	8,24	20,3	4,805	4,02	370	340	0,92
54	2016.02.20								
55	2016.02.21								
56	2016.02.22	2,92	8,8	20,3	1,48	0,795	330	285	0,86
57	2016.02.23								
58	2016.02.24								
59	2016.02.25	2,11	8,68	19,9	1,495	1,265			
60	2016.02.26								
61	2016.02.27								
62	2016.02.28								
63	2016.02.29								
64	2016.03.01								
65	2016.03.02	0,6	8,04	19,6	1,69	1,235	260	260	1,00
66	2016.03.03								
67	2016.03.04		8,37	19,5	4,905	2,83	375	350	0,93
68	2016.03.05								
69	2016.03.06								
70	2016.03.07								
71	2016.03.08								
72	2016.03.09	0,2	8,47	19,7	4,375	2,81	410	355	0,87
73	2016.03.10								
74	2016.03.11								
75	2016.03.12								
76	2016.03.13								
77	2016.03.14	0,9	8,42	20,5	4,81	3,03	795	760	0,96
78	2016.03.15								
79	2016.03.16								
80	2016.03.17	1,76	8,45	20,4	3,555	1,865	855	795	0,93
81	2016.03.18								
82	2016.03.19								
83	2016.03.20								
84	2016.03.21	1,3	8,52	21,1	3,725	2,27	800	675	0,84
85	2016.03.22								
86	2016.03.23								
87	2016.03.24								
88	2016.03.25								
89	2016.03.26	0,3	8,02	20,6	5,735	2,75	1133	1116	0,98
90	2016.03.27								

Figure 34: Data for CFIC® of Lab Reactor(2)

91	2016.03.28								
92	2016.03.29								
93	2016.03.30								
94	2016.03.31								
95	2016.04.01	1,2	7,94	19,8	4,335	1,85	1022	1011	0,99
96	2016.04.02								
97	2016.04.03								
98	2016.04.04								
99	2016.04.05								
100	2016.04.06								
101	2016.04.07	0,8	6,6	20,7	6,89	5,27	470	405	0,86
102	2016.04.08								
103	2016.04.09								
104	2016.04.10								
105	2016.04.11								
106	2016.04.12								
107	2016.04.13	3,3	7,48	20,4	7,065	4,84	730	690	0,95
108	2016.04.14								
109	2016.04.15								
110	2016.04.16								
111	2016.04.17								
112	2016.04.18								
113	2016.04.19								
114	2016.04.20								
115	2016.04.21	0,5	7,51	20,2	7,205	5,21	1610	1440	0,89
116	2016.04.22								
117	2016.04.23								
118	2016.04.24								
119	2016.04.25								
120	2016.04.26								
121	2016.04.27								
122	2016.04.28	0,3	7,15	19,8	7,035	6,245	467	460	0,99
123	2016.04.29								
124	2016.04.30								
125	2016.05.01								
126	2016.05.02								
127	2016.05.03								
128	2016.05.04								
129	2016.05.05								
130	2016.05.06	0,3	6,62	21,5	7,27	5,995	393	386	0,98

Figure 35: Data for CFIC® of Lab Reactor(3)

Date	Days	AD		CFIC	
		TSS (mg/L)	VSS (mg/L)	TSS (mg/L)	VSS (mg/L)
2015.12.28	0	340	330	105	100
2015.12.29	1				
2015.12.30	2				
2015.12.31	3	330	290	220	195
2016.01.01	4				
2016.01.02	5				
2016.01.03	6				
2016.01.04	7	560	510	325	260
2016.01.05	8				
2016.01.06	9	280	270	155	140
2016.01.07	10				
2016.01.08	11				
2016.01.09	12				
2016.01.10	13				
2016.01.11	14				
2016.01.12	15	590	510	210	185
2016.01.13	16				
2016.01.14	17				
2016.01.15	18	410	380	300	260
2016.01.16	19				
2016.01.17	20				
2016.01.18	21				
2016.01.19	22				
2016.01.20	23				
2016.01.21	24	520	520	70	70
2016.01.22	25				
2016.01.23	26				
2016.01.24	27				
2016.01.25	28	400	390	285	250
2016.01.26	29				
2016.01.27	30				
2016.01.28	31	420	460	320	295
2016.01.29	32				
2016.01.30	33				
2016.01.31	34	1150	1100		
2016.02.01	35				
2016.02.02	36				
2016.02.03	37				
2016.02.04	38	880	850	375	370
2016.02.05	39				
2016.02.06	40				
2016.02.07	41				
2016.02.08	42	420	510	260	295
2016.02.09	43				

Figure 36: Data for TSS and VSS of Lab Reactor(1)

2016.02.10	44				
2016.02.11	45	480	500	165	210
2016.02.12	46				
2016.02.13	47				
2016.02.14	48				
2016.02.15	49	310	340	125	150
2016.02.16	50				
2016.02.17	51				
2016.02.18	52				
2016.02.19	53	1070	930	370	340
2016.02.20	54				
2016.02.21	55				
2016.02.22	56	550	470	330	285
2016.02.23	57				
2016.02.24	58				
2016.02.25	59				
2016.02.26	60				
2016.02.27	61				
2016.02.28	62				
2016.02.29	63				
2016.03.01	64				
2016.03.02	65	340	390	260	260
2016.03.03	66				
2016.03.04	67	770	750	375	350
2016.03.05	68				
2016.03.06	69				
2016.03.07	70				
2016.03.08	71				
2016.03.09	72	640	570	410	355
2016.03.10	73				
2016.03.11	74				
2016.03.12	75				
2016.03.13	76				
2016.03.14	77	1400	1280	795	760
2016.03.15	78				
2016.03.16	79				
2016.03.17	80	860	1030	855	795
2016.03.18	81				
2016.03.19	82				
2016.03.20	83				
2016.03.21	84	2533	2233	800	675
2016.03.22	85				
2016.03.23	86				
2016.03.24	87				
2016.03.25	88				
2016.03.26	89	2683	2683	1133	1117
2016.03.27	90				

Figure 37: Data for TSS and VSS of Lab Reactor(2)

2016.03.28	91				
2016.03.29	92				
2016.03.30	93				
2016.03.31	94				
2016.04.01	95	1400	1500	1022	1011
2016.04.02	96				
2016.04.03	97				
2016.04.04	98				
2016.04.05	99				
2016.04.06	100				
2016.04.07	101	590	580	470	405
2016.04.08	102				
2016.04.09	103				
2016.04.10	104				
2016.04.11	105				
2016.04.12	106				
2016.04.13	107	950	950	730	690
2016.04.14	108				
2016.04.15	109				
2016.04.16	110				
2016.04.17	111				
2016.04.18	112				
2016.04.19	113				
2016.04.20	114				
2016.04.21	115	1740	1660	1610	1440
2016.04.22	116				
2016.04.23	117				
2016.04.24	118				
2016.04.25	119				
2016.04.26	120				
2016.04.27	121				
2016.04.28	122	670	650	467	460
2016.04.29	123				
2016.04.30	124				
2016.05.01	125				
2016.05.02	126				
2016.05.03	127				
2016.05.04	128				
2016.05.05	129				
2016.05.06	130	700	660	393	386

Figure 38: Data for TSS and VSS of Lab Reactor(3)



Date	Day	Feed		Anaerobic			CFIC		
		HRT	Organic load	sCOD removal(%)	tCOD removal(%)	tCOD removal		COD removal(%)	sCOD removal(%)
2015.12.28	0,00	27,33	3,08	27,73	69,94	54,84	15,21	60,54	62,68
2015.12.29	1,00	27,33							
2015.12.30	2,00	27,33							
2015.12.31	3,00	27,33	3,02	27,14	70,01	63,90	17,34	49,78	68,56
2016.01.01	4,00	27,33							
2016.01.02	5,00	27,33							
2016.01.03	6,00	27,33							
2016.01.04	7,00	27,33	2,99	26,94	62,17	47,21	12,72	64,66	65,10
2016.01.05	8,00	27,33							
2016.01.06	9,00	27,00	2,20	19,51	61,74	42,51	8,30	50,81	57,69
2016.01.07	10,00	27,00							
2016.01.08	11,00	27,00							
2016.01.09	12,00	27,00							
2016.01.10	13,00	27,00							
2016.01.11	14,00	27,00							
2016.01.12	15,00	27,00	2,67	23,74	67,55	35,44	8,41	55,24	63,73
2016.01.13	16,00	27,00							
2016.01.14	17,00	27,00	2,43	21,57	56,41	48,17	10,39	48,17	68,13
2016.01.15	18,00	27,00							
2016.01.16	19,00	27,00							
2016.01.17	20,00	27,00							
2016.01.18	21,00	27,00	3,30	29,35	60,83	53,84	15,80	55,18	67,16
2016.01.19	22,00	27,00							
2016.01.20	23,00	27,00							
2016.01.21	24,00	27,00	2,87	25,48	60,16	38,45	9,80	55,50	61,40
2016.01.22	25,00	27,00							
2016.01.23	26,00	27,00							
2016.01.24	27,00	27,00							

Figure 39: Data for Mass Balance of Lab Reactor(1)

2016.01.25	28,00	27,00	8,89	79,00	29,10	24,45	19,32	25,90	27,60
2016.01.26	29,00	27,00	27,00						
2016.01.27	30,00	27,00	27,00						
2016.01.28	31,00	27,00	10,73	95,35	30,78	28,13	26,82	26,47	27,84
2016.01.29	32,00	27,00							
2016.01.30	33,00	27,00							
2016.01.31	34,00	27,00							
2016.02.01	35,00	27,00							
2016.02.02	36,00	27,00							
2016.02.03	37,00	27,00							
2016.02.04	38,00	27,00	10,32	91,72	43,07	35,44	32,51	39,32	44,57
2016.02.05	39,00	27,00							
2016.02.06	40,00	27,00							
2016.02.07	41,00	27,00							
2016.02.08	42,00	27,00	7,57	67,31	24,53	19,72	13,27	26,06	26,12
2016.02.09	43,00	27,00							
2016.02.10	44,00	27,00							
2016.02.11	45,00	27,00	6,35	56,41	31,23	18,35	10,35	26,05	29,90
2016.02.12	46,00	27,00							
2016.02.13	47,00	27,00							
2016.02.14	48,00	27,00							
2016.02.15	49,00	27,00	6,31	56,09	43,59	26,55	14,89	28,66	61,83
2016.02.16	50,00	27,00							
2016.02.17	51,00	27,00							
2016.02.18	52,00	27,00							
2016.02.19	53,00	27,00	5,58	49,61	24,92	-5,89	-2,92	23,49	35,99
2016.02.20	54,00	27,00							
2016.02.21	55,00	27,00							
2016.02.22	56,00	27,00	2,24	19,91	64,09	35,12	6,99	41,27	68,45
2016.02.23	57,00	27,00							

Figure 40: Data for Mass Balance of Lab Reactor(2)

2016.02.24	58,00	27,00	27,00	4,56	40,49	68,10	57,95	23,46	70,83	75,32
2016.02.25	59,00	27,00	27,00	4,56	40,49	68,10	57,95	23,46	70,83	75,32
2016.02.26	60,00	27,00	27,00	4,56	40,49	68,10	57,95	23,46	70,83	75,32
2016.02.27	61,00	27,00	27,00	4,56	40,49	68,10	57,95	23,46	70,83	75,32
2016.02.28	62,00	27,00	27,00	4,56	40,49	68,10	57,95	23,46	70,83	75,32
2016.02.29	63,00	27,00	27,00	4,56	40,49	68,10	57,95	23,46	70,83	75,32
2016.03.01	64,00	27,00	27,00	7,07	62,81	79,69	71,07	44,64	78,74	84,47
2016.03.02	65,00	27,00	27,00	7,07	62,81	79,69	71,07	44,64	78,74	84,47
2016.03.03	66,00	27,00	27,00	7,07	62,81	79,69	71,07	44,64	78,74	84,47
2016.03.04	67,00	27,00	27,00	7,07	62,81	79,69	71,07	44,64	78,74	84,47
2016.03.05	68,00	27,00	27,00	7,07	62,81	79,69	71,07	44,64	78,74	84,47
2016.03.06	69,00	27,00	27,00	7,07	62,81	79,69	71,07	44,64	78,74	84,47
2016.03.07	70,00	27,00	27,00	7,07	62,81	79,69	71,07	44,64	78,74	84,47
2016.03.08	71,00	27,00	27,00	7,07	62,81	79,69	71,07	44,64	78,74	84,47
2016.03.09	72,00	27,00	27,00	6,88	61,11	63,87	44,67	27,29	43,44	63,67
2016.03.10	73,00	27,00	27,00	6,88	61,11	63,87	44,67	27,29	43,44	63,67
2016.03.11	74,00	27,00	27,00	6,88	61,11	63,87	44,67	27,29	43,44	63,67
2016.03.12	75,00	27,00	27,00	6,88	61,11	63,87	44,67	27,29	43,44	63,67
2016.03.13	76,00	27,00	27,00	6,88	61,11	63,87	44,67	27,29	43,44	63,67
2016.03.14	77,00	27,00	27,00	6,88	61,11	63,87	44,67	27,29	43,44	63,67
2016.03.15	78,00	27,00	27,00	6,88	61,11	63,87	44,67	27,29	43,44	63,67
2016.03.16	79,00	27,00	27,00	6,88	61,11	63,87	44,67	27,29	43,44	63,67
2016.03.17	80,00	27,00	27,00	5,76	51,15	67,34	42,24	21,61	45,10	71,20
2016.03.18	81,00	27,00	27,00	5,76	51,15	67,34	42,24	21,61	45,10	71,20
2016.03.19	82,00	27,00	27,00	5,76	51,15	67,34	42,24	21,61	45,10	71,20
2016.03.20	83,00	27,00	27,00	5,76	51,15	67,34	42,24	21,61	45,10	71,20
2016.03.21	84,00	27,00	27,00	5,76	51,15	67,34	42,24	21,61	45,10	71,20
2016.03.22	85,00	27,00	27,00	5,76	51,15	67,34	42,24	21,61	45,10	71,20
2016.03.23	86,00	27,00	27,00	5,76	51,15	67,34	42,24	21,61	45,10	71,20
2016.03.24	87,00	27,00	27,00	5,76	51,15	67,34	42,24	21,61	45,10	71,20

Figure 41: Data for Mass Balance of Lab Reactor(3)

2016.03.25	88,00	27,00	27,00	9,40	83,50	70,62	47,92	40,01	45,74	73,98
2016.03.26	89,00	27,00	27,00	9,40	83,50	70,62	47,92	40,01	45,74	73,98
2016.03.27	90,00	27,00	27,00	9,40	83,50	70,62	47,92	40,01	45,74	73,98
2016.03.28	91,00	27,00	27,00	9,40	83,50	70,62	47,92	40,01	45,74	73,98
2016.03.29	92,00	27,00	27,00	9,40	83,50	70,62	47,92	40,01	45,74	73,98
2016.03.30	93,00	27,00	27,00	9,40	83,50	70,62	47,92	40,01	45,74	73,98
2016.03.31	94,00	27,00	27,00	9,40	83,50	70,62	47,92	40,01	45,74	73,98
2016.04.01	95,00	27,00	27,00	5,29	47,01	64,37	18,15	8,53	27,14	68,91
2016.04.02	96,00	27,00	27,00	5,29	47,01	64,37	18,15	8,53	27,14	68,91
2016.04.03	97,00	27,00	27,00	5,29	47,01	64,37	18,15	8,53	27,14	68,91
2016.04.04	98,00	27,00	27,00	5,29	47,01	64,37	18,15	8,53	27,14	68,91
2016.04.05	99,00	27,00	27,00	5,29	47,01	64,37	18,15	8,53	27,14	68,91
2016.04.06	100,00	27,00	27,00	5,29	47,01	64,37	18,15	8,53	27,14	68,91
2016.04.07	101,00	27,00	27,00	8,77	77,97	47,06	29,43	22,95	30,19	46,61
2016.04.08	102,00	27,00	27,00	8,77	77,97	47,06	29,43	22,95	30,19	46,61
2016.04.09	103,00	27,00	27,00	8,77	77,97	47,06	29,43	22,95	30,19	46,61
2016.04.10	104,00	27,00	27,00	8,77	77,97	47,06	29,43	22,95	30,19	46,61
2016.04.11	105,00	27,00	27,00	8,77	77,97	47,06	29,43	22,95	30,19	46,61
2016.04.12	106,00	27,00	27,00	10,16	90,30	57,74	38,45	34,72	38,19	57,66
2016.04.13	107,00	27,00	27,00	10,16	90,30	57,74	38,45	34,72	38,19	57,66
2016.04.14	108,00	27,00	27,00	10,16	90,30	57,74	38,45	34,72	38,19	57,66
2016.04.15	109,00	27,00	27,00	10,16	90,30	57,74	38,45	34,72	38,19	57,66
2016.04.16	110,00	27,00	27,00	10,16	90,30	57,74	38,45	34,72	38,19	57,66
2016.04.17	111,00	27,00	27,00	10,16	90,30	57,74	38,45	34,72	38,19	57,66
2016.04.18	112,00	27,00	27,00	10,16	90,30	57,74	38,45	34,72	38,19	57,66
2016.04.19	113,00	27,00	27,00	10,16	90,30	57,74	38,45	34,72	38,19	57,66
2016.04.20	114,00	27,00	27,00	10,16	90,30	57,74	38,45	34,72	38,19	57,66
2016.04.21	115,00	27,00	27,00	10,97	97,49	59,52	40,11	39,11	41,61	57,78
2016.04.22	116,00	27,00	27,00	10,97	97,49	59,52	40,11	39,11	41,61	57,78
2016.04.23	117,00	27,00	27,00	10,97	97,49	59,52	40,11	39,11	41,61	57,78

Figure 42: Data for Mass Balance of Lab Reactor(4)

2016.04.24	118,00	27,00							
2016.04.25	119,00	27,00							
2016.04.26	120,00	27,00							
2016.04.27	121,00	27,00							
2016.04.28	122,00	27,00	10,97	97,49	53,53	37,80	36,85	42,99	49,39
2016.04.29	123,00	27,00							
2016.04.30	124,00	27,00							
2016.05.01	125,00	27,00							
2016.05.02	126,00	27,00							
2016.05.03	127,00	27,00							
2016.05.04	128,00	27,00							
2016.05.05	129,00	27,00							
2016.05.06	130,00	27,00	9,26	82,32	42,08	29,27	24,10	30,23	42,47

Figure 43: Data for Mass Balance of Lab Reactor(5)