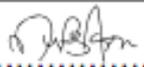




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**BIOGAS POTENTIAL OF MUNICIPAL WASTEWATER
TREATMENT USING LABORATORY SCALE UP- FLOW
ANAEROBIC SLUDGE BLANKET (UASB) REACTORS
UNDER PSYCHROPHILIC CONDITION**

MASTER'S THESIS

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**WATER SCIENCE AND TECHNOLOGY
ENVIRONMENTAL TECHNOLOGY STUDY PROGRAM
DEPARTMENT OF MATHEMATICS AND NATURAL
SCIENCES UNIVERSITY OF STAVANGER**

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Abstract

The operation of two laboratory scale of Up-flow Anaerobic Sludge Blanket (UASB) reactors on treating medium to high strength wastewater (i.e. 500 – 1200 mg COD/l) under psychrophilic conditions (5.5°C) were studied. The reactors were equipped with active granular sludge from three different sources. The aim of this study is to evaluate and investigate capability of anaerobic treatment of medium to high strength municipal wastewater to convert organic matter into methane production using UASB reactors under psychrophilic condition. Hydraulic retention time (HRT) was applied between 6.7 – 1.3 hours with gradually increased organic loading rate (OLR) from 3.11 ± 0.10 – 8.35 ± 0.16 g COD/l.d. The observed results present a fluctuated COD removal efficiency and methane production in the UASB reactors. High effluent COD was also discovered indicating high dissolved concentration of gaseous compounds at low temperatures. Apart from those, COD removal efficiency was able to be achieved a maximum of 58.5% in the reactor I with such a high OLR of 8.9 g COD/l.d with 3.3 h of HRT and yield of $0.62 \text{ gCOD}_{\text{CH}_4} / \text{gCOD}_{\text{removed}}$ (62% of COD removed converted into methane). Overall methane yields observed were $0.224 \text{ l CH}_4/\text{g COD}$ and $0.186 \text{ l CH}_4/\text{g COD}$ in reactor I and II, respectively. The performance of anaerobic treatment systems using UASB reactor for treating medium to high strength municipal wastewater under psychrophilic conditions show a good alternative as a pre-treatment with some parameters which need to be considered.

Keywords: *anaerobic treatment, UASB reactor, psychrophilic condition, COD removal efficiency, methane production.*

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Table of Contents

Abstract	i
Acknowledgement.....	ii
Table of Contents.....	iii
List of Figures.....	v
List of Tables	vi
Abbreviations	vii
1. Introduction.....	1
1. 1. Scope of Work	2
1. 2. Objectives	4
1. 3. Thesis Outline	4
2. Literature Review and Theoretical Background	5
2. 1. Anaerobic Treatment	5
2. 1. 1. Hydrolysis	6
2. 1. 2. Acidogenesis	6
2. 1. 3. Acetogenesis.....	6
2. 1. 4. Methanogenesis	6
2. 2. Anaerobic Stoichiometry	7
2. 2. 1. Chemical Oxygen Demand (COD)	7
2. 2. 2. COD Fractions.....	7
2. 2. 3. Methane Production	9
2. 2. 4. COD Balance.....	10
2. 3. Considerations in the Application of Anaerobic Treatment Processes.....	11
2. 3. 1. Characteristic of Wastewater.....	11
2. 3. 1. 1. Flow rate, hydraulic and OLR Variations	12
2. 3. 1. 2. Organic Concentration	12
2. 3. 1. 3. Temperature.....	13
2. 3. 1. 4. Fractions of Non-Dissolved Organic Matter	14
2. 3. 1. 5. pH	14
2. 3. 1. 6. Nutrients and Trace Elements	14
2. 3. 1. 7. Inorganic and Organic Toxic Compounds	15
2. 3. 2. Pretreatment of Wastewater	15
2. 3. 2. 1. Screening.....	16
2. 3. 2. 2. Solids Conditioning or Reduction	16
2. 3. 2. 3. pH Adjustment	16
2. 3. 2. 4. Fats, Oil, and Grease Control	16

2. 3. 2. 5. Toxicity Reduction	17
2. 4. Up-Flow Anaerobic Sludge Blanket (UASB)	17
2. 4. 1. General Concept	17
2. 4. 2. Applications of UASB under Psychrophilic Condition	19
2. 5. Knowledge Gaps	20
3. Materials and Methods.....	21
3. 1. The Configuration of UASB Reactors.....	21
3. 2. Starting-up UASB Reactors and Operational Conditions.....	25
3. 3. Analytical Methods.....	27
3. 3. 1. Conductivity and pH Measurement.....	27
3. 3. 2. Alkalinity and VFA Measurement	27
3. 3. 3. COD Measurement.....	28
4. Results	29
4. 1. Reactor Performance.....	29
4. 1. 1. COD Removal Efficiency	29
4. 1. 2. Methane Production	31
4. 1. 3. COD Balance.....	34
4. 2. pH, VFA and Alkalinity Variations.....	34
5. Discussions	36
5. 1. Reactor Performance.....	36
5. 1. 1. COD Removal Efficiency	36
5. 1. 2. Methane Production	37
5. 1. 3. COD Balance.....	39
5. 2. Environmental Factors.....	39
5. 3. Economy and Energy Recovery	40
5. 4. Hydrodynamic Condition	41
6. Conclusions.....	43
7. Recommendations.....	45
Appendixes	a
Appendix 1 : Daily Data of Reactor I and Reactor II	a
Appendix 2 : Energy Recovery Calculation	k

List of Figures

Figure 1.1 The processes configuration of IVAR Grødalund	2
Figure 2.1 Anaerobic digestion processes [3]	7
Figure 2.2 COD balance of anaerobic reactor [3].....	11
Figure 2.3 Upflow anaerobic sludge blanket (UASB) bioreactor [14]	18
Figure 3.1 Photo of the laboratory scale UASB reactors	22
Figure 3.2 Flow diagram of the laboratory scale UASB reactors	24
Figure 3.3 The flowchart of general operation.....	26
Figure 4.1 Profiles in Reactor I and II: OLR and COD inlet (a); COD outlet (b); and COD removal efficiency (c).....	30
Figure 4.2 The methane production rate with function of; OLR (a); and CODs Inlet (b)	31
Figure 4.3 Graphical determination of methane production of reactor I and II.....	32
Figure 4.4 Graphical determination of theoretical methane production reactor I (a) and II (b)	33
Figure 4.5 Methane production rate in function of OLR in reactor I (a) and II (b).	33
Figure 4.6 The COD balance and OLR profiles of Reactor I and II	34
Figure 4.7 pH, alkalinity, and VFA accumulation profiles of; Inlet (a), reactor I (b), reactor II (c); and CODs Inlet (d)	35

List of Tables

Table 1.1 Wastewater sources of Grødalaland WWTP	3
Table 1.2 The average flow and composition of wastewater in and out the DAF.....	4
Table 2.1 Main results obtained in a 6m³ UASB plant with raw domestic sewage as influent (9.5 - 19°C) [5]	19
Table 3.1 The properties of equipment used for the UASB reactors	25
Table 4.1 Methane yield, theoretical methane yield and COD conversion to methane at operating condition.....	32

Abbreviations

COD	Chemical Oxygen Demand
DAF	Dissolved Air Flotation
FOG	Fats, Oil and Grease
EGSB	Expanded Granular Sludge Blanket
DO	Dissolved Oxygen
HAc	Acetic Acid
HRT	Hydraulic Retention Time
IVAR	<i>Interkommunalt Vann Avløp og Renovasjon</i>
LCFA	Long Chain Fatty Acid
OLR	Organic Loading Rate
SBR	Sequencing Batch Reactor
SCFA	Short Chain Fatty Acid
SRT	Solid/Sludge Retention Time
STP	Standard Temperature and Pressure
UASB	Up-flow Anaerobic Sludge Blanket
VFA	Volatile Fatty Acid
WWTP	Wastewater Treatment Plant

1. Introduction

Wastewater produced by community might originate from three different sources: (a) domestic wastewater; (b) industrial wastewater, and; (c) rain-water [1]. Untreated sewage and industrial wastewater pose a serious and potential health risk due to the pathogenic microorganisms, nutrients and toxic compounds. Direct discharge was the common way of handling the sewage and domestic wastewater thousands of years ago, especially in developing countries. Various technologies are available today for wastewater treatment, such as conventional aerobic treatment, direct anaerobic treatment and combination of anaerobic and aerobic treatment [2].

In the energy crisis in the 1970s aerobic treatment lose its attractiveness, linked with the increased demand for industrial wastewater treatment leading to the shifting attention of the treatment from aerobic treatment to anaerobic treatment [3]. Anaerobic wastewater treatment has then evolved into competitive wastewater treatment technology. This technology has been used for treating the concentrated domestic and industrial water for over a century. It has various positive characteristics, such as capable to be operated under high solids retention times, require low energy consumption, low production of sludge, produce biogas and require low land and low construction cost.

Lettinga and co-workers (2001) introduced the Up-Flow Anaerobic Sludge Treatment (UASB) as breakthrough for anaerobic treatment. These days, various types of wastewater have been extensively treated by UASB reactors [4]. This technology was claimed to be a robust technology and the most widely used high rate anaerobic process for sewage treatment [2]. UASB reactors are also proven for treating the wastewater treatment under wide range of temperature conditions [5]. Treating the wastewaters at ambient temperatures would be beneficial because of reduced resources. At tropical temperatures, this treatment has been successfully applied and receives huge acceptance for domestic wastewater treatment, while at lower temperatures, the application has been studied only in the lab and pilot scale plants (Lew et. al., 2004; [2]). Despite of the deterioration of activity of anaerobic microorganisms at temperatures below 20 °C, the propitious results have already been reported in a few studies at 10 - 15°C [6, 7, 8, 9]. The development of psychrophilic anaerobic treatment will have a great economic and ecological impact [10] and will be beneficial for some high latitudes countries, thus, this psychrophilic anaerobic treatment still requires further study.

1. 1. Scope of Work

This study was part of master project in collaboration with IVAR and Environmental Technology Study Program, University of Stavanger. IVAR (Interkommunalt vann, avløp og renovasjon) is a Norwegian public company that constructs and operates municipal facilities for water, waste water and general waste. The effluent wastewater from dissolved air flotation (DAF) units at Grødaland wastewater treatment plant (WWTP) were treated by using laboratory scale UASB reactors for this study. The whole processes configuration and wastewater sources of IVAR Grødaland are presented in **Figure 1.1** and **Table 1.1**, respectively. As shown in **Figure 1.1**, this plant consists of pre-treatment with 3 mm opening bar screening, sand and fat removal followed by DAF unit with the addition of polymer. The flotation unit consists of two tanks with a surface area of 48 m² for each tanks and designed for 7.5 m/h surface loading and can withstand with maximum load of 200 l/s. This unit is capable to remove around 61.5% of suspended solid and can achieve approximately 80% suspended solid removal with the addition of polymer, depending on the dosing concentration.

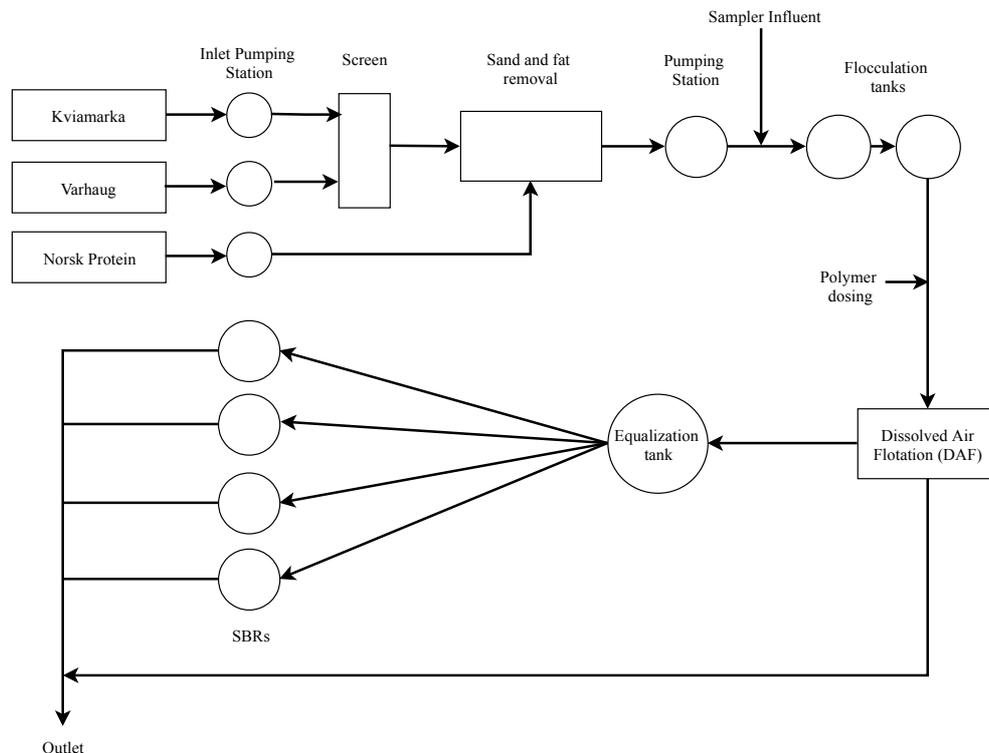


Figure 1.1 The processes configuration of IVAR Grødaland

Table 1.1 Wastewater sources of Grødaland WWTP

Wastewater Sources	Average Loading (m³/d)
a. Dairy and chicken slaughtering in Kviemarka	3284
b. Municipal wastewater of 3000 houses in Varhaug and food industry in Fjordland	1680
c. Animal destruction in Norsk Protein	167
Total Loading	5131

On the other hand, these DAF units are not able to remove the high fractions of dissolved COD, only 14 % of dissolved COD is removed, as reported by IVAR Grødaland in **Table 1.2**. This high concentration of dissolved COD influences the performance of sequencing batch reactor (SBR), the next stage of treatment. The SBR units at IVAR Grødaland are not able to function properly due to high fraction of dissolved COD followed by oxygen deficiency (high oxygen demand). This condition leads to the filamentous microorganisms grow rapidly, causing the sludge struggling to settle. One approach to solve this problem is to remove the dissolved COD before the SBR units through anaerobic granular sludge treatment using UASB reactors as pre-treatment which is expected to remove high portion of dissolved COD and convert them into biogas (methane). This has been done on the laboratory scale at University of Stavanger using UASB reactors under mesophilic conditions i.e. at 25 °C and has shown satisfying results with COD removal efficiency above 70 % [11]. Treating wastewater at ambient temperatures would be beneficial because of reduced costs (e.g. no heating or cooling required). For high latitudes countries, i.e. Norway and Canada, wastewater treatment under psychrophilic condition would be advantageous. For this reason, wastewater treatment laboratory scale using UASB reactors under psychrophilic condition, i.e. at 5.5°C was conducted in the university.

Table 1.2 The average flow and composition of wastewater in and out the DAF

Parameters	Inlet Flotation unit	Outlet Flotation unit
SS (mg/L)	510	196
Dissolved COD (mg/L)	959	825
Total COD (mg/L)	1862	1184
BOD (mg/L)	1501	927
Total Phosphorus (mg/L)	25,9	23,1
Conductivity ($\mu\text{S/cm}$)	1938	1904
pH	7,73	7,54
Flow (m^3/d)	4728	4330

1. 2. Objectives

This study is the continuation of examining the removal efficiency from the effluent of DAF unit from Grødaland WWTP using laboratory scale Up-flow Anaerobic Sludge Blanket (UASB) reactors under psychrophilic condition i.e. 5.5 °C. Thus, the main objective of this study is to investigate and evaluate the effectiveness of anaerobic treatment of medium to high strength municipal wastewater to convert organic matter into methane production using UASB reactors under psychrophilic condition.

1. 3. Thesis Outline

This master thesis is entitled: “**Biogas Potential of Municipal Wastewater Treatment using Up-Flow Anaerobic Sludge Blanket (UASB) reactors under psychrophilic condition**” and divided into seven chapters.

1. Introduction;
2. Literature Review and Theoretical Background;
3. Materials and Methods;
4. Results;
5. Discussions;
6. Conclusions; and
7. Recommendations including further research.

Appendixes are included to present supporting materials of the whole study.

2. Literature Review and Theoretical Background

Theoretical explanation based on relevant scientific literature of anaerobic treatment are explained in this chapter. This also includes the fundamental considerations in the application of anaerobic treatment processes, anaerobic stoichiometry, as well as general concept of UASB. Additionally, the applications of UASB for treating the wastewater under psychrophilic condition from previous studies also presented in this chapter. The knowledge gaps are described in the last of this chapter are based on this literature review and theoretical background explained.

2. 1. Anaerobic Treatment

Anaerobic treatment involves specialized bacteria and archaea, using variety of electron acceptors in the nonappearance of molecular oxygen for energy production [12]. The important advantages of anaerobic treatment include energy considerations, lower biomass yield, less nutrients required, higher volumetric organic loadings and effective pre-treatment process. This also can be conducted in the plain system and applied in any scale and any place. In opposition with anaerobic treatment, which is defined by high operational costs (energy) and converting large fraction of waste into another type of waste (sludge), anaerobic treatment produces useful energy (biogas) and converts waste into small amount of excess sludge (90% sludge reduction).

Lettinga et. al. (1984) described four metabolic groups of bacteria that can be distinguished in anaerobic digestion;

1. Hydrolytic bacteria, which break down polymers for instance proteins and carbohydrates into their monomers.
2. Fermentative bacteria which ferment these monomers to organic acids, alcohol, carbon dioxide, hydrogen and ammonia;
3. Acetogenic bacteria which convert VFA and alcohols into acetic acid and hydrogen;
4. Methanogenic bacteria utilize methanol, acetate and hydrogen and carbon dioxide to produce methane. These bacteria are located at the end of anaerobic food chain and due to their activity, only small quantities of organic matter accumulate in the anaerobic environments.

The anaerobic digestion processes are multi step process of series and parallel reactions (**Figure 2.1**), which are subdivided and described shortly into the following four stages;

2. 1. 1. Hydrolysis

Hydrolysis can be described as a process that converts complex polymeric substrates, particulate or dissolved into monomeric and dimeric compounds which are ready for use for acidogenic bacteria. Polymeric particles are degraded by the action of exo-enzymes to produce smaller molecules which is able to pass over the cell barrier. Proteins are hydrolysed into amino acids, polysaccharide to simple sugars and lipids to LCFA. This process is very susceptible to temperature.

2. 1. 2. Acidogenesis

Hydrolysis products, which are, amino acids, simple sugars, LCFAs) are diffused inside the bacterial cells through cell membrane and fermented. the products of this process consist of a variety of small organic compounds, mainly VFAs (acetate, propionate and butyric), H_2 , CO_2 , some lactic acids, ethanol and ammonia. If methanogenesis is limited and instead, H_2 accumulates, propionate and butyrate are likely to appear and possibly lactate and alcohols [3].

Acidogenesis is the most rapid conversion step in the anaerobic food chain. With the highest AG° of all anaerobic conversions, resulting ten to twenty fold higher bacterial growth rates and fivefold higher bacterial yields and conversion rates compared to methanogens [3]. Consequently, anaerobic reactors tend to be acidified with the sudden pH drop. Severe inhibition of methanogens could occur when the higher concentration of non-dissociated VFAs appear due to the consumed alkalinity by the produced acids.

2. 1. 3. Acetogenesis

In this process, acetogenic bacteria converts SCFA which is produced in the acidogenesis into acetate, hydrogen gas and carbon dioxide. Despite of the importance of propionate and butyrate as acetogenic substrates, lactate, ethanol, methanol and even H_2 and CO_2 are also converted to acetate, as shown in **Figure 2.1**.

2. 1. 4. Methanogenesis

This conversion process is the final stage of anaerobic conversion of organic matter into methane and carbon dioxide. A group of methanogenic archaea using hydrogen as electron donor and decarboxylate acetate to form CH_4 . The influent COD is converted into gaseous form and leaves the reactors. Generally, about 70% of the produced methane derives from acetate which is converted by aceticlastic methanogenesis process, while the rest may come from H_2 and CO_2 by hydrogenotrophic methanogenesis.

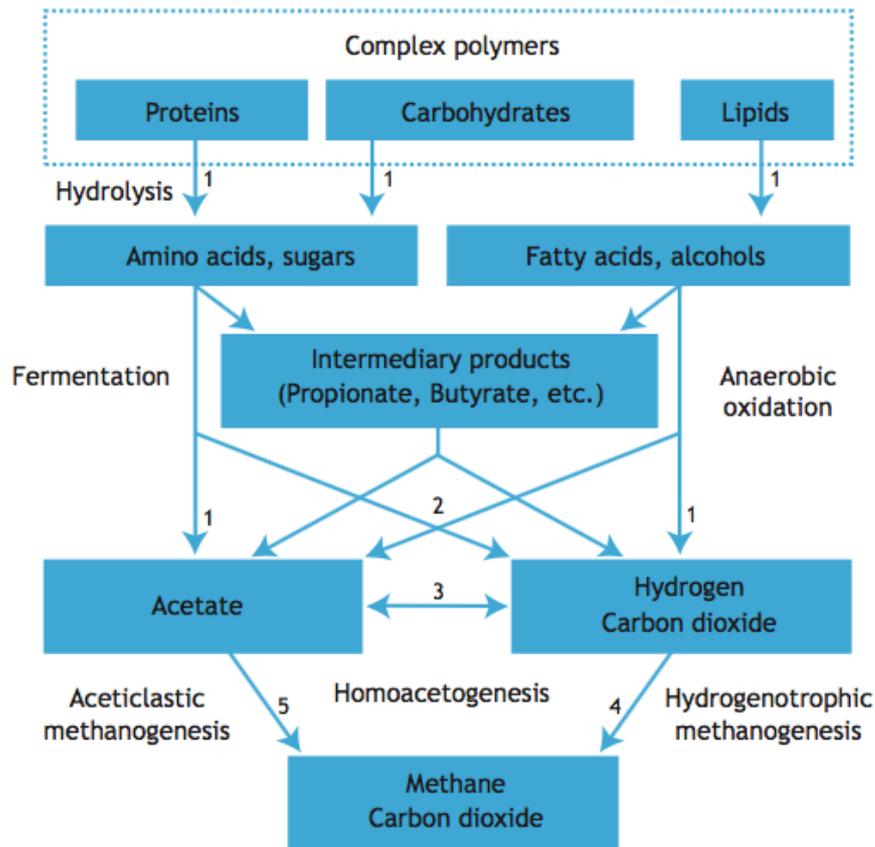


Figure 2.1 Anaerobic digestion processes [3]

2. 2. Anaerobic Stoichiometry

2. 2. 1. Chemical Oxygen Demand (COD)

COD is the most important parameter for the concentration of contaminants in wastewater, particularly for industrial wastewater [3]. Chemical oxidation resulted by dichromate the majority of organic matter available in the sample is the basic concept of measurement COD analysis. This COD analysis is required for mass balances in wastewater treatment as it can be subdivided into fractions that useful for consideration regarding the design of the treatment processes, that is discussed in following sub-chapter.

2. 2. 2. COD Fractions

Based on the ability to degrade, the total amount of COD can be divided into two fractions, biodegradable and non-biodegradable COD. Biodegradable COD is expressing the sewage treatability, which is defined as the total COD portion present in the waste that can be

biologically degraded in anaerobic conditions. Both biodegradable and non-biodegradable COD are also divided into two fractions, particulate and dissolved COD. Dissolved biodegradable COD is readily degraded by microorganisms while particulate biodegradable COD must be hydrolysed into smaller molecules so that can be used for the growth of microorganism. this dissolved COD can be further subdivided into readily and slowly biodegradable components. Slowly biodegradable components consists of high molecular weight that requires significant metabolism to convert them into simple monomers which are substrates for acidogenic bacteria, thus, long SRT may be required to metabolize these materials [13]. While particulate non-biodegradable COD cannot be used by microorganisms, thus, will adsorb to biomass and will be accumulated in the sludge. Meanwhile, dissolved non-biodegradable COD will not also be degraded by microorganisms, in the other hand, it will not be accumulated to sludge, instead, it will pass through the effluent.

Some anaerobic processes are better to treat wastewaters containing primarily particulate matter, while others are best with soluble substrates. For example, particulate organic matter is not retained as effectively in UASB and hybrid UASB/AF systems, which allow it to pass through the bioreactor with little hydrolysis and stabilization, hence, UASB is better with soluble wastes [14].

Measurement made for determining the soluble COD in the feed (S_{CO}) and in the bioreactor S_C , non-biodegradable COD, which is the substrate. The concentrations of biodegradable COD can be observed from measurement soluble COD values by subtracting the inert soluble COD, S_I , which passes through bioreactor:

$$S_{SO} = S_{CO} - S_I \quad \text{Eq. 2.1}$$

$$S_S = S_C - S_I$$

where,

S_{SO} = Biodegradable COD concentration in the feed

S_{CO} = Soluble in the feed

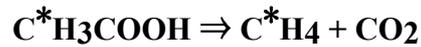
S_S = Biodegradable COD concentration in the bioreactor

S_C = Soluble in the bioreactor

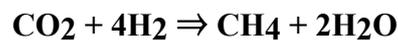
S_I = Inert soluble COD

2. 2. 3. Methane Production

Chernicharo (2007) described that cleavage of acetic acid and reduction of carbon dioxide are two basic mechanisms for methane formation. Cleavage of acetic acid leads to the formation of methane and carbon dioxide in the absence of hydrogen. The methyl group of the acetic acid is reduced to methane, while the carboxylic group is oxidised to carbon dioxide:

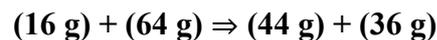
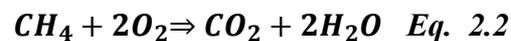


While with the presence of hydrogen, most of the remaining methane is formed from the reduction of carbon dioxide. CO_2 acts as an acceptor of the hydrogen atoms removed from the organic compounds by the enzymes. Since carbon dioxide is always present in excess in an anaerobic reactor, its reduction to methane is not the limiting factor in the process. The methane formation from the reduction of the carbon dioxide is shown below:



The overall composition of the production of the biogas during anaerobic digestion varies depending on the environmental conditions prevailing in the reactor. The composition changes quickly during the initial start-up of the system and also when the digestion process is inhibited. For the stable operation, the composition of the production of the biogas is reasonably uniform. However, the carbon dioxide/methane ratio can vary substantially, depending on the characteristics of the organic compound to be degraded. In the anaerobic treatment of domestic sewage, typical methane and carbon dioxide fractions present in the biogas are 70 to 80% and 20 to 30%, respectively [15].

One method of evaluating the production of methane is from COD degradation in the reactor, as shown in the following equation:



As shown in Equation 2.2, one mole of methane required two moles of oxygen for the complete oxidation to carbon dioxide and water. Thus, every 16 grams of CH_4 produced and lost to atmosphere corresponds to the removal of 64 grams of COD from the waste. Under STP, this corresponds to 0.35 m^3 of CH_4 for each gram of degraded COD using Equation 2.3. At

temperature 5.5°C, this corresponds to 0.36 m³ of methane produced per kg of COD converted to methane.

$$K(t) = \frac{P \times K}{R \times (273 + T)} \quad \text{Eq. 2.3}$$

where:

- P = atmospheric pressure (1 atm)
- K = COD corresponding to one mole of CH₄ (64 gCOD/mole)
- R = gas constant (0.08206 atm.L/mole.K)
- T = operational temperature of the reactor (°C)

While the general equation that demonstrate the theoretical production of methane per gram of COD removed from the waste is shown in Equation 2.4.

$$V_{CH_4} = \frac{COD_{CH_4}}{K(t)} \quad \text{Eq. 2.4}$$

- V_{CH₄} = volume of methane produced (L)
- COD_{CH₄} = load of COD removed from the reactor and converted into methane (gCOD)
- K(t) = correction factor for the operational temperature of the reactor (gCOD/l)

The solubility of gaseous compounds increases as the temperature decreases below 20°C. This implies that the dissolved concentrations of methane, hydrogen sulphide and hydrogen will be higher in the effluent reactors operated at low temperature than those at high temperature. High increase of solubility of CO₂ indicates that a slightly lower reactor pH might prevail under psychrophilic conditions [16].

2. 2. 4. COD Balance

COD balance is an important consideration for anaerobic degradation process. In contrary with aerobic treatment, there is no COD destruction in an anaerobic reactor, instead, the COD is only re-arranged. Thus, the COD and COD balance can be very useful as the control parameter for anaerobic systems. All COD that entered the system end up in the end product of CH₄, minus the COD that is incorporated in the biomass, as shown in **Figure 2.2**. Since a mass balance can be made by only using the COD as a parameter (Equation. 2.5), the COD therefore generally taken as a control tool to operate an anaerobic system.

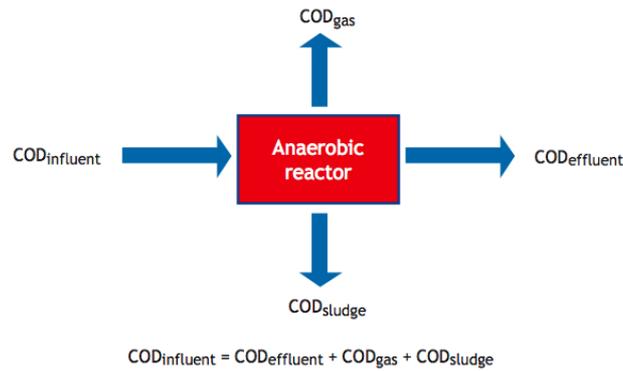


Figure 2.2 COD balance of anaerobic reactor [3]

$$COD_{in} = COD_{out}$$

$$COD_{Influent} = COD_{effluent} + COD_{gas} + COD_{sludge} \quad Eq. 2.5$$

Thus,

$$\%COD \text{ Balance} = \frac{COD_{effluent} + COD_{gas} + COD_{sludge}}{COD_{influent}} \times 100\% \quad Eq. 2.6$$

Using COD balance as a tool to monitor reactor performance, gives the important information about the functioning of the system. Also, the impact alternative of electron acceptors on the CH₄ production rate can be easily evaluated. An estimation of the amount of newly grown and entrapped biomass can also be obtained based on gas production and effluent COD values [14].

2. 3. Considerations in the Application of Anaerobic Treatment Processes

The focus of this sub-chapter is to explain parameters that need to be considered for the application of the anaerobic treatment process. This sub-chapter is further subdivided into characteristic of wastewater and pre-treatment of wastewater.

2. 3. 1. Characteristic of Wastewater

The type of wastewater and its characteristics are important in the implementation, design, and evaluation of anaerobic processes. A wide variety of wastewaters have been treated by anaerobic processes. Other parameters within the type of wastewater need to be considered, as described in following sub-chapter.

2. 3. 1. 1. Flow rate, hydraulic and OLR Variations

Wide variations in influent flowrate and loading rate affect the balance between acid fermentation and methanogenesis in anaerobic processes. According to van Lier, et al., (2001) strong variations in flow and concentration may also affect the efficiency of an anaerobic reactor. The acidogenic reactions can be much faster to reduce pH and increase the VFA that can further inhibit methanogenesis [17, 12]. More uniform feeding provides more stable anaerobic process operation and also allows higher average organic loading rates. Anaerobic processes have shown to be able to respond quickly to the incremental increases in wastewater feed after periods without substrate addition.

OLR is linked to SRT through the active biomass concentration in the bioreactor. It is an easy parameter to calculate and has been used to characterize the loading on anaerobic treatment systems. OLR provides useful information for the design and operation of anaerobic processes. According to Tchobanoglous, et al. (2014), OLR for UASB systems may range from 5 to 15 kg COD/m³ and can be calculated using formula;

$$OLR = \frac{Q(S_o)}{V_{OLR}} \quad Eq. 2.7$$

where V_{OLR} = reactor process volume controlled by the organic loading rate, m³.

2. 3. 1. 2. Organic Concentration

A wastewater strength greatly affects the economics and choice of anaerobic treatment over aerobic treatment. Generally, biodegradable COD concentrations higher than 1500 to 2000 mg/l are needed to provide sufficient methane production to heat the wastewater assuming it is in ambient temperature [12]. Lettinga et. al (2001) reported that low substrate levels (50-100 mgCOD/l) and low biogas production rate will be produced by low COD_{influent}. According to Rebac et. al., (1995), anaerobic treatment of low strength wastewaters at low temperatures may give numerous problems. Low influent COD concentration results in very low substrate levels inside the reactor as well as low biogas production rate. Consequently, a lower mixing intensity and a poor substrate-biomass contact can be expected. The treatment of very low strength wastewater at the maximum possible organic loading rate with respect to the maximum COD conversion capacity of the sludge, might cause severe hydraulic wash out of the sludge [18].

2.3.1.3. Temperature

Temperature affects the growth of microorganisms significantly. Growth rate that conducted in higher temperature has maximum value than those conducted at lower range. Generally, the used terms on describing microorganisms' classes based on temperature are; psychrophilic below 15°C, mesophile for 15 °C - 40 °C, thermophile at 40 °C - 70 °C and hyperthermophile at 70 °C - 110 °C. Chemical and biological reactions under psychrophilic conditions will be much slower than those reactions under mesophilic conditions because the reactions in the biodegradation of organic matter require more energy to be proceed at low temperature than at optimum temperature of 37 °C [16]. 25 to 35°C of reactor temperature is generally preferred to support more optimal biological rates and provide more stable treatment. A drop in temperature is accompanied with a change of physical and chemical properties of the wastewater, which can affect the design and operation of the treatment system. Most studies with the effect of temperature on anaerobic digestion show a strong negative effect on the metabolic activity of mesophilic anaerobic bacteria methanogenic bacteria at decreasing temperature [16]

Lower temperatures can still apply to the anaerobic treatment and can continue its operation at 10 – 20 °C in granular sludge blanket, suspended growth and attached growth reactors. Promising results have been reported for temperature 10 - 15°C in a few studies [19, 7]. Slower reaction rates occur in at lower temperature, thus longer SRT, larger reactor volumes, and lower organic COD loadings are needed. Bowen et. al. (2014) reported for anaerobic treatment of domestic wastewater at 8°C anaerobic activity was established following a lag period after which the dynamics of acidogenesis and methanogenesis were similar to those at 15°C. Even lower temperature also reported, which the methanogenesis was inhibited due to inhibition of activity rather than the absence of methanogen population, while the acidogenic reactions still occur at all temperatures [20].

Under psychrophilic conditions, chemical and biological reactions proceed much slower than at optimum temperature of 37°C. The solubility of gaseous compounds increases as the temperature decrease below 20°C. This leads to the higher concentration of dissolved methane, hydrogen sulphide and hydrogen in the effluent of reactors. At low temperatures, particles will settle more slowly because of a decreased liquid-solid separation at low temperatures [16].

2. 3. 1. 4. Fractions of Non-Dissolved Organic Matter

Particulate and soluble fractions affect the type of anaerobic reactor selected and its design. Wastewater with high solids concentration are treated more appropriately in suspended growth and UASB reactors. Where more particulate conversion is required, longer SRT values may be needed if the solids hydrolysis is the rate limiting step as compared to acid fermentation or methanogenesis in anaerobic treatment.

2. 3. 1. 5. pH

pH has a significant impact on the performance of anaerobic processes, with the decreasing as the pH deviates from the optimum value. Variation of influent pH value can affect reactor performance, but it depends on the buffer capacity of mixed liquor. Methanogenic activity will decrease as the pH deviates from the optimum value [14]. A pH range between 6.3 to 7.8 [4] or 6.5 to 7.5 [17] generally provides optimum conditions for methanogens. Unlike methanogens, acidogenic bacterial are less sensitive to higher or lower pH values, thus methanogenic activity can be inhibited at lower pH values while VFA can still be produced particularly propionic and butyric acid, this is caused by the build-up of H_2 in the system that accumulated as methanogens activity is slowed, which then slows down the production of acetic acid by acidogens and transform their metabolism toward other VFAs which may acidify the reactor [17, 14]. Based on experimental experience by Lettinga et al. (2000), the process recovers almost immediately from pH shocks once the influent pH is back to the optimal range. Thus, the recovery the process depends on the extent and duration of imposed change and concentration of VFA during the event, in case of sudden changes [21]

2. 3. 1. 6. Nutrients and Trace Elements

Nutrients are required by anaerobic processes because they are essential components of their growth otherwise the growth will not be satisfied. Nutrient addition may be required when carbon rich industrial wastes are being treated. Such wastewaters may be deficient in the macronutrients nitrogen and phosphorus, as well as limiting micronutrients such as, iron, nickel, cobalt, sulfur and calcium. According to Demirel and Scherer (2011) the availability or lack of trace elements such as Fe, Co, Ni, Zn, Mo, W and Se definitely play a significant role in maintaining a stable and an efficient conversion process of organic wastes to biogas-methane. The favorable concentrations of Cr, Ni, Co, Mo and W were reported to vary between 2.2 mg

m³ and 21.2 mg m³, 801 mg m³ and 5.362 g m³, 148 mg m³ and 580 mg m³, 44 mg m³ and 52.94 g m³, and 658 mg m³ and 40.39 g m³, respectively [22].

2. 3. 1. 7. Inorganic and Organic Toxic Compounds

To assure that serious transient toxicity does not exist for wastewater treated by anaerobic process, proper analysis and treat-ability studies are needed. The appearance of the toxic compounds does not always indicate that processes are unable to operate. For instance, some toxic compounds inhibit anaerobic methanogenic reaction rates, but with high biomass inventory and low enough loading, the process can be continued. Methanogens are quite vulnerable to a large variety of components. The key factor in application of anaerobic treatment of toxic wastewater is adaptation [4].

Dissolved oxygen is a presumed inhibitory compound for methanogens and acetogens. In a very low strength wastewater, dissolved oxygen concentration might appear up to 10 m/l [4]. Its presence can preclude the activity of methanogenesis, in addition, might deteriorate the granular sludge due to growth of filamentous aerobic or facultative organisms. However, studies from Kato (1994) reveal that any risk of serious upset does not exist. The facultative organisms will consume the dissolved oxygen and grow rapidly, hence, protecting the methanogens from the effect of oxygen [23].

Detergents belong to the compounds that discharged down to the drain into municipal sewer systems and eventually enter the treatment plants. Detergents contain surfactants, which decrease the surface tension when added to mixed system, for instance, water and air or water and soil. A harmful effect which is inhibition of methanogenic activity and increasing VFA concentration observed on lab-scale UASB reactor's performance that treating brewery wastewater, however, the system recovered easily as soon as after the shock.

2. 3. 2. Pretreatment of Wastewater

Pre-treatment considerations include in this sub-chapter are screening, solid conditioning or reduction, pH and temperature adjustment, nutrient addition, fats, oil and grease (FOG) control and toxicity reduction.

2. 3. 2. 1. Screening

Screening is normally used as removal of objectionable material that could cause the interference with the flow distribution in a granular sludge reactor, mixing problems in the suspended reactors or plugging of attached growth reactors. A 2-3 mm screening should be taken as consideration for anaerobic membrane reactors in order to prevent membrane fouling problems [12].

2. 3. 2. 2. Solids Conditioning or Reduction

This pre-treatment may be considered in the processing of wastes that are high in solids content and lignin material to enhance methane production and anaerobic degradation reaction rates. Two-step process of solid reduction with solids removal or solids removal and hydrolysis in the first step before the downstream granular sludge and attached growth anaerobic processes can be beneficial to COD removal performance and the stability of the operation. High suspended solids concentration in the influent may create clogging and channelling in the sludge blanket, thus, reduce the treatment effectiveness.

Van Haandel and Lettinga (1994) proposed two step processes with solid contact and hydrolysis in the first step for both industrial and domestic wastewater treatment with downstream UASB, EGSB and packed bed anaerobic processes [2]. A greater solids accumulation occurs under low temperature due the slower hydrolysis rates and may require solids wasting and further treatment.

2. 3. 2. 3. pH Adjustment

Little variation in pH and temperature of the reactor can lead to more stable operation and better efficiency. Alkalinity is needed for treating dilute wastewaters with lower gas production rates, relatives to the wastewater flowrates, as the percent CO₂ can be minimal. This influent alkalinity should be controlled to maintain pH values of reactors between 6.3 and 7.8 for which the favourable methanogens activity occurs [4].

2. 3. 2. 4. Fats, Oil, and Grease Control

The presence of FOG could cause the inhibition of methanogenesis due to inhibition by long chain fatty acids (LCFAs) and also cause sludge flotation. FOG can also destructive to the

granular sludge particles in the UASB and EGSB and can cause fouling of the synthetic membranes used in the anaerobic reactors. Because of this reason, source control and dissolved air flotation pre-treatment are needed to be considered especially for wastewater with high FOG concentration.

2. 3. 2. 5. Toxicity Reduction

As discussed in previous sub-chapter, wide range of inorganic and organic substances can be toxic to anaerobic. This also includes heavy metals, high dissolved solids, chlorinate organic compound, amino acids, proteins and industrial chemical products. One way to remove or biodegrade toxic substance before ta final methanogenic treatment stage is to use a two-step process in the anaerobic treatment system [12].

2. 4. Up-Flow Anaerobic Sludge Blanket (UASB)

2. 4. 1. General Concept

The UASB reactor is the most widely and successfully used high rate anaerobic technology for treating several type of wastewater. The anaerobic process through UASB reactors presents several advantages compare to conventional aerobic processes, especially when applied in warm temperature such as [15];

- compact systems
- low land requirements, low construction and operating costs
- low sludge production
- low energy consumption
- satisfactory COD and BOD removal efficiencies, amounting to 65 to 75%
- high concentration and good dewatering characteristics of the excess sludge

Although the UASB reactors present many advantages, there are still some disadvantages or limitations:

- possibility of release of bad odours
- low capacity of the system in tolerating toxic loads
- long start-up periods
- need for a post-treatment stage

The presence of sulfur compounds and toxic materials usually are at very low levels and being well handled by the treatment system, when the wastewater is predominantly domestic. If the system is well designed, constructed and operated, the bad smell and failure problems due to the presence of toxic elements and/or inhibitors should not be occurred.

Figure 2.3 shows the schematic diagrams of UASB process. Influent wastewater enters the bottom of the bioreactor through distribution system that is designed to provide relatively uniform flow. In the lower portion of the bioreactors, a dense slurry of granules forms and the combined effects of the influent wastewater distribution and gas production result in mixing of the influent with granules. Within these dense blanket of granules, the treatment occurs. For some wastewaters, the flocculent sludge also develops and accumulates on top of the blanket of granules provided the up flow velocity is insufficient to carry it away. Wastewater that has been treated exits the granular and flocculent sludge zones and flows upward into the gas-liquid-solids separator. Gas bubbles and the upward flowing liquid cause some granular and flocculent solids to rise through the bioreactor and enter the gas-liquid-solids separator.

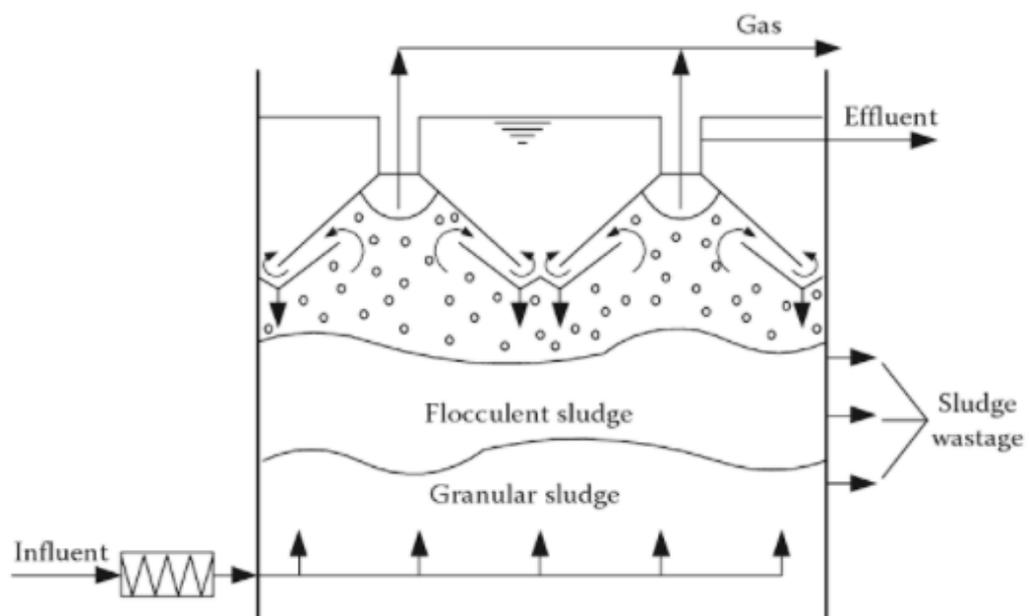


Figure 2.3 Upflow anaerobic sludge blanket (UASB) bioreactor [14]

The success of the UASB concept relies on the establishment of the dense sludge bed in the bottom of the reactor, in which all the biological occurs [2]. This sludge bed is formed basically by accumulation of incoming suspended solids and bacterial growth. It is also observed that bacteria can naturally aggregate in flocs and granules, in the up-flow anaerobic systems, under

certain conditions [24]. These aggregates are not sensitive to wash-out from the system under practical reactor conditions due to good settling properties. Retention of granular or flocculent sludge within the UAS reactor enables good treatment performance at high loading rates. UASB reactor may replace the primary settler, the anaerobic sludge digester and the aerobic step and the secondary settler of a conventional aerobic treatment plant. However, the effluent from UASB reactors still needs the further treatment.

2. 4. 2. Applications of UASB under Psychrophilic Condition

UASB reactors can be using to treat wastewater under wide range of temperature conditions [5] In warm temperatures or in tropical countries, the application UASB reactors have been used and give promising and satisfactory results [25, 26, 27]. The application of UASB reactors to sewage treatment under low temperature conditions have been studied in Netherland since 1976 by Lettinga [2]. The results of the treatment temperature as low as 9.5°C show COD reduction obtained up to 30% and 55% of the filtered and raw effluent, as shown in table 2.1 [5]. From the table it is shown that the removal of SS is fairly poor, particularly at temperature below 12°C. However, from the table by Seghezzi et. al. (1998), efficiencies up to 80% were observed at 10-20°C when treating sewage from separated sewer system (domestic wastewater separated from rain-water).

Table 2.1 Main results obtained in a 6m³ UASB plant with raw domestic sewage as influent (9.5 - 19°C) [5]

Measurement	Week number		
	1-11	16-22	24-26
Temperature (°C)	19-15	11-12	9.5 -10
Effl. COD _{intr.} (mg/ℓ)	100-200	150-175	175-250
COD reduction (%) _{filtr. eff.}	65-80	55-70	55
COD reduction (%) _{raw eff.}	40-55	30-50	30
CH ₄ production (m ³ /kg COD _{intr.})	0.130	0.090	0.050
Excess sludge production (kg DS/kg COD _{intr.})	0.195	0.172	0.271

According to Lettinga et. al. (2001), the feasibility of high-rate anaerobic wastewater treatment systems under psychrophilic condition depends on; (1) the quality of the seed material used and its development under sub-mesophilic conditions; (2) an extremely high sludge retention under high hydraulic loading conditions; (3) an excellent contact between retained sludge and wastewater to utilize all the available capacity within the bioreactor; (4) types of organic pollutant in the wastewater and (5) the configuration of the reactor.

2. 5. Knowledge Gaps

This study aimed to contribute to the development methane potential of anaerobic treatment systems. Furthermore, the main focus was to investigate the effectiveness of UASB reactors to convert organic matter to methane under low temperature. To achieve this objective, in-house designed laboratory scale, UASB reactor systems were set up for treating medium to high strength municipal wastewater treatment ranged 500 mgCOD/l - 1200 mgCOD/l under psychrophilic condition (5.5°C). Based on literature review and theory background, the anaerobic treatment using UASB at very low temperature has been limited to few studies [16] [18] [28] [8] [19] [6], due to the complication i.e. inhibition of methanogens and low degradation rate. In the other hand, treating the wastewater at initial temperatures would be beneficial because of reduced costs (e.g. no heating or cooling required), therefore, wastewater treatment under low temperature would be useful for cold countries such as Norway and Canada. The important factors affecting performance of UASB reactors were also identified so that by maintaining optimal operating conditions, optimum efficiency could be achieved. Therefore, the OLR capacity, pH variability, VFA production, and alkalinity also investigated in this study. COD removal efficiency, methane production rate, including methane yield and fraction and COD balance were investigated in this study to monitor and evaluate the reactors performance.

3. Materials and Methods

This chapter describes the materials and methodology of laboratory-scale experiment of anaerobic treatment of industrial wastewater from DAF effluent at psychrophilic condition, IVAR Grødaland, using UASB reactor. Two UASB reactors were used in the study to investigate UASB reactor performance and their potential for COD removal and biogas production. These two reactors (Reactor I and Reactor II) used active granular sludge. This chapter also includes operational, maintenance and control procedures, and the analytical methods used in this the study. All laboratory tests for this master's thesis project were performed and conducted at the University of Stavanger.

3. 1. The Configuration of UASB Reactors

In the laboratory, two UASB reactors were used to treat medium to high strength wastewater, as shown in Figure 3.1. These two reactors were already set and in operation since 2016 and just previously finished the project with temperature of 8.5 °C, therefore, similar granules have been used since 2016, which were made from mixed sources; (a) pulp and paper company treating cellulose and lignin containing (Norske Skog, Moss); (b) agriculture pilot plant treating swine and cow manure supernatant (farm in Skien, Norway); and (c) hydrocarbon oil containing wastewater at Bamble Industrial Park, Telemark (Safitri, 2016). The flow diagram of the laboratory scale reactors is shown in **Figure 3.2**.



Figure 3.1 Photo of the laboratory scale UASB reactors

As shown in **Figure 3.2**, the two reactors, which both has volume of 1 L, were connected to the 30 L feed wastewater stored in the fridge by using peristaltic pump with adjustable flow rate. The effluent and the gas from the reactors were first filtered and went out to gas trap/collector caused by liquid height, pressure and suction force given by the recirculation pump. The filter for each reactor was installed in order to retain the granules or biomass from washed out with the effluent that could clog the pump and tubes.

Small glass tubes were installed in order to trap the humidity of the biogas and stop the biogas from evaporating before it goes to the gas counters. Two gas counters were also installed for each reactor for measuring produced gas and biogas respectively. CO₂ gas absorber was installed in between two gas counters in order to absorb CO₂ using 3 M NaOH solution. When the CO₂ from produced gas is absorbed in this solution the methane could go to the next gas counter. In total, four gas counters were used for two UASB reactors.

These two reactors were equipped with cooling or heating system and circulation tubes. This cooling system allowed the liquid to be cooled inside the external layer of the reactors through circulation tubes. External insulator was also installed in order to help maintaining the psychrophilic condition in the reactors at temperature of 5.5 °C. The pH of the liquid inside the

reactors should be maintained around 7 due to the optimum growth of the methanogens. Sodium hydrogen bicarbonate (NaHCO_3) needed to be added into the feed if the pH of the liquid was around 6.5 or less. This buffer was chosen because it does not affect the physical and chemical properties of the wastewater. All of important equipment used for UASB reactors were shown in **Table 3.1**.

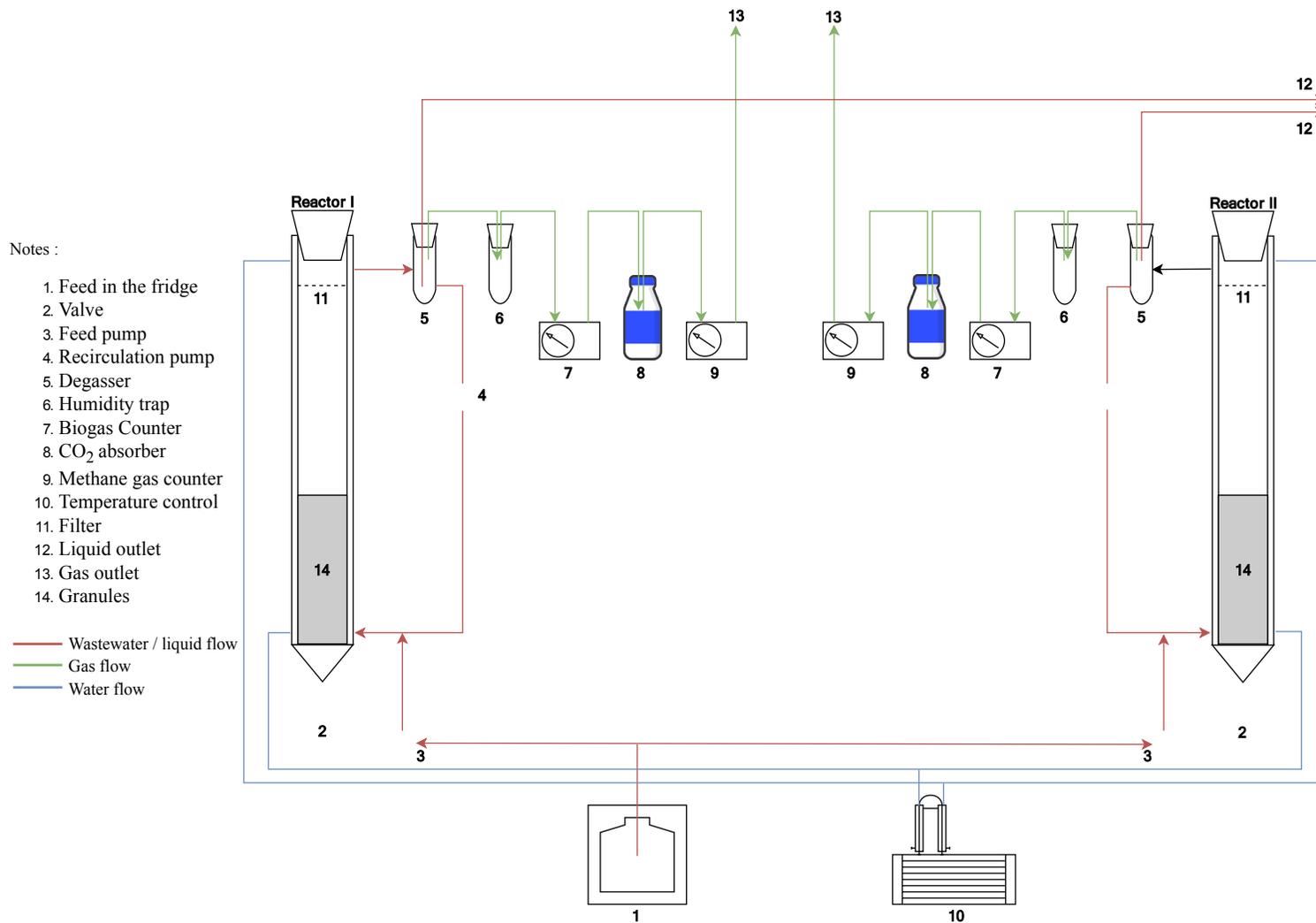


Figure 3.2 Flow diagram of the laboratory scale UASB reactors

Table 3.1 The properties of equipment used for the UASB reactors

Equipment	Manufacturer	Specification
Filters	Sefar	Type : peristaltic pump
		Model : Reglo ICC
		Flowrate : 0 - 43 ml/min
Feed and recirculation pump	Ismatec	Type : peristaltic pump
		Channel : 4 independent adjustable channels
		Model : Reglo ICC
		Flowrate : 0 - 43 ml/min
Gas Counter	Ritter	Model : MGC - 1 V3.3 PMMA
		Gas flowrate : 1 - 1000 ml/h
		Max. pressure : 100 mbar
		Min. pressure : 5 mbar
		Vol. measurement : Reactor I → 3,34 ml (biogas); 3,26 ml (Methane) Reactor II → 3,29 ml (biogas); 3,10 ml (Methane)
		Measurement accuracy : less than approx. ± 1%
Thermo heating circulator	Lauda Alpha	Model : RA 8 LCK 1907
		Temperature range : -25 - 100 °C
		Heater capacity : 230 B; 50/60 Hz; 1.5 kW
		Max. pressure : 0.2 bar
		Max. flowrate : 15 l/min
		Bath volume : 20 liters

3. 2. Starting-up UASB Reactors and Operational Conditions

The UASB reactors were already in the operation with temperature (8.5°C), therefore, the re-activation of the granules was not needed. Before started this project, all of the tubes, degasser and humidity traps were washed and cleaned and the temperature were then decreased to 5.5 °C. These reactors were also then covered with the insulator to maintain the low temperature. This thesis work started on 21st December 2017 and finished on 05th May 2018.

The first stage of starting up the reactors was to ensure the reactors were in pseudo-steady state condition and worked hydraulically well. A pseudo-steady state was achieved in the reactors when the parameters, e.g. the effluent soluble COD and the daily gas production remained constant at the same OLR. The flowchart of general operation of UASB reactors was shown in Figure 3.3. Key parameters such as OLR, flow rate, and pH were controlled daily to ensure the conditions were conducive for anaerobic bacteria during the anaerobic treatment processes. The UASB reactor was started-up at OLR of 3.11±0.10 g COD/l.d and gradually increased until to 8.35±0.16 g COD/l.d. Changes in OLR were made when the reactors was assumed in pseudo-steady state condition. Sometimes the liquid level in the degasser was too high or low during the operation which either caused the liquid went to the humidity trap and to the gas counter or

the gas went to the liquid outlet (this gas therefore not counted). This problem was solved by cleaning the tubes and the gas counter and adjusting the pressure by increasing or decreasing the height of the liquid outlet tube until the liquid in the degasser was in its normal level. The flowrate of the reactors was adjusted depended on the inlet COD and in accordance with the desired OLR. The flowrate was taken manually by measuring the volume of the effluent liquid in the measuring cylinder per specific time. The flowrate of each reactor needed to be kept equal as possible although this was sometimes difficult as clogging occurred unequally. This section describes the starting-up process and operation conditions of two reactors used in this study.

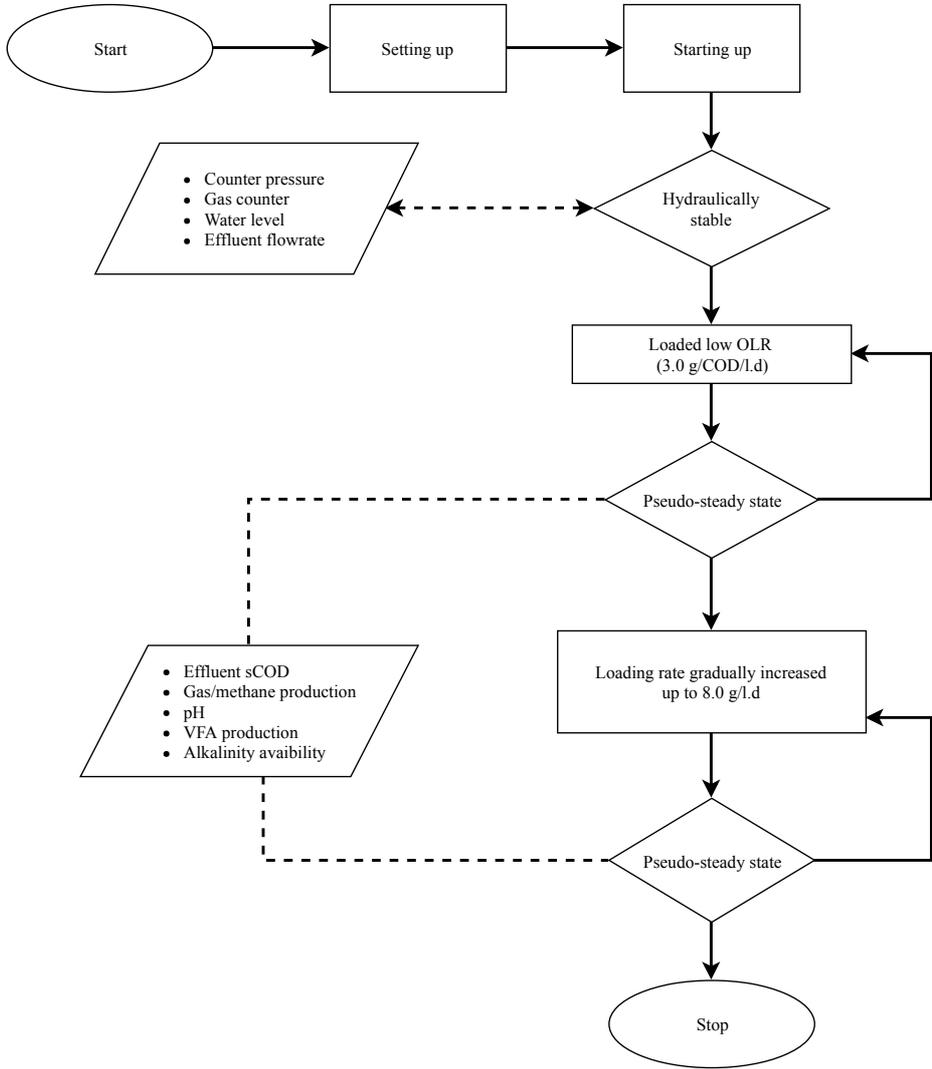


Figure 3.3 The flowchart of general operation

3. 3. Analytical Methods

This sub-chapter explains about how the analytical methods were obtained in the laboratory in order to estimate the effectiveness of anaerobic treatment of wastewater under psychrophilic condition. The following parameters were measured;

1. Conductivity and pH measurement
2. Alkalinity and VFA measurement
3. COD measurement

Before doing analytical experiment, the sample needed to be homogenised by shaking it to ensure the particles and solids are well distributed. In this study, diluting/washing water used was deionized water of 18 M Ω -cm resistances or higher (DI water type 1). The analytical methods used are described as follows.

3. 3. 1. Conductivity and pH Measurement

Conductivity was measured using WTW Multi340i which was connected to conductivity probe (WTW Tetra Con® 325). The probe was immersed into the samples until the value was constant. The conductivity unit was also noted as μ S/cm or mS/cm then converted to mS/m, a unit consistent with the TITRA 5 software for measuring alkalinity and VFA concentration. This conductivity meter was regularly calibrated with conductivity calibration solution, 0.01 mol/l KCl, once in one or two weeks. The pH was measured automatically by alkalinity and VFA instrument, TitroLine® 5000 Auto-Titrator. This pH meter was calibrated with buffer standard solutions (4.01 and 7.00) regularly once in one or two weeks.

3. 3. 2. Alkalinity and VFA Measurement

In this study, TitroLine® 5000 Auto-titration (Instrument-teknikk AS, Oslo) was the instrument used for measuring alkalinity and VFA. VFA and alkalinity analysis were measured using five pH point titration method. These analyses were done for the influent and effluent of the bioreactor, in total 3 samples were needed to be analysed. To do so, 20 ml of each sample was taken and diluted into 50 ml with deionized water (DI water type 1), then placed on a magnetic stirring for a mixing at a low rotation speed. The initial pH of the diluted sample was recorded, if pH of sample was less than 6.7, NaOH 0.1 M (Product No. 106498, Merck) was added to the sample until reached pH = 6.7. The diluted sample was then titrated with 0.1 M HCl (Product No. 100317, Merck) to four different selected pH values (6.7, 5.9, 5.2 and 4.3) and the volume

of acid added to reach those pH values for each titration was noted. These data were input into computer software (TITRA 5) and automatically calculated the total VFA as mg acetic acid/l and alkalinity concentration as mg CaCO₃/l.

3.3.3. COD Measurement

COD analysis was done for feed wastewater and bioreactor effluent, both total and dissolved COD. Total COD tests were done once or twice every OLR to observe non-biodegradable COD and biomass produced. Dissolved COD tests were daily measured as parameter control of reactor performance. To determine dissolved COD, the wastewater was collected in the filtration device using 1.5 µm particle retention of glass microfiber filters (Whatman™ CAT No. 1822-047 and VWR European Ca. No. 516-0876). If using dilution for the sample, it is important to compensate the COD value with correction factor of washing/diluting water

In this study, COD test kits were used to carry out the wastewater analysis. COD test kits product number used were Product Number 109772 (10 - 150 mg/l of COD concentration range, Hg-Free) and 109773 (100 - 1500 mg/l of COD concentration range). This COD test kit is a small glass tube (known as vial or reaction cell) on which there are digestion and catalyst solutions that could react with the samples

The procedure of COD test was carried out based on the COD test kit manual. 2.0 ml sample of wastewater for total COD and 2.0 ml sample of filtrated wastewater for dissolved COD were added into the reaction cell and vigorously mixing the contents of the reaction cell until well mixed, then the cell was heated in thermo reactor (Model TR 620), at 148 °C for 120 mins. After that the cell was cooled for about 30 minutes until it reached room temperature. Then the cell was placed into the cell compartment in the spectrometer (Spectroquant Pharo 300), and aligning the orientation mark on the vial with mark on the spectrometer to measure COD based on the method selected on display. The reading is equivalent to COD concentration.

4. Results

All results obtained from the lab experiment were presented in this chapter which is divided into two sub-chapters; (1). Reactor performance, including COD removal efficiency, methane production and COD balance analysis, and; (2) pH, VFA and Alkalinity variations. All data, tables and figures given in this chapter are summarized, while the raw data are presented in appendixes.

4. 1. Reactor Performance

This sub-chapter explains the reactor performance during lab experiment was conducted in total 135 days which consists of three series of OLR; OLR 3.11 ± 0.10 g COD/l.d was taken 39 days, OLR 5.02 ± 0.15 g COD/l.d was 28 days and 67 days for OLR 8.35 ± 0.16 g COD/l.d. these UASB reactors' performance were characterized by COD removal efficiency and COD conversion to methane gas.

4. 1. 1. COD Removal Efficiency

In- and outlet COD concentrations, and the COD removal efficiency with function of time and OLR are shown in **Figure 4.1**. HRT 6.7 – 1.3 h were applied on Reactor I and Reactor II. The OLR increased gradually from 3.11 ± 0.10 g COD/l.d to 8.35 ± 0.16 g COD/l.d. During periods of constant OLR, COD was fluctuated without really achieving pseudo-steady state, especially at OLR 8.35 ± 0.16 g COD/l.d, this results the fluctuating effluent COD as well as COD removal efficiencies. The lowest COD removal efficiencies were observed at day 97 (6.2%) in reactor I and day 83 (6.9%) and 97 (7.4%) in reactor II because of low pH and power blackout at the university.

At day 117, highest COD removal efficiencies for both reactors were achieved, 58.5% and 57% in reactor I and II respectively at OLR 8.9 g COD/l.d and HRT of 3.3 h. 57.1% of COD removal efficiency was also achieved in reactor II at OLR 5.02 ± 0.15 g COD/l.d at HRT of 3.5 h. At OLR 3.11 ± 0.10 g COD/l.d, the HRT was applied in the range 3.9 – 6.7 h with COD removal efficiencies varied between 22.5 – 49.8% and 23.2 - 54.1% for reactor I and II respectively. COD removal efficiencies at OLR 5.02 ± 0.15 were observed between 13.7 – 48.7% for reactor I and 18.3 – 57.1% for reactor II with HRT ranged between 1.9 – 3.8 h. While for OLR 8.35 ± 0.16 g COD/l.d, HRT applied was between 1.3 – 3.8h at OLR 8.0 g COD/l.d and the COD

removal efficiency achieved were fluctuated in the range of 6.2 - 58.5 % and 6.9 – 57.0 % in reactor I and reactor II, respectively. It also shows that COD removal efficiency corresponds with CODs inlet, especially at OLR 8.35 ± 0.16 g COD/l.d, where high COD removal efficiency could be achieved with high CODs inlet.

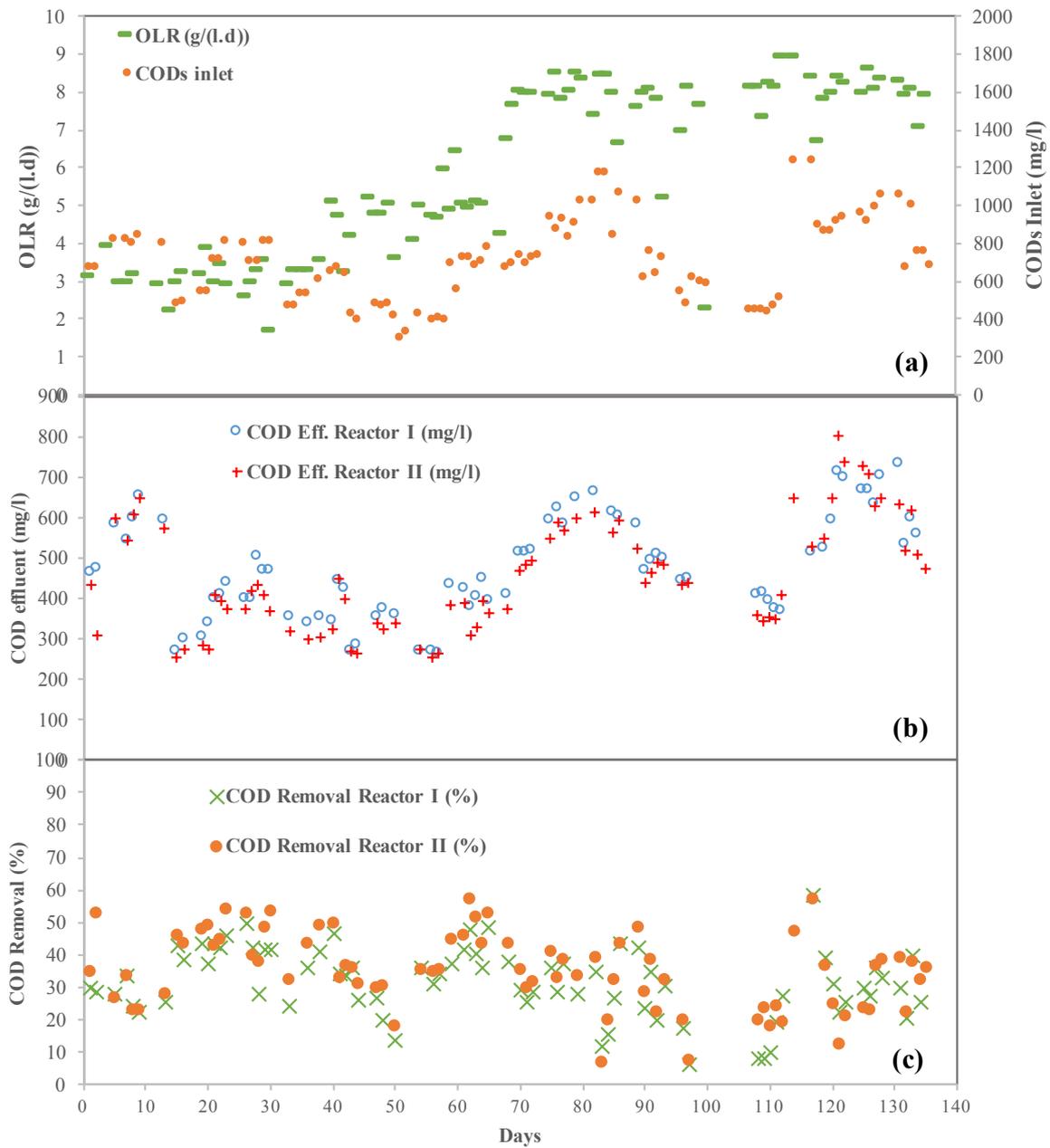


Figure 4.1 Profiles in Reactor I and II: OLR and COD inlet (a); COD outlet (b); and COD removal efficiency (c).

4. 1. 2. Methane Production

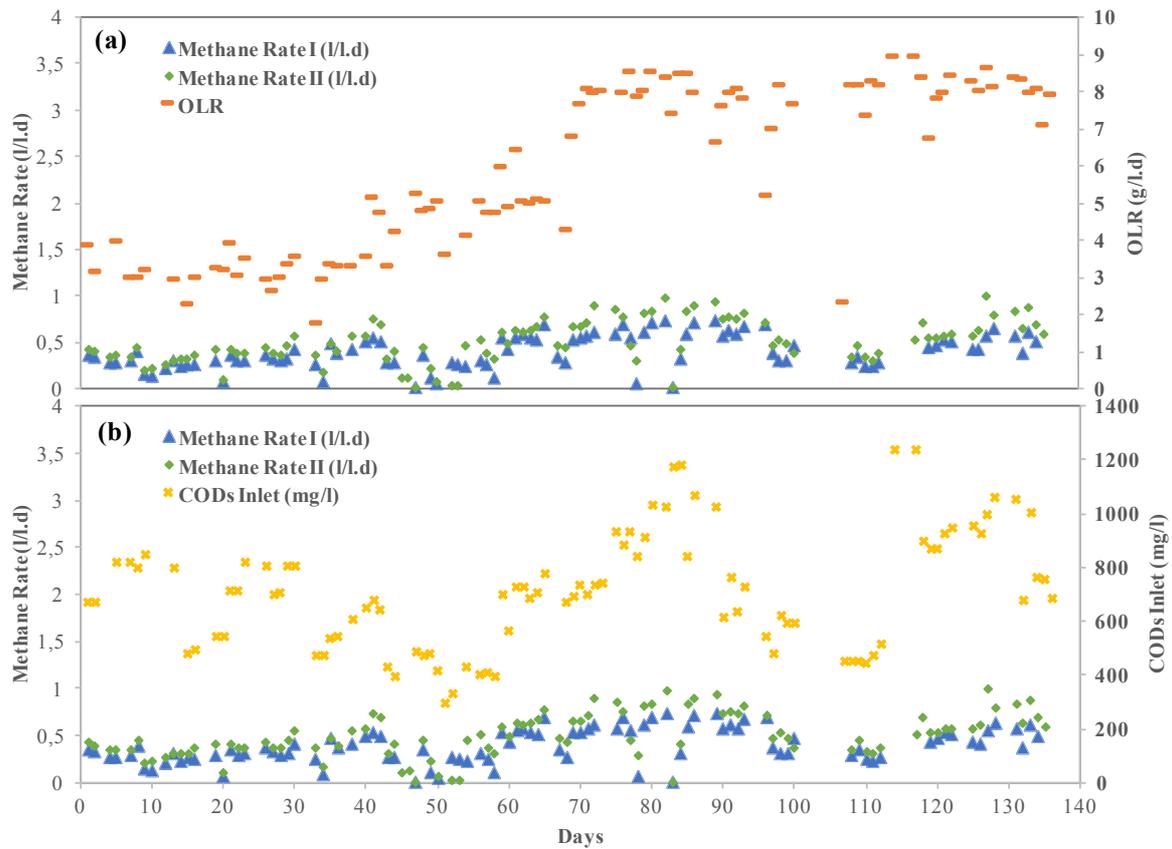


Figure 4.2 The methane production rate with function of: OLR (a); and CODs Inlet (b)

Figure 4.2 represents the methane production rate of volume specific (l/l.d) in function of operation periods and OLR (gCOD/l.d) and inlet COD. As shown in **Figure 4.2**, methane production rate was fluctuated especially after washed out granules, cleaning and increased OLR. Methane production rate in Reactor I and II were observed decreased during the low concentration of inlet COD with relatively high OLR (8 gCOD/l.d). Methane production rate also corresponds with the concentration of inlet COD. In general, methane production rate increased with increasing OLR only with high concentration of inlet COD.

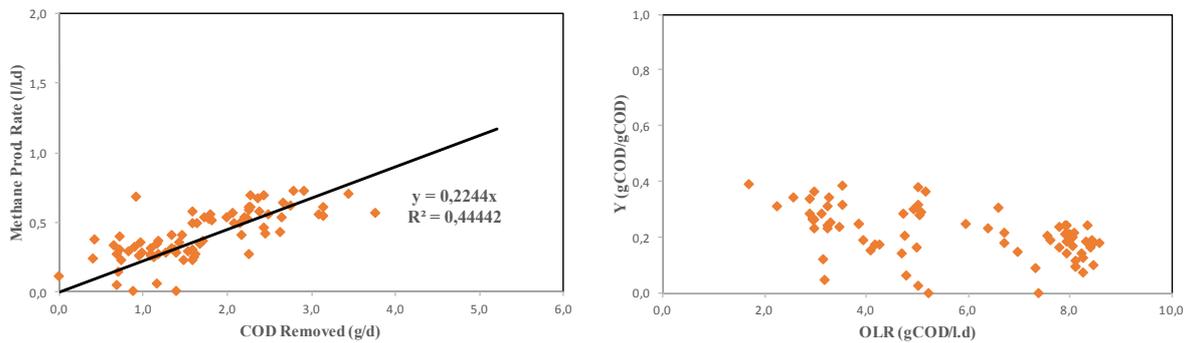
Figure 4.3 shows methane production rate in function of COD removed that can determine the values of methane yield. Methane yields in reactor I and reactor II were taken linearly by $y = 0.224x$ ($R^2 = 0.444$) and $y = 0.186x$ ($R^2 = 0.273$) respectively. Methane yields obtained during operation, theoretical methane yield and COD conversions are presented in **Table 4.1**. The theoretical methane yield was also determined from values of theoretical methane production rate in function of COD removed, as shown in **Figure 4.4**. From **Table 4.1**, the observed methane yields were quite far to the theoretical methane yields. The COD conversion were obtained 62% and 52% for reactor I and II respectively. These numbers reflect the percentage

of COD in wastewater that were converted to methane.

Table 4.1 Methane yield, theoretical methane yield and COD conversion to methane at operating condition

Reactor	Methane Yield l CH ₄ /gCODremoved	Methane Yield gCOD CH ₄ /gCODremoved	Theo. Methane Yield l CH ₄ /gCODremoved	COD Conversion %
Reactor I	0.224	0.622	0.284	62
Reactor II	0.186	0.517	0.269	52

(a) Reactor I



(b) Reactor II

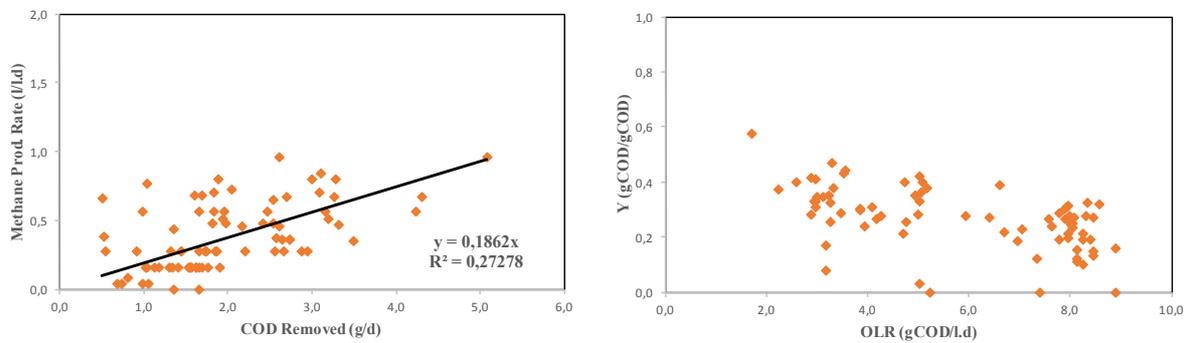


Figure 4.3 Graphical determination of methane production of reactor I and II

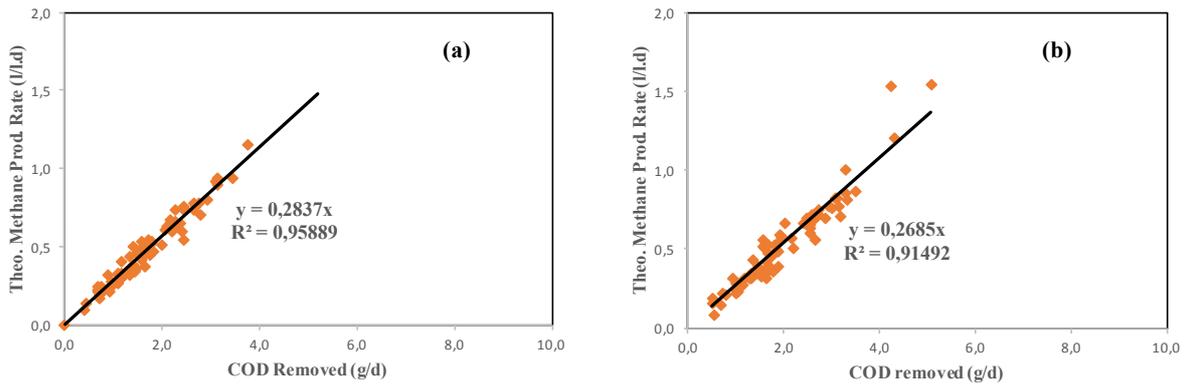


Figure 4.4 Graphical determination of theoretical methane production reactor I (a) and II (b)

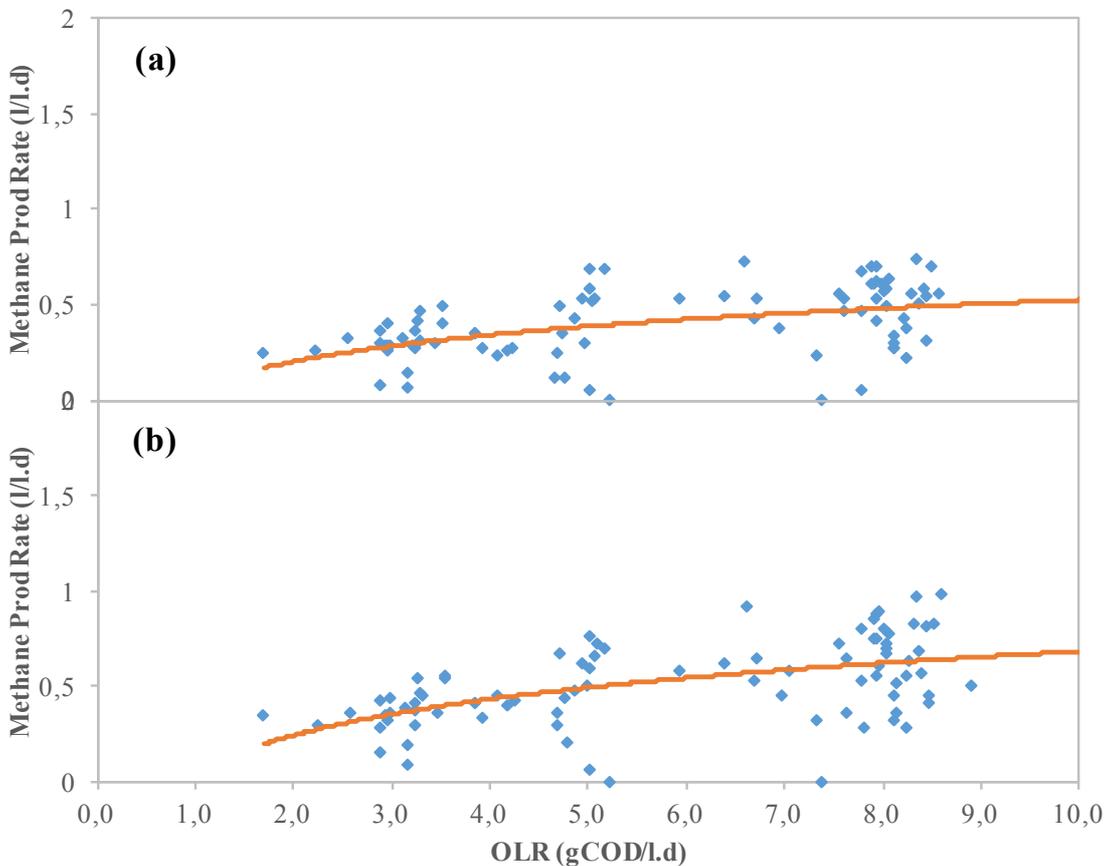


Figure 4.5 Methane production rate in function of OLR in reactor I (a) and II (b).

Methane yield profile (gCOD/gCOD) in function of OLR was shown in **Figure 4.3**. It shows methane yield was decreased with increasing OLR generally. The uniformity and fluctuated methane yield also appear in reactor I and reactor II. **Figure 4.5** shows methane production rate in function of OLR in both reactors. The power trend line was applied and the trend lines do not follow Monod kinetic curve.

4. 1. 3. COD Balance

In this section, COD balance is presented. COD balance was calculated based on dissolved COD feed, outlet, and corresponding methane production as COD by using Equation 2.6. The corresponding methane production as COD obtained using Equation 2.3 by $0.36 \text{ l CH}_4/\text{g COD}$ for temperature 5.5°C in reactor I and reactor II (Equation 2.3). **Figure 4.6** presents the graphs illustrate the COD balance profile in function of OLR profile.

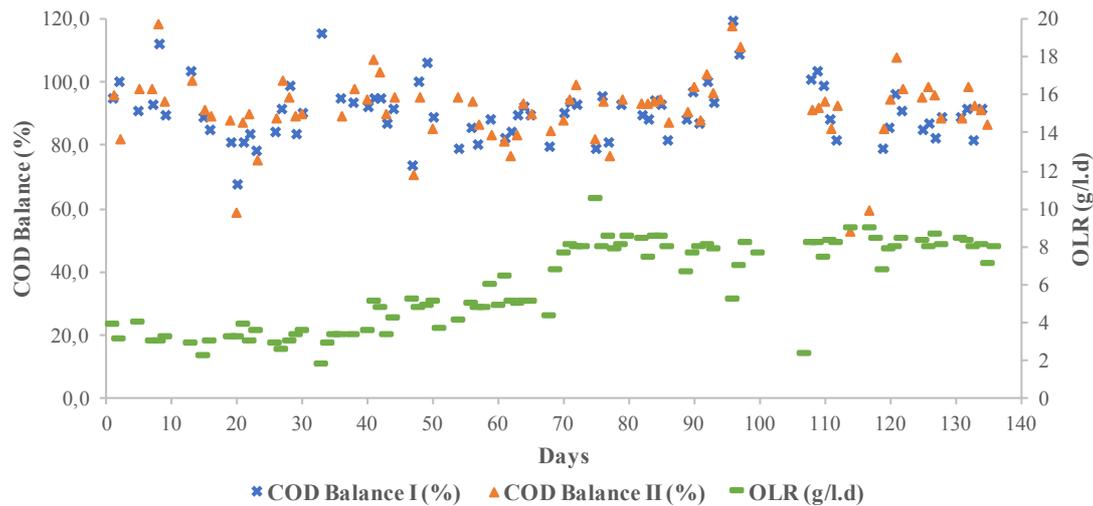


Figure 4.6 The COD balance and OLR profiles of Reactor I and II

Figure 4.6 shows the COD balance observed for both reactors in function of OLR. The averages of COD balances were $90.56 \pm 1.09 \%$ and $91.22 \pm 1.27 \%$ for reactor I and II, respectively. As shown in figure 4.6. The highest COD balance was achieved at day 69 (121,5%) and day 33 (124.9%) for reactor I and reactor II, respectively. While the lowest COD balances were observed at day 47, 73.3% and 70.2% for reactor I and reactor II, respectively. COD biomass/sludge was excluded in the calculation of COD balance because the COD biomass was not obtained during experiment.

4. 2. pH, VFA and Alkalinity Variations

Figure 4.7 shows pH, alkalinity, and VFA variability profiles of reactor I and II during experiment in function of operating period. It is shown that at first 82 days of operation, the pH was quite stable at $\text{pH} = 7$ until drop to $\text{pH} = 6$ at day 83 followed by significant increase of VFA directly after COD feed was relatively high ($>1000 \text{ mg/l}$). 2 g/l of NaHCO_3 was then added to the inlet wastewater for 2 days and decreasing the amount as the pH increased until it was stable around $\text{pH} = 7$. Generally, the VFA increased with increasing OLR.

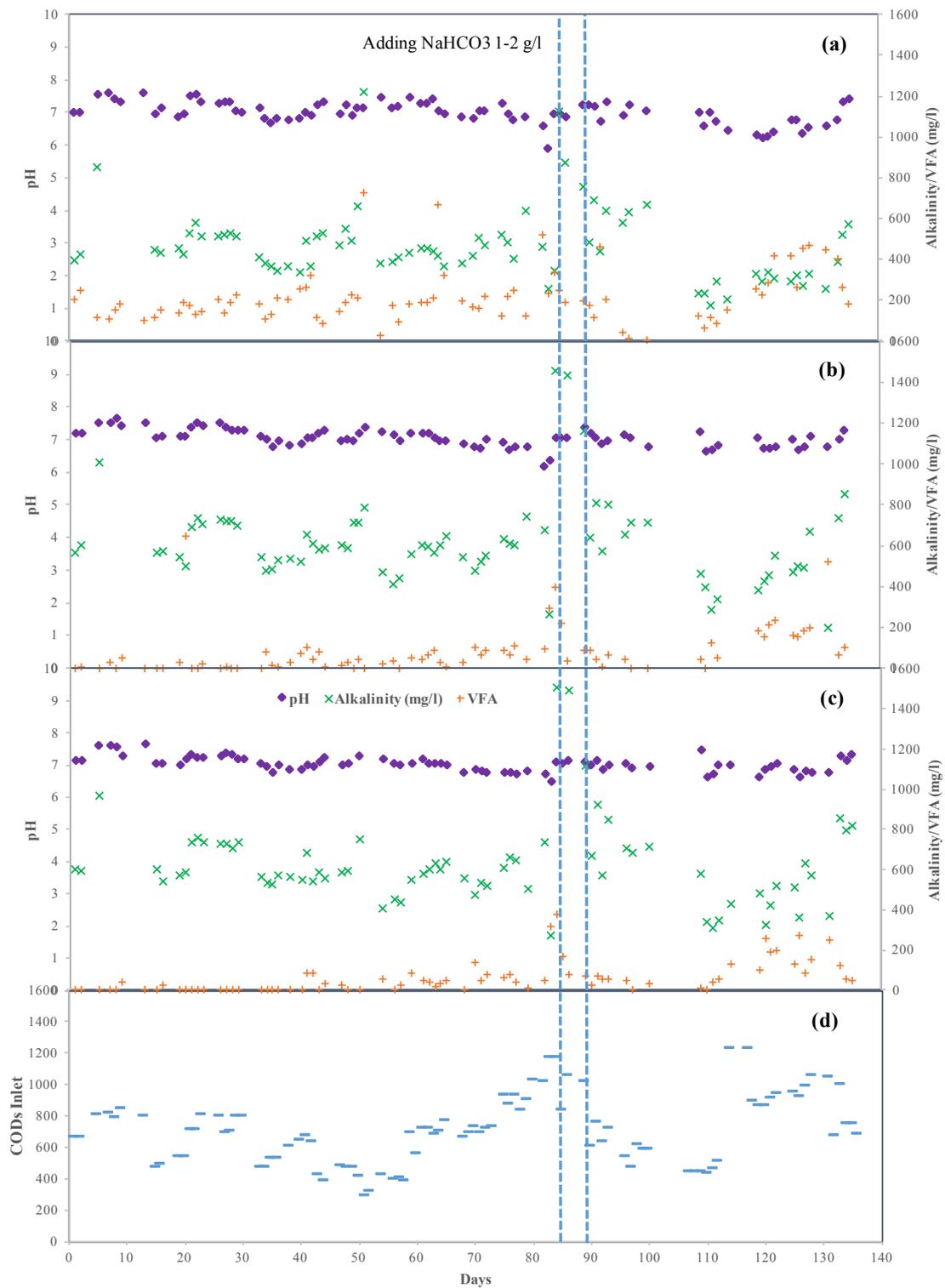


Figure 4.7 pH, alkalinity, and VFA accumulation profiles of; Inlet (a), reactor I (b), reactor II (c); and CODs Inlet (d)

5. Discussions

This chapter discusses the results observed from previous chapter, divided into four sub-chapter: (1) reactor performance, including COD removal efficiency, methane production and COD balance; (2) environmental factors including pH variations, VFA and alkalinity availability; (3) economy and energy recovery; (4) and hydrodynamic conditions.

5. 1. Reactor Performance

Reactor performance will be analysed by evaluating the COD removal efficiency and methane production. In this sub-chapter, reactor performance will be evaluated and analysed by investigating two main parameters; COD recovery and methane production as these parameters reflected the capability of anaerobic treatment for treating medium to high strength municipal wastewater under psychrophilic condition.

5. 1. 1. COD Removal Efficiency

The profiles of COD concentration at the inlet and outlet of the reactors, followed by the COD removal efficiency in function of time and OLR are shown in **Figure 4.1**. During periods of constant OLR, despite of much lower COD removal efficiency than in mesophilic conditions (with optimum of 70% of COD removal efficiency [11]), the COD removal efficiency was very fluctuated without really achieving pseudo-steady state, especially at OLR 8.35 ± 0.16 g COD/l.d. Under psychrophilic conditions, much longer period was required in order to achieve the pseudo-steady state than under mesophilic conditions. This is linked to the low temperature conditions, which affecting the hydrolysis rate. At low temperature, the hydrolysis rate is low and high amount of suspended solids accumulates on the biomass bed, resulting the reduction of volume for active biomass and its specific methanogenic activity [20][28]. Under psychrophilic conditions, biochemical reactions proceed much slower than in mesophilic conditions, as it requires more energy [16], thus deterioration of treatment efficiency also occurred [2].

The lowest COD removal efficiencies were observed at day 97 (6.2%) in reactor I and day 83 (6.9%) and 97 (7.4%) in reactor II respectively, associated to low pH values of the inlet at day 83 and power blackout at the university at day 96 which was a night before. The low pH values at day 83 caused by sudden increased of inlet VFA of 552.2 mg/l at day 82. After adding 2 g/l

of NaHCO_3 the reactors performance was back into normal and start increasing the COD removal efficiency.

Generally, at higher temperature, with decreasing HRT and increasing OLR, the COD removal efficiency is decreased. Decreasing HRT leads to declining of contact time of wastewater with the granules while increasing OLR means increase the turbulence. These two factors lead to less organic matter that is utilized, thus the decreasing COD removal efficiency was also observed. The increasing OLR represents increasing biomass concentration and biomass growth in the reactor until it reached the maximum growth rate (μ_{max}), which is associated with the increasing substrate concentration. This increasing substrate concentration could also lead to the reduction of COD removal efficiency.

Despite of the theory mentioned above, surprisingly, highest COD removal efficiencies achieved at the highest OLR conducted during this study. The highest COD removal efficiencies observed were 58.5% and 57% in reactor I and II, respectively, at day 117 and OLR 8.9 gCOD/l.d with HRT of 3.3 h. 57.1% of COD removal efficiency was also achieved in reactor II at OLR 5.02 ± 0.15 g COD/l.d at HRT of 3.5 h. Overall, despite of the fluctuation, highest COD removal efficiencies achieved were 49.8% and 54.1% for reactor I and II, respectively, at lowest OLR applied, 3.11 ± 0.10 g COD/l.d (39 days), with applied HRT in the range 3.9 – 6.7. 58.7% and 57.1% of COD removal efficiencies were the highest at OLR 5.02 ± 0.15 gCOD/l.d (28 days) with HRT ranged between 1.9 – 3.8 h. While at OLR 8.35 ± 0.16 gCOD/l.d (67 days), HRT applied was between 1.3 – 3.8h, highest COD removal efficiencies observed were 58.5 % and 57.0 % in reactor I and reactor II, respectively. It is shown that relatively high COD removal efficiencies are still able to be achieved in longer period, in such very low temperature, besides the slower physical and chemical reactions in anaerobic degradation, the system also required longer acclimatization period with the decreasing temperature [29]. This was proved by a long term experiment (1243 days), achieving more than 85% of COD removal efficiencies at temperature range 4 – 15 °C with OLRs applied of 3.75 – 10 gCOD/l.d [8].

5. 1. 2. Methane Production

Methane produced, as CH_4 , is equivalent to a certain amount of COD, for this reason, evaluating it is also important regarding the reactor performance. In general, despite of the fluctuations, methane production rate (l/l.d) is increased with the increasing OLR as presented in **Figure 4.2**.

the significant decreased of methane rates were observed when using low concentration of COD inlet or diluted wastewater as feed (day 45-53 and 95-110) and day 83 when the sudden drop of pH followed by significant increase of VFA. Methane yield was determined linearly from the graph of CH₄ production rate and COD removed, adjusted by the method of least squares. From figure 4.3, methane yields observed were 0.224 l CH₄/g COD (0.622 g COD CH₄/g COD removed), 0.186 l CH₄/g COD (0.517 g COD CH₄/g COD removed in reactor I and reactor II, respectively).

Theoretical values were observed; 0.284 l CH₄/g COD and 0.269 l CH₄/g COD for reactor I and reactor II, respectively, which quite higher from the observed methane yield. Despite of the unexpected gas leakage and errors from the analytical method, the methane loss could be due to the high solubility of the methane gas at low temperature which lead to the higher concentration of dissolved concentrations of gaseous compounds, such as dissolved methane in the effluent of reactors [16]. The growth of methanogens was reduced up to 35% from mesophilic condition into psychrophilic condition could also be a reason of methane loss [30]. In addition, the liquid level in the degasser was too low thus the gas passed directly through to the effluent of reactors. If this condition happened, the liquid outlet was adjusted into higher position until the liquid in the degasser back to normal.

The maximum methane yield was observed by 0.6 gCOD CH₄/gCOD_{in}, at 5.1 h of HRT with 2.2 gCOD/l.d of OLR in reactor II. Compared to previous studies with similar settings but higher temperature done by Safitri (2016), the maximum methane yield was 0.6 gCOD CH₄/g COD_{in} at 3.3 h of HRT with 6.0 g COD/l.d of OLR also in reactor II. Generally, methane yield decreased with decreasing HRT with increasing OLR. This is due to the decreasing of contact time of wastewater and granules with decreasing HRT.

Figure 4.5 shows methane production rate in function of OLR in reactor I and reactor II. The trend lines do not follow Monod kinetic, this might be due to a strong temperature effect on the maximum substrate utilization rates of microorganisms [18, 6, 10], which a decreased in maximum specific growth and substrate utilization rates occurred when lowering the operational temperature [16]. Methane production rate increased with OLR, proportional to the amount of organic matter removed in the UASB reactors. Therefore, if COD removal efficiencies diminished, methane production rate also decreased; contrarily, if the efficiency increased then the methane production rate also increased.

5. 1. 3. COD Balance

COD balance is important to be monitored in anaerobic reactor performance as it is a tool reflecting the functioning of the system [3]. **Figure 4.6** shows the COD balance observed for both reactors in function of OLR. The averages of COD balances were 90.56 ± 1.09 % and 91.22 ± 1.27 % for reactor I and II, respectively. As also shown in **Figure 4.6**, the highest COD balance was achieved at day 69 (121,5%) and day 33 (124.9%) for reactor I and reactor II, respectively. While the lowest COD balances were observed at day 47, 73.3% and 70.2% for reactor I and reactor II, respectively. and day 114 for reactor II (52.3%). COD biomass/sludge was excluded in the calculation of COD balance because the COD biomass was not obtained during experiment. The proportion of COD biomass can be assumed to be very low or 0% due to the inhibition of methanogens at low temperature. By using this assumption, the average of COD balance would be still under 100% for both reactors. Generally, the contribution of the COD ‘gaps’ is caused by LCFA-containing wastewater which lead to the high removal efficiency but low CH₄ production rates [3] [11], however, this theory was not really applicable to this case. Despite of the assumption of low to zero COD biomass due to the inhibition of the methanogens under low temperature, under psychrophilic condition, the gaps are highly attributed by the higher concentration of dissolved gaseous compounds, i.e. methane and H₂S appeared in the effluent of the reactor [3] [16].

5. 2. Environmental Factors

In anaerobic process, various VFAs are formed by fermentation process, which then converted into methane in methanogenesis stage. In the other hand, the accumulation of pH can greatly affect the pH, which has a significant impact on the performance of anaerobic process. The decreased of pH will be followed by deterioration of the anaerobic activity. This results to diminishing activity of methanogens as methanogens is the most susceptible to the change of pH. Therefore, the concentrations of VFAs need to be measured and monitored to ensure the concentration do not cross the line of the optimum level. In this study, the concentration of VFA was measured by titration method in daily basis in the unit of mg HAc/l, however, other various VFAs such as propionic and butyric were not conducted.

As shown in **Figure 4.7**, in the first 81 out of 136 days of operation, the pH was settled in the range of 6.8 – 7.4 with average of 7.13 ± 0.03 and average VFA of 193.86 ± 28.64 mgHAc in the inlet, while in both reactors were in the optimum range of 6.7 – 7.7 with average of

7.14±0.03 and 7.09±0.03 and for reactor I and II, respectively. At day 82, the inlet pH suddenly dropped to pH of 6.6 followed by the increase of inlet VFA up to 522.2 mgHAc/l at OLR of 8.5 gCOD/l.d and concentration of inlet COD of 1027.5 mg/l, which the highest in the first 80 days. Decreased pH also occurred in both reactors. At day 83, the methane rates were completely zero in both reactors when the inlet pH drop to 5.9. it is might be due to deviations in the environments provided or the presence of toxic or inhibitory materials in the influent [14]. Long period of storage was also believed contributing to the increase of VFA in the container. It is also presumed to be triggered by a high concentration of inlet COD at high organic loadings that results in an increase the production of VFAs by the acidogenic bacteria. When the increased VFA production rate exceeds the maximum capacity of methanogens to use acetic acid and H₂, excess VFA will start to be accumulated, decreasing the pH. This decreased pH results the reduction of activity of the methanogens, thereby decreasing their use of acetic acid and H₂, leading to a further accumulation of VFAs and further decreased in pH. After zero production of methane, 2 g/l of NaHCO₃ were then added to the inlet wastewater to avoid “sour” anaerobic process that cause the imbalance between the acidogenic bacteria and the methanogens or to assure the stability of the reactors [31].

The inhibition methanogenesis due to the low pH and high VFA also led by the decreased alkalinity. As shown in **Figure 4.7**, the alkalinity in the inlet, and both reactors are also decreased. In consequence, the deterioration of COD conversion to methane occurred, hence, the COD efficiencies during those periods were significantly low. After the pH drop at day 82, the COD removal efficiencies dropped up to 23.1% (35% to 11.9%) and 32.7% (39.6% to 6.9%) in reactor I and II, respectively. Right after the addition of external alkalinity, the COD removal efficiencies started to increase from 11.9% to 15.9% and from 6.9% to 19.9% in reactor I and II, respectively. The performance of both reactors kept increasing ever since despite of the fluctuations. Other than that, the alkalinity in inlet, reactor I and II were relatively stable due to the recirculation [11]. The alkalinity is generated itself as a result of carbonate/bicarbonate buffering system within the anaerobic process. The CO₂ reacted with water to form carbonic acid (H₂CO₃), H₂CO₃ dissociates into bicarbonate (HCO₃⁻) and then to carbonate (CO₃²⁻), thus the addition of NaHCO₃ was no longer necessary.

5. 3. Economy and Energy Recovery

The estimation of electricity and heat generation from the methane produced in this experiment

will be presented in this section. In contrast with aerobic treatment which is generally characterised by high operational cost and energy, and the large fraction of sludge of the waste is converted to another type of sludge, anaerobic treatment produced useful energy in the form of biogas. According to Henze, M. *et. al.* (2008) the production of about 13.5 MJ CH₄ energy/kg COD removed to methane gives 1.5 kWh electricity by assuming 40% electric conversion efficiency and the rest turns into heat. Assuming the optimum COD removal efficiency is also the highest, which was achieved at day 117 in reactor I (58.5% of COD removal efficiency) with 3.3 h of HRT and 8.9 g COD/l.d of OLR. Methane yield of reactor I observed was 0.224 l CH₄/g COD removed, which is equivalent with 0.62 g COD CH₄/g COD removed. With 5.21 gCOD/l.d of COD removed, hence, COD removed that converted to methane was 3.23 g COD/l.d. The average loading of IVAR Grødaland is assumed 5000 m³ per day (4728 m³). Thus, energy generation will be 87210 MJ (24.2 MWh/d) of electricity and 130815 MJ of heat. The detailed energy recovery calculation is presented in Appendix 2.

According to previous study at higher temperature (25°C) by Anissa (2016), which achieved 70% of optimum COD removal efficiency at 6.0 gCOD/l.d of OLR and 3.3 of HRT, the electricity and heat obtained were 81000 MJ (22.5MWh/d) and 121500 MJ, respectively. Compared to these findings, the electricity and heat observed in this study is implausible as the heat and electricity produced in higher temperature should be much higher than those in low temperature. This is due to the high solubility of gaseous compounds i.e., dissolved methane in the effluent of reactors at low temperature as described in section 5. 1. 2. above. High dissolved methane lead to high COD effluent which affect the value of COD removal and thus affecting the heat and electricity calculation.

5. 4. Hydrodynamic Condition

In this section, the overall condition including the behaviour the granules that affected the reactor I and II performances will be discussed. As well as evaluated design and operational condition during experiment.

Sludge wash-out is common occurred in anaerobic process. Generally, the washed out occurred only once in a while. The exception happened at day 99 - 101 which washed out sludge happened three days in a row at such low COD influent. This is due very low F/M ratio. The ratio of food-to-microorganism (F/M) refers to the balance between the food supply and the mass of microorganism in the system [32]. F/M ratio is also called process loading factor, where

defined as the mass of substrate applied per unit time divided by the mass of microorganisms contained in the bioreactor [33]. A low F/M ratio enhances organic removal efficiency and sludge flocculation, thereby increasing the settleability of biomass in the reactor. In the other hand, if the F/M ratio becomes too low, cell growth is limited and sludge de-flocculation may also occur [34]. In addition to that, the lower mixing intensity and poorly substrate-biomass contact resulted from the low COD concentration of influent, which cause low substrate, at low temperature [18], where mixing is a critical part of the anaerobic process as it can reduce resistance to mass transfer and stabilize environmental condition [14]. Rebac et.al. (1995) also added, the treatment of very low strength wastewater at the maximum possible organic loading rate might cause severe hydraulic wash out of the sludge. The reactors' performances were then enhanced after being fed with relatively high influent COD concentration.

Anissa (2016) mentioned sludge blanket expansion occurred in reactor I and reactor II due to the buoyancy of accumulated and entrapped biogas, and liquid friction due to up-flow from inlet and recirculation flow rate. Similar cases also appeared in this study with relatively more frequent. Similar reason mentioned above also applied for this case. When this occurred, gently mix to avoid more washed out granules and eliminate the floating granules, which could result in removing dead zone and improved contact between wastewater and sludge. In addition, at low temperature, too narrow or too high height/diameter ratio in reactor I and reactor II made it worse due to the accumulation of suspended solids (SS) in biomass bed and also dead spaces in the bottom part of the reactors that resulted from the poor distribution of inlet and recirculation liquid, this liquid came into the reactors from the bottom side, instead of the very end bottom part [11].

6. Conclusions

Under psychrophilic condition, i.e. 5.5°C, COD removal efficiency achieved in the range of 6.2% - 58.5% in UASB reactors at the applied HRT between 6.7 h - 1.3 h with gradually increased OLR from 3.11±0.10 to 8.35±0.16 g COD/l.d. Despite of the fluctuation, the maximum biogas potential was achieved in reactor I; 58.5% of COD removal efficiency was achieved at 3.3 h of HRT with 8.9 g COD/l.d of OLR and 3.23 g COD/l.d of COD removed was converted to methane. Overall methane yields obtained were 0.224 l CH₄/g COD, and 0.186 l CH₄/g COD in Reactor I and Reactor II, respectively. These methane yield were not so close to the calculated theoretical values; 0.284 l CH₄/g COD and 0.269 l CH₄/g COD. Using this data, approximately 24.2 MWh/d of electricity and 130815 MJ/d of heat were determined. In low temperature, high dissolved concentration of gaseous compounds i.e. methane and H₂S would be expected in the effluent of the reactors. For similar reason, COD balances obtained in this study have relatively significant “gaps”, with averages of 90.56±1.09 % and 91.22±1.27 % for reactor I and II, respectively.

In this study, pH, VFA and alkalinity concentration affected the methane production in UASB reactors. With the sudden drop of inlet pH to 5.9 followed by significant increase of VFA at high OLR of 8.3gCOD/l.d and relatively high inlet COD concentration, the methane production was completely zero. After addition of 2 g/l of external alkalinity i.e. NaHCO₃, the methane productions started to increase. The addition of this external alkalinity was reduced as the pH was back to the optimum range which the methane production and COD removal efficiency started to increase. As the capability of alkalinity to recover itself from carbonate system and recirculation, the addition of NaHCO₃ was no longer needed.

The performance of the UASB reactors were also affected by the design and operational condition of the reactors. Due to the low temperature, severe sludge wash-out occurred during this study. Sludge blanket expansion also occurred more frequently in both reactors than in high temperatures, this is due the lower mixing intensity and poorly substrate-biomass contact resulted from low COD concentration of the influent which cause low substrate, at low temperature where mixing is a critical part of the anaerobic process as it can reduce resistance to mass transfer and stabilize environmental condition. In addition, re-design of laboratory scale reactors, or increased recirculation flow distribution should be considered.

Anaerobic treatment systems using UASB reactor for treating medium to high strength municipal wastewater under psychrophilic conditions showing a good result as an alternative as pre-treatment. Apart from the fluctuation, in order to achieve optimum COD efficiency and methane production, high HRT and SRT, low organic loading rate and longer period are required.

7. Recommendations

In this study, despite of the fluctuation, relatively high COD removal efficiencies can still be achieved under psychrophilic condition. When even higher COD removal efficiencies and methane production rate are expected, the longer HRT, low organic loading rates and longer period of time need to be considered.

For further studies, measuring the size distribution and the amount of granules at starting and finishing the project are also suggested to estimate the effectiveness of the amount of the granules regarding COD removal efficiency. In addition, the size distribution as well as the community analysis of granules are also beneficial to study, in order to discover the development of the granules. It also important to calculate the methane yield with specific to volume granules.

Apart from the manual calculation of methane fraction, the measurement of gas fraction using gas chromatography is also suggested in order to avoid uncertainties regarding biogas specification. This experiment could also be helpful to detect other possible gases, such as, H₂S. The concentration of other VFAs, such as, propionic and butyric acid are also advantageous to study in order to identify the anaerobic processes with more detail.

Due to high dissolved concentration of gaseous compound at low temperature, it is also important to check into detail the fraction of each compound particularly dissolved methane fraction in the effluent of the reactors. The recommended instrument to measure the dissolved methane is Ruska Gasometer [35] This will be helpful on determining the COD balance during the experiment.

In term of temperature, the treatment at even lower temperature i.e. <5°C is also interesting to investigate as it would be beneficial for extreme cold places such as north pole.

In field application, a post-treatment is required to remove residual COD. By far, anaerobic treatment by UASB reactors perform sufficiently as an alternative pre-treatment during winter time. Last, in order to generate optimum mixing in the reactors, re-design of laboratory scale reactors, or increased recirculation flow distribution should be consider

8. References

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Appendixes

Appendix 1 : Daily Data of Reactor I and Reactor II

Appendix 2 : Energy Recovery Calculation

Appendix 1 : Daily Data of Reactor I and Reactor II

The daily data of reactor I are summarised in Table A.1

Table A.1 Daily measurement of reactor I

Day	pH	sCODin (mg/l)	OLR (gCOD/L.d)	HRT (h)	VFA (mg/l)	Alk (mg/l)	sCODeff (mg/l)	COD rem. (%)	Meth. Rate (l/l.d)	COD Balance (%)	COD removed (g/L.d)	Y (gCODCH4/gCOD)	Theo.meth (l/l.d)
1	7,2	667	3,8	4,2	0	567,4	465	30,3	0,34	114	1,16	0,25	0,32
2	7,2	667	3,1	5,1	3,6	604,9	474	28,9	0,32	120	0,90	0,29	0,23
4									0,26				
5	7,5	815	3,9	5,0	0	1005,5	586	28,1	0,27	107	1,10	0,19	0,32
6													
7	7,5	820	3,0	6,7	31		544,5	33,6	0,28	109	0,99	0,26	0,26
8	7,7	794	3,0	6,4	0		600	24,4	0,39	129	0,73	0,36	0,17
9	7,4	844	3,2	6,4	50		654	22,5	0,14	105	0,71	0,12	0,23
10									0,12				
12									0,20				
13	7,5	799	2,9	6,7	0		596	25,4	0,30	120	0,73	0,29	0,19
14									0,22				
15	7,1	477,5	2,2	5,1	0	569,5	272,5	42,9	0,25	117	0,96	0,31	0,24
16	7,1	490	3,0	4,0	0	571,4	301,5	38,5	0,25	112	1,14	0,23	0,32
19	7,1	543	3,2	4,0	30,4	543,3	306,5	43,6	0,28	106	1,40	0,24	0,38
20	7,1	543	3,2	4,1	646,7	503,9	341	37,2	0,05	92	1,18	0,05	0,40
21	7,4	713,5	3,9	4,4	0	694,8	402	43,7	0,34	100	1,68	0,24	0,46
22	7,5	713	3,0	5,7	0	733,6	409,5	42,6	0,28	102	1,27	0,26	0,34
23	7,4	814	3,5	5,6	19,4	709,8	440,5	45,9	0,29	94	1,59	0,24	0,44
26	7,5	800,5	2,9	6,7	0	732,2	401,5	49,8	0,35	101	1,44	0,34	0,34
27	7,4	698,5	2,6	6,5	8,8	724,7	401	42,6	0,31	111	1,09	0,34	0,26
28	7,3	704	2,9	5,7	0	719,2	505	28,3	0,28	118	0,83	0,27	0,22

Day	pH	sCODin (mg/l)	OLR (gCOD/l.d)	HRT (h)	VFA (mg/l)	Alk (mg/l)	sCODeff (mg/l)	COD rem. (%)	Meth. Rate (l/l.d)	COD Balance (%)	COD removed (g/l.d)	Y (gCODCH4/gCOD)	Theo.meth (l/l.d)
29	7,3	804	3,3	5,8	2,7	697,7	468	41,8	0,30	100	1,38	0,25	0,37
30	7,3	804	3,5	5,5			468	41,8	0,40	106	1,48	0,31	0,36
33	7,1	471	1,7	6,7	0	545,2	357	24,2	0,24	144	0,41	0,39	0,09
34	7,0	471	2,9	3,9	78	480,2			0,07				
35	6,8	531	3,3	3,9	13,5	483,7			0,46				
36	7,0	537	3,2	4,0	9,6	529,2	342	36,3	0,36	120	1,18	0,31	0,29
38	6,8	606	3,3	4,4	29	538,8	357	41,1	0,40	115	1,34	0,34	0,32
40	6,9	648	3,5	4,4	74,8	520,5	345	46,8	0,49	113	1,65	0,39	0,37
41	7,1	675	5,1	3,2	102,1	657,4	444	34,2	0,53	115	1,74	0,29	0,45
42	7,1	639	4,7	3,3	46,1	608,3	423	33,8	0,48	116	1,59	0,29	0,41
43	7,2	429	3,2	3,2	77,4	585,4	273	36,4	0,27	118	1,18	0,23	0,33
44	7,3	390	4,2	2,2	7,6	592,4	288	26,2	0,26	126	1,09	0,17	0,33
45													
46													
47	7,0	483	5,2	2,2	11,1	600,4	354	26,7	0,00	101	1,39	0,00	0,50
48	7,0	471	4,7	2,4	28,4	590,8	375	20,4	0,35	129	0,97	0,20	0,28
49	7,0	474	4,8	2,4	0	715,9			0,10	135	0,00	0,06	0,00
50	7,2	416	5,0	2,0	43,1	711,6	359	13,7	0,04	121	0,69	0,02	0,24
51	7,4	296	3,6	2,0	0	789,1							
52		327							0,26				
53									0,25				
54	7,2	426	4,1	2,5	21,8	467,9	271	36,4	0,22	110	1,48	0,15	0,45
56	7,2	395,5	5,0	1,9	36,3	414,5	273	31,0	0,29	119	1,54	0,16	0,47
57	7,0	407,5	4,7	2,1	0	441,4	267,5	34,4	0,24	113	1,61	0,14	0,50
58		393,5	4,7	2,0					0,11				
59	7,2	694,5	5,9	2,8	47,7	558,9	436	37,2	0,53	107	2,21	0,25	0,60

Day	pH	sCODin (mg/l)	OLR (gCOD/L.d)	HRT (h)	VFA (mg/l)	Alk (mg/l)	sCODeff (mg/l)	COD rem. (%)	Meth. Rate (l/l.d)	COD Balance (%)	COD removed (g/L.d)	Y (gCODCH4/gCOD)	Theo.meth (l/l.d)
60		559	4,9	2,8					0,42				
61	7,2	725	6,4	2,7	46,2	605,1	424	41,5	0,53	100	2,65	0,23	0,73
62	7,2	725	5,0	3,5	68,6	594,9	379	47,7	0,57	103	2,40	0,32	0,59
63	7,1	685,5	4,9	3,3	91,1	569,9	407,5	40,6	0,53	109	2,00	0,30	0,51
64	7,0	702,5	5,1	3,3	26,7	604,2	450	35,9	0,51	111	1,82	0,28	0,47
65	7,0	774,5	5,0	3,7	8,6	650,9	397,5	48,7	0,68	107	2,44	0,38	0,55
67									0,34				
68	6,9	667,5	4,2	3,8	28	548,3	411,5	38,4	0,27	99	1,63	0,17	0,48
69		692	6,7	2,5					0,52			0,22	0,00
70	6,8	730,5	7,6	2,3	100,5	480,8	516	29,4	0,52	108	2,24	0,19	0,65
71	6,8	696,5	8,0	2,1	64,3	520,2	516,5	25,8	0,56	113	2,07	0,19	0,60
72	7,0	728,5	7,9	2,2	84,3	553,7	518,5	28,8	0,60	111	2,29	0,21	0,65
73		736	7,9	2,2									
75	6,9	933	10,4	2,2	88	635,8	595	36,2	0,57	93	3,77	0,15	1,15
76	6,7	878	7,9	2,7	64,3	613	623,5	29,0	0,69	111	2,29	0,24	0,63
77	6,8	930,5	8,5	2,6	109,7	600,4	584,5	37,2	0,54	95	3,15	0,18	0,93
78		834,5	7,8	2,6					0,05				
79	6,8	907	8,0	2,7	43,4	744	650,5	28,3	0,60	107	2,26	0,21	0,64
80		1027,5	8,5	2,9					0,69				
82	6,2	1019,5	8,3	2,9	96,6	678,7	662,5	35,0	0,73	102	2,92	0,24	0,80
83	6,4	1172	7,4	3,8	293,3	266,4	1032,5	11,9	0,00	100	0,88	0,00	0,32
84	7,1	1175	8,5	3,3	397,7	1459,1	988,5	15,9	0,30	106	1,34	0,10	0,44
85	7,1	838	8,4	2,4	221,3	1636,8	612,5	26,9	0,58	108	2,27	0,19	0,66
86	7,1	1064	7,9	3,2	35	1438,9	602,5	43,4	0,70	94	3,45	0,24	0,94
89	7,4	1019	6,6	3,7	91,4	1164,1	586,5	42,4	0,72	101	2,80	0,30	0,70
90	7,2	613,5	7,6	1,9	90,2	641,7	467,5	23,8	0,56	119	1,80	0,20	0,52

Day	pH	sCODin (mg/l)	OLR (gCOD/L.d)	HRT (h)	VFA (mg/l)	Alk (mg/l)	sCODeff (mg/l)	COD rem. (%)	Meth. Rate (l/l.d)	COD Balance (%)	COD removed (g/l.d)	Y (gCODCH4/gCOD)	Theo.meth (l/l.d)
91	7,1	761	7,9	2,3	46,2	806,8	497	34,7	0,61	104	2,76	0,21	0,78
92	6,9	634	8,0	1,9	10,3	571,8	508	19,9	0,57	121	1,60	0,20	0,46
93	7,0	721,5	7,8	2,2	64,4	801,5	501,5	30,5	0,66	112	2,38	0,24	0,65
96	7,2	542,5	5,2	2,5	43,3	658,4	446,5	17,7	0,68	144	0,92	0,37	0,21
97	7,1	477,5	7,0	1,6	0	711,4	448	6,2	0,37	137	0,43	0,15	0,13
98		619,5	8,1	1,8					0,29				
99		593							0,30				
100	6,8	590	7,6	1,9	0	712,9			0,46				
107		451	2,3	4,8									
108		451	8,1	1,3			412,5	8,5	0,27	131	0,69	0,09	0,23
109	7,3	451	8,1	1,3	45	465	414	8,2	0,33	133	0,67	0,11	0,21
110	6,7	438	7,3	1,4	0	397,4	393	10,3	0,23	129	0,75	0,09	0,25
111	6,7	467,5	8,2	1,4	127,9	288	377,5	19,3	0,22	117	1,59	0,07	0,53
112	6,8	513	8,1	1,5	49,7	335,1	370,5	27,8	0,27	108	2,26	0,09	0,74
114		1236	8,9	3,3									
117		1236	8,9	3,3			512,5	58,5					
118		893	8,4	2,6									
119	7,1	865,5	6,7	3,1	181,7	383,4	525	39,3	0,43	94	2,64	0,18	0,78
120	6,8	865,5	7,8	2,7	151,4	429,6	594,5	31,3	0,46	101	2,44	0,16	0,73
121	6,7	919,5	7,9	2,8	209,9	453,4	712	22,6	0,52	110	1,79	0,18	0,53
122	6,8	942	8,4	2,7	233,2	552,9	700	25,7	0,49	105	2,16	0,16	0,65
125	7,0	954	8,2	2,8	162,2	471,6	669,5	29,8	0,42	98	2,46	0,14	0,76
126	6,7	920,5	8,0	2,8	156,6	502,9	668	27,4	0,41	101	2,18	0,14	0,67
127	6,8	994	8,6	2,8	182,7	496,9	636	36,0	0,55	95	3,09	0,18	0,92
128	7,1	1055	8,1	3,1	199,9	667	706	33,1	0,63	101	2,67	0,22	0,75

Day	pH	sCODin (mg/l)	OLR (gCOD/l.d)	HRT (h)	VFA (mg/l)	Alk (mg/l)	sCODeff (mg/l)	COD rem. (%)	Meth. Rate (l/l.d)	COD Balance (%)	COD removed (g/l.d)	Y (gCODCH4/gCOD)	Theo.meth (l/l.d)
131	6,8	1049,5	8,3	3,0	519,1	199,3	734,5	30,0	0,56	101	2,49	0,19	0,73
132		675	8,3	2,0			534,5	20,8	0,37	111	1,72	0,12	0,54
133	7,0	997,5	7,9	3,0	64,8	735,2	600,5	39,8	0,60	95	3,14	0,21	0,89
134	7,3	757,5	8,0	2,3	101,1	854,4	561	25,9	0,49	109	2,09	0,17	0,62
135		751,5	7,0	2,6									
136		685	7,9	2,1									
MEAN	7,06	708,68			77,15	631,23				90,56		0,21	0,48
SE¹	0,03	21,96			12,73	27,62				1,09		0,01	0,03

¹ Standard Error

The daily data of reactor II are summarised in Table A.2

Table A.2 Daily measurement of reactor II

Day	pH	sCODin (mg/l)	OLR (gCOD/l.d)	HRT (h)	VFA (mg/l)	Alk (mg/l)	sCODeff (mg/l)	COD rem. (%)	Meth. Rate (l/l.d)	COD Balance (%)	COD removed (g/l.d)	Y (gCODCH4/gCOD)	Theo.meth (l/l.d)
1	7,1	667	3,8	4,2	0	603,3	433	35	0,42	116	1,35	0,30	0,34
2	7,1	667	3,1	5,1	0	596,8	312,5	53	0,39	102	1,66	0,35	0,39
4									0,33				
5	7,6	815	3,9	5,0	0	966,1	598	27	0,34	114	1,05	0,24	0,29
6													
7	7,6	820	3,0	6,7	0		545,5	33	0,33	114	0,99	0,31	0,25
8	7,6	794	3,0	6,4	0		610	23	0,44	135	0,69	0,41	0,15
9	7,3	844	3,2	6,4	39		647,5	23	0,19	110	0,74	0,17	0,22
10									0,21				
12									0,25				
13	7,7	799	2,9	6,7	0		575,5	28	0,29	117	0,80	0,28	0,21
14									0,3				
15	7,0	477,5	2,2	5,1	0	603,3	256,5	46	0,3	120	1,03	0,37	0,23
16	7,1	490	3,0	4,0	26,2	542,9	276	44	0,35	117	1,29	0,33	0,31
19	7,0	543	3,2	4,0	0	568,4	283,5	48	0,41	113	1,54	0,35	0,36
20	7,2	543	3,2	4,1	1,9	584,1	275	49	0,09	84	1,56	0,08	0,52
21	7,3	713,5	3,9	4,4	0	733,9	408	43	0,41	106	1,65	0,30	0,42
22	7,2	713	3,0	5,7	0	756,6	393,5	45	0,37	109	1,33	0,35	0,31
23	7,2	814	3,5	5,6	0	735,6	373,5	54	0,36	92	1,87	0,29	0,48
26	7,3	800,5	2,9	6,7	0	731,5	377	53	0,43	106	1,52	0,41	0,32
27	7,4	698,5	2,6	6,5	0	728,7	421,5	40	0,37	120	1,02	0,40	0,22
28	7,3	704	2,9	5,7	0	705	434	38	0,35	114	1,13	0,33	0,27

Day	pH	sCODin (mg/l)	OLR (gCOD/l.d)	HRT (h)	VFA (mg/l)	Alk (mg/l)	sCODeff (mg/l)	COD rem. (%)	Meth. Rate (l/l.d)	COD Balance (%)	COD removed (g/l.d)	Y (gCODCH4/gCOD)	Theo.meth (l/l.d)
29	7,2	804	3,3	5,8	0	733,8	411	49	0,45	106	1,61	0,38	0,36
30	7,2	804	3,5	5,5			372	54	0,55	107	1,90	0,43	0,39
33	7,1	471	1,7	6,7	0	559,7	318	32	0,35	154	0,55	0,57	0,08
34	7,0	471	2,9	3,9	0	533,7			0,16				
35	6,8	531	3,3	3,9	0	528,7			0,47				
36	7,0	537	3,2	4,0	0	569	303	44	0,38	114	1,42	0,33	0,34
38	6,8	606	3,3	4,4	0	561,8	306	50	0,55	120	1,62	0,47	0,31
40	6,9	648	3,5	4,4	0,8	549,7	324	50	0,56	115	1,77	0,44	0,36
41	7,0	675	5,1	3,2	80,8	679,3	450	33	0,73	127	1,69	0,40	0,37
42	7,0	639	4,7	3,3	82,8	543,1	402	37	0,68	124	1,75	0,40	0,38
43	7,1	429	3,2	3,2	0,2	586	273	36	0,3	121	1,18	0,26	0,32
44	7,2	390	4,2	2,2	31,3	552,8	267	32	0,4	130	1,32	0,27	0,35
45									0,1				
46									0,11				
47	7,0	483	5,2	2,2	22,1	588	339	30	0	99	1,56	0,00	0,56
48	7,0	471	4,7	2,4	0	594,8	327	31	0,44	124	1,45	0,26	0,39
49		474	4,8	2,4					0,21				
50	7,3	416	5,0	2,0	0	748,7	340	18	0,06	118	0,92	0,03	0,32
51		296	3,6	2,0									
52		327							0,02				
53									0,02				
54	7,2	426	4,1	2,5	50,8	404	274,5	36	0,45	127	1,45	0,31	0,36
56	7,0	395,5	5,0	1,9	1,1	454,4	258	35	0,51	128	1,73	0,28	0,45
57	7,0	407,5	4,7	2,1	21	436,3	263,5	35	0,36	120	1,66	0,21	0,47
58		393,5	4,7	2,0					0,3				
59	7,1	694,5	5,9	2,8	81,5	551,2	383	45	0,59	102	2,66	0,28	0,69

Day	pH	sCODin (mg/l)	OLR (gCOD/l.d)	HRT (h)	VFA (mg/l)	Alk (mg/l)	sCODeff (mg/l)	COD rem. (%)	Meth. Rate (l/l.d)	COD Balance (%)	COD removed (g/l.d)	Y (gCODCH4/gCOD)	Theo.meth (l/l.d)
60		559	4,9	2,8					0,48				
61	7,2	725	6,4	2,7	50,1	578,4	391,5	46	0,62	100	2,94	0,27	0,77
62	7,1	725	5,0	3,5	38,7	603,9	311	57	0,6	95	2,87	0,33	0,69
63	7,0	685,5	4,9	3,3	14,5	633,7	331	52	0,62	103	2,55	0,35	0,60
64	7,1	702,5	5,1	3,3	29,5	597,2	396,5	44	0,66	112	2,20	0,36	0,51
65	7,0	774,5	5,0	3,7	43,5	640,6	365,5	53	0,76	107	2,65	0,42	0,55
67									0,46				
68	6,8	667,5	4,2	3,8	0	555,8	376	44	0,425	105	1,85	0,28	0,48
69		692	6,7	2,5					0,65				
70	6,8	730,5	7,6	2,3	133,7	473	468,5	36	0,65	107	2,74	0,24	0,75
71	6,8	696,5	8,0	2,1	44,5	531,5	486,5	30	0,7	114	2,42	0,24	0,66
72	6,8	728,5	7,9	2,2	74,3	521,5	495	32	0,88	118	2,54	0,31	0,63
73		736	7,9	2,2									
75	6,8	933	10,4	2,2	58,9	608,6	547,5	41	0,85	96	4,30	0,23	1,20
76	6,8	878	7,9	2,7	76,9	657,3	588	33	0,75	109	2,61	0,26	0,69
77	6,7	930,5	8,5	2,6	43,1	645,8	571	39	0,45	91	3,27	0,15	1,00
78		834,5	7,8	2,6					0,28				
79	6,8	907	8,0	2,7	11,7	501,2	601	34	0,8	109	2,70	0,28	0,70
80		1027,5	8,5	2,9					0,83				
82	6,7	1019,5	8,3	2,9	50	736,6	615,5	40	0,97	106	3,31	0,32	0,81
83	6,5	1172	7,4	3,8	319,1	269	1091,5	7	0	105	0,51	0,00	0,18
84	7,1	1175	8,5	3,3	376,7	1502,6	941	20	0,41	105	1,68	0,13	0,52
85	7,1	838	8,4	2,4	164,8	1650,2	566	32	0,82	111	2,74	0,27	0,72
86	7,1	1064	7,9	3,2	74,6	1492	596,5	44	0,89	100	3,49	0,31	0,87
89	7,1	1019	6,6	3,7	70,2	1113,3	526	48	0,92	104	3,19	0,39	0,70
90	7,0	613,5	7,6	1,9	25,3	666,2	438	29	0,73	121	2,16	0,27	0,57

Day	pH	sCODin (mg/l)	OLR (gCOD/l.d)	HRT (h)	VFA (mg/l)	Alk (mg/l)	sCODeff (mg/l)	COD rem. (%)	Meth. Rate (l/l.d)	COD Balance (%)	COD removed (g/l.d)	Y (gCODCH4/gCOD)	Theo.meth (l/l.d)
91	7,1	761	7,9	2,3	68,8	924	465	39	0,75	105	3,09	0,26	0,82
92	6,9	634	8,0	1,9	52,2	573,3	489	23	0,73	124	1,84	0,25	0,50
93	7,0	721,5	7,8	2,2	53,6	849,5	486	33	0,8	115	2,54	0,29	0,65
96	7,1	542,5	5,2	2,5	45,4	707,9	433	20	0,7	143	1,04	0,38	0,23
97	6,9	477,5	7,0	1,6	0	680,4	442	7	0,46	140	0,52	0,18	0,15
98		619,5	8,1	1,8					0,52				0,00
99		593							0,47				
100	6,9	590	7,6	1,9	28,9	716,1			0,37				0,00
107		451	2,3	4,8									0,00
108		451	8,1	1,3			359	20	0,33	121	1,66	0,11	0,53
109	7,5	451	8,1	1,3	9	578	343,5	24	0,45	122	1,94	0,15	0,59
110	6,7	438	7,3	1,4	0,11	338,1	357,5	18	0,32	125	1,35	0,12	0,43
111	6,7	467,5	8,2	1,4	37	312,5	352	25	0,29	114	2,04	0,10	0,66
112	7,0	513	8,1	1,5	51,4	344,5	411,5	20	0,36	119	1,61	0,12	0,51
114	7,0	1236	8,9	3,3	126,6	432,1	647	48				0,00	0,00
117		1236	8,9	3,3			531	57	0,51	70	5,08	0,16	1,54
118		893	8,4	2,6					0,69				
119	6,7	865,5	6,7	3,1	100,6	479	547	37	0,53	101	2,47	0,22	0,69
120	6,8	865,5	7,8	2,7	255,2	322,4	649	25	0,53	110	1,95	0,19	0,57
121	7,0	919,5	7,9	2,8	190,2	418,3	805	12	0,56	122	0,99	0,20	0,29
122	7,1	942	8,4	2,7	198	519,2	739	22	0,57	112	1,81	0,19	0,53
125	6,9	954	8,2	2,8	129,5	509,8	726,5	24	0,56	109	1,97	0,19	0,57
126	6,6	920,5	8,0	2,8	268,2	362,9	707,5	23	0,61	113	1,84	0,21	0,52
127	6,8	994	8,6	2,8	85,1	626,9	628,5	37	0,99	109	3,16	0,32	0,77
128	6,8	1055	8,1	3,1	153,7	570,1	649	38	0,78	101	3,11	0,27	0,82

Day	pH	sCODin (mg/l)	OLR (gCOD/l.d)	HRT (h)	VFA (mg/l)	Alk (mg/l)	sCODeff (mg/l)	COD rem. (%)	Meth. Rate (l/l.d)	COD Balance (%)	COD removed (g/l.d)	Y (gCODCH4/gCOD)	Theo.meth (l/l.d)
131	6,8	1049,5	8,3	3,0	249,9	365,2	635	39	0,83	101	3,28	0,28	0,85
132		675	8,3	2,0			521	23	0,63	119	1,88	0,21	0,53
133	7,3	997,5	7,9	3,0	125,5	851,7	618,5	38	0,86	106	3,00	0,30	0,75
134	7,1	757,5	8,0	2,3	56,9	795,9	511,5	32	0,68	109	2,61	0,23	0,72
135	7,3	751,5	7,0	2,6	44,7	818,9	477	37	0,58	105	2,57	0,23	0,71
136		685	7,9	2,1									
MEAN	7,04	708,68			57,31	636,57				91,22		0,27	0,49
SE¹	0,03	21,96			8,99	28,23				1,27		0,01	0,03

Appendix 2 : Energy Recovery Calculation

Energy recovery was calculated under optimum biogas potential condition in reactor I, as shown below.

Table A.2 Parameter conditions used in energy recovery calculation

Parameter	Mark(s)
Conversion a)	13.5 MJ CH ₄ energy/kg COD removed to methane gives 1.5 kWh electricity t 40% electric conversion efficiency and the rest turns into heat
OLR b)	8.9 g COD/l.d
HRT b)	3.3 H
COD removal efficiency b)	58.5%
Methane yield c)	0.224 l CH ₄ / COD (0.62 g COD CH ₄ /g COD removed)
COD Removed b)	5.21 g COD/l.d (3.23 g COD/l.d was converted to methane)
Q d)	5000 m ³ /d
Electricity generation e)	81000 MJ/d (22.5 MWh/d)
Heat generation f)	121500 MJ/d

a) Conversion was referred to Henze *et.al* (2008).

b) The data was taken from daily measurement reactor I in Table A.1 (day 117).

c) Methane yield was determined graphically in Figure 4.3 from values of methane production rate in function of COD removed.

d) The value is the approximate average hydraulic loading at IVAR Grødalund.

e) Electricity generation was calculated using equation below.

$$Electricity \left(\frac{kWh}{d} \right) = COD_{removed\ to\ CH_4} \times Q_v \times 1.5\ kWh = 3.23 \frac{kgCOD}{m^3.d} \times 5000\ m^3 \times 1.5\ kWh$$

$$Electricity \left(\frac{kWh}{d} \right) = 24225 \frac{kWh}{d} = 24.2\ MWh/d$$

$$Electricity \left(\frac{MJ}{d} \right) = \frac{40\% \times 24225\ kWh/d}{1.5\ kWh} \times 13.5\ MJ = 87210\ MJ/d$$

f) Heat generation was calculated using equation below.

$$Heat \left(\frac{MJ}{d} \right) = \frac{60\% \times 24225\ kWh/d}{1.5\ kWh} \times 13.5\ MJ = 130815\ MJ/d$$