



UNIVERSITI PUTRA MALAYSIA

***ENHANCEMENT OF METHANE PRODUCTION FROM ANAEROBIC
DIGESTION OF GREASE TRAP WASTE***

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DIGESTION OF GREASE TRAP WASTE**

By

NAZAITULSHILA BINTI RASIT

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in
Fulfilment of the Requirements for the Degree of Doctor of Philosophy**

June 2016

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

ENHANCEMENT OF METHANE PRODUCTION FROM ANAEROBIC DIGESTION OF GREASE TRAP WASTE

By

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June 2016

Chairman : Azni Bin Idris, PhD
Faculty : Engineering

Research on waste recovery option as part of waste management strategy has become increasingly important for environmental sustainability. In anaerobic digestion, less attention has been focused on the digestion of grease trap waste (GTW) as a single substrate may be due to high lipid content contained in GTW that may cause inhibition effects resulted from long chain fatty acids (LCFA) accumulation. The inhibition behaviour of GTW as a single substrate in anaerobic digestion using continuous stir tank reactor (CSTR) with appropriate operating conditions are investigated. The aim of this study is to investigate various strategies to enhance methane production from anaerobic digestion of GTW. Thus, the objectives set up were to evaluate the influence of acclimated and non-acclimated inoculum, investigate the feasibility of glycerine supplementation, evaluate the performance of different feeding pattern strategy and model the reaction kinetics of grease trap waste anaerobic digestion. Four reactors were set-up as R_{AB} for acclimated (LCFA) to biomass reactor, R_{GS} for glycerine supplementation reactor, R_{12H} for feeding every 12 hours reactor and $R_{control}$ for control and comparison with other reactors. The experimental works were conducted in two phases, which is the first phase is a start-up operation, where the reactors were run in batch experiments. Then, the second phase is the semi-continuous feeding operation with increasing organic loading rate (OLR) ranging from 1.3-3.6 gCOD/L.day. The performance of all reactors was evaluated based on methane composition, production rate and yield.

The strategy of acclimated inoculum to LCFA (R_{AB}) and glycerine supplementation (R_{GS}) were demonstrated to enhance methane production performance. In R_{AB} reactor, there is no lag phase was observed during the start-up of the process, where the other reactors using non-acclimated inoculum were observed (R_{AB} , R_{12H} and $R_{control}$). Without lag phase, R_{AB} reactor showed the highest methane production performance as compared to $R_{control}$ when methane composition recorded was 71% with 0.455 LCH₄/L.day of methane production rate and 0.22 LCH₄/gCOD_{removed} of methane yield at OLR of 2.2 gCOD/L.day. In R_{GS} reactor, a reduction of 5 days lag phase was observed as compared to $R_{control}$ which experienced 9 days lag phase with 67% methane composition, 0.376 LCH₄/L.day of methane production rate and 0.19

$\text{LCH}_4/\text{gCOD}_{\text{removed}}$ of methane yield at OLR of 2.2 gCOD/L.day. $R_{12\text{H}}$ (2 times feeding per day) reactor has shown less efficiency in terms of methane enhancement as compared to R_{control} (1 time feeding per day) reactor. Increasing feeding frequency does not enhance methane production when methane composition recorded was 57% with 0.269 $\text{LCH}_4/\text{L.day}$ of methane production rate and 0.14 $\text{LCH}_4/\text{gCOD}_{\text{removed}}$ of methane yield during OLR of 2.2 gCOD/L.day as compared to R_{control} when methane composition recorded was 60% with 0.287 $\text{LCH}_4/\text{L.day}$ of methane production rate and 0.14 $\text{LCH}_4/\text{gCOD}_{\text{removed}}$ of methane yield at the same OLR. The experimental results were well fitted in Monod and Contois kinetic models. High relationship between experimental and simulated results were obtained with high correlation coefficients (R^2) ranging from 0.96-0.98. Overall, the efficient strategies to enhance methane productions were evaluated from R_{AB} and R_{GS} reactor when overall methane enhancement were 42% and 25%, respectively. Accordingly, the kinetic models used in the study can be used to foresee the performance of the reactor for anaerobic digestion system treating GTW.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENINGKATAN PENGHASILAN METANA DARIPADA PENCERNAAN ANAEROBIK PERANGKAP SISA GRIS

Oleh

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Penyelidikan mengenai opsyen pemulihan sisa sebagai sebahagian daripada strategi pengurusan sisa telah menjadi semakin penting untuk kelestarian alam sekitar. Di dalam pencernaan anaerobik, fokus terhadap pencernaan sisa perangkap gris (GTW) sebagai substrat tunggal kurang diberi perhatian mungkin kerana kandungan lemak yang tinggi terkandung di dalam GTW yang boleh menyebabkan kesan perencatan hasil daripada pengumpulan asid lemak rantai panjang (LCFA). Tingkah-laku penghalang GTW sebagai substrat tunggal dalam pencernaan anaerobik menggunakan reaktor teraduk berterusan (CSTR) dengan keadaan operasi yang sesuai dikaji. Kajian ini bertujuan untuk mengkaji pelbagai strategi untuk meningkatkan pengeluaran metana daripada pencernaan anaerobik GTW. Oleh itu, objektif yang telah ditetapkan adalah untuk menilai pengaruh inokulum diaklimatisasi dan tidak diaklimatisasi, menyiasat kebolehsanaan tambahan gliserin, menilai prestasi bagi strategi corak pemakanan yang berlainan dan memodelkan tindakbalas kinetik pencernaan anaerobik GTW. Empat buah reaktor telah disediakan sebagai R_{AB} bagi reaktor diaklimatisasi (LCFA) terhadap biomas, R_{GS} bagi reaktor tambahan gliserin, R_{12H} bagi reaktor pemakanan setiap 12 jam dan $R_{control}$ bagi kawalan dan perbandingan dengan reaktor-reaktor lain. Kerja-kerja ujikaji telah dijalankan dalam dua fasa, fasa yang pertama iaitu operasi permulaan, di mana reaktor telah dijalankan dalam ujikaji berkelompok. Kemudian, fasa kedua adalah operasi pemakanan separa berterusan dengan peningkatan kadar beban organik (OLR) dari lingkungan 1.3-3.6 gCOD/L.day. Prestasi kesemua reaktor telah dinilai berdasarkan komposisi, kadar pengeluaran dan penghasilan metana.

Strategi inokulum diaklimatisasi terhadap LCFA (R_{AB}) dan tambahan gliserin (R_{GS}) telah menunjukkan prestasi peningkatan pengeluaran metana. Di dalam reaktor R_{AB} , tiada fasa sela diperhatikan semasa proses permulaan, di mana telah diperhatikan terdapat di dalam reaktor lain yang menggunakan inokulum tidak diaklimatisasi (R_{AB} , R_{12H} and $R_{control}$). Tanpa fasa sela, R_{AB} reaktor menunjukkan prestasi pengeluaran methana tertinggi dibandingkan dengan $R_{control}$ apabila komposisi metana direkodkan adalah 71% dengan kadar pengeluaran metana sebanyak 0.455 LCH₄/L.hari dan penghasilan metana sebanyak 0.22 LCH₄/gCOD_{disingkirkan} pada OLR 2.2 gCOD/L.hari. Di dalam R_{GS} reaktor, pengurangan selama 5 hari fasa sela telah diperhatikan apabila

dibandingkan dengan R_{control} yang telah mengalami fasa sela selama 9 hari dengan komposisi metana sebanyak 67%, kadar pengeluaran metana sebanyak 0.376 $\text{LCH}_4/\text{L.hari}$ dan penghasilan metana sebanyak 0.19 $\text{LCH}_4/\text{gCOD}_{\text{disingkirkan}}$ pada OLR 2.2 $\text{gCOD}/\text{L.hari}$. Reaktor $R_{12\text{H}}$ (pemakanan 2 kali sehari) telah menunjukkan kurang kecekapan dari segi penambahan metana apabila dibandingkan dengan reaktor R_{control} (pemakanan 1 kali sehari). Peningkatan frekuensi pemakanan tidak menambahkan pengeluaran metana apabila komposisi metana direkodkan adalah 57% dengan kadar pengeluaran metana sebanyak 0.269 $\text{LCH}_4/\text{L.hari}$ dan penghasilan metana sebanyak 0.14 $\text{LCH}_4/\text{gCOD}_{\text{disingkirkan}}$ pada OLR 2.2 $\text{gCOD}/\text{L.hari}$ dibandingkan dengan R_{control} apabila komposisi metana direkodkan adalah 60% dengan kadar pengeluaran metana sebanyak 0.287 $\text{LCH}_4/\text{L.hari}$ dan penghasilan metana sebanyak 0.14 $\text{LCH}_4/\text{gCOD}_{\text{disingkirkan}}$ pada OLR yang sama. Keputusan ujikaji sangat sesuai bagi model Monod dan Contois. Hubungkait yang tinggi di antara keputusan ujikaji dan simulasi telah diperolehi dengan pekali kolerasi (R^2) dalam lingkungan 0.96-0.98. Secara keseluruhan, strategi kecekapan untuk menambah pengeluaran metana telah dinilai daripada reaktor R_{AB} dan R_{GS} apabila keseluruhan penambahan metana masing-masing adalah 42% dan 25%. Selaras dengan itu, model kinetik yang digunakan dalam kajian ini boleh digunakan untuk meramal prestasi reaktor bagi sistem pencernaan anaerobik merawat GTW.

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“May Allah bless us with his Taufik and Hidayah.

May we benefit from the knowledge He has given us.

May we always be under His Protection and Guidance.

May He forgive us for our sins, those we know and those we do not know.

May He place us on the righteous path and steadfast our Imans.

May He shower our one and true Prophet Muhammad Alaihisalam and his family and followers, with eternal blessings.

Amin ya rabbal-alamin”

I certify that a Thesis Examination Committee has met on 30 June 2016 to conduct the final examination of Nazaitulshila binti Rasit on her thesis entitled "Enhancement of Methane Production from Anaerobic Digestion of Grease Trap Waste" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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LIST OF ABBREVIATIONS AND SYMBOLS

Abbreviations

AD	Anaerobic digestion
BMP	Biochemical methane potential
C	Carbon
COD	Chemical oxygen demand
CSTR	Continuous stir tank reactor
FID	Flame ionization detector
FOG	Fat, oil and grease
GC	Gas chromatography
GTW	Grease trap waste
H	Hydrogen
HRT	Hydraulic retention time
IWK	Indah water consortium
LCFA	Long chain fatty acid
O&G	Oil & grease
OLR	Organic loading rate
POMS	Palm oil mill sludge
SRT	Sludge retention time
TAN	Total ammonia carbon
TCD	Thermal conductivity detector
TS	Total solid
UASB	Upflow anaerobic sludge blanket
VFA	Volatile fatty acid
VS	Volatile solid

Symbols

X	Microorganism concentration
t	Time
μ	Specific growth rate
μ_{\max}	Maximum specific growth rate
K_d	Specific microorganism decay rate
Y	Growth yield coefficient
q	Specific utilization rate
Q	Flow rate
V	Volume of the reactor
X_0	Influent biomass concentration
X	Effluent biomass concentration
S	Effluent substrate concentration

S_0	Influent substrate concentration
Y	Yield coefficient
B	Kinetic parameter
K_B	Saturation value constant



CHAPTER 1

INTRODUCTION

This chapter describes the introduction to anaerobic digestion overview focusing on the digestion of grease trap waste (GTW). Interest and concern for current study have been thoroughly described in detail in problem statement. Then, the objectives are defined and scope of study is further elaborated in this chapter.

1.1 Research Background

Addressing environmental pollution problems is a challenge that needs to be addressed effectively when conventional methods such as treatment and waste disposal only meant for pollutants elimination. An approach towards sustainable environment in the future is now focusing on waste recovery option when the waste is no longer to be disposed but it is transformed into useful resources (Williams, 2013). Waste recovery option is part of a waste management strategy that can reduce waste and one of the ways to achieve this goal is through biological treatment methods. Anaerobic digestion is one of the sustainable methods when the waste is utilized as a useful resource to produce beneficial by-product and renewable energy (Evans, 2013). In addition, the other advantages obtained from anaerobic process are the low comparative level of energy requirement, investment cost and sludge production (Khanal, 2008; Williams, 2013). The implementation of anaerobic digestion technology has been practiced worldwide to reduce and stabilize wastes such as wastes from domestic, agriculture, livestock, and municipal (Evans, 2013; Wheatley, 1991).

In Malaysia, the development of anaerobic treatment is more focused on the palm oil sector and the research on the anaerobic treatment of palm oil mill effluent is abundant (Abdurahman et al., 2012; Chin et al., 2013). The study of the potential use of other substrates available in Malaysia other than palm oil mill effluent is also increasing. For instance, the anaerobic digestion of landfill leachate (Ghasimi et al., 2009) and food waste (Tanimu et al., 2014) have been reported for their potential of producing biogas. However, study on the digestion of GTW is still less reported. In Malaysia, GTW management for commercial areas is the responsibilities of the municipal council and for domestic area; it is under the national sewerage company. Currently, there is lack of proper treatment of the entire content of GTW. Usually, the content of oil and grease are separated and will be discarded as solid waste and the other content is removed through drainage. Poor maintenance of grease trap has led to the deposition of high lipid wastewater and the flow into watercourses will result in adverse effects to human health, water courses and the environment. Indah Water Konsortium (IWK), a national

sewerage company, in their sustainability report (Year 2012-2013) stated that 20499 blockages due to oil and grease deposition were reported in 2013 (IWK, 2013). Therefore, the current study is significant to investigate the GTW potential in order to reduce the volume of waste produced and to serve as the useful resources for beneficial use.

Many studies reported the consumption of waste lipid with two or more substrates to increase biogas production known as co-digestion. For co-digestion of other substrate with waste lipid such as slaughterhouse waste (Martínez et al., 2012), grease interceptor waste (Wang et al., 2013) and food industry wastewater (Fernandez et al., 2005), the resultant showed the potential of higher methane production but the selection of suitable inoculum and substrate should be attentively selected and since the characteristics of each resources used in the study are vary considerably, therefore it is important to investigate other suitable resources. The digestion of waste lipid as a single substrate may be less favourable due to inhibition factors and risk as described. Furthermore, the studies are still lacking on information regarding the digestion of grease trap waste (GTW) as a single substrate. To provide additional views if anaerobic waste processing plant receives different types of waste which sometimes contains a high lipid content, it is therefore important to find improved strategy to deal with the lipid inhibition in GTW and improve its degradability prior for process improvement and to reduce the above mentioned risk.

In theory, when complex organic substrates degraded anaerobically, lipid is a substance that can yield more biogas with higher methane content as compared with glucose and protein (Alves et al., 2009). Based on standard temperature and pressure (STP), a comparison was made between 1 g of oleate and 1 g of glucose. It showed methane productions were 1.01 L and 0.37 L, respectively (Cavaleiro et al., 2008). Nevertheless, the exploitation of waste with high lipid content is not widely used as a main substrate because of several inhibition factors. For instance, in order to enhance biogas production of low biodegradability substrate, waste lipids is ideal for implementation as co-substrate. Several researchers proved that high lipid waste such as grease trap waste (GTW) when co-digested with low biodegradability substrate yielded higher biogas production than single substrate. For example, GTW when co-digested with municipal wastewater sludge showed an improvement of 67% of biogas production (Razaviarani et al., 2013a), while an improvement of 77% of methane yield when co-digested with thickened waste activated sludge (Wang et al., 2013). Moreover, co-digestion of GTW with primary sludge resulted in 2.95 times higher methane yield than primary sludge alone (Kabouris et al., 2009). Even though the ability of waste lipid to enhance anaerobic co-digestion in producing biogas has long been discovered, recent studies are being projected to enhance lipid degradation, overcome the inhibition effects and increase methane production of single digestion of waste lipid such as GTW.

1.2 Problem Statement

Anaerobic digestion is one of the potential processes of waste lipid recovery for beneficial use to produce biogas. However, despite its promising approach in increasing biogas yield, lipid hydrolysis may be inhibited by the formation of long chain fatty acid (LCFA) and destabilization occur during methanogenesis phase and hence reduces its biodegradability potential (Angelidaki & Ahring, 1992; Pereira et al., 2005). Oleate and palmitate became known as the culprit when their accumulation known as the main cause of inhibition. It was reported that their accumulation in low level over than 100 mg/L was inhibited anaerobic digestion of sewage sludge (Martinez et al., 2012), and cattle manure (Angelidaki and Ahring, 1992; Palatsi et al., 2009). The destabilization is identified by the presentation of lag phase during methane formation and the formation of biomass floatation (Hwu et al., 1998; Long et al., 2011; Palatsi et al., 2010). As a result, the methane production is low and slows which leads to potential failure of the digester; the risk that the plant operator would not be willing to take.

The inhibition scenario occurred when dealing with substrate with high lipid content was reported by several researchers. Hidalgo and Martin-Marroquin (2014) experienced a 13 days of lag phase and low methane yield (0.04 LCH₄/gVS.d) when waste vegetable oil was treated in batch anaerobic process. Pereira et al. (2001) treated skim milk in their study observed that oleate concentration of 100 mg/L was inhibited batch process when lag phase occurrence of 5 days with methane composition of 69%. Shock load of fat-rich dairy wastewater at 900 mg/L of oleate and palmitate does not exhibit any methane production. The methane production affected the start-up of the process by having a lag phase and affected the overall process performance. These finding suggested that methane production is low and slow due to the lipid inhibition during hydrolysis process and thus substrate with high lipid content is less favourable substrate to be used in anaerobic digestion. Therefore, it is important to investigate a method to deal with lipid inhibition in order to reduce the inhibition scenario and enhance methane production using high lipid substrate.

There are many methods dealing with lipid inhibition such as inoculum acclimatization, feeding sequence, supplementation addition, enzymatic hydrolysis, easily degradable substrates addition and saponification. Nielsen & Ahring (2006) studied oleate pulses onto thermophilic anaerobic reactors and reported the pulses effects on degradation of volatile fatty acids (VFA) and also reduction of the lag phase during hydrolysis stage. In reactor experiments with semi-continuously feed, the lag phase as the consequences of long chain fatty acids (LCFA) accumulation has reduced from 4-5 days to 2-3 days with the addition of bentonite and fibers and indicate higher LCFA precipitation (Palatsi et al., 2009). Using lipase as pre-treated enzyme, an anaerobic digester treating dairy effluents was found to increase biogas production approximately 445±29 mL as compared to effluents not exposed to enzyme pre-treatment (Mendes et al., 2006). Battimeli et al. (2010) studied the biodegradability of saponified fatty wastes which exhibits an improvement on biogas production during hydrolysis stage in batch tests as compared to unsaponified wastes with biogas yield

was 4 times higher. However, the studies on several factors, such as the usage of acclimatization inoculum, feeding patterns and supplementation of glycerine on GTW anaerobic treatment, were less reported.

Glycerine is a by-product produced in a large quantity from biodiesel production industry (López et al., 2009). It is estimated approximately 10 kilogram of impure glycerine will be produced from 100 kilogram of biodiesel production (Chi et al., 2007). Because of its mass production in the industry, market value of glycerine was low. Besides its numerous applications such as soaps and pharmaceutical products, currently, crude glycerine conversion to beneficial product is being actively researched. Therefore, this study was motivated by its low price, high availability and beneficial values. Not exceeding 6% (v/v), glycerine supplementation was found to increase methane production from anaerobic digestion of pig manure and maize silage (Amon et al., 2006). Razaviarani et al (2013a) researched on the same application with municipal wastewater as substrate and Fountoulakis et al. (2010) with sewage sludge as substrate also reported positive findings on methane production enhancement. However, recent research on application of crude glycerine to enhance methane production from GTW anaerobic digestion is less reported.

Oily effluents and sludge such as GTW is considered as potential substrates to increase biodegradability and bioavailability in methane production of anaerobic treatment. The reported inhibition effects such as lag phase during hydrolysis stage and sludge floatation do not restrain the exploitation of lipid wastes in anaerobic digestion to produce biogas. Additional research is needed to determine the lipid degradation of single substrates. It was found that lipid waste characteristics are diverse depending on its origin. Thus, this study is needed to investigate the wide range of lipid wastes characteristics as the results will provide an overview of potential waste recovery, besides being considered as a waste and then discarded to landfill or flow into watercourses. To accomplish the concept of low cost waste recovery strategy, resources used in this study were taken from the commercial area that are no longer used and appropriate strategies and operating condition parameters were used to study the effects in enhancing the production of methane. Therefore, the current study is a noteworthy effort to evaluate recent trends in this field and will contribute to additional information to the anaerobic plant operators when dealing with high lipid

1.3 Research Objectives

This study aims to investigate various strategies to enhance methane production from anaerobic digestion of grease trap waste (GTW). The specific objectives of this study are as follows:

- (i) To evaluate the influence of acclimated and non-acclimated inoculum on methane production from anaerobic digestion of grease trap waste.
- (ii) To investigate the feasibility of glycerine supplementation for the anaerobic digestion of grease trap waste.
- (iii) To evaluate the performance of different feeding strategies for the anaerobic digestion of grease trap waste.
- (iv) To model the reaction kinetics of grease trap waste anaerobic digestion.

1.4 Scope of the Study

The following criteria form the principal for the scope of the study:

1. For each abovementioned objective (i) to (iii), the anaerobic digestion of grease trap waste (GTW) was performed by continuous stir tank reactor (CSTR) under controlled condition as the following:
 - i. pH ranging from 6.8-7.2.
 - ii. Temperature $37\pm 1^{\circ}\text{C}$.
 - iii. Agitation 100 rpm.
2. The materials selection of this study were grease trap waste (GTW) as main substrate and palm oil mill sludge (POMS) as inoculum for the anaerobic digestion processes.
3. The characterization of materials will be analysed based on pH, total solids (TS), volatile solid (VS), chemical oxygen demand (COD), lipid content, volatile fatty acids (VFAs) and long chain fatty acids (LCFA).
4. Four reactors used in the study were tagged as acclimatized biomass reactor (R_{AB}), glycerine supplementation reactor (R_{GS}), 12-hour feeding reactor (R_{12H}) and control reactor ($R_{control}$).

5. The efficiency of reactor for each experiment was evaluated based on methane composition, methane production rate and methane yield. While the reactor stability was determined based on chemical oxygen demand (COD), pH, alkalinity, volatile fatty acids (VFA) and long chain fatty acids (LCFA).
6. Subsequently, kinetic parameters and kinetic models determination based on objective (iv) were described using Monod, Contois and Stover-Kincannon equation.



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