UNIVERSITI PUTRA MALAYSIA

EFFECTS OF CARBON TO NITROGEN RATIO AND ORGANIC LOADING RATE ON BIOGAS METHANE PRODUCTION FROM ANAEROBIC DIGESTION OF FOOD WASTE

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By

MUSA IDRIS TANIMU

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Master of Science

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DEDICATION

Dedicated to my beloved parents.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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September 2014

Chairman: **Tinia Idaty Mohd Ghazi, PhD**
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This study has been concerned with an investigation of biogas methane generation from the abundant food waste (FW) available in Malaysia by anaerobic digestion (AD) process at mesophilic (35^{o}C) conditions. Methane recovered from FW digestion is a source of renewable energy for heat and electricity generation. The biogas generation was carried out in batch digesters and then in 2 semi-continuous mode digesters. The semi-continuous mode digesters consists of a 1000L pilot scale (PS) digester up-scaled from a 10L laboratory scale (LS) anaerobic digester. The key parameters investigated were the effect of carbon to nitrogen (C/N) ratio and the organic loading rate (OLR) of the FW on the biogas methane production. The FW was fed to the digesters at different OLR of 0.5-5.5 g VS /L d in the semi-continuously fed LS and PS digesters and at OL of 0.5-7.5 g VS /Ld in the batch digester. Using a constant hydraulic retention time (HRT) of 12.5 days, each OLR was investigated during the semi-continuous process. Three C/N ratio values of 17, 26 and 30 were studied at batch scale while two C/N ratio values of 17 and 30 were studied during the semi-continuous AD in the LS and PS. The available FW gathered from restaurants around Taman Sri Serdang was at C/N ratio 17. The C/N ratio 26 and 30 fed were formulated by mixing the available FW with calculated proportions of Fruits, vegetables and meat wastes to increase its C/N ratio value. Performance evaluation parameters for AD process such as the COD removal, VS destruction, volatile fatty acids, alkalinity, ammonia-nitrogen and the biogas methane yield were studied during the 30 days batch and 168 days semi-continuous AD process. The highest methane yield obtained from the batch digestion was 0.557 L/g VS fed at an OLR of 5.5 g VS /Ld during the digestion of FW of C/N=30. This compares with the maximum methane yield of 0.510 L/g VS fed obtained at OLR of 5.5 g VS /Ld in the LS digester during the semi-continuous digestion of FW at C/N=30. The maximum methane yield obtained in the PS digester was 0.392 L/gVS at OLR of 4.5gVS/Ld during the digestion of FW at C/N=30. The methane yield attained in the up-scaled PS digester was about 80% of that obtained in the LS. Results showed that process stability and digester treatment performance during semi-continuous digestion was higher during the digestion of FW at C/N=30 than at C/N=17. Foaming potential tests revealed that foaming was initiated during digestion at OLR of 1.5 g VS/Ld in

all the feedstock digested. However, the highest foaming volume of 26% recorded was during the digestion of FW of C/N=17 at OLR of 7.5 g VS/Ld. The feedstock with C/N=30 had the least foaming tendency with foaming volume of 10% at OLR of 7.5 g VS/Ld.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Master sains

KESAN NISBAH KARBON KEPADA NITROGEN DAN KADAR PEMUATAN ORGANIK TERHADAP PENCERNAAN ANAEROBIK SISA MAKANAN

Oleh

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Kajian ini berkenaan dengan penyiasatan penjanaan biogas metana daripada sisa makanan yang banyak (FW) terdapat di Malaysia oleh proses pencernaan anaerobik (AD) pada keadaan mesophilic (35^{o}C). Metana terhasil daripada penghadaman FW adalah sumber tenaga yang boleh diperbaharui untuk penjanaan haba dan elektrik . Penjanaan biogas telah dijalankan dalam pencerna kumpulan dan kemudian dalam 2 pencerna mod separa-berterusan. pencerna mod separa berterusan terdiri daripada skala perintis 1000L (PS) dan pencerna skala makmal 10L (LS). Parameter utama yang dikaji ialah kesan nisbah karbon kepada nitrogen (C/N) dan kadar pemuatan organik (OLR) daripada FW kepada pengeluaran biogas metana. Parameter lain disiasat di LS dan reaktor PS ialah pepejal meruap (VS) dan kestabilan proses. FW telah dimasukkan kepada pencerna di OLR berjulat antara 0.5-5.5 g VS / L d dalam LS separa berterusan dan pencerna PS dan di OL daripada 0.5-7.5 gVS/Ld dalam pencerna kumpulan. Menggunakan masa yang tetap hidraulik pengekalan (HRT) sebanyak 12.5 hari, setiap OLR telah disiasat selama 14 hari dalam proses semi-berterusan. Tiga C/N nilai nisbah 17, 26 dan 30, telah dikaji di skala kumpulan manakala dua nilai nisbah C/N 17 dan 30 telah dikaji semasa AD separa berterusan dalam LS dan PS. FW yang dikumpul dari restoran di sekitar Taman Sri Serdang adalah pada nisbah C/N nisbah 17. C/N=26 dan 30 yang dimasukkan telah dirumuskan dengan mencampurkan FW yang disediakan dengan kadar yang dikira. Buah-buahan, sayur-sayuran dan sisa daging didapati akan meningkatkan nilai nisbah C/N. Parameter penilaian prestasi untuk proses AD seperti penyingkiran COD, VS kemusnahan, asid lemak meruap, kealkalian, ammonia-nitrogen dan hasil biogas metana telah dikaji dalam kelompok 30 hari dan 168 hari untuk proses AD separuh berterusan. Hasil metana tertinggi diperolehi daripada penghadaman kumpulan adalah 0.557 L/gVS yang diberi pada OL 5.5gVS/Ld dan C/N=30. Ini berbanding dengan hasil metana maksimum 0.510L/ gVS didapati di OLR 5.5 gVS/ Ld dalam pencerna LS semasa penghadaman separuh berterusan FW di C/N = 30. Hasil metana maksimum diperolehi dalam pencerna PS adalah 0.392L/gVS di OLR daripada 4.5gVS/Ld semasa pencernaan FW di C/N=30. Hasil metana diperolehi semasa penghadaman FW yang meningkat dengan peningkatan nisbah C/N dari 17 hingga 26 dan 30. Keputusan menunjukkan bahawa kestabilan proses dan prestasi rawatan pencerna semasa penghadaman separuh berterusan adalah lebih tinggi semasa pencernaan FW di C/N = 30 daripada di C/N=17. ujian buih berpotensi

mendedahkan bahawa buih telah dimulakan semasa penghadaman di OLR 1.5gVS/Ld dalam semua bahan mentah yang dicerna. Bagaimanapun, jumlah buih tertinggi 26% yang dicatatkan adalah semasa pencernaan FW C/N=17 di OLR sebanyak 7.5 g VS / Ld. Bahan mentah dengan C / N = 30 mempunyai isipadu yang tidak berbuih daripada 10% pada OLR sebanyak 7.5 g VS / Ld.

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LIST OF ABBREVIATIONS

AD	Anaerobic digestion
ADF	Acid detergent fibre
ANOVA	Analysis of Variance
APHA	American Public Health Association
BOD	Biological Oxygen Demand
CF	Crude fibre
C/N	Carbon to Nitrogen ratio
COD	Chemical Oxygen Demand
CP	Card Packaging
CM	Cow Manure
CS	Cattle Slurry
CSTR	Continuously Stirred Tank Reactor
DMD	Dimethyldisulphide
F1	Food waste mixture with carbon to nitrogen ratio 17
F2	Food waste mixture with carbon to nitrogen ratio 26
F3	Food waste mixture with carbon to nitrogen ratio 30
FAO	Food and Agricultural Organization
FOG	Fat oil and grease
FVW	Fruit and Vegetable waste
FW	Food Waste
FW/I	Food waste to Inoculum ratio

FWD	Food Waste Digester
GC	Gas Chromatograph
GE	Gross energy
GHG	Green House Gas
GW	Green Waste
GW/I	Green waste to inoculum ratio
HASL	Hybrid anaerobic solid-liquid system
HPLC	High Performance Liquid Chromatography
HRT	Hydraulic retention Time
LCFA	Long Chain Fatty Acid
LFS	Landfill soil
LS	Lab Scale
MC	Moisture Content
MSW	Municipal Solid waste
MTS	Modified three stage reactor
NDF	Neutral detergent fibre
NR	Not Reported
OFMSW	Organic Fraction of Municipal Solid Waste
OLR	Organic Loading Rate
PS	Pilot Scale
PVC	Polyvinylchloride
SBHR	Sonicated biological hydrogen reactor
SS	Source separated
STP	Sludge Treatment Plant
TAN	Total Ammonia-Nitrogen

TCD	Thermal Conductivity Detector
TE	Trace Element
TS	Total Solids
UNEP	United Nation Environmental Protection Agency
UV	Ultraviolet rays
VD	Vegetable Digester
VFA	Volatile Fatty Acid
Vis	Visible ray
VS	Volatile Solids
VW	Vegetable Waste
WAS	Waste activated sludge
WWTPD	Waste Water Treatment Plant Digester
YE	Yeast Extract

CHAPTER 1

INTRODUCTION

1.1 Background

Recently, food processing, food consumption and food waste (FW) have been counted among the sectors producing high emissions of greenhouse gas (Wiedmann and Minx, 2007; UNEP, 2009; Bentley, 2008; Guinée et al., 2009; Padfield and Chris, 2012). Globally, 1.3 billion tons of food is wasted per annum amounting to about 310 billion US dollars in developing countries and about 680 billion US dollars in industrialized nations (FAO, 2014). More than 40% of FW occur at consumer or retail level in industrialized nations while 40% FW occur during processing and post-harvest periods in developing nations (FAO, 2014). Reduction of GHG emanating from these sectors require priority attention especially in south-east Asia where increase in population and economic growth is leading to higher food consumption yearly (Hassan et al., 2001; Padfield and Chris, 2012).

Anaerobic digestion (AD) has been considered a viable option because it not only serves as the most economically viable method (Poh and Chong, 2009) of treatment for FW but also yields biogas methane as a source of renewable energy. Biogas contains but not limited to methane (40-75%), carbon-dioxide (19-38%) and water (6%). The remnant digestate can be applied as soil fertilizer, in composting and land-filling. AD is a process whereby complex organic materials or the organic contents of a material is broken down naturally (in a process often known as fermentation) by a consortia of micro-organisms in the absence of oxygen (Christensen, 2011). FW obtained from households, restaurants or other commercial sources are most suited to the AD because of its high moisture content (up to 90%), high organic content (80-97%), and ease of biodegradability (Lay et al., 1999; Zhang et al., 2007; Neves et al., 2009).

1.2 Food waste challenges in Malaysia

FW disposal into the already overstretched 176 landfill sites in Malaysia is one of the largest sources of greenhouse gases (GHG) in the country. Statistics showed a FW amounting to 50% of solid wastes at source and 70% as disposed at the landfill sites in Malaysia (Ministry of Housing and Local Government, 2013; Periathamby et al., 2009). Out of 17,000 tons/day of solid waste generated in Malaysia in 2002, about 7650 tons/day was FW. This represents 45% of the municipal solid waste (MSW). With an estimated per capital waste generation of 1kg/ person /day (Ministry of Housing and Local Government, 2013), FW is currently 50% of total MSW generated. Experts have proposed that FW generation may increase to 13,500 tons/day by the year 2020 (Papargyropoulou, 2010; Ministry of Housing and Local Government, 2013). The FW are mainly food remnants and kitchen wastes from the residential homes, industrial sources, commercial restaurants, school cafeteria etc.

1.3 Strategic plan for waste reduction in Malaysia

Innovative strategic plans have been developed for FW management in Malaysia alongside solid waste management strategies. This strategy emphasizes reduction of FW to the barest minimum at source, reuse and recycling in order to divert FW from landfills (Ministry of Housing and Local Government, 2013). As part of a national strategic plan for FW management, Malaysian government have proposed the establishment of FW treatment centers in addition to the programs already put in place for the reduction of FW at source (Ministry of Housing and Local Government, 2013). This will involve energy recovery method such as AD and the use of recycling method such as composting. Composting enables FW to be recycled as soil fertilizer while AD help to recover biogas as a source of renewable energy for heat and electricity generation besides using the remnant digestate as soil fertilizer.

At the United Nations Climate Change Conference held in Copenhagen in 2009, Malaysia government announced to adopt a voluntary carbon emissions intensity reduction of up to 40% by the year 2020. Although the waste reduction, reuse and recycle programs (Ministry of Housing and Local Government,2013) put in place have been largely successful especially in reducing other aspects of MSW but these strategies need to be intensified especially in the aspect of FW reduction.

1.4 Impacts of food waste

Apart from impacting negatively on the environment, increasing FW problems can affect government policy and finances. Some of the impacts of FW generation are discussed below.

1.4.1 Production of odour

The nature of food subjects it to easy spoilage resulting in the production of odourous substances. The characteristics of food waste which makes it easily degradable are: low pH (below 6), high moisture content, presence of nitrates and volatile organic carbon content. This provides a good medium for both aerobic and anaerobic microbes such as *Lactobacteria*, *Clostridia*, *Bacillales* or *Actinobacteria* (Epstein and Wu, 2000; Coker, 2012). The majority of food components are made up of carbohydrates, protein, vitamins and fats or oils consisting mainly of compounds of nitrogen, oxygen, hydrogen and sulfur. Due to chemical changes and microbes acting on a combination of these complex organic compounds, each is reduced to its various components. In this process, odourous volatile intermediate compounds are produced. The type of odourous compounds produced depends on the composition of the FW. These intermediate compounds include: dimethyldisulphide (DMD), methyl mercaptan, hydrogen sulphide, tarpenes, limonene, acetaldehyde, volatile fatty acids (VFA), methylamine etc (Coker, 2012; Epstein and Wu, 2000). Therefore, littering of FW or disposal in open landfills can cause a mixture of unpleasant odours.

1.4.2 Greenhouse and landfill gas emissions

Green House Gases (GHG) are the various gases that block outgoing long-wave infrared from easily leaving our atmosphere. The greenhouse effect is the process, or mechanism, by which greenhouse gases interfere with the direct radiation of heat energy leaving the surface of the planet. In other words GHG can get in the way of heat escaping and thus prevent some of the heat from escaping quickly or easily thereby causing global warming. The dominant natural GHG are H_20 (water), CO_2 (carbon dioxide), CH_4 (methane), and Nitrous oxide (N_2O). There are also industrial Chlorofluorocarbons. The final products of FW decomposition are: carbon dioxide, methane and water. The first two are sources of GHG which could emanate from landfills or littered FW. Global activity in the food sector such as agriculture, processing, transportation, storage and retail contributes an impact of about 22% global warming (UNEP, 2009; Papargyropoulou, 2010). Contribution of landfill to global warming is placed at about 3%. This in total makes 25% contribution from the food sector.

1.4.3 Water contamination

FW obtained from food processing industries and activities arising from FW management can lead to soil and surface water (rivers, lakes, ponds etc) contamination. Leachate percolation in landfills could also lead to ground water contamination if the landfill is not properly designed to remove leachate. Water contaminated with FW leachates may contain toxins, pathogens and other pollutants which have negative health impacts on humans, animals and the environment.

1.5 Problem statement

The yield of biogas methane in FW digesters depend on factors such as the FW composition (Sosnowski et al., 2003; Gomez et al., 2006), Organic Loading Rate (OLR) fed to the digesters (Jiang et al., 2012), carbon to nitrogen (C/N) ratio of the FW (Zeshan et al., 2012) and adequate nutrition to microbes (Zhang et al., 2012; Gonzalez-Gil et al., 2003). Challenges due to the last three factors could be mitigated through co-digestion with complementary wastes (Yen and Brune, 2007; Zhang et al., 2012). Co-digestion of FW with wastes that could release balanced nutrients for better microbial growth and activity also help to increase C/N ratio of feed mixture leading to the attainment of higher OLR and biogas methane yield during AD (Zhang et al., 2012; Zeshan et al., 2012). There has been little research on the deliberate increase in C/N ratio of available restaurant FW mixture by co-digestion with other FW of higher C/N ratio. Consequently, the influence on biogas methane production, of increasing C/N ratio of the digested FW from 17 to 26 and 30 was first tested by experimenting in batch digesters operating at stepwise OLR increase of 0.5-7.5 kg VS/m^3d. Based on the batch tests which indicate a high methane yield, the FW digestion study was further carried out simultaneously in semi-continuous mode digesters. This was done in order to ensure if a good methane yield can be obtained with daily feeding from the FW generated. In order to find out if the test results of the batch digester (1L) and LS digester (10L) could be transferred to commercial scale, the performance of a commercial 1000L capacity pilot scale (PS) digester which was scaled up from the LS digester were compared at stepwise OLR increase of 0.5 to 5.5 g VS/Ld under the same operating condition. This was done to study, compare and contrast the behavior of the two identical semi-continuous digesters on the basis of their treatment performance and operating parameters during the digestion of the different feedstock compositions.

1.6 Objectives

The overall objective of this research was to investigate methane yield from available and formulated restaurant FW at different C/N ratio of feedstock by co-digestion method and at different OLR using a PS digester up-scaled from a LS digester. The specific objectives are:

1. To carry out batch AD feasibility tests on the FW feedstocks in order to determine the effect of C/N ratio of each FW on methane yield at varying OLR under mesophilic AD condition.

2. To determine the possibility of daily feeding of the FW into the digester by semi-continuous mode of operation through comparison of the methane yields of a 1000L commercial PS digester scaled up from a 10L LS digester operating at the same incremental OLR, start-up composition and mesophilic condition.

3. To determine the foaming potential of the feed during the batch test and also monitor AD performance parameters such as VS destruction, COD removal, pH and digester stability (Ripley ratio) during the semi-continuous operation.

1.7 Scope of the research

The FW digestion was carried out in duplicate at C/N ratio 17, 26, and 30 and at OLR of 0.5, 1.5, 3.5, 5.5 and 7.5gVS/Ld respectively. The batch test was carried out to ascertain the feasibility of the experimental study and the biogas production potential at each of the specified C/N ratio. The semi-continuous study was carried out by comparing the process parameters of the LS and the PS digester at OLR of 0.5, 1.5, 2.5, 3.5 4.5, and 5.5 g VS/Ld using FW at C/N ratio of 17 and 30 only. The semi-continuous mode of operation is necessary in order to accommodate the daily FW treatment as they are generated daily. The PS which is a scale-up of the LS digester has been designed for the purpose of daily treatment of FW. Therefore, the comparison of the two digesters will help to assess the performance of the PS digester with the LS as the reference. Consequently, comparison of operating and performance parameters between the PS and LS will help to determine whether the good treatment and high methane yield obtained in the LS could be transferred to the commercial scale (Sell et al., 2011;Wang et al., 2005). The parameters measured during the AD where: TS, VS, COD removal, VS destruction (or digester performance), methane yield, volatile fatty acid (VFA) content of digestion medium, methane composition in biogas, and process stability which can be determined by calculating Ripley ratio (i.e VFA/Alkalinity) (Jiang et al., 2012; Banks et al., 2012).

Both the batch and semi-continuous studies were carried out at mesophilic temperature only and in duplicates. Digestion at thermophilic temperature was not investigated during this study. In order to ensure adequate comparisons of the LS and PS digestion, both digesters were operated as much as possible under the same conditions both at start-up and during the AD process.

1.8 Contributions

This research mainly focused into the biogas methane production potential from daily FW feeding of both the LS and PS digester. Methane recovered from FW digestion is a source of renewable energy which can be used for heat and electricity generation and as fuel. This will help to reduce the greenhouse gas (GHG) emissions arising from FW disposal in landfills which accounts for the largest source of carbon emissions in Malaysia. FW anaerobic digestion is in line with the Malaysian government strategic goal and action plan for FW treatment, management and diversion (or minimization) from the already over-stretched landfills (Ministry of Housing and Local Government, 2013; Papargyropoulou, 2010). The digestate or stabilized mass obtained after the AD is lower in volume and can easily be disposed in landfills, can be further utilized as fertilizer in soil applications or further treated in a composting process before application. The results obtained from the comparison study between the LS digester and the up-scaled PS could be helpful in terms of providing preliminary data for the design of centralized commercial FW digestion treatment plants for household FW and the growing food service sector in Malaysia.

1.9 Thesis Organization

This thesis was written in five chapters followed by appendices. Chapter 1 is a brief introduction highlighting the general importance and justification for FW anaerobic digestion especially in Malaysia. The advantage as well as the scope of research has also been mentioned. Necessary literatures on FW have been reviewed in Chapter 2. These include general basic concepts of AD, various techniques applied by researchers to enhance the production, yield and methane content of biogas. Factors influencing AD such as OLR, hydraulic retention time (HRT), carbon to nitrogen (C/N) ratio, temperature, pH etc were discussed. Materials and methods used in this research have been described in Chapter 3. The composition of the FW substrate, reagents and various equipment used have been described. Experimental procedures, equipment and tests used in monitoring and evaluating the performance of the digester medium have also been described in the same chapter. Chapter 4 presents the essential results including discussion of these results. Chapter 5 covers the conclusions and recommendations based on the inferences derived from the results obtained during the study.

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