UNIVERSITI PUTRA MALAYSIA

# PRETREATMENT STUDY OF FOOD WASTE USING SONICATION FOR ENHANCEMENT OF METHANE PRODUCTION

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**Chairman: Assoc. Prof. Ir. Wan Azlina Wan Abdul Karim Ghani, PhD**
**Faculty: Engineering**

The application of low-frequency ultrasonic pretreatment to the organic fraction municipal solid waste (OFMSW), particularly food waste (FW), is able to overcome slow hydrolysis rate by disintegrating the substrate into a biodegradable substrate. In addition, the process promotes solubilization of organic matter resulting in high amount of substrate readily available within the digestion process and thus enhancing high methane production. The main objective of this study was to obtain high methane production through anaerobic digestion of sonicated food waste. To accomplish this aim, the following objectives were pursued namely correlation between ultrasonication with the physicochemical property changes and anaerobic biodegradability, evaluation of the digester performance and system stability of the anaerobic digestion of the sonicated food waste at different organic loading rate (OLR) and the determination of the kinetic parameters for the performance of the anaerobic digestion of sonicated food waste.

The anaerobic digestion experiment on sonicated food waste was conducted in two phases, where in Phase 1, the ultrasonic pretreatment was performed by sonicating 200 mL of food waste slurry at different sonication duration of 2, 4, 6, 8 and 10 minutes at 20 kHz frequency and specific energy input ranged from 5,396 to 25,997 kJ/kg TS, and then anaerobic batch tests were carried out on the sonicated food waste samples. While in Phase 2, the anaerobic digestion was conducted on the sonicated food waste (SFW) and non-sonicated food waste (NSFW) or control in a 13 L laboratory scale anaerobic digester (working volume of 10 L) with increasing organic loading rate (OLR) ranging from 1.5-3.5 gCOD/L.day. The experimental works were carried out in two stages (batch start-up and then continued with semi-continuous feeding). The performance of all digesters was evaluated based on the methane composition, methane production rate as well as methane yield.

In the ultrasonic pretreatment (Phase 1), when the specific energy inputs increased from 0 to 25,997 kJ/kg TS, analysis of chemical properties found that the percentage of soluble COD (SCOD) values have increased to 34-40 %. The range of $COD_{solubilization}$ and degree of disintegration (DD) values were between 11.4-13.4% and 57.15-71.08%, respectively. A linear relationship with an $R^2$ of 0.907 was obtained for the correlation between $COD_{solubilisation}$ and DD. The application of sonicated food waste in the anaerobic digestion process has increased the cumulative $CH_4$ production about 40.3-70.5%. The volume of methane produced was increased from 4.2 liters (non-sonicated food waste) to 7.9 - 14.5 liters in the sonicated food waste. While for Phase 2, the methane composition generated during the process was higher in SFW digester at OLR of 1.5 g COD/L.day and 3.5 g COD/L.day compared to NSFW digester. There was an increase of 9.54-41.28% in methane composition when SFW digester was operated at OLR of 3.5 g COD/L.day. Methane production rate was enhanced by 20.8 -75.7% or 1.26-4.12 times in SFW digester when operated at an OLR of 3.5 g COD/L.day. Methane yields did not show significant changes at the OLR of 1.5 and 2.5 g COD/L.day in both digesters, but started to show a significant increase when SFW digester was operated at OLR of 3.5 g COD/L.day. The methane yield was enhanced by 42.87-82.83% or 1.75-5.82 times at the stated OLR.

The process performance as well as acceptable stability in SFW digester provided satisfactory predictions with Monod, Modified Stover-Kincannon, Grau second-order multicomponent substrate removal and Contois kinetic models. The experimental data were well fitted with the models with high correlation coefficients ($R^2$) ranging from 0.914-0.996. The kinetic coefficients such as yield coefficient (Y), maximum specific growth rate ($\mu_{max}$), death rate ($k_d$), half velocity coefficient ($K_s$), saturation value constant ($K_B$), maximum utilization rate ($U_{max}$), multicomponent Grau second-order substrate removal coefficient ($k_s$), dimensionless Grau second order constant (a and b), $\mu_{max}$ and B (for Contois model) recorded 1.580 gVSS/gCOD, 1.219 day$^{-1}$, 0.06 day$^{-1}$, 617.59 g/L, 1.928 gCOD/L.day, 1.667 gCOD/L.day, 0.103 day$^{-1}$, 48.121, 1.156, 10.76 day$^{-1}$ and 361.31 gCOD/gVSS, respectively. Furthermore, a significant relationship was observed between the predicted and experimental data with correlation coefficients ($R^2$) ranging from 0.893-0.996. In this study, the Monod model with $R^2$ = 0.996 indicates the most appropriate model for interpreting the kinetic parameters of the anaerobic system in the CSTR treating sonicated food waste (SFW) slurry.

Overall, the implementation of ultrasonic pretreatment prior to anaerobic digestion of food waste can increase the production of methane by recording overall enhancement in methane production rate of 18.99%. The overall enhancement of methane yield was 23.86% with the highest methane composition of 79.6%. Therefore, the kinetic model used in this study can be applied to predict the performance of the anaerobic digestion system treating sonicated food waste slurry.

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

**KAJIAN PRARAWATAN SISA MAKANAN MENGGUNAKAN ULTRASONIK UNTUK PENINGKATAN PENGHASILAN METANA**

Oleh

**NOR HABSAH MD SABIANI**

**Januari 2019**

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Penggunaan kaedah rawatan ultrasonik berfrekuensi rendah terhadap pecahan organik sisa pepejal perbandaran, terutamanya sisa makanan (FW), mampu mengatasi kadar hidrolisis yang perlahan dengan memecahkan substrat menjadi substrat yang tersedia untuk tindakan secara biologi. Di samping itu, proses ini menggalakkan proses solubilisasi bahan organik yang menghasilkan banyak substrat yang sedia ada dalam proses pencernaan dan dengan itu meningkatkan pengeluaran metana yang tinggi. Objektif utama kajian ini adalah untuk menghasilkan metana yang tinggi melalui pencernaan anaerobik sisa makanan yang telah dirawat dengan gelombang ultrasonik. Bagi mencapai matlamat ini, beberapa objektif berikut telah diikuti iaitu kolerasi di antara ultrasonik dengan perubahan sifat fizikal dan kimia dan biodegradabiliti anaerobik, penilaian prestasi reaktor dan kestabilan sistem pencernaan anaerobik sisa makanan yang dirawat pada kadar beban organik yang berbeza dan penentuan parameter kinetik untuk pencernaan anaerobik sisa makanan yang dirawat dengan gelombang ultrasonik.

Eksperimen pencernaan anaerobik pada sisa makanan yang telah dirawat dengan gelombang ultrasonik dilakukan dalam dua fasa, di mana dalam Fasa 1, gelombang ultrasonik telah dikenakan terhadap 200 mL sisa makanan pada tempoh masa yang berbeza iaitu 2, 4, 6, 8 dan 10 minit pada frekuensi 20 kHz dengan input tenaga tertentu di antara 5,396 hingga 25,997 kJ/kg TS, dan kemudiannya ujian pencernaan anaerobik kelompok telah dijalankan ke atas sampel sisa makanan yang dirawat. Dalam Fasa 2, pencernaan anaerobik dilakukan ke atas sisa makanan yang dirawat dengan gelombang ultrasonik (SFW) dan sisa makanan tidak dirawat dengan gelombang ultrasonik (NSFW) atau kawalan di dalam reaktor anaerobik berskala makmal berisipadu 13 L (dengan isipadu kerja 10 L) dengan peningkatan kadar beban organik (OLR ) dari 1.5-3.5 gCOD/L.hari. Eksperimen telah dijalankan dalam dua peringkat (operasi permulaan secara kelompok dan kemudian diteruskan dengan mod separa berterusan). Prestasi kedua-dua pencerna dinilai berdasarkan komposisi metana, kadar pengeluaran metana serta hasil metana.

Dalam rawatan ultrasonik (Fasa 1), apabila input tenaga tertentu meningkat dari 0 ke 25,997 kJ/kg TS, analisis sifat kimia yang dijalankan mendapati bahawa peratusan nilai COD larut (SCOD) telah meningkat kepada 34-40 %. Julat nilai $COD_{solubilization}$ dan darjah perpecahan (DD) adalah di antara 11.4-13.4% dan 57.15-71.08%, masing-masing. Hubungan linear dengan $R^2$ = 0.907 diperolehi untuk korelasi di antara $COD_{solubilisation}$ dan DD. Penggunaan sisa makanan yang telah dirawat dalam proses pencernaan anaerobik telah meningkatkan pengeluaran $CH_4$ kumulatif kira-kira 40.3-70.5%. Isipadu metana yang dihasilkan telah ditingkatkan daripada 4.2 liter (sisa makanan tidak dirawat dengan gelombang ultrasonik) kepada 7.9 - 14.5 litres di dalam sisa makanan yang dirawat dengan gelombang ultrasonik. Dalam Fasa 2, komposisi metana yang dihasilkan semasa proses berlangsung lebih tinggi di reaktor SFW pada OLR 1.5 g COD/L.hari dan 3.5 g COD/L.hari berbanding reaktor NSFW. Terdapat peningkatan sebanyak 9.54-41.28% dalam komposisi metana apabila reaktor SFW dikendalikan pada OLR 3.5 g COD/L.hari. Kadar pengeluaran metana telah ditingkatkan sebanyak 20.8 -75.7% atau 1.26-4.12 kali ganda dalam reaktor SFW apabila dikendalikan pada OLR 3.5 g COD/L.hari. Hasil metana tidak menunjukkan perubahan ketara pada OLR 1.5 dan 2.5 g COD/L.hari dalam kedua-dua reaktor, namun mula menunjukkan peningkatan ketara apabila reaktor SFW dikendalikan pada OLR 3.5 g COD/L.hari. Hasil metana ditingkatkan sebanyak 42.87-82.83% atau 1.75-5.82 kali ganda pada OLR yang dinyatakan.

Prestasi proses serta kestabilan reaktor SFW memberikan ramalan yang memuaskan dengan model kinetik Monod, Modified Stover-Kincannon, Grau second-order dan Contois. Data eksperimen menunjukkan penyesuaian terbaik dengan semua model dengan julat pekali korelasi ($R^2$) adalah tinggi dalam julat 0.914-0.997. Pekali kinetik seperti pekali hasil (Y), kadar pertumbuhan spesifik maksimum ($\mu_{max}$), kadar kematian ($k_d$), pekali separuh halaju ($K_s$), pekali nilai ketepuan ($K_B$), kadar penggunaan maksimum ($U_{max}$) dan pekali penyingkiran substrat ($k_s$), pekali tidak berdimensi Grau second (a dan b), $\mu_{max}$ dan B (untuk Contois model) mencatatkan 1.580 gVSS/gCOD, 1.219 hari$^{-1}$, 0.06 hari$^{-1}$, 617.59 g/L, 1.928 gCOD/L.hari, 1.667 gCOD/L.hari dan 0.103 hari$^{-1}$, 48.121, 1.156, 10.76 day$^{-1}$ and 361.31 gCOD/gVSS, masing-masing. Selain itu, hubungan yang ketara diperhatikan di antara data ramalan dan eksperimen dengan pekali korelasi ($R^2$) dalam julat 0.893-0.996. Dalam kajian ini, model Monod dengan $R^2$ = 0.996 merupakan model yang paling sesuai untuk mentafsirkan parameter kinetik bagi sistem anaerobik dalam merawat sisa makanan di dalam reaktor pengaduk berterusan (CSTR) yang telah melalui rawatan ultrasonik.

Secara keseluruhannya, pelaksanaan rawatan ultrasonik sebelum proses pencernaan anaerobik dapat meningkatkan pengeluaran metana dengan mencatatkan peningkatan keseluruhan dalam kadar pengeluaran metana sebanyak 18.99%. Peningkatan keseluruhan hasil metana mencatatkan 23.86% dengan komposisi metana tertinggi sebanyak 79.6%. Oleh itu, model kinetik yang digunakan dalam kajian ini boleh digunakan untuk meramalkan prestasi sistem pencernaan anaerobik yang merawat sisa makanan yang telah didedahkan dengan gelombang ultrasonik.

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May ALLAH S.W.T rewards your kindness with the best of rewards. Thank you all.

**Declaration by graduate student**

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| | |
|---|---|
| AD | Anaerobic digestion |
| AAS | Atomic absorption spectrophotometer |
| BMP | Biochemical Methane Potential |
| CSTR | Continuous stirred tank reactor |
| COD | Chemical oxygen demand |
| DS | Dewatered sludge |
| DM | Dairy manure |
| DD | Degree of disintegration |
| FW | Food waste |
| FOG | Fat, oil and grease |
| GC | Gas chromatography |
| HRT | Hydraulic retention time |
| ICP-AES | Inductively Coupled Plasma Atomic Emission Spectrometry |
| kV | kilovott |
| KHz | kilohertz |
| MC | Moisture content |
| MH | Maize husk |
| MHz | Megahertz |
| NSFW | Non-sonicated food waste |
| OLR | Organic loading rate |
| OFMSW | Organic fraction municipal solid waste |
| POME | Palm oil mill effluent |

| | |
|---|---|
| SFW | Sonicated food waste |
| SEM | Scanning electron microscope |
| SCOD | Soluble chemical oxygen demand |
| SE | Specific energy input |
| TCD | Thermal conductivity detector |
| UD | Ultrasonic density |
| $UD_o$ | Ultrasonic dose |
| UI | Ultrasonic intensity |
| VS | Volatile solid |
| t | Time |
| μ | Specific growth rate |
| $k_d$ | Death rate |
| Y | Growth yield coefficient |
| Q | Specific utilization rate |
| Q | Flow rate |
| V | Volume of reactor |
| $S_o$ | Influent substrate concentration |
| S | Effluent substrate concentration |
| $X_o$ | Concentration of biomass in influent |
| X | Concentration of biomass in influent |
| $\mu_{max}$ | Maximum specific growth rate |
| $K_s$ | Half velocity constant |
| $U_{max}$ | Maximum utilization rate constant |
| $K_B$ | Saturation value constant |
| $k_s$ | Grau second-order multicomponent substrate removal rate constant |

| | |
|---|---|
| $\theta_H$ | Hydraulic retention time |
| a | Dimensionless Grau second-order constant |
| b | Dimensionless Grau second-order constant |

# CHAPTER 1

# INTRODUCTION

## 1.1 Research overview

Food waste (FW), which is known as a complex heterogeneous organic material consisting of highly recalcitrant material up to extremely biodegradable compounds becomes major concerns globally. In United States, the generation of food waste has reached 188 kg/capita per year with total losses reaching $165.6 billion at consumer and retail levels. According to Garrone et al. (2014), estimated food waste generation between 280-300 kg/capita per year has been reported in Europe and North America. Furthermore, approximately 33% of food is wasted in Southeast Asia (Yang et al., 2016). For Malaysia, Bong et al. (2017) reported that the average household in Malaysia being thrown was about 0.5-0.8 kg of non-consumable food daily which indirectly contributed up to 40% of the municipal solid waste (MSW). The remaining 60 % comprised inorganic waste such as plastics, papers, diapers/napkins, textile, metal, glass, rubber and leather, garden or yard waste and others (SWCorp, 2016). Ineffective FW disposal will contribute to severe environmental problems including odour emission, leachate production, greenhouse gas emissions (GHG) and groundwater pollution (Han and Shin, 2004). Landfilling, incineration and aerobic composting are some of the traditional approaches that are often taken into consideration in the FW management in Malaysia. There are several factors that make these three methods unfavourable to dispose of FW, namely the tendency to create environmental problems, space constraints and characteristics of FW.

In FW management, anaerobic digestion (AD) is considered as the best alternative of treatment compared to landfilling, incineration and composting. Limited environmental impacts and high potential for energy recovery make this technology best suited for treating FW (Ariunbaatar et al., 2014) as well as having great potential in reaching 40 to 60% of waste reduction. In AD process, the decomposition of organic material is performed by groups of bacteria and microorganism in an oxygen-depleted environment (Ward et al., 2008), involving 4 stages of processes namely hydrolysis, acidogenesis, acetogenesis and methanogenesis. This method offers several environmental benefits over other forms of treatment technology, for example it produces biogas and useful fertilizer, reduces organic content of the waste (COD), preserves fertilizer value, eliminates biodegradable components that produce odour, reduces pathogen in the waste and reduces greenhouse gas emission (methane and carbon dioxide) which may lead to global warming (Wilkie, 2005).

Several previous literatures have reported that FW is easily biodegradable in nature, but the situation is different from the FW produced in Malaysia where the structure is more complex compared to the FW generated in other countries or regions around the world. The composition of the feedstock used during the digestion process affects the degradability rate of FW (Kondusamy and Kalamdad, 2014). There are difficulties and challenges in estimating and measuring the percentage of carbohydrates, proteins and lipids in a heterogeneous substrate such as FW. The inconsistency in feedstock in terms of composition, shape and size are also among the problems encountered in the anaerobic digestion of food waste that may contribute to low biogas or methane yield. Besides that, most researchers have reported that the rate-limiting step for complex organic substrates particularly FW was the hydrolysis step and needed to be improved to enhance the performance of an anaerobic digester (Khalid et al., 2011; Raposo et al., 2011; Elliot and Mahmood, 2012). Nathao et al. (2013) and Yang et al. (2015) reported that the relatively low methane yield was between 100-250 L $CH_4$/kg $VS_{added}$. It has been reported by Shen et al. (2013), Khairuddin et al. (2015) and Zeynali et al., (2017), that by using either batch and semi-continuous systems, low methane yield was obtained ranging between 54-220 L $CH_4$/kg $VS_{added}$ and 223 L $CH_4$/kg $COD_{removed}$ while conducting AD experiment involving FW (i.e. kitchen FW, synthetic FW, canteen FW, restaurant FW, household FW as well as fruits and vegetables waste). To overcome this problem, substrate pretreatment and co-digestion are frequently preferable methods. However, this study only focuses on substrate pretreatment in order to enhance biogas or methane yield from the AD process.

Mechanical grinding, ultrasonic, thermal, chemical, biological and combined pretreatments are among the pretreatments methods that can be used to pretreat the FW. In this study, the ultrasonic pretreatment method was selected to increase the production of biogas and methane during the AD process based on several advantages over other pretreatment methods. Ultrasonic is a mechanical pretreatment method that can be used for substrate pretreatment before hydrolysis occurs in the AD process. Deepanraj et al. (2017) stated that when high-intensity ultrasonic waves emitted, a large hydro-mechanical shear force will be produced by the cavitation bubbles and this is a predominant effect for FW disintegration. This extremely fast treatment method will increase soluble microbial products when ultrasonic waves are imposed on the FW which leads to the solubilization of both extracellular and intracellular substances. The increase in biogas and methane yield between 7.7 - 88% can be observed in AD process involving sonicated FW conducted by Elbeshbishy et al. (2012), Li et al. (2013) and Naran et al. (2016). Most of AD studies involving FW in Malaysia have focused on the following areas including reactor configuration and operating conditions (Zakarya et al., 2016), co-digestion (Khairuddin et al., 2015) as well as factors affecting the AD process (Tanimu et al., 2014) in order to enhance biogas or methane production. The motivation to carry out this study is due to scarcity of previous studies and reports associated to the implementation of ultrasonic pretreatment on FW prior to AD process in Malaysia. So far, only a study related to chemical pretreatment has been reported as one of the substrate pretreatment methods conducted on the FW in Malaysia (Junoh et al., 2015). Nevertheless, ultrasonic pretreatment also has been used to pretreat other organic materials such as palm oil mill effluent or POME (Wong et al., 2018) and petrochemical wastewater (Siddique et al., 2017) in Malaysia prior to AD in order to enhance biogas and methane production.

Overall, this study seeks effect of using ultrasonic pretreatment on FW (which is complex, has an inconsistent composition and varied in size) in the perspective of Malaysia before it is used as a feedstock in the AD process. This pretreatment method has a great potential in breaking their complex structure into simpler structure, providing a greater surface area for microbial action and enzymatic attack, softening the materials in the FW, enhancing solubilization of FW and thus increasing hydrolysis rate before fermentation, and subsequently increasing the production of biogas and methane yield.

## 1.2 Problem statement

Municipal solid waste (MSW) is one of the most frequently debated environmental issues around the world. Malaysia, like most developing countries, is facing an increase in waste generation and accompanying problems associated with the disposal of MSW. According to Ouda and Raza (2014), current generation of MSW worldwide is 1.3 billion tonnes per year and this volume will continue to increase by 2.2 billion tonnes annually by 2025. The increasing population as well as solid waste generation per capita from 1.2 to 1.42 kg/capita/day is the main factor contributing to the worldwide MSW generation rate (Hoonwerg and Bhada-Tata, 2012). According to Kamaruddin et al. (2017), the generation of municipal solid waste per capita in Malaysia is currently about 1.1 kg/capita/day where about 40% of MSW consisted of food waste.

Food waste (FW) is the largest and most problematic organic waste component in the solid waste management system in Malaysia. In promoting renewable energy production, food waste has enormous potential and is one of the most promising sources as it can be converted into energy due to high organic matters found in this source. The main components of food waste consist of carbohydrate polymers (starch, cellulose and hemicellulose), lignin, other organics (proteins, lipids, acids) and other inorganic substances (Vavouraki et al., 2013). Generally, food waste produced is acidic with a pH value of 3.84 - 6.50 (Adhikari et al., 2008; Zhang et al., 2011; Cheerawit et al., 2012; Tanimu et al., 2014). The value of moisture content is more than 70% (Nazlina et al., 2009; Hafid et al., 2010; Cheerawit et al., 2012). Food waste has high COD value of more than 200,000 mg/L, indicating that the food waste contains high organic pollutants (Kubaska et al., 2010; Bodik et al., 2014; Tanimu et al., 2014). The ranges of TS and VS values for food waste are between 14.8 - 29.6% and 89.5-94.3% (Malakahmad et al., 2008; Tanimu et al., 2014), indicating that the food waste is rich in organic solids content that can be converted into biogas during the anaerobic digestion process. Lipids found in food waste are usually characterized as fat, oil (liquid) and grease (solid) which vary between 11.6-28.6% (Hafid et al., 2010; Tanimu et al., 2014). In addition, food waste also contains macronutrients and micronutrients such as Ni, Zn, Cu, Pb, Fe, Mn, Cd, Al, M, P and K as required by methanogen bacteria in trace quantities (in correct ratio and concentration) for robust growth.

In general, food waste treatment in Malaysia is very limited. Most of the food waste produced throughout the country is disposed of together with other MSWs collected and sent to the landfill for final disposal. In solid waste management, incineration, landfilling, aerobic composting and anaerobic digestion can be implemented. Biogas production through anaerobic digestion is an attractive and effective option to solve this problem. The characteristics of food waste such as high moisture content ranges between 75-85 % (Han and Shin, 2004; Zhang et al., 2007; Omar et al., 2009) and high organic content (Zakarya et al., 2008) have made anaerobic digestion as an attractive option to reduce food waste. The application of anaerobic digestion to treat food waste offers several benefits such as volume reduction, waste stabilization and biogas recovery (Shin et al., 2001; Han and Shin, 2004).

Although anaerobic digestion is seen as an attractive and most effective option in solving problems related to food waste problems faced worldwide and also in Malaysia, there are some issues related to the selection of this treatment method. These include inconsistent feedstocks (in terms of composition, shape, and size) and complex food waste characteristics used in the anaerobic digestion process. Some previous studies showed that anaerobic digestion process on food waste produced low biogas or methane yields (Nathao et al., 2013; Shen et al., 2013; Khairuddin et al., 2015; Yang et al., 2015). The inconsistent feedstocks in terms of composition, shape and size are also among the problems encountered in the anaerobic digestion of food waste that may contribute to low biogas or methane production. The composition of food wastes generated from the household, housing area, commercial area and food services industries (restaurants, and canteens) varies which is influenced by the origin of waste produced, eating habits as well as different countries and regions around the world. In Europe, vegetables and fruits (40%) are the largest compositions of FW produced, followed by pasta and bread (33%), dairy products including eggs (17%) and meat and fish (9%). Meanwhile, in Asia (especially in Japan, China and South Korea), FW consists of 56% vegetables and fruits, 34% rice and noodles, and only a small portion (about 10%) represents fish, meat and dairy products (Braguglia et al., 2018). In Malaysia, about 40% of municipal solid waste generated by Malaysians consists of food waste. Approximately, 77% of the food waste produced consists of cooked rice, noodles, bread and pastries (carbohydrate group) produced from the cafeteria, commercial restaurants, meat and market industries (Tanimu et al., 2014). Most of the polymeric carbohydrates and protein are present in solid form, for example rice, bread, noodles, vegetables and meat. For countries in Southeast Asia such as Malaysia, the composition of food waste containing carbohydrates is usually derived from the meal preparations and food leftovers such as cooked and uneaten rice, noodles, bread, vegetables and fruits. In addition, the presence of cellulose and hemicellulose from vegetables, fruits and wet tissue papers (normally present in food waste collected from restaurants or diners) is very difficult to be segregated from other discarded food waste. This will also cause problems if the food waste is not pretreated prior to the digestion process.

The aims of using pretreatment to FW are to soften the materials found in the food waste, break complex structure into smaller sizes in order to provide larger surface areas for microbial action and enzymatic attack, enhance FW solubilization and increase hydrolysis rate prior to fermentation and subsequently increase biogas production and methane yield (Xu et al., 2012). In anaerobic digestion, hydrolysis is the rate-limiting step in the whole process. Hydrolysis rate needs to be improved to enhance the performance of an anaerobic digester. Factors such as raw materials, particle sizes and structural properties can affect the hydrolysis rates particularly during decomposition of high molecular compounds and granular substrates.

Zeynali et al. (2017) stated that ultrasonic pretreatment has been recognized as the most powerful method to disintegrate complex organic matter into soluble compounds prior to hydrolysis compared to thermal, chemical or biological pretreatment methods. The ultrasonic effect is based on monolithic cavitation with the effects of physical and chemical changes in the slurry (Dehghani, 2005). Gronroos et al. (2004) explained that the collapse of cavitation bubble will modify the chemical structure with the formation of free radicals during the sonication process. Complex structure will be broken up, and changes occur on the physical, chemical and biological properties of the substrate. This is also supported by Kwiatkowska et al. (2011) whom agreed that this physical disintegration will lead to an increase in biogas production and methane yield. Based on literature studies, there are many effects of ultrasonic pretreatment on FW which include able to increase soluble COD, total dissolved protein, soluble reducing sugars and increase the total amount of VFAs (Jiang et al., 2014; Deepanraj et al., 2017). In terms of physical changes, ultrasonic pretreatment has a great potential to convert the complex structure of feedstock such as FW into a simpler form to increase the reaction rate and thus reduce hydrolysis time. In terms of anaerobic biodegradability of FW, ultrasonic pretreatment shows a high percentage of TS, VS and COD removal which in turn increases the production of biogas and methane yield. In this study, detailed investigation have been conducted to see the effect of ultrasonic pretreatment to enhance the biodegradability of food waste including the impact on the particle size distribution, degree of disintegration and COD solubilization, morphological properties of food waste as well as anaerobic biodegradability of food waste.

In anaerobic digestion, digester performance and system stability are two main components that need to be considered to ensure high biogas and methane production. Digester performance includes methane yield, methane production rate and methane content. Meanwhile, the system stability for a digester is monitored through changes in pH, total alkalinity and total VFA. Maranon et al. (2016) found that CSTR showed good performance when recording the maximum value of methane production of 603 $LCH_4$/kg $VS_{feed}$ when co-digesting a mixture of 70% manure, 20% food waste and 10% sewage sludge ( TS 4 %) at 36 $^oC$ in a CSTR, for an OLR of 1.2 g VS/ L.day. The increase of OLR to 1.5 gVS/L.day caused a decline of 20-28% in methane production. Lower methane yields are observed when the digester operates at 55$^oC$. High volatile fatty acids are recorded in the reactor (820 mg/L) but no inhibition due to unionized ammonia (224 mg/L) indicates that the system is in stable condition. However, the increase in methane caused by the use of ultrasonic pretreatment against feed mixtures does not compensate for energy used during pretreatment. Elbeshbishy and Nakhla (2011) found that there was an increase in methane production rate 31.25% and 13% in single stage and two stage CSTR, respectively, during an anaerobic digestion on pulp waste (sonicated at 20 kHz, 500W, 24 min). Both digester show a relatively low performance but high efficiency in TCOD removal (43-73%) and VSS removal (36-59%). In the present study, the process performance is determined through the methane composition, methane production rate and methane yield. While the system stability is indicated by several parameters such as total alkalinity, pH and volatile fatty acid (VFA). The process efficiency is determined in terms of COD removal and VS removal.

The kinetic study is conducted to provide knowledge in order to predict the performance of biological treatment digesters (Senturk et.al, 2013). To describe and predict the performance of the system, the developed mathematical model can be used as a useful tool for conducting the kinetic analysis. Li et al. (2011) stated that mathematical models are the tools needed in the anaerobic treatment process to control and optimize the operations in addition to improve the efficiency, stability and operational costs. There are numerous other kinetic models that have been used for this purpose and reported in previous studies for anaerobic digestion such as Monod, Contois, Chen-Hashimoto, Michaelis-Menten, Modified Stover-Kincannon, Grau second-order multicomponent substrate removal, Haldane and others. In this study, kinetic parameters determination were described using Monod, Contois, Modified Stover-Kincannon and Grau second-order multicomponent substrate removal models on the performance of anaerobic digestion of sonicated food waste using CSTR.

Food waste, the largest composition of organic waste generated has the potential to improve biodegradability and bioavailability in the production of methane gas through anaerobic treatment. Although it is characterized as highly complex and inconsistent as feedstock in terms of composition, shape and size that may affect the amount of biogas or methane to be produced, this does not limit the effort to exploit food waste for producing beneficial products such as biogas and liquid fertilizer. To accomplish the concept of low cost waste recovery strategy, this study has been conducted using a single stage continuous stirred tank reactor (CSTR), as it is simple to construct and operate, low cost of capital needed compared to the other reactor designs, and it is better than multistage reactors due to the complex FW characteristics. Energy-saving measures can also be taken into consideration when ultrasonic pretreatment is carried out on FW where a low ultrasonic frequency of 20 kHz has been used and the short duration of sonication was also applied during pretreatment. The semi-continuous system is preferred in this study because this operation mode is still very limited and less reported in FW pretreatment as most previous studies using a batch system in their studies related to anaerobic digestion of sonicated FW.



In this study, the palm oil mill effluent (POME) anaerobic sludge collected from a 500 $m^3$ semi-commercial closed anaerobic digester system at Felda Serting Hilir, Negeri Sembilan was used as the source of inoculum to start-up the digester. Its ability to produce biogas within a short period of time once an anaerobic digestion operation is initiated can minimize lag phase and thereby improve digester performance and stability (Yaacob et al., 2006; Saidu et al., 2013). Two methanogen species present in POME sludge i.e *Methanosaeta sp.* and *Methanosarcina sp*. with *Methanosaeta concilii* was found as a dominant species in the digester (Tabatabaei et al., 2009). This causes POME anaerobic sludge to be a source of methanogens consortia in the anaerobic digestion process. Likewise in the kinetic study, none of literatures so far has documented the determination of kinetic parameters using the Monod, Contois, Modified Stover-Kincannon and Grau second-order multicomponent substrate removal kinetic models on the performance of anaerobic digestion of sonicated food waste using CSTR.

## 1.3 Research hypothesis

The hypotheses of this study are as follows:

1. The implementation of ultrasonic pretreatment prior to anaerobic digestion of food waste would enhance the solubilization of FW and thus increasing hydrolysis rate before fermentation, and subsequently increasing the production of biogas and methane yield.
2. Higher biogas and methane production can be obtained if the digester performance (methane yield, methane production rate, methane composition) and system stability (pH, total alkalinity and VFA) of the anaerobic digestion of sonicated food waste using CSTR were monitored and evaluated.
3. Kinetic parameters such as yield coefficient (Y), maximum specific growth rate ($\mu_{max}$), death rate ($k_d$), half velocity coefficient ($K_s$), saturation value constant ($K_B$), maximum utilization rate ($U_{max}$), multicomponent Grau second-order substrate removal coefficient ($k_s$), dimensionless Grau second order constant (*a* and *b*) and Y, $k_d$, $\mu_{max,}$ B (for Contois model) for the performance of anaerobic digestion of sonicated food waste using CSTR for biogas and methane production can be determined using the developed mathematical model such as Monod, Modified Stover-Kincannon, Grau second-order multicomponent substrate removal and Contois kinetic models.

## 1.4 Research objectives

The aim of this study is to obtain high biogas yield particularly methane through anaerobic digestion of sonicated food waste. Thus, the following objectives will be pursued:

1. To investigate the effect of ultrasonic pretreatment towards the physicochemical property changes and anaerobic biodegradability enhancement for methane production.
2. To evaluate the digester performance and system stability of the anaerobic digestion of sonicated and non-sonicated food waste at different organic loading rate (OLR).
3. To determine the kinetic parameters for the performance of the anaerobic digestion of sonicated food waste.

## 1.5 Scope of study

To achieve the above objectives, scope of the study are given as follows:

1. The anaerobic digestion of sonicated food waste and non-sonicated food waste was performed by continuous stirred tank reactor (CSTR) under controlled condition as the following:
   i. Temperature 35± 2$^{o}$C
   ii. Mixing 80 rpm
   iii. pH ranging from 6.8-7.2
2. Two different types of feed were introduced into the digester, namely sonicated and non-sonicated (control) food waste slurry. For sonicated sample, food waste slurry was sonicated for 10 minutes prior to feeding. The frequency and power of the ultrasonic processor used were 20 kHz and 500W, respectively.
3. The digestion process was conducted at 3 different organic loading rates (1.5, 2.5, 3.5 gCOD/L.day).
4. Food waste used throughout the study was collected from a cafeteria located in Malaysian Technology Development Corporation (MTDC) in Universiti Putra Malaysia (UPM) area.
5. The palm oil mill effluent (POME) anaerobic sludge collected from a 500m$^3$ semi-commercial closed anaerobic digester system at Felda Serting Hilir, Negeri Sembilan, Malaysia was used as a source of the inoculums.
6. The chemical characteristics of food waste will be analysed based on total solids (TS), volatile solids (VS), moisture content (MC), chemical oxygen demand (COD), pH, lipid/fat content, macronutrients (phosphorus, potassium), micronutrients (Ni, Fe, Zn, Cu, Cd, Pb, Mn, Al, Mg), percentage of carbon (C), hydrogen (H), nitrogen (N), oxygen (O) and sulphur (S). The physical characteristics of the food waste were analysed based on particle size distribution and morphological structure of the food waste sample.
7. The semi-continuous study was performed to achieve the following:
   i. The system performance in terms of methane composition, methane production rate and methane yield.
   ii. The system stability in terms of total alkalinity, pH and volatile fatty acids.
   iii. The process efficiencies in terms of COD removal and VS removal.
8. Kinetic parameters determination were described using Monod, Modified Stover-Kincannon, Grau second-order multicomponent substrate removal and Contois kinetic models.

## 1.6 Organization of thesis

This thesis is separated into five (5) contrast parts which portray briefly the general research work that have been conducted. Chapter 1 covers the overview or research background of the thesis, problem statements which discusses the rational and basis to identify the research directions to be followed, scope of study and specific objectives to be achieved. Chapter 2 reviews the technical aspects of anaerobic digestion process involved and factors affecting anaerobic digestion process. Ultrasonic pretreatment as one of the pretreatment technologies in anaerobic digestion is highlighted in details including mechanisms of ultrasonic disintegration, delivery of ultrasonic energy and expressions for sample disintegration as well as the merits and demerits of ultrasonic pretreatment. The kinetics of anaerobic digestion involving microbial growth rate, substrate utilization rate and kinetic models such as Monod, Modified Stover-Kincannon and Grau second-order multicomponent substrate removal and Contois are also discussed in detail. Previous results and analysis are discussed and compared for better understanding of the directions of this present work.

A general overview of methodologies used throughout the research is presented in Chapter 3. In this chapter, an overall experimental design flowchart with detailed experimental set up is elaborated which involves anaerobic biodegradability test on the sonicated food waste, start-up of the continuous stirred tank reactor (CSTR) and operating condition of the biodigester throughout the study. Detailed explanation on inoculum, feedstock, chemicals, apparatus and equipment used as well as analytical techniques involved throughout the whole experimental process is also highlighted. Chapter 4 further outlines the results obtained as well as discussion of the results that are obtained from the experiments with regards to the objectives of the research. This chapter comprises three (3) main sections. First section discusses in detail the composition of food waste being discarded and characterization of food waste chemically and physically and elaborates the effect of ultrasonic pretreatment that has been used to enhance the biodegradability of food waste including the impact on the particle size distribution, degree of disintegration and COD solubilisation, morphological properties of food waste and anaerobic biodegradability of food waste. The overall picture regarding the ongoing anaerobic digestion of sonicated food waste (using anaerobic biodigester) has been evaluated based on the process performance and system stability and this topic is explained in detail in the second section. The process performance is determined through the methane composition, methane production rate and methane yield. While the system stability is indicated by several parameters such as total alkalinity, pH and volatile fatty acid (VFA). In the third section, the kinetic parameters involved in the anaerobic digestion process are determined and discussed. Chapter 5 presents the conclusion of overall findings from the current studies. In addition, some recommendations for future research related to this area in order to give the implication and importance for further study will be covered in this chapter.

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**LIST OF PUBLICATIONS**

**N.H.M Sabiani,** A. Idris, W.A.W.A.K Ghani & A.S Baharuddin (2015) The Effect of Ultrasonic Energy on the Enhancement of the Biodegradability of Food Waste, Energy Sources, Part A: Recovery, Utilization and Environmental Effects, 37:13, 1397-1405, doi:10.1080.15567036.2014.915362

**N.H.M Sabiani,** W.A.W.A.K Ghani, A. Idris, & A.S Baharuddin (2019) Characterization of food waste as a feedstock for methane production via anaerobic digestion: Malaysia perspective. Energy Sources, Part A: Recovery, Utilization and Environmental Effects. Article submited

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