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Recovery of biogas from food waste using treated and untreated anaerobic digestion

N Wid^{1,2*} and N Raudin¹

¹Faculty of Science and Natural Resources, Universiti Malaysia Sabah, Jalan UMS, 88400, Kota Kinabalu, Sabah, Malaysia

²Water Research Unit, Faculty of Science and Natural Resources, Universiti Malaysia Sabah, Jalan UMS, 88400, Kota Kinabalu, Sabah, Malaysia

*Corresponding author: newati@ums.edu.my

Abstract. Biogas is an environmentally friendly, clean, and low-cost renewable energy source. This alternate sustainable source can be used for heat, power, or fuel and making it a good alternative for renewable energy. Biogas can be produced through anaerobic digestion of biodegradable organic materials without oxygen and can be improved by performing thermal treatment. In this study, biogas was recovered using treated and untreated food waste. The characterisation of food waste for its suitability for an anaerobic digestion process was studied by its total solids, volatile solids, pH value, and C/N ratio, following the APHA standard. The anaerobic digestion was treated in a water bath at a temperature of 70 °C for 45 minutes. The Biochemical Methane Potential method was conducted to determine the biogas production from treated and untreated anaerobic digestion. The treated and untreated anaerobic digestion were anaerobically digested at a mesophilic temperature of 37 °C and at a speed of 100 rpm for 15 days. The biogas produced was measured daily by using water displacement method. The total production of biogas for treated anaerobic digestion was 198.70 mL, meanwhile for untreated anaerobic digestion was 185.50 mL. Treated anaerobic digestion showed a slightly higher production of biogas in 15 days by 13.2 mL. The biogas yield obtained for treated and untreated anaerobic digestion was 51.21 mL/g and 47.80 mL/g for every 1 g of total wet sample. This study found that thermal treatment of anaerobic digestion helps in the degradation of organic content in food waste for anaerobic digestion, which leads to higher biogas recovery.

1. Introduction

Food waste (FW) generation in Malaysia shows an increasing trend mostly due to economic growth, urbanisation and increase in population which is predicted to reach 37.4 million by the year of 2030 [1]. All the food wasted is mainly from different daily sources such as fish, seafood, vegetables, fruits, meat, dairy products, and cereals. Malaysians are reported producing 16,688 tonnes of food waste daily, enough to feed 2.2 million people for daily meals for a day [2]. FW is generated at home or in cafeterias, canteens, restaurants, residential areas and food industries. Some FW compositions contain of non-edible material, such as fruit or vegetable peels, shells, and bones [3].

In this study, anaerobic digestion (AD) technique was used to manage FW in the right way at once to recover biogas. The total amount of biogas produced from AD process depends on the amount and type of organic waste fed into the digester. It has been stated that temperature and pH are more likely to be the most important factors in the rate of digestion and biogas production [4]. The AD process also



affected by total solids (TS) and volatile solids (VS) content of substrates. Biogas including methane and carbon dioxide are formed as by-product of degradation of organic materials by hydrolysis, acidogenesis, acetogenesis, and methanogenesis processes of AD [4]. Hence, a treatment of substrate before the AD process can be applied to optimal digestion and biogas production [4]. The treatment process of AD for wastes can be classified into a few, for instance, thermal, alkali, mechanical, biological, ultrasonic, ozonation, and co-digestion [4]. Out of all, the treatment by thermal happens to be the most commonly used for AD. Therefore, the effect treatment by thermal on FW was investigated. From previous findings, the thermal treatment at temperature of 70 °C with retention time of 45 minutes have shown the most effective digestion process for FW in producing high biogas using BMP technique [5]. In this study, the recovery of biogas was investigated by using kitchen waste from expired and left-over food as AD substrate, and primary sludge was used as inoculum to boost the reaction. The FW was classified as treated and untreated before starting the AD process. The thermal treatment method from previous study [5] was applied to treat the FW sample prior to AD. The characterisation of FW samples, such as pH, total solids (TS), volatile solids (VS) and C/N ratio were studied and then the biogas recovery was investigated.

2. Materials and methods

2.1. Collection and preparation of substrate and inoculum

The FW sample was collected from a household kitchen containing of rice, stale bread, vegetables and fruit peels. The non-bio-degradable types of waste, such as plastics, bottles, cans, iron, and glass, were separated and discarded before the FW materials were weighed. FW samples were stored in a freezer and maintained at a temperature of 4 °C prior to use.

Primary sludge (PS) from municipal wastewater was collected from the Water Treatment Plant in Papar, Sabah. It was used as inoculum to boost up the AD process. The inoculum was pre-incubated at 37 °C for degasification of organic matter content for 5 days prior to use [6]. This was to ensure the organic matter contained in the inoculum will not affect the results of this study [7]. Both FW and PS samples were collected in November 2021 or when needed.

2.2. Determination total solids (TS) and volatile solids (VS) content

The determination of TS and VS content was carried out according to the standard method described by [8] in triplicates. The FW sample was weighed, placed in a crucible, and put in an oven at 105 °C for 24 hours. After 24 hours, the crucible was cooled down in a desiccator for 1 hour before being weighted and the TS (%) of the FW sample was calculate using equation (1).

$$\text{Total Solid (TS) \%} = \frac{W_t - W_c}{W_s - W_c} \times 100 \quad (1)$$

Where W_t is weight of dry sample and crucible (g) W_c is weight of crucible (g) and W_s is weight of wet sample and crucible (g).

The process was continued with determination of VS from the TS content in FW. The FW was heated in a furnace at a temperature of 550 °C for 4 hours. After 4 hours, the crucible was cooled down in a desiccator for 1 hour before being weighted [8]. The sample was weighted and the total VS (%) of the FW sample was calculated using equation (2).

$$\text{Volatile Solid (VS)\%} = \frac{W_t - W_{vs}}{W_t - W_c} \times 100 \quad (2)$$

2.3. Determination of pH

The determination of pH was carried out according to the method described by [8] in triplicates. The FW sample was mixed with distilled water in a ratio of 1:10 in a 250 mL bottle. Next, it was put on a shaking orbital at 130 rpm for 24 hours. After 24 hours, the mixture was then filtered using vacuum filtration. The filtrate liquid of FW was collected and used to determine the pH value using a pH meter.

2.4. Determination of C/N ratio

The determination of total carbon (TC) was conducted using a method concentration by sulphuric acid-UV as described by [9]. The spectrometric absorbance of glucose, $C_6H_{12}O_6$ (Merck) solutions was used to evaluate the TC and then was examined using Ultraviolet-Visible (UV-Vis, Perkin Elmer) spectrophotometer at 315 nm. The TC of FW was determined using a ratio of 1:10 (w/v) of FW to distilled water. Then was placed in a shaking orbital (MMM-Group) for 24 hours at speed of 130 rpm. Then, it was filtered to obtain the liquid form of FW. In 100 mL volumetric flask, the FW filtrate and glucose then were mixed with 3 mL of H_2SO_4 and diluted till the mark was reached using distilled water before being left for 20 minutes. The sample concentration was determined using the equation $y = mx + C$.

The concentration of Total Kjeldhal Nitrogen (TKN) was determined using three main steps: digestion, distillation, and titration [10]. About 2 mL of raw FW was added to the digester tube for digestion. Then, 10 mL of concentrated sulphuric acid was added into the digester tube, followed by a tablet as catalyst which consists of about 7 g of potassium sulphate (K_2SO_4) and 0.2 g of copper sulphate ($CuSO_4$). Then, the tube was fitted onto the digester, including the tube with the blank sample. It was heated in the digester and digestion was considered completed once a clear transparent or a clear turquoise colour product was obtained. For distillation, the process was carried out by inserting the digester tube into the Kjeldhal machine and the tube was fitted with a blank sample into a holder to calibrate the machine, followed by the sample tubes. For titration, 3 drops of methyl red indicator were added into a 250 mL conical flask or the receiving end prior to titration process. A 0.1 M HCl was titrated with the collected distillate solution until the colour changed to pale pink.

2.5. Preparation of treated and untreated food waste

About 1.6 g VS of wet samples of FW were prepared separately into two parts: treated and untreated. The ratio of FW to inoculum of primary sludge used for AD process was 1.5:1 with a working volume of 200 mL in a 250 mL of Duran bottle. Therefore, 120 mL was the amount of wet FW sample and distilled water, and another 80 mL was the inoculum of primary sludge. The treated FW was done by thermal at a temperature of 70 °C for 45 minutes in a water bath [5] prior to AD process. Meanwhile, the untreated of wet samples of FW was ready to be used to carry out the AD process along with the treated FW. Before AD, both treated and untreated FW were added with 80 mL of inoculum in the 250 mL of Duran bottle and underwent a simultaneous AD process for 15 days for biogas recovery investigation.

2.6. Preparation of BMP method

A Biomethane Potential (BMP) method was performed in laboratory scale using a 250 mL Duran bottle with a working volume of 200 mL [11]. The ratio of inoculum of primary sludge to the raw FW sample was 1.5:1 [12]. The optimum temperature of the digester reactor was set for mesophilic condition at 37 °C [13]. The optimum pH was set in the range of 6.8 to 7.2 [12] and was maintained by using 0.1 M of NaOH and 0.1 M of HCl, respectively, to ensure the highest production of biogas. The BMP method was carried out in a shaker incubator for 15 days.

2.7. Water displacement method

The water displacement method was used to determine the volume of biogas produced [7]. The biogas released was displaced equally with the water inside the burette. After that, the amount of water displaced inside the burette was recorded daily for 15 days and was corrected to standard temperature

and pressure of 0 °C and 1 atm. In theory, methane gas production accounted for 70% of total production of biogas, and about 30% is in the form of carbon dioxide and other traces number of gases [14].

3. Results and Discussion

3.1. Characterisation of food waste

The physical characterisation of FW was described using TS (%), VS (%), pH value and C/N ratio (Table 1). The TS (%) content of FW in this study was found to be 47.26% (± 1.38), which is considered high compared to previous study with 23.7% (± 0.15) [5]. The high TS content may be due to high solid materials in the FW used in this study, such as vegetable and fruit peels, bread and rice. Previous study has also mentioned that an ideal concentration of TS content of FW ranged from 20% to 50% [5] to carry anaerobic digestion.

The VS (%) content of FW was 87.17% (± 1.49), slightly lower as reported by previous study with 96.00% (± 0.20) [5]. The VS content is not only can be used to evaluate the biogas potential of the substrate and its degree of degradation, but also the efficiency of performance in the AD system [15]. According to [15] the yield production of biogas was directly proportional with the percentage of volatile solid content. In AD systems, changes in TS content will result in changes in microbial morphology and performance of AD [15,16]. Therefore, the TS and VS in FW employed in this study were still appropriate to use as substrates in the AD process.

The pH value of FW sample was 5.93 (± 0.04), within the range as reported by previous study with 6.86% (± 0.06) where they used kitchen FW [17]. For the AD process to work effectively, the ideal pH is in the range of 7.0 to 8.5 [5]. It is suggested, a buffer, for example sodium bicarbonate (NaHCO_3), can be employed to treat substrates with pH value less than 7.0 [17].

The C/N of FW was 6.82 possessed a low C/N value due to unbalanced type of FW selected in this study. The type of FW sample used mostly consists of vegetables and fruit peels. Basically, vegetables are one of the contributors to source nitrogen content in food [18], resulting in a low C/N ratio of FW obtained in this study. The ideal C/N ratio for AD optimal condition is between 20 to 35 [19]. To prevent ammonia accumulation from excess nitrogen content during AD due to low C/N ratio, it is recommended to use large amount of FW sample with high carbon content such as carbohydrate and lipid. A previous study suggests to co-digest FW or organic substrate with crop residue or agricultural waste to increase or obtain the optimum C/N ratio [20]. However, during the AD process in this study, FW was mixed with inoculum of primary sludge with ratio of 1.5:1. Inoculum of primary sludge had high carbon content, 43.20% (± 3.0) [21]. Thus, low C/N ratio in FW sample was then stabilized by the addition of inoculum of primary sludge. Therefore, the possibility of an increase in the C/N ratio for the AD in the digester is higher. Table 1 shows the physical characterisation of FW.

Table 1. Physical characterisation of FW.

Parameter	Unit	Value
Total Solids (TS)	%	47.26 $\pm$ 1.38
Volatile Solids (VS)	%	87.17 $\pm$ 1.49
pH	-	5.93 $\pm$ 0.04
Total Kjeldahl Nitrogen (TKN)	mg/L	140
Total Carbon (TC)	mg/L	956
C/N ratio	-	6.82

3.2. Determination of biogas recovery

The observation of the recovery of biogas for treated and untreated AD of FW was conducted for 15 days by adapting the thermal treatment method for the treated AD which is at 70 °C for 45 minutes [5]. The treated AD reactor showed a slight increase in biogas production, with 198.70 mL compared to 185.50 mL for the untreated AD reactor. This shows the difference of 13.2 mL in the total production

of biogas. According to a previous study [5], thermal treatment of FW able to increase the rate of AD biodegradation which resulted in the enhancement of methane in the biogas production [22]. Treatment by thermal induces macromolecule deflocculation, which increases the surface area of the substrates, as reported by previous study [23]. It has been demonstrated that high surface area of substrate will improve the interaction with the microbial population, which resulting in more organic matter being digested into biomethane [23]. This indicates that the treatment by thermal induces the destruction of complex compounds as well as an increase in the soluble organic materials. Since the sample of FW used for the treated FW was also an easy biodegradable material, a higher thermal treatment at temperature exceeding 120 °C and long treatment period may result in loss of potential methane in biogas production from FW [24]. Thus, it was recommended to have a mixed and balanced content of FW for better degradation process such as carbohydrates, lipids, and proteins in the FW substrate [24]. Figure 1 shows the cumulative production of the biogas (mL).

The pH value in both AD reactors showed significant decrease in the pH range of 3.5 to 5.5 started from day-3 to day-6 due to the occurrence of acidogenesis phase of AD. Volatile fatty acids (VFA) in a form of acetate was produced in acidogenesis phase associate with small amount production of lactate and ethanol inside the AD reactor [25]. This resulted in slightly acidic condition due to low pH inside the AD reactor and lead to disruption in the production of biogas.

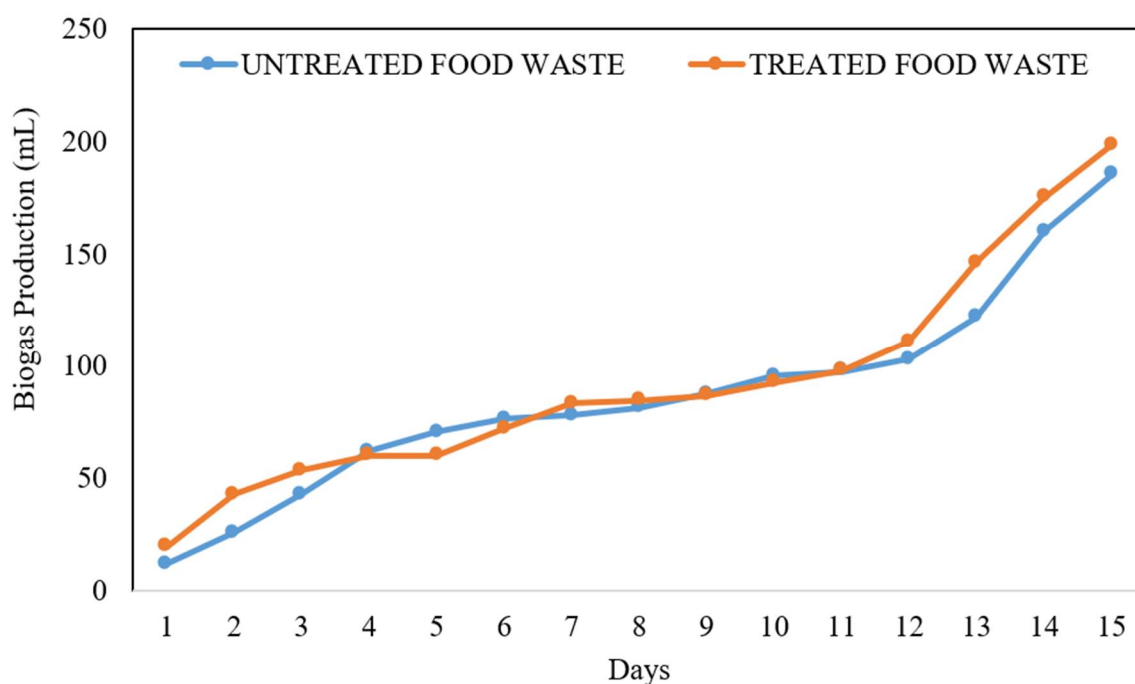


Figure 1. Graph of Cumulative Biogas Production in 15 Days.

The cumulative production in figure 1 shows that treated and untreated FW of AD were steadily increasing in the biogas production. Methanogenesis phase was occurred starting from day-11, as shown by the rapid increase of biogas in both treated and untreated AD reactors. The bacteria in this phase was responsible to convert the hydrogen and acetic acid into methane and carbon dioxide [26]. However, the low recovery of biogas due to low C/N ratio or high nitrogen level obtained from FW characterisation continuously increases the microbial population, resulting in a low biogas yield during the methanogenesis phase of AD [27].

4. Conclusion

The characterisation of FW for its suitability for anaerobic digestion was studied by its total solids ($47.26\% \pm 1.38$), volatile solids ($87.17\% \pm 1.49$), pH value (5.93 ± 0.04), and C/N ratio (6.82). The BMP method was used to conduct the AD process for both treated and untreated AD in 15 days. Treated AD produced 198.70 mL of biogas, while untreated AD produces 185.50 mL of biogas. Treated AD shows slightly higher biogas production in 15 days by 13.20 mL. The yield of biogas obtained for treated and untreated AD is 51.21 mL/g and 47.80 mL/g of wet sample of FW, respectively. Therefore, the treated FW using 70 °C for 45 minutes helps in the degradation of organic content in FW prior to AD and enhanced the production of biogas.

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