

Enhancement of Methane Production in Anaerobic Digestion of Food Waste using Thermal Pretreatment

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ABSTRACT

Anaerobic digestion (AD) is an energy production process and food waste is a potential feedstock. The main biochemical reactions are Hydrolysis, Acidogenesis, Acetogenesis, and Methanogenesis. The hydrolysis step acts as the rate-limiting reaction and the pretreatment of the feedstocks can be used to support this step. In this research, thermal pretreatment was used as a potential method for food waste pretreatment. Six different pretreatment conditions were used: two different temperatures (80°C and 100°C) and three different pretreatment times (30, 60, and 90 min). The Bio-Methane Potential (BMP) test was conducted using 120 mL serum bottles for 20 days to determine the most suitable pretreatment conditions. An experiment was also conducted at the selected optimal conditions (80°C for 90 min) using a small-scale bioreactor against the control with a NaHCO₃ buffer solution. The highest Soluble Chemical Oxygen Demand (SCOD) was observed at 100°C for 90 min. The optimal pretreatment was selected as 80°C for 90 min, which produced 14.75 mL/g VS of methane while the control produced 8.64 mL/g VS in BMP test. After a few days, the methane production started to slow down due to a decrease in pH. When a buffer was added, a specific methane yield of 120.13 mL/g VS was observed in the small-scale bioreactor. This was an 11.24% increase compared to the buffered control without thermal pretreatment. In conclusion, thermal pretreatment has a potential to enhance the AD but it is economical to use with less biodegradable waste than food waste.

1. INTRODUCTION

At present, the world generates about 2.01 billion tonnes of solid waste per year (known as Municipal Solid Waste (MSW)), and it is projected to increase to 3.40 billion tonnes by 2050. The average personal waste generation is about 0.74 kg/day (Kaza et al., 2018). Food waste accounts for the largest fraction of MSW, and one-third of the food produced goes to waste. Several methods are used to manage organic waste such as landfilling, incineration, composting, and AD. According to the World Bank, the most practiced method of dealing with MSW is landfilling. Landfilling causes many environmental effects such as groundwater contamination, the release of Greenhouse gases (GHGs), odors, and acts as a breeding ground for rats, insects, and other scavenging animals (Hoornweg and Bhada-Tata, 2012). Current food waste generation is estimated at 1.3 billion tonnes

per year, and the GHGs emission is estimated at 3.3 billion tonnes of CO₂/year (FAO, 2021). About 18% of global warming occurs due to these processes that can be easily avoided by diverting landfilling to AD or composting (Ward et al., 2008). Therefore, it is important to divert organic waste, especially food waste from landfills to AD processes.

AD is a complex biological reaction with four distinguishable steps: Hydrolysis, Acidogenesis or Fermentation, Acetogenesis, and Methanogenesis. The hydrolysis step slows down the AD because (in hydrolysis) complex molecules are broken down into simpler molecules (such as carbohydrates into glucose, proteins into amino acids, and lipids into fatty acids). Therefore, hydrolysis requires much more time than the other steps (Zhang et al., 2014; Merlin et al., 2014). Slow processes require larger reactors, and higher initial and operating costs. These issues can be

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addressed by employing pretreatment methods (Merlin et al., 2014). There are several methods to improve AD, and pretreatment can assist in the hydrolysis step. Many researchers have reported different pretreatment methods such as biological, mechanical, chemical, and thermal methods on different substrates such as organic-portion of MSW, activated sludge, lignocellulose biomass, and manure (Carrere et al., 2016). Only a few researchers have focused on food waste using thermal pretreatment as a potential method. Moreover, these researchers focused on high temperatures for short duration or low temperatures for long duration (Ariunbaatar et al., 2014; Krishna and Kalamdhad, 2014; Naran et al., 2016). In this research, high and low temperatures were examined for short pretreatment times using food waste as the substrate.

Thus, the objective of this study is to determine the optimal thermal pretreatment conditions that are suitable for food waste, to enhance methane production. A bio-methane potential test was conducted to measure the effects of pretreatment temperature and time. The scope of this study was limited to food waste and two thermal pretreatments with three short time pretreatment durations. The potential use of thermal pretreatment was further investigated using small-scale anaerobic reactors.

2. METHODOLOGY

2.1 Substrate and inoculum

Food waste was used as the feedstock and granular sludge was used as the inoculum. The feedstock was collected from the engineering faculty canteen at Thammasat University, Thailand, and indigestible materials such as bones and seashells were sorted out. The waste samples were then ground and stored at 4°C until use. Granular sludge was collected from an upflow anaerobic sludge blanket anaerobic digester (UASB) at Pathum Thani Brewery Co., Ltd (Pathum Thani, Thailand). The TS content was maintained at 5% by diluting to maintain a lower VS content so that the digestion can be carried out easily in a small-scale reactor.

2.2 Characterization

The C, H, N, and S elemental contents of the food waste and inoculum were determined using an elemental analyzer (THERMO FLASH 2000), and the C:N ratio was calculated following USDA (2015). The Total Solid Content (TS) and Volatile Solid content (VS) were measured according to the Environmental

Protection Agency (United State) method 1684 (USEPA, 2001).

2.3 Thermal pretreatment

Two different pretreatment temperatures were used with three different durations, which were 80°C and 100°C for 30 min, 60 min, and 90 min. These conditions were selected to determine the suitable thermal pretreatment condition at low temperatures and short treatment time. Lower pretreatment thermal conditions are economically viable compared to higher temperature or longer pretreatment time (Zhang et al., 2015). Furthermore, these conditions are selected so that it also can be used as conditioning process which agrees to the EU Regulation EC1774/2002 (Ariunbaatar et al., 2014). Treatments were conducted in glass bottles and 200 mL of sample were used for each pretreatment. The thermal pretreatment was conducted in a preheated forced air oven. The samples were stored at 4°C after pretreatments until used further.

2.4 Solubilization

SCOD was measured to determine the effects of pretreatment on solubilization. The pretreated samples were filtered using GF/C filter papers. The chemical oxygen demand (COD) of the filtrate was measured according to the closed reflux titrimetric method, following the standard methods for water and wastewater treatment (APHA, 2017).

2.5 Bio-methane potential test

BMP tests were conducted to determine the effectiveness of pretreatments on enhancing methane generation. BMP tests were conducted in 120 mL serum bottles. Substrate and inoculum were mixed in a 1:1 ratio (g VS to g VS). After mixing, the pH was adjusted to 7 by adding 1 M NaOH or 1 M HCl. The sample size was 75 mL, leaving 45 mL of headspace. The serum bottles were sealed with rubber stoppers and aluminum (Al) crimpers. The headspace was replaced by N₂ gas to facilitate AD. Then, the serum bottles were covered with Al foil and placed in a shaking incubator at 37°C. Two replicates were performed for each sample. The BMP tests were conducted for 20 days. Biogas production in each day was measured by the displacement method using a disposable syringe. The methane content was measured by a gas chromatograph (GC) (PerkinElmer, USA), equipped with a thermal conductivity detector (TCD) with a Porapak Q, 50/80 mesh column. The

operating temperatures of the column, detector, and injector were 45, 100, and 100°C, respectively. Helium gas was used as the mobile phase with a flow rate of 25 mL/min. The produced methane amount was calculated accordingly (Naran et al., 2016; Ta and Babel, 2019). The conditions with the highest cumulative methane yield were selected as the optimal pretreatment.

2.6 Small-scale bioreactor

After selecting the optimal thermal pretreatment conditions, small-scale bioreactors (2 L) were used to determine the effects of the thermal pretreatment on

food waste. In this experiment, the pH was controlled in both control and the reactor with thermally pretreated substrate by adding NaHCO_3 buffer (300 mg/g VS) (Habarakada and Babel, 2020). As described in the BMP tests, 1:1 was used for the inoculum to feedstock ratio. A final working volume of 1 L was used for the AD. The headspace was purged with N_2 gas. The experiment was conducted at room temperature (28-30°C). The experimental setup is shown in Figure 1. For comparison, a control reactor was also run in a similar way where the substrate was not thermally pretreated.

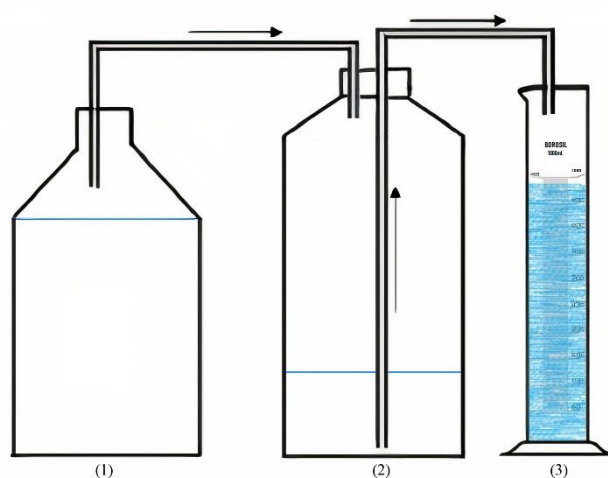


Figure 1. Schematic diagram of the bioreactor and the gas collecting apparatus. (1) 2 L glass reactor; (2) gas collecting bottle; (3) volumetric flask

3. RESULTS AND DISCUSSION

3.1 Characterization of food waste and inoculum

The characteristics of the substrate and inoculum are shown in Table 1. The food waste mainly consisted of cooked rice, noodles, pasta, meats, eggs, seafood, and vegetables. Occasionally raw vegetables and green leaves were also found. The composition of food waste varies as food preferences are fluctuating. The VS and TS of the substrate and feedstock were $90.94 \pm 0.89\%$ and $21.91 \pm 0.8\%$, respectively, while the VS and TS of the inoculum were $89.01 \pm 3.8\%$ and $4.54 \pm 0.5\%$, respectively. The TS of the feedstock was decreased to $5 \pm 0.5\%$ by diluting with distilled water. The pH of the feedstock and inoculum were 5.2 ± 0.4 and 7.83 ± 0.10 , respectively. The sample pH was measured between 4.5 and 6, after mixing with the inoculum. The final pH was adjusted to 7 by adding 1 M NaOH. The C/N ratio of the feedstock and inoculum was calculated as 18.03:1 and 4.76:1, respectively. The C/N ratio of the mixture was

determined as 10.95:1. According to Divya et al. (2015), a suitable C/N ratio for AD is from 10 to 35.

Table 1. Characteristics of substrate and inoculum

Parameter	Substrate	Inoculum
C	$53.57 \pm 0.23\%$	$47.02 \pm 0.52\%$
H	$8.06 \pm 0.03\%$	$6.62 \pm 0.08\%$
N	$2.97 \pm 0.42\%$	$9.87 \pm 0.14\%$
S	0	$1.11 \pm 0.26\%$
pH	5.2 ± 0.4	7.83 ± 0.10
C:N	18.03:1	4.76:1
TS	$21.91 \pm 0.8\%$	$4.54 \pm 0.5\%$
VS	$90.94 \pm 0.89\%$	$89.01 \pm 3.8\%$

3.2 Effects of pretreatment on solubilization

The solubilization of organic compounds was measured after pretreatment. Pretreatment aids AD, either by increasing the SCOD through solubilization or breaking down the larger molecules into smaller

molecules or both (Kim et al., 2003). Figure 2 shows the measured SCOD values after each pretreatment. The highest SCOD was observed in the 100°C for 90 min pretreatment which was a 5% increase, compared to the control. The SCOD values increased with an increase in the pretreatment time and temperature. According to the T-test, there is no significant difference ($p>0.05$) between the SCOD values at 100°C for 90 min pretreatment and the control (Table S1). Previous researchers also have observed the same pattern where higher temperatures had higher SCOD

values in food waste (Kumar et al., 2020). Menon et al (2016) has obtained the highest SCOD increase for the 130°C pretreatment. The increase of SCOD level was due to the solubilization of extracellular matter and extracellular polymeric substances which leads to the increase of SCOD in the waste (Elbeshbishy and Nakhla, 2011). The increase of SCOD increases the nutrient content in the soluble state, which means that bacteria can utilize these nutrients much easier and quicker.

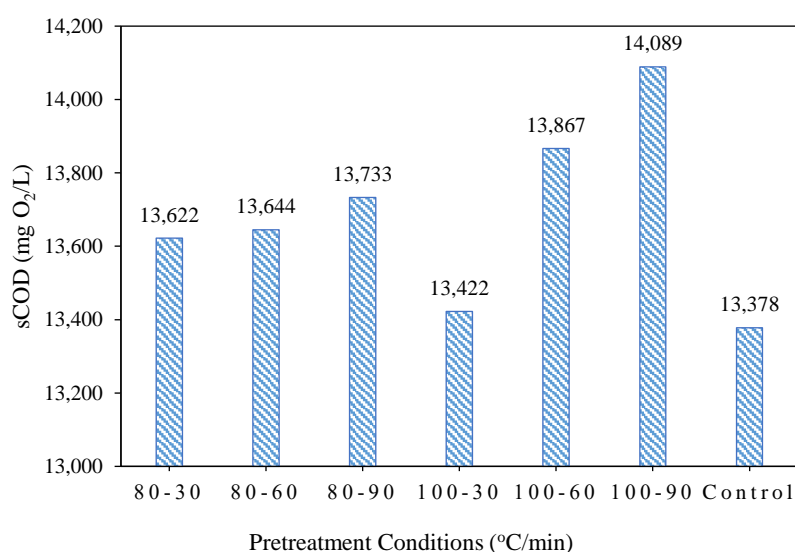


Figure 2. Changes in SCOD after thermal pretreatment

El Gnaoui et al. (2020) observed the highest solubilization in food waste at the 100°C pretreatment. The solubilization increased with the increase of temperature. Their study observed higher solubilizations in shorter pretreatment durations compare to this research where the highest solubilization was observed at 30 min pretreatment. The composition of waste plays a major role as optimum solubilization can be achieved with different pretreatment conditions. However, higher temperature pretreatments give higher SCOD levels and depending on the type, composition, and digestibility of the waste, the treatment duration can vary. A recent research study reported higher SCOD levels at pretreatment conditions of 170°C for 60 min with an initial TS of 150 g/L. This indicates that the substrate TS level is also a major factor when determining the most suitable thermal pretreatment conditions for food waste AD (Zhang et al., 2021).

3.3 Bio-methane potential

The results of methane generation after thermal pretreatment are shown in Figure 3. Figure 3(a) shows the cumulative methane production measured for 20 days, Figure 3(b) shows the cumulative methane production at the end of the 20-day BMP test, and Figure 3(c) compares the daily methane generation between the best pretreatment and the control.

As shown in Figures 3(a) and 3(b), the highest methane yield was observed in the 80°C for 90 min pretreated samples, followed by 80°C for 60 min. This increase can be explained by the deflocculation of macromolecules. Due to the deflocculation of macromolecules by the thermal pretreatment, the surface area is increased for the reactions, causing an increase in methane production (Ariunbaatar et al., 2014). Pretreated samples at 100°C for 60 min and 90 min did not show any (substantial) improvement compared to the control. No visible lag phase was observed in all pretreated samples and the control.

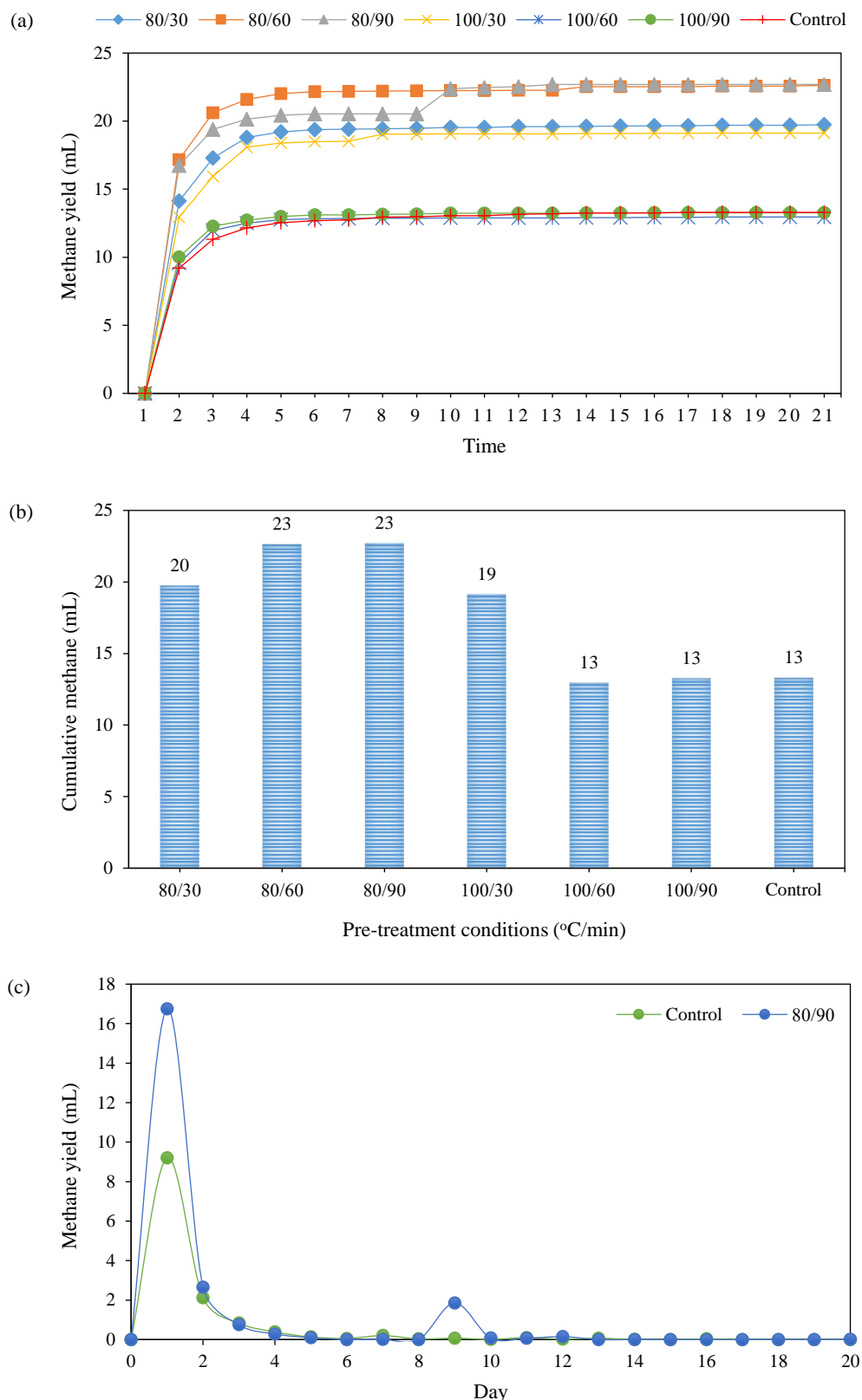


Figure 3. (a) Cumulative methane production with time at different pretreatment conditions, (b) Cumulative methane production after 20 days, (c) Daily methane production comparison between the 80°C/90 min and control

Several BMP tests were performed to verify the data. In all the BMP tests, the same pattern was observed. The high biodegradability of food waste may be the reason for no visible lag phase. More than

65% of the cumulative methane yield was observed within the first 24 h. In all cases, more than 90% of methane was observed within the first 4 days. The methane production drastically decreased after a few

days (Figure 3(c)), due to a decrease in pH as methanogenic bacteria are susceptible to changes in media pH. The average pH at the end of the BMP test of the control and the 80°C for 90 min pretreated sample was 4.07 and 4.04, respectively. A pH decrease was observed in all the pretreated samples at the end of the BMP tests. This may be due to the initial high VS content of the feedstock. Although the initial TS content was decreased to 5%, the available VS content was high enough for the reactor to produce a high amount of Volatile Fatty Acids (VFA). The accumulation of VFA in anaerobic media can result in a pH decrease (Cho et al., 1995). The pH was adjusted to pH 7 at the beginning of the BMP test. However, this was not enough to maintain the AD process, as the production of VFA caused a decrease in the media pH. In AD, different reactions prefer different pH levels,

as pH is a governing factor in microbial growth. Hydrolytic bacteria prefer a pH level of 5.5-6.0, while acidogenic and acetogenic bacteria prefer a pH level of 6.0-7.0. Methanogenic bacteria prefer a pH level of 6.5-7.5 (Leung and Wang, 2016). Therefore, it is clear that the methane generation was affected by the pH variation after a few days of the BMP test. This can also be observed in Figure 3(a) and 3(c) where there is no substantial amount of methane produced after few days.

Figure 4 shows the cumulative biogas production after 20 days of BMP tests. The highest cumulative biogas production was observed in the 80°C for 30 min pretreatment. The 80°C for 30 min pretreatment and 80°C for 90 min pretreatment had a 12% and 3% increase in cumulative biogas yield respectively, compared to control.

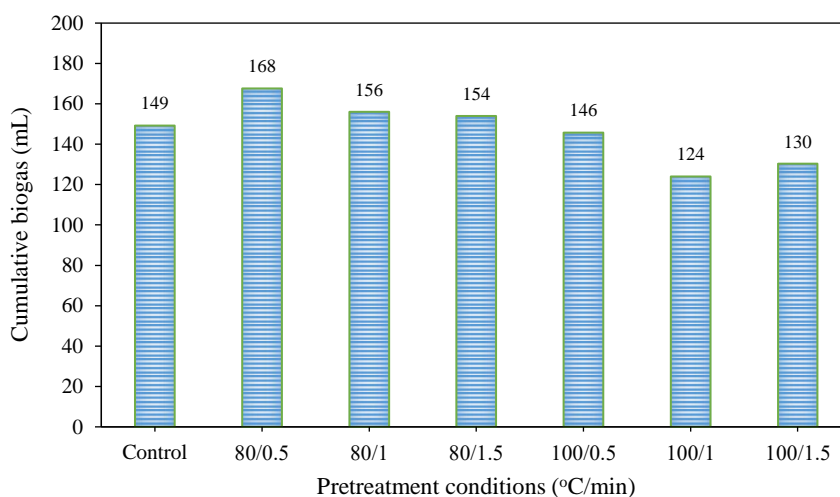


Figure 4. Cumulative biogas production at different pretreatment conditions after 20 days

Although the increase of biogas production was less in the 80°C for 90 min pretreatment compared to the 80°C for 30 min pre-treatment, the cumulative methane yield was higher. In the 100°C pretreatments, the cumulative biogas yield was less than the control. This biogas decrease may be due to the occurrence of the Maillard reaction. Researchers have reported that the Maillard reaction can occur between amino acids and reducing sugars at temperatures above 80°C (Menon et al., 2016). The end products of this reaction are not easily biodegradable. Hence, this can decrease methane production. The possibility of the Maillard reaction increases with an increase in the pretreatment temperature and time. This could be the reason for low biogas generation in the 100°C treatments (Ariunbaatar et al., 2014; Eskicioglu et al., 2007). The daily methane yield (Figure 3(a)) in this study also

showed no improvement compared to control at higher temperatures. Some researchers have reported a slower methane generation after pretreatments using higher temperatures compared to the control. Also, they have observed higher Maillard reaction products when feedstock concentrations are high (Ortega-Martínez et al., 2021).

In the 80°C for 90 min pretreatment, the volume of methane produced per one gram of VS is 14.75 mL, and the control produced 8.64 mL. These results are lower than those reported by Cho et al. (1995). They observed more than 300 mL/g VS of methane during the AD of food waste. According to Cho et al. (1995), the initial VS content should be less than 10 g/L to obtain a higher methane yield from food waste when using a 1:4 inoculum to feedstock ratio. The authors have observed rapid production of VFAs in high initial

VS samples, causing a pH decrease, with inhibition of methane production. In this research, the initial VS content was estimated to be 35 to 45 g/L.

As the next step, NaHCO_3 buffer solution was used to control the pH changes. Although more dilution can also be used to decrease the initial VS, this was not used as a higher dilution factor requires a large reactor and a larger storage area to manage the same amount of waste.

3.4 Small-scale bioreactor

The BMP assay is a commonly used method to predict the theoretical methane yield of a substrate and for dimensioning anaerobic reactors. A small-scale anaerobic reactor was used to determine the behavior of AD of food waste after thermal pretreatment.

The selected optimal thermal pretreatment was used along with a control experiment. The AD was conducted for 20 days, as in the BMP tests. The pH in the reactor was regulated by adding NaHCO_3 300 mg/g VS. The optimal NaHCO_3 concentration was

determined by a separate set of experiments (Habarakada and Babel, 2020). Figure 5 shows the cumulative methane generation for the 20-day experiment.

The highest cumulative methane yield was observed in the thermally pretreated (80°C 90 min) food waste, which was 120.13 mL/g VS, while the control produced 107.99 mL/g VS. The thermal pretreatment had an increased cumulative methane yield of 11.24% compared to the control. Unlike in the previous BMP test, the methane production did not decrease, as the added NaHCO_3 acted as a buffer and maintained the pH. The reactors continued to produce methane after 20 days of AD. The cumulative biogas yield of the thermal pretreatment was not significantly different from the control ($p>0.05$) after 20 days (Table S2). However, the cumulative methane yield increased after 20 days. Therefore, a pretreated sample may have better performance (long-term), compared to the control.

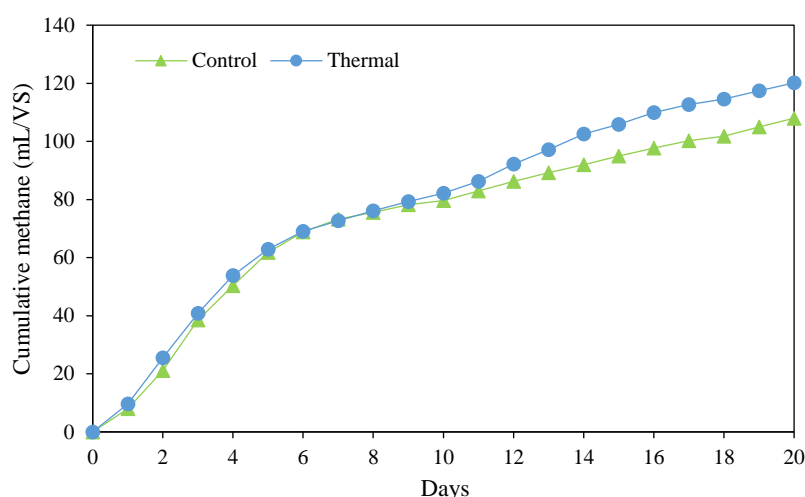


Figure 5. Cumulative methane production by food waste, with and without thermal pretreatment in a small-scale anaerobic reactor

As in Figure 6(a), methane was produced from the 1st day, and it continued to increase until day 3 in both conditions. After day 3, the daily methane production decreased and by days 7-8 of the digestion, the daily methane production became stable for the control and the reactors with a pretreated substrate. This can be described as the stationary phase. As these experiments were conducted in ambient conditions, small fluctuations can be observed (Figure 6(b)) in the daily methane yield. After 20 days of AD, the average biogas yield for the control bioreactor was 35.34% while the thermal pretreatment had an average methane yield of 39%. In the thermal pretreatment, the

maximum daily methane yield was 50.97% (on day 13) while the control had a maximum daily methane yield of 48.47% (on day 5). The overall reaction has improved in the thermal pretreatment compared to the control but it was not much different. This might be due to several reasons such as the relatively easy digestibility of food waste and the initial bacterial activity in the food waste before adding it to the bioreactor.

In the initial BMP tests, the 80°C for 90 min pretreated food waste produced only 14.75 mL/g VS of methane while the bioreactor with the same conditions produced 120.13 mL/g VS of methane.

Therefore, the added buffer played a major role in controlling and stabilizing the pH of the anaerobic media for better digestion. Also, a larger reactor better shows the AD process compared to the BMP test. Increased cumulative methane generation can be observed even after 20 days (Figure 6). Fadzil et al.

(2020) has observed higher methane production at 80°C for 90 min pretreatment compared to control. The increase of methane production was due to the improved biodegradability of food waste due to the thermal pretreatment.

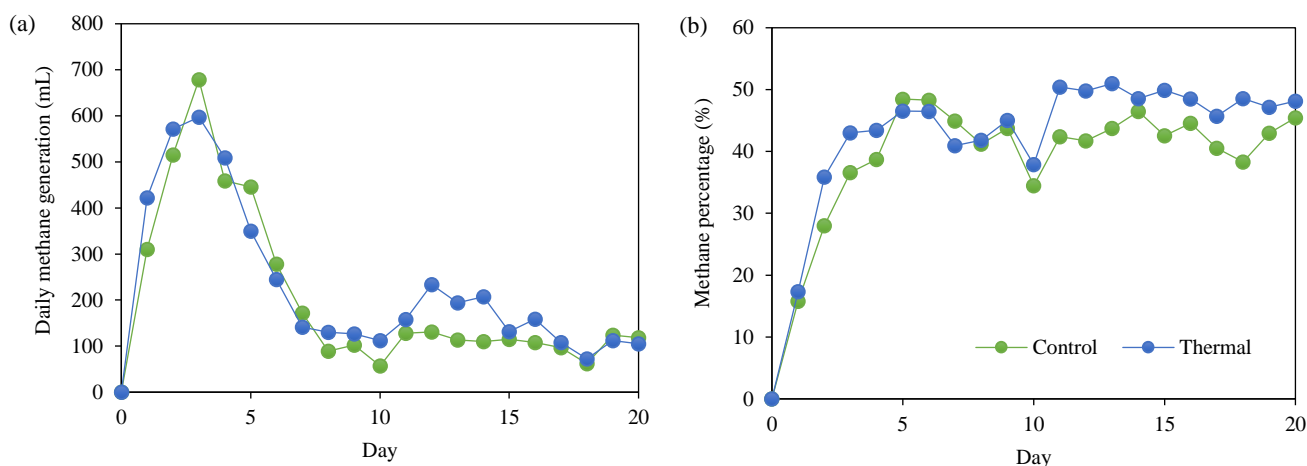


Figure 6. Small scale bioreactor (a) Daily methane generation, (b) Daily percentage of methane in biogas

4. CONCLUSION

The use of thermal pretreatment to increase methane production was examined in this research. The SCOD of sludge increased after thermal treatment and the highest SCOD was observed at 100°C for 90 min pretreatment. The BMP test results showed an initial methane generation in each sample but after a few days the methane production was drastically decreased due to the accumulation of VFA that causes a reduction in pH. These pH reductions are not suitable for methanogenesis. The highest methane yield was observed in the 80°C for 90 min thermal pretreated sample. The methane production increased by 11.24% compared to the control in small-scale bioreactor where NaHCO_3 buffer solution was used to control the pH level. Therefore, thermal pretreatment can be used as a method to increase the methane yield. From an economical standpoint, it is more suitable for less digestible feedstock such as garden waste or agricultural waste than food waste as it requires energy.

The potential use of thermal pretreatment has two main benefits. One is the increase of methane production and the other benefit is that the thermal pretreatment can act as a conditioning process for the substrate (Ariunbaatar et al., 2014). Conditioning is vital for substrates that contain pathogenic bacteria, and in some countries, they require sterilization before or after AD. Furthermore, NaHCO_3 can be used as a

buffer in AD to stabilize pH variations without decreasing the initial VS level.

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