Jeanger Juanga, Periyathamby Kuruparan and Chettiyappan Visvanathan*

Environmental Engineering and Management, Asian Institute of Technology, P.O. Box 4, Klong Luang, Pathumthani 12120, Thailand

*Corresponding author: Telephone: +66 2 524 5640, Fax: +66 2 5245625

E-mail: visu@ait.ac.th

Abstract. Anaerobic digestion was potentially viewed as attractive method for waste stabilization prior to landfills as pre-treatment to reduce significant pollution load to the environment. Optimizing anaerobic digestion process, aim to maximize organic waste conversion to biogas at short digestion period. This paper presents the combined process of anaerobic digestion in solid state batch system which involves enhanced pre-stage (hydrolysis and acidification) and methane phase. Early flushing with microaeration was found to optimize the overall pre-stage performance with hydrolysis yield of 129 g C/kg TS and acidification yield of 193 g VFA/kg TS. A reduced substrate particle size of 30 mm showed an optimized biogas production at short digestion period when compared to 60 mm particle size. Moreover, the importance of thermophilic condition in digestion process further enhanced the operation. A methane yield of 320 L CH₄/kg VS was obtained by thermophilic pilot scale digester and compared with lab-scale biochemical methane potential test (BMP) of 400 L CH₄/kg VS corresponds to a process efficiency of 80%. Nevertheless, significant waste stabilization with mass and volume reduction of 74% and 58% was obtained respectively, with VS reduction of 71%. This study highlights the importance of optimum pre-stage flushing with microaeration, reduced substrate particle size of 30 mm, and thermophilic condition in optimizing the process.

Keywords: anaerobic digestion, hydrolysis, biogas, solid waste, flushing, microaeration

1. INTRODUCTION

The triggering potential environmental problems linked to municipal solid waste landfills and its disposal, diminishing land resources and depletion of fossil fuels have fostered the need for biological pre-treatment of solid waste prior to landfill. The anaerobic digestion process is considered as innovative and attractive technology for waste stabilization with significant mass and volume reduction with the generation of valuable by products such as biogas and fertilizer. Moreover, this process is attractive method especially in Asian countries because of its suitable waste characteristics. Municipal solid waste (MSW) stream in Asian cities is composed of high fraction of organic material of more than 50% with high moisture content (ARRPET, 2004).

So far, the available technologies for anaerobic digestion of MSW are varied from wet to dry, from single-phase to multi-phase, from batch to continuous and within a variety of feedstock. The specific features of batch process includes simple design and process control, lower investment cost, small water consumption, etc. make them attractive for developing economies (Mata-Alvarez, 2003). To maintain a stable high solids digestion process, the chemical value, pH, volatile fatty acid, ammonia and moisture content should be considered as the important environmental factors affecting the efficiency (Lay *et al.*, 1997). In the complex process of anaerobic digestion, the hydrolysis/acidification and methanization are considered as rate-limiting steps. Since hydrolytic/acidogenic bacteria and methanogens have different growth requirements, it may not be

possible to use single-phase system especially in high-solid digestion where substrates are concentrated and VFA are produced in high amount inhibiting the growth of methanogens. Thus, separation of hydrolysis/acidogenesis and methanogenesis would possibly enhance the process. Growth of hydrolytic and acidogenic bacteria can be optimized in the first stage where methanogenesis can be optimized in the second stage. In parallel, the rate of pre-stage reaction can be optimized by applying microaeration (Capela *et al.*, 1999; Wellinger *et al.*, 1999).

This paper presents different strategies to optimize anaerobic digestion of solid waste in combined process in which early flushing and microaeration were conducted to improve pre-stage (hydrolysis and acidification) performance. In order to maximize methanogenic phase, pH adjustment and inoculum addition were conducted. Moreover, this study asses the influence of substrate particle size reduction and the advantage of thermophilic system over mesophilic in the overall digestion process. Moreover, process efficiency evaluation based on biochemical methane potential test was performed. The study was conducted in solid state batch system in pilot scale bioreactors.

2. MATERIALS AND METHODS

2.1 Solid waste preparation and characterization

The substrate used was collected from Rangsit Market in Bangkok, Thailand and mainly comprised of mixed vegetables waste. Fresh waste was manually sorted to remove bulky and inorganic fractions and was subjected to size reduction to less than 60 mm and 30 mm for run 1 and run 2, respectively. Representative waste sample was taken for solid analysis and was characterized to contain high moisture content (90%), high volatile solids (78%) and total solids (10%). The shredded waste was loaded into the reactor together with bamboo cutlets as bulking agent. The purpose of adding bulking material was to create void space to facilitate the flow and distribution of flushing water and microaeration/aeration throughout the waste bed. At the end of the process, bamboo cutlets were separated from the digested waste and the waste was subjected for solid analysis.

2.2 Equipment

The study was performed in three pilot scale digesters. The equipment associated with the reactor allows leachate recirculation, measurement of biogas production, collection of leachate and gas samples, and addition of water. The experimental set-up is illustrated in figure 1. Each vessel is a double walled stainless-steel with total volume of 375 L and the designated volume for waste bed is 260 L, leaving the available headspace and bottom space for biogas generation and gravel support, respectively. The top removable cover of the reactor was equipped with several connector pipes, valves, screws and rubber seals which ensure gas tightness of the reactor. Optimum temperature condition of mesophilic and thermophilic were maintained by a digital temperature controller wherein hot water from water bath was pumped within the water jacket.

Each reactor was provided with two leachate tanks of 200 L and 60 L capacity. The former designed for pre-stage leachate storage during flushing while the latter for main-stage leachate storage during percolation. Centrifugal liquid pumps were used in pre-stage flushing and peristaltic pumps in main stage percolation. Air compressor was used to provide microaeration/aeration. The operation of pumps and air compressor was controlled at certain rate and interval by flow meters and timers. The leachate recirculation system consist of the outlet at the bottom of the reactor, water/leachate storage tank which is connected to the pump and liquid distribution line which drive the water up to the top inlet of the reactor. The sprinkler placed at 3 cm below the top cover distributes the water throughout the waste surface.



Figure 1: Schematic diagram of combined anaerobic digestion experimental set-up

2.3 Operation

A combined process involves three stages (Figure 2). The first stage consists of enhanced pre-stage (hydrolysis and acidification) operation with microaerophilic (very low air flow rate) application. This was viewed as beneficial to partly removed VFA and other dissolved organics from the waste to reduce the organic load of the system and to prepare the system for methanogenic phase. The second stage involved start-up of methanization which employed pH adjustment, inoculum addition, and mature leachate percolation so that the inoculum can be disseminated in the system. The system was allowed undisturbed while the biogas composition was constantly monitored. Mature methanogenesis can be detected when the methane content in the biogas reached 50%, then acidified leachate percolation was started until the biogas production decrease and consecutive batches of leachate were fed until the biogas production and enhance methanogenic phase. Finally, after the waste was completely stabilized, aeration was applied to wash out the remaining biogas from the digester before unloading. This study was conducted in two runs. Three parallel digestion systems ran to optimize the overall process.



Figure 2: Schematic representation of combined anaerobic digestion in 3 stages

Run 1

In run 1, the digesters were loaded with fresh waste intermittently (2 days interval) during flushing in order to utilize the headspace resulting from the settlement of the waste by using 60 mm substrate particle size. Initially, 150 kg of waste was loaded at a compaction density of 630 kg/m³

together with bulking agent (10% of loaded waste). In day 2 and 4, the vessels were opened and 30 kg and 20 kg of waste were added, respectively. The flushing rate was performed at 5 L/min for 4 hours run for every 4 hours stop. Additionally, the flushing operation and the influence of the application of microaeration were evaluated at pre-stage ambient condition. Daily water replacement was applied in reactor 1, whereas in reactor 2 and 3 water was replaced in day 1 and 3. As a result a total of 1000 L of water was used for reactor 1, but only 600 L water was used for reactor 2 and 3. After pre-stage operation, the pH of the system was adjusted to 6.5 by using NaOH solution and inoculum was added at the top portion of the waste and the reactors were ensured for anaerobic condition. Also, the temperature of the system was brought into mesophilic condition and mature leachate percolation was conducted for 2 days.

Run 2

The pre-stage operation for this run follows the optimized condition observed from run 1. An optimum pre-stage performance was exhibited by reactor 3 (run 1), for this reason the flushing operation with microaeration was employed in run 2. Reduced substrate particle size of 30 mm was used in three reactors at different reaction temperature during pre-stage. Ambient condition was maintained for reactor 1 while mesophilic and themophilic conditions were employed for reactor 2 and 3, respectively. After pre-stage operation, similar strategy was followed to start-up methanogenic phase by pH adjustment and inoculum addition. It is worthwhile to note that the inoculum used in reactor 3 (thermophilic reactor) was acclimatized to thermophilic condition before adding. Moreover, the main stage temperature condition for reactor 2 and 3 was maintained as mesophilic and thermophilic condition respectively, while reactor 1 was increased from ambient to mesophilic condition. Figure 3 and 4 represents the overall pre-stage and main stage optimization process involved in this study, respectively.



Figure 3: Pre-stage optimization



Figure 4: Main stage optimization

2.4 Analytical methods

Waste characteristics before and after digestions were determined in terms of moisture content (MC), total solids (TS), and volatile solids (VS). The biochemical methane potential (BMP) test was conducted on fresh waste based on the method established by Hansen *et al.* (2004). The parameters applied to the leachate were pH, alkalinity, dissolve organic carbon (DOC), total kjeldahl nitrogen (TKN), and volatile fatty acids (VFA: acetic, propionic, butyric and valeric) based on the analytical procedures in Standard Methods (APHA, AWWA, and WEF, 1995). In addition, the biogas composition was measured by using gas chromatograph. All the parameters were measured daily.

3. RESULTS AND DISCUSSIONS

3.1 Run 1

Pre-stage

Figure 5 exhibits the variation of various parameters of leachate. The result shows the DOC, VFA, and TKN trend concentration in daily leachate reduced with run time. Reactor 1, which used a total of 1000 L of water for flushing, showed the highest loads of both carbonaceous and nitrogenous materials. However, when compared to reactor 2 and 3 which used lower volume of 600 L of water the result does not significantly differ. Flushing the waste bed with higher volume of water may dilute the waste bed and wash out necessary microorganisms. The pre-stage performance of reactor 3 with microaeration does not show considerable effect when compared to reactor 2 without microaeration. Thus, microaeration shows equivocal result in terms of enhancing hydrolysis.

The summary of pre-stage performance is presented in table 1. Since the objective of this stage is to partly removed organic fraction in preparation for methane phase, the overall result suggests that lower volume of 600 L of tap water with the application of microaeration offer a comparable hydrolysis yield of 129 g C/kg TS and high acidification yield of 193 g VFA/kg TS, respectively. After pre-stage operation, the pH of the system remain at 5.5, in order to stimulate the development of methanogenic phase pH adjustment and inoculum addition seems necessary. The alkalinity of the system decreases with run time which corresponds to pH value. This implied that flushing operation enhances the extraction of organic acids from the waste bed.



Figure 5: Variations of DOC, VFA, TKN, alkalinity, and pH in pre-stage leachate in run 1 [Continuous line corresponds to primary Y-axis, broken line corresponds to secondary Y-axis; -**n**- reactor 1

(without microaeration, 1000 L water); -•- reactor 2 (without microaeration, 600 L water); - \blacktriangle - reactor 3 (with microaeration, 600 L)].

Run/reactor no.	Hydrolysis yield	% C removal	Acidification yield
	(g C/kg TS)	into leachate	(g VFA/g TS)
Run 1: 5 days; 60 mm; ambient			
Reactor 1: 1000 L	139.59	31.75	181.53
Reactor 2: 600 L	128.06	29.12	174.67
Reactor 3: 600 L; with microaeration	129.17	29.38	193.23
Run 2: 5 days; 30 mm; 600 L; with microaeration			
R1: Ambient	143.2	33.3	251.6
R2: Mesophilic	169.4	39.4	313.4
R3: Thermophilic	180.5	42.0	355.4

Table 1: Pre-stage performance (Hydrolysis and acidification yield)

Main-stage

According to Gerardi (2003), the enzymatic activity of methanogens does not occur below pH 6.3 and the favored pH value is between 6.4 and 7.2 (Chugh *et al.*, 1999). Thus, the pH of waste bed was adjusted to 6.5 in order to prepare the environment to be suitable for the growth of methanogens prior adding the seeding material. This procedure may significantly aid the system in starting up the process of methanogenesis.

Figure 6a depicts biogas composition in methanogenic phase. During start-up the gas production was low and methane content increased gradually to 50% in all three digesters. The system was successfully started up after 30 days. Gas production rate in reactor 3 is considered as the highest among other reactors. One possible explanation maybe due early microaeration during pre-stage which might have resulted in better acidification stage during start-up of methanization period providing substrate for methanogens.

Different behaviors were observed after start-up as shown in figure 6b. In reactor 3, it was observed that the cumulative gas production increased immediately after lag phase, when methane composition was stable and leachate percolation was practiced.



Figure 6: Cumulative biogas production in run 1

3.2 Run 2

The optimum pre-stage operation which showed better performance in overall digestion process was exhibited by reactor 3 from run 1. It suggests that lower volume of 600 L of flushing water with the application of microaeration does not only promote enhanced pre-stage operation but in methanogenic phase as well. Pre-stage operation in run 2 is inspired by the result from run 1

with new variables such as reduced substrate particle size under different reaction temperature.

Pre-stage

Table 1 also represents the significance of reduced substrate particle size of 30 mm over 60 mm in terms of hydrolysis and acidification yields by comparing the results between reactor 3 in run 1 and reactor 1 in run 2, signifying that all parameters were similar except only for the particle size of substrate used. It shows that enhanced pre-stage performance is exhibited by 30 mm over 60 mm. Nevertheless, the data highlights the importance of thermophilic condition over mesophilic and ambient conditions. Figure 7 shows the trend of DOC and VFA in daily leachate concentration and the cumulative load. Reactor 1 operates under ambient condition gives lowest load. Whereas, reactor 3 operated in thermophilic condition display the highest load. After 1 day of flushing, reactor 3 generates 5 g/L of DOC and after 5 days it reduced to 2 g/L. The concentration approximately reduced by half after 5 days of operation. This statement also conforms to the result of reactor 1 and 2.

The hydrolysis is often the slowest and limiting step in anaerobic digestion for solid substrate (Schieder *et al.*, 2000). Since the substrate is high in moisture content and flushing mechanism is employed it was observed that hydrolysis process was enhanced and not inhibited. The biodegradable organics was removed from the waste bed during early pre-stage operation and the hydrolysis of particulate was insignificant. The highest hydrolysis yield of 180.5 g C/kg TS was displayed by reactor 3 which corresponds to 42% of C removed from the waste bed into leachate. According to Veeken and Hamelers (1999) and Tong *et al.* (1990), the hydrolysis rate has a direct relation with biodegradability and the rate of hydrolysis enables the estimation of the rate of release of the intermediates (Liebetrau *et al.*, 2003). Importantly, this study reveals that the increase of temperature enhanced the hydrolysis and biodegradability as well. It is worthwhile to note that the DOC in terms of cumulative load that was generated by reactor 1 after 5 days of pre-stage was approximately generated by reactor 3 in just 3 days.

To explain the effect of temperature in acidification process, it is important to examine the VFA concentration. Similarly, the highest concentration was generated after day 1 of flushing and the concentration gradually reduces with run time. Generally, at the end of flushing, a VFA yield of 252 g VFA/kg TS, 313 g VFA/kg TS, and 335 g VFA/kg TS were obtained in reactor 1, 2 and 3 respectively. Emphasizing the idea that higher temperature generates higher VFA yield.



Figure 7: Variation of DOC and VFA in daily and cumulative load leachate in run 2. (Continuous line corresponds to primary Y-axis, broken line corresponds to secondary Y-axis; - \blacksquare - reactor 1 (without microaeration, 1000 L water); - \blacktriangle - reactor 2 (without microaeration, 600 L water); - \blacktriangle - reactor 3 (with microaeration, 600 L).

Figure 8 represents DOC and DOC equivalent of VFA. DOC includes VFA and un-acidified hydrolyzed material. For three reactors, more than 80% of soluble organic carbon in the total leachate was acidified during pre-stage and the un-acidified hydrolysate was accounted for small fraction of less than 20%. Thus, during early stage of operation, acidification was robust over hydrolysis. Importantly, the acetic acid was accounted for more than half of the VFA and it is the predominant acid present during pre-stage.



Figure 8: Load of DOC and DOC equivalent of VFA in pre-stage leachate (run 2)

Main stage

The biogas production and composition were monitored daily. It was found out that long lag phase time was exhibited by mesophilic reactors; it took 20 and 24 days of lag phase for reactor 1 and 2, respectively. Thermophilic reactor took only 14 days for mature methane phase (50% CH₄ in biogas) to develop. Examining the importance of 30 mm over 60 mm particle size in methanogenic phase, figure 9 shows the variation.



Figure 9: Variation of daily and cumulative biogas production between 60 mm and 30 mm substrate

The result showed that 60 mm substrate took longer lag phase period than 30 mm. Consequently, early leachate percolation was provided for 30 mm substrate reactor. Importantly, a total of 4700 L of biogas was produced after 60 days of operation by 60 mm, whereas, it could be achieved by 30 mm after 45 days only. In short, an improve digestion performance with the reduction of digestion time are possible at reduced substrate size. This finding conforms to the findings of Palmowski and Muller (2000) who studied the influence of size reduction of organic waste in anaerobic digestion.

The performance of mesophilic and thermophilic methane phase in terms of biogas production (Figure 10) can be evaluated by comparing the results among the reactors. Generally, the data suggests that the thermophilic condition offers short lag phase period with highest volume of biogas produced. After 45 days of operation, reactor 1 which showed better mesophilic performance during methane phase compared to reactor 2 was used to compare with reactor 3. The methane phase performance of reactor 2 was inhibited (data not presented) and might be due to the low quality of mature leachate used for percolation as it was found out to contain high propionic concentration of more than 3000 mg/L which is considered to be toxic and can cause digester failure (Hanaki et al., 1994).

Thermophilic reactor produced highest volume of biogas than mesophilic reactor. After 45 days, mesophilic digester produced only 4700 L while thermophilic digester generated 5400 L of

biogas. This suggests that a thermophilic condition enhances metabolic activities and thereby improve the rate of anaerobic digestion. It is important to mention that the total of 4700 L of biogas can be produced by thermophilic digester after 30 days only while mesophilic reactor produced this much of biogas after 45 days. Eventually, digestion at thermophilic temperature reduces the required retention time. This finding agrees with the statement of Gunaseelan (1997), higher biogas production at lower retention time can be obtained by high solids thermophilic digestion.



Figure 10: Daily and cumulative biogas production between mesophilic and thermophilic reactors

Lab-scale BMP test was also conducted to investigate the maximum methane generation of the waste in which the methane yield from pilot scale experiment were compared to this test in order to determine the process efficiency. The BMP of fresh market waste is 400 L CH₄/kg VS. After evaluation, it was found that the well performed operation was exhibited by thermophilic reactor (run 2) which obtained methane yield of 320 L CH₄/kg VS which corresponds to the process efficiency of 80% based on methane conversion with 71% VS reduction. Lower process efficiency of 71% obtained by mesophilic reactor with 65% VS reduction.

4. CONCLUSIONS

Application of microaeration at early stage showed equivocal results in terms of enhancing prestage performance. However, the flushing mechanism was beneficial for removing as much as 42% of total carbon content from fresh waste into leachate. Conducting pH adjustment and inoculum addition for methanogenic phase start-up was viewed necessary. The overall optimization process highlights the importance of reduced substrate particle size of 30 mm to stabilize the waste at short period. In addition, thermophilic condition offers better result over mesophilic condition. With 71% VS reduction, the calculated methane yield for thermophilic digester was 320 L CH_4/kg VS and an improve process efficiency of 80% was obtained based on the lab-scale BMP test of 400 L CH_4/kg VS.

5. **REFERENCES**

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