High Solid Anaerobic Co-Digestion of Household Organic Waste with Cow Manure for Mass and Energy Recovery

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Abstract

This paper describes a batch study on four different mixture ratios of household organic waste and cow manure. The biomethane potential test is used to evaluate the suitability of high solid anaerobic co-digestion of household organic waste and cow manure and its biodegradability. The maximum methane yield was observed for both co-digestions in R_3 (247 mL/g VS) and R_4 (244 mL/g VS). The percentages of mass recovery were 65-80% (based on VS removal). The biodegradability of each reactor was recorded as 97.7%, 10.7%, 71.6% and 76.8% for R_1, R_2, R_3, and R_4, respectively. High solid co-digestion of household organic waste and cow manure in different mixture ratios increase the specific methane yields compared to mono-digestion. Hence, this clearly demonstrates a synergistic effect on the stability of anaerobic digestion. Upon correcting the results of the theoretical method with experimental data, an agreement ranged 70-90% was achieved.

Introduction

Today, anaerobic digestion is widely applied to treat a diverse range of organic waste that promotes betterment in landfill management and produces a potential renewable energy source. Organic waste (mainly food waste) is very attractive and a potential feedstock for anaerobic digestion due to it high fraction in waste generation, especially in Malaysia. Moreover, the high composition of fat and lipids in organic waste contributes to higher conversion of organic matter in the waste fraction for biogas production. However, degradation of lipids and fats produces long-

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chain fatty acids (LCFAs) that inhibit and suppress the process. Anaerobic digestion of cow manure also is seen as unstable due to its low carbon:nitrogen ratio (C:N). Hence it is important to look forward the potential of co-digestion approaches to organic waste – especially food waste and cow manure – for optimum and stable biogas production.

Lately, high solids digestion has gained rapid interest for treatment technology (HS-AD). High solid anaerobic digestion (HS-AD) is preferable because it requires minimal pre-treatment and added water compared to low solid anaerobic digestion (LS-AD) [1]. High solid anaerobic digestion is regularly described with the total solid (TS) greater than 15% (w/w) [2]. A comprehensive review discussed on the advantages of HS-AD over LS-AD supports HS-AD being able to operate under minimal water addition with technical simplicity, smaller reactor volume, and high quality of digested residue due to minimal nutrient loss during digestion [3]. Some studies are related to the application of HS-AD on different types of organic substrates, including organic fraction of municipal solid waste (OFMSW) [4-6], industrial organic residue [7-8], animal manure [9-10], biomass [11-12], and sewage sludge [13]. Although there is increasing interest in anaerobic co-digestion, so far less has been reported on the synergistic effect of co-digestion of high solid anaerobic digestion on source-sorted household organic waste (15% TS) with cow manure (5% TS).

The present work involves the application of co-digestion of HS-AD in a single-phase system of source-sorted household organic waste – mainly household organic waste (HOW) with cow manure (CM). The principal objective of this paper is to access the levels of material and energy recovery from household organic waste. We seek to evaluate the feasibility and performance of co-digestion HS-AD of HOW. We also have paid attention to the impact of implementing HS-AD for promoting recycling and reuse of organic substrate prior to sustainable landfill management.

Materials and Methods

Preparation of Feedstock

The HOW used in the current study is basically sampled from source-sorted household OFMSW (mainly food waste). Samples were collected, manually sorted, and shredded using a laboratory blender. Water was added to the desired total solid (15% TS). CM was sampled from the Universiti Putra Malaysia (UPM) agricultural park. The feedstock was kept frozen (-2ºC) until required for feeding. Anaerobic inoculums used in this study were supplied by SPmultitech (SP Multitech Renewable Energy Sdn Bhd). The inoculum were thermophilic (55ºC) treated in an air-tight reactor before being introduced to the feedstock. The chemical characteristics of the feedstock and inoculums operated in this study are prescribed as in Table 1.

Table 1. Characteristics of feedstock and inoculums.

Parameter	HOW	CM	Inoculum
pH	5.3	7.5	8.3
TS (%)	40.4	15.2	26.3
VS (%)	30.6	13.8	22.5
Moisture Content (%)	84.5	50.4	44.0
TOC (g)	40	78.3	27.6
C:N	11.0	11.2	25.3
Ammonia (g/L)	4.3	26.88	14.8
Alkalinity (g/L)	5.1	7.7	16.5
Total C (%)	59.2	35.4	35.4
Total H (%)	9.4	6.3	-
Total N (%)	5.4	5.8	1.4
Total S (%)	0.4	1.0	-
Total P (%)	18.5	0.18	-
Total K (%)	65.6	1.27	-

Experimental Design and Operation

In this study, batch experiments were designed to evaluate the performance of high solid co-digestion of HOW and CM in four different mixing ratios. The assays were conducted in a 1 L batch scale reactor as depicted in Fig. 1 with total working volume of 800 mL incorporated with three separate ports for different functions of feeding inlet, biogas measurement, and biogas collection connected to a 1 L standard FlexFoil gas bag with single polypropylene fitting (SKC brand). The experimental designs and scheme are summarized in Table 2. Before starting anaerobic digestion, each reactor headspace was purged with pure N_2 gas (99.9%) for at least three minutes, and sealed with a rubber septum prior to incubation. Each digester was manually mixed once daily. The biogas production is measured periodically by the water displacement method and calculated as volume at standard temperature and pressure (STP) conditions.

Analytical Procedures

Substrate samples were analyzed twice a week to monitor total solids (TS), volatile solids (VS), alkalinity, total nitrogen (TKN), total organic carbon (TOC), chemical oxygen demand (COD), and ammonium nitrogen (N_4^+-N). All the analytical monitoring was determined according to Standard Methods (2005) [14]. Elemental analysis such as total carbon, total nitrogen, and total sulfur were analyzed using AAS based on the liquid extraction method, whereas nitrogen, phosphorus, and potassium (N, P, and K) were measured by the soil extraction method using a Mac CNS analyzer, and crude protein was determined by multiplying total nitrogen values by 6.25. The biogas composition (CH_4
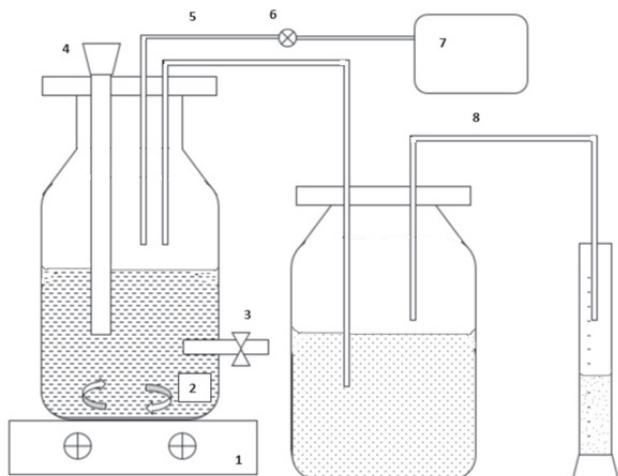
Fig. 1. Schematic diagram on batch experiments set-up.
Hot plate with magnetic stirer 2) 1L Schott bottle 3) Effluent port
4) Feeding port 5) Gas-pipeline 6) Gas-pump 7) 1L Biogas bag
8) Water displacement measurement.

Table 2. Batch experimental design and scheme of feedstock and inoculum.

Reactor	Feedstock		5% Inoculum (mL)
	HOW (mL,15%TS)	CM (mL, 5%TS)	
R_1	-	760	40
R_2	760	-	40
R_3	380	380	40
R_4	506	254	40

and CO_2) analysis was carried out by gas chromatography separation (6890N Agilent Technologies, CA, USA) using a thermal conductivity detector (GC-TCD) equipped with a Hay sep N 80/100 and a molecular sieve column (5A 60/100). Argon (Ar) was used as carrier gas at a flow rate of 12 mL/min. The injector, oven, and detector temperatures were 105ºC, 60ºC, and 150ºC, respectively. Methane generation yield was normalized by correcting the gas volume to STP.

Theoretical Methane Yield (TMY_{ele}) and Biodegradability (BD_{ele})

Theoretical methane yield (TMY) is commonly expressed as mL CH_4 at STP conditions per amount of organic material added (VS- or COD-based). The assessments are basically estimated using the stoichiometric equation as simplified in Equation 1 based on Buswell formula and (STP = 0ºC, 1 atm) to determine the energy value of the feedstock [15-16]:

$$TMY_{ele} \frac{mL\ CH4}{g\ VS} = \frac{\left(\frac{a}{2} + \frac{b}{8} - \frac{c}{4} - \frac{3d}{8} - \frac{e}{4}\right).22400}{(12a + b + 16c + 14d + 32e)} \quad (1)$$

...where a is the number of atoms of carbon, b is the number of atoms of hydrogen, c is the number of atoms of oxygen, d is the number of atoms of nitrogen, and e is the number of atoms of sulphur. Biodegradability (BD_{ele}) of the feedstock was measured based on measured experimental methane yield (EMY) and theoretical methane yield (TMY) as presented in Eq. (2) [17].

$$BD_{ele} = \frac{Measure\ BMP}{TMYele} \quad (2)$$

Statistical Analysis

To define the relationship between operational parameter and process performance on the synergistic effect, *Pearson* correlation coefficient (R^2) was determined and the significance of the study was verified using *SPSS version 22.0* (SPSS, Inc, Chicago, USA).

Results and Discussion

The Stability of High Solid Anaerobic Digestion

pH Monitoring

Fig. 2 shows pH trends for solid phase functioning of each reactor. At the beginning of digestion pH dropped drastically, causing the substrates to be suppressed as the organic substance was hydrolyzed and converted to fatty acids. Hydrolysis took place at this point, when pH started to show decrement. pH inconstancy was observed as a result of the production of fatty acids in the reactor, especially at the early stage of functioning. This is supported by a previous study that reported methanogenisis occurring effectively at pH 6.5-8.2 while hydrolysis and acidogenisis occur at pH 5.5 and 6.5 [18]. R_1 is maintained at the optimum pH values whereas R_2 only reaches 6.4 for three consecutive days and drops dramatically, caused sourness. The pH values in R_2 fell instantly at the start-up phase and reached a pH value of 5.3 on day 15. The extreme pH decrement in R_2 results in
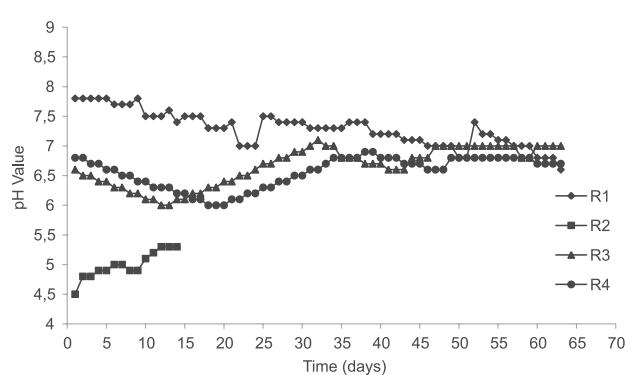

Fig. 2. Temporal pH variations with digestion times.

methane production to suppression. Hence, the digestion process in R_2 was mainly limited to the first 15 days of functioning. At the short period of the acetogenesis stage the pH started to increase gradually as fatty acids were consumed by acetogens, and later at the methanogenesis stage by methanogens. At this phase, pH values from R_3 and R_4 during days 15-30 are stable and maintained while R_1 showed drastic decrement. The average pH values in the study period were 4.5-7.

Removal of VS and COD

The periodical VS evolutions in each reactor are presented in Fig. 3. The degradation of feedstock was presented with the variation of VS patterns in this study. R_2 showed a slight descent of VS removal during days 15-40 and reaching constant values until the finishing phase. R_3 and R_4 showed significant VS removal in several consecutive days of functioning (40-45 days). The percentage of VS removal attained 50% for R_2 and more than 70% for R_1, R_3, and R_4 in 60 days of operation. These results support the fact that approximately 65-80% of organic matters were removed in R_1, R_3, and R_4 within 45 days, whereas R_2 needed an additional 15 days of operation to achieve the same percentage of removal. COD concentration patterns in each of the reactors are reported in Fig. 4. High COD values were described in the initial process and started to decrease as organic matter in the substrate was utilized by methanogens R_3 and R_4. Satisfactorily, the COD values in each reactor decreased to approximately more than 80% of COD elimination during the functioning time. The initial COD concentrations were 6,879 mg COD/L, 10,587 mg COD/L, 7,380 mg COD/L, and 6,541 mg COD/L for R_1, R_2, R_3, and R_4, respectively. At the beginning of the process, slight decrements of COD concentrations were measured in the first week for R_1, R_3, and R_4, and R_2 in the first two weeks. The decrement is due to the degradation of complex organic substrates at the initial phase of digestion without effecting COD concentration. The stability of anaerobic digestion mainly depends on the removal of soluble organics. High solid reduction is the indicator to measure efficiency and stability of a treatment system and operation. Elimination of organic matter is important to meet the standard
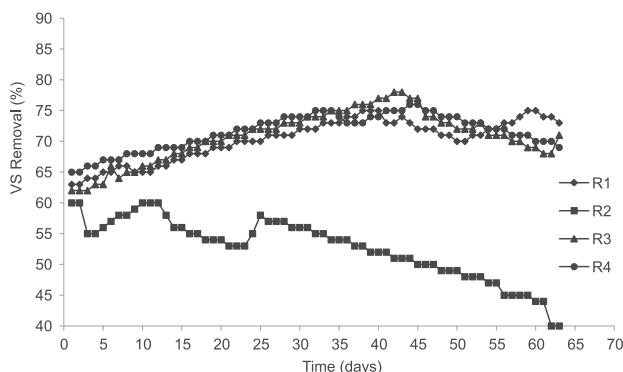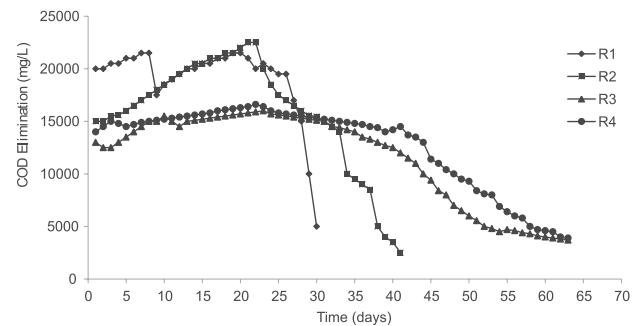

Fig. 4. Temporal COD eliminations with digestion times.

operation in a sanitary landfill. Hence, from monitoring, this study supports the stable operation of the co-digestion of high solid HOW and CM.

Evolution of Methane Production

Daily methane yield from each treatment was depicted as in Fig. 5. Methane production in R_3 is the highest (247 mL CH_4/g VS) followed by R_4 (244 mL/g VS). All these mixtures obtained higher CH_4 production than sole substrate in R_1 (CM) and R_2 (HOW), with 223 mL/g VS and 54mL/g VS, respectively. The digestion suppressed at the beginning of the process in R_1 (11 days) and R_2 a week (21 days) later caused the digestion to stop. Co-digestion in R_4 increased 9% for CM and 78% for HOW in CH_4 yield. Anaerobic digestion of monosubstrate was found to be unstable due to insufficient nutrient content [19]. The alkaline components of CM as co-substrate might have neutralized the pH in R_3 and R_4, thus improving the buffer capacity potential in both reactors. During stable phase methane production there were 237.9 mL, 74.4 mL, 313.5 mL, and 240.3 mL in approximately 15-35 days of operation. The batch assays ended with a CH_4 production less than 1% in 40 days of operation. Pearson correlation coefficient (R^2) between operational parameters and specific methane yield shows as described in Table 3. VS and COD are strongly correlated with specific methane yield followed by pH, ammonium nitrogen, and total nitrogen. A previous study reported as total solid in substrate increases and that organic matter conversion by microorganism induced higher methane production [20]. R^2 indicates a slightly stronger relationship between
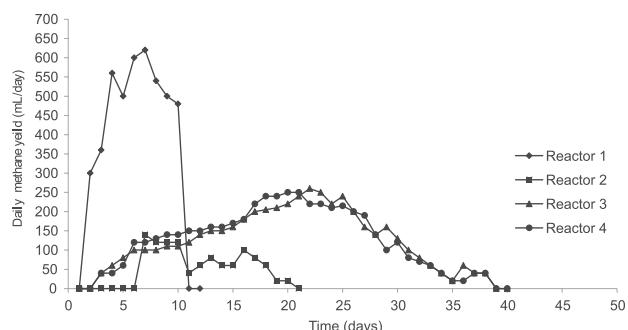

Fig. 3. Temporal VS patterns with digestion time.

Fig. 5. Daily methane production in the reactors.

Table 3. Relationships between main operational parameter and specific methane yield (R^2).

Parameter	R^2 Pearson
pH	0.670
COD	0.965
VS	0.960
N_4^+-N	0.407
TKN	0.233

Correlation is significant at < 0.01 level (2-tailed)

pH and specific methane yield due to the variation of HOW chemical characteristics. The optimum pH for methane production is 6.8-7.2. However, HOW is vulnerable to higher VFA production that promotes a decrease in pH.

Mass and Energy Recovery

The potential of theoretical methodology to precisely estimate methane yield of anaerobic co-digestion was evaluated by comparing the experimental methane yields (EMY) with theoretical methane yields (TMY). Table 4 represents experimental production (EMY) and biodegradability (BD_{exp}). The methane yield from the experimental process is at more than 90% agreement with the theoretical methane yield estimation for R_3 and R_4. The maximum CH_4 purity percentages were 65% and 55% for R_3 and R_4, respectively. The results showed different behaviors for R_1 and R_2 (sole-digestion), indicating that the synergistic effect is significant for anaerobic co-digestion. Overall, it is ascertainable that theoretical estimation methods based on stoichiometric composition with biodegradability values have the potential to evaluate specific methane yields R_3 and R_4.

Mass recovery is widely calculated in two ways: by wet weight and on a VS basis. This study focuses on VS removal. HS-AD seems to be efficient in treating organic waste and significant mass and volume reduction of 60-80% in each reactor. Higher VS reduction was observed in reactors with higher methane yield (R_3 and R_4). More than 50% of VS removal was observed in the failed reactors, even though there was least methane production (<10%). The estimation of important elements such as macromolecules (N, P, and K) is crucial for further utilization of digestate. The AD added value to the digestate converts the organic portion of waste to compost, fertilizer, and soil amendments. Almost 5-30% of nitrogen content was analyzed in digestate from each reactor. Complex nitrogen compounds are mineralized to ammonium (N_4^+-N) during the digestion process. Most of the N_4^+-N is utilized by microorganisms for AD metabolism. In the other hand, there is almost no effect of AD on phosphorus (P) availability in digestate (0.01-1.00%). Increasing the pH enhanced the formation of phosphate ($HPO_4^{-2} \rightarrow PO_4^{3-}$) in the reactor. However, the mineralization stage during AD accumulates with a suspended solid, leading to precipitation of P. Moreover, the highest increment of potassium (K) content in digestate was observed in R_2 (33%), followed by R_3 (4.34%), R_1 (2.36%), and R_4 (2.1%).

Conclusion

Adapting the co-digestion of high-solid HOW with CM by using different mixture experimental design was established over a period of 9-10 weeks. The experimental methane yield was enhanced by 70-90% (227-247 mL/g VS) from theoretical methane yield for R_1, R_3, and R_4, respectively. The maximum methane percentage was observed in a range of 55-75% from each reactor. The varied methane content

Table 4. Potential material and energy recovery.

Recovery	Parameters	R_1(CM)	R_2 (HOW)	R_3 (1HOW:1CM)	R_4(2HOW:1CM)
Mass	VS Elimination (%)	80	67.3	77.3	71.2
	COD Removal (%)	27.3	76.4	52.6	46.5
	N (%)	28.6	7.4	14.6	17.5
	P (%)	0.01	0.01	1.1	0.01
	K (%)	2.36	33.3	4.34	2.1
	Biodegradability	97.7	10.7	71.6	76.8
Energy	Daily CH_4 (mL)	90 – 600	11 – 149	20 – 371	20 – 307
	Maximum CH_4 (%)	75	<10	65	55
	TMY (mL/g VS)	232.29	505.17	345.17	317.7
	EMY (mL/g VS)	227	54	247	244

TMY: Theoretical Methane Yield (Eqs. 1 and 2)
EMY: Experimental Methane Yield

in all reactors was likely due to homogeneity of the feedstock.

The experiment ended after day 40 of operation with VS elimination ranging 65-80% from each reactor.

Acknowledgements

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