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Dry mesophilic and thermophilic semi-continuous anaerobic digestion of cow manure: effects of solid loading rate on the process performance

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ABSTRACT

Four solid loading rates (SLRs) of 3-9 kg TS/m3 day at fixed 20% total solids (TS) content were chosen in this study and assessed under two distinct circumstances: mesophilic and thermophilic under dry semi-continuous cow manure (CM) digestion. This research aimed to investigate the production of biogas, volatile solid reductions and volatile fatty acids (VFAs) concentration effects on process efficiency. This was done in 100 days of operation. The findings indicated that the production of biogas and volatile solid reduction in both digesters showed similar patterns of decrements alongside increasing SLRs. In all test cases, the best performance of the digesters was found at the SLR of 3 kg TS /m3 day, which was under thermophilic conditions. The methane yielded 0.39 m3 CH4/kg VS (65 % CH4). Meanwhile, the mesophilic reactor recorded the production of 0.25 m3 CH4/kg VS (60 % CH4). A 35.89 % increment in methane production was observed in the thermophilic state. About 67 % and 62 % of volatile solids reduction were observed in the thermophilic and mesophilic digesters, respectively. In contrast, it was observed that volatile fatty acids showed inhibitory effects at the SLR of 9 kg TS/m3 day, which in turn decreased the digesters' performance.

Keywords: *Dry Anaerobic Digestion; Mesophilic and Thermophilic Digester; Biogas Production; Cow Manure; Volatile Fatty Acids*

1. INTRODUCTION

The world has been experiencing an astonishing rise in surface temperature in recent years. This phenomenon is generally attributed to the increment in greenhouse gas emissions into the atmosphere. One of the contributory factors to such a problem is the rapid development of industry and agriculture sectors. As an example, Malaysia has recorded about 35 – 40% of total emission that is sourced from livestock [1,2]. Based on the statistical trend for the growing demand of livestock in Malaysia [3], there is a very high likelihood for methane (CH_4) emissions to exceed twice the current level unless serious reductive measures are put in place [2].

The current strategies employed nowadays are tailored towards eliminating or reducing environmental pollution by converting the Waste to Energy (WtE) [4]. Anaerobic digestion (AD) technique has therefore been classified as an efficient technique and has subsequently been commonly used in practice for the management of organic waste and the restoration of renewable energy [5,6,7]. AD technique is theoretically split into three classifications based on dry (≥20% TS), semi-dry (10-20% TS) and wet (≤10% TS) [8]. Dry AD, is a technology intended to process more organic waste per reactor with a total solid content exceeding 20% [9]. This technology has increasingly attracted the attention of worldwide scientists in recent year years, primarily owing to the advantages of dry AD in comparison with wet AD.

Several studies have been done under mesophilic (20-45 °C) or thermophilic (41-70 °C) conditions via dry AD, such as lignocellulosic biomass [10], organic fraction of municipal solid waste [11,12] and food waste [13]. These studies found that the best temperature that produced good biogas production amount and quality are at 38 °C and 55 °C. Besides, these studies have highlighted various characteristics dry AD offers, which include: high rates of biogas production [14], high biogas volumetric efficiency [15], low water content [16], wide-ranging applicability for organic wastes [17], capacity for operating at high OLRs [18], and cost-effective technology as it offers better economic feasibility since the volume of the reactor volume is minimized and it is easier to handle [14,37]. This process also lowers the production of leachate and it contains higher nutrients, mainly nitrogen and phosphorus, which can be further used as organic fertilizers [19]. However, it is also associated with shortcomings requiring improvements, such as lengthy start-up and degradation time [20], VFA accumulation impacts [17] and sensitivity towards any minor changes of the operating parameters (e.g. temperature, pH, nutrients, and others) [21].

Many types of research have been carried out regarding the co-digestion of dry AD, such as CM co-digested with agricultural waste [22], animal manure co-digested with sweet potato vine [23], pig manure co-digested with rice straw [24] and cow manure with food waste [38]. However, there is limited literature on the evaluation for CM dry AD alone under mesophilic and thermophilic conditions. The objective of this study was, therefore, to assess the mesophilic and thermophilic on CM under dry semi-continuous anaerobic digestion processes, particularly in terms of their practical applicability to treat CM at different concentrations of SLR. Moreover, this study comprehensively compared both digester's performance specifically in terms of biogas production and methane composition as well as solids removal. Also, the influence of the intermediate products on the operational performance of AD processes was thoroughly investigated.

2. MATERIALS AND METHODS

2.1. Cow manure and inoculation sludge.

The Dry CM used as feedstock in this study were gathered from nearby feedlot around Universiti Malaysia Terengganu (UMT). Then, they were grinded to the diameter of 1mm [25]. After crushing, the dry grinded CM was stored in a tight container. A 10 kg of CM was collected in a sample collection icebox and was preserved at 4° C in the sample preservation room of UMT. The inoculum sludge was collected from the Palm Oil Mill Sludge, POMS (Serting, Negeri Sembilan, Malaysia). The characteristics of CM and inoculum used in this research are summarized in Table 1.

Table 1. Physical characteristics of the cow manure and inoculum used in this study.

Parameters	Unit	Cow Manure	Inoculum
pH	-	6.82 ± 0.01	7.28 ± 0.01
Total Solid (TS)	%	90.29 ± 0.03	72.91 ± 0.02
Volatile Solid (VS)	%	72.65 ± 0.04	39.57 ± 0.03
Ash	%	17.64 ± 0.03	33.34 ± 0.02
VS/TS	-	0.80	0.54

2.2. The digesters setup and operational conditions.

Figure 1 illustrates the experimental setup used in this study. The experiments were executed in a dry semi-continuous anaerobic digester system with a total capacity of 7 L and a working volume of 4 L. The reactor was made of borosilicate glass (Sartorius, Melsungen, Germany), which possessed a cylindrical geometry with a diameter of 16 cm and a height of 25 cm. Mixing for each vessel was done by mechanical stirring using the Rushton impeller and built into the vessel. Meanwhile, other equipment attached to the fermenters included an electrical heating jacket for temperature control, an EasyFerm plus K8 325 pH sensor (both Hamilton, Bonaduz, Switzerland), and a Pt-100 temperature sensor (Sartorius, Melsungen, Germany). Process temperature was maintained at 38 ± 1 °C for M_R and 55 ± 1 °C for T_R by using the heating jacket. The pH control was maintained at pH 6.8 – 7.2 automatically by the addition of 1M HCl and 1M NaOH as per propriety, while the stirring was maintained at a speed of about 500 rpm. Then, the systems were flushed with nitrogen gas for 10 min to ensure the anaerobic conditions before sealing. During the start-up phase, about 0.34 kg substrate supplemented with 0.06 L inoculum was added into each digester for 10 consecutive days in the batch mode process. After the start-up phase, the digesters were operated with increasing SLRs starting from 2 kg TS/ L.d to 9 kg TS/ L.d. This corresponded to the hydraulic retention time (HRTs) from 29 days to 10 days, respectively. During phases 1 and 2, both digesters were operated using identical operational conditions including temperature (38 ± 2 °C), as summarized in Table 2. Subsequently, during phase 3, the T_R digester was progressively shifted at a rate of 1°C per two days from the mesophilic condition of 38 ± 2 °C to the thermophilic condition of 55 ± 2 °C. This was an improved transition strategy to minimize the shock due to rising temperatures [8,26].

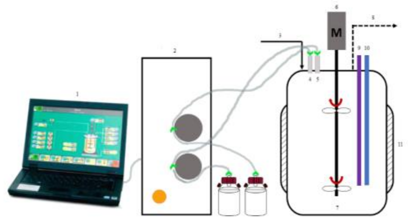


Figure 1. Schematic diagram of the 7-L continuous stirred tank reactor (CSTR); (1) PC with SCADA software MFCS/DA; (2) Control unit; (3) Feeding and sampling port; (4) Acid port; (5) Base port; (6) Stirrer; (7) Glass reactor; (8) Biogas collector; (9) pH probe; (10) Temperature sensor; (11) Water Heating jacket

Table 2. Operational conditions of mesophilic and thermophilic semi-continuous dry anaerobic digestion.

Phase -	Solid retention time (d)	Operation time (d)	Solids loading rate (kg TS/ L.d)	Flow rate (L/d)
1	21	30	3	0.188
2	13	20	5	0.314
3	9	15	7	0.440
4	7	11	9	0.563

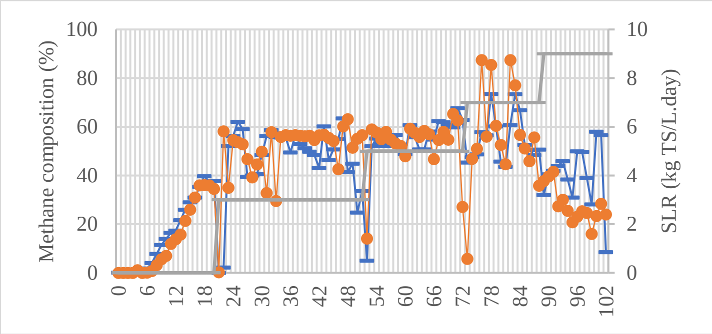
2.3. Analysis.

To evaluate the stability, efficiency and the production of biogas from both digesters, the effluent of the samples were collected and evaluated daily. The determination of pH, Total Solids (TS), Volatile Solids (VS), and alkalinity were performed based on the Standard Methods [27]. The biogas production was collected using a Tedlar® gas bag and its volume determined using the water displacement method. Meanwhile, the analysis of methane composition was carried out using gas chromatography (GC; Agilent 7890A, Agilent Technologies, Inc., USA) equipped with a thermal conductivity detector and GDX-01 column. The temperature of the injection port and detector was 100 °C, while the column temperature was maintained at 80 °C. Hydrogen at 8.78 mL min^{-1} was assigned as the carrier gas. The amount number of volatile fatty acids (VFAs) was determined by using a gas chromatograph (GC, 2010 Plus, Shimadzu), which was equipped with a flame ionization detector and SP2560 column (100 m length X 0.25 mm ID X 0.2 μm film thickness) with nitrogen as the carrier gas. Approximately 1μL of each sample was injected into the GC. The initial temperature of the GC column was 150 °C, which increased at the rate of 1°C /min to reach 171 °C. Once it reached 171 °C, the temperature increment was maintained at the rate of 0.5 °C/min to result in the final temperature of 176 °C. The injector temperature was set at 240 °C, while the flame ionization detector was set at 250 °C.

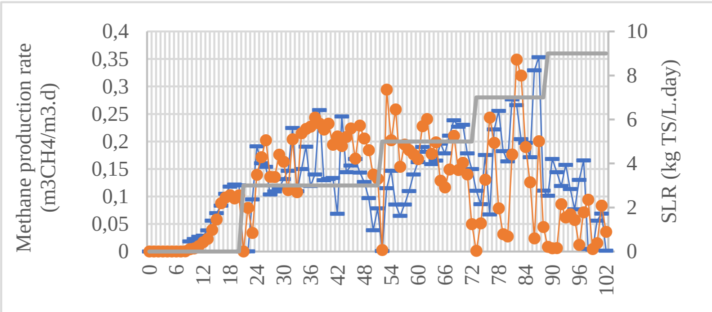
3. RESULTS

3.1. Effect of SLR on biogas production.

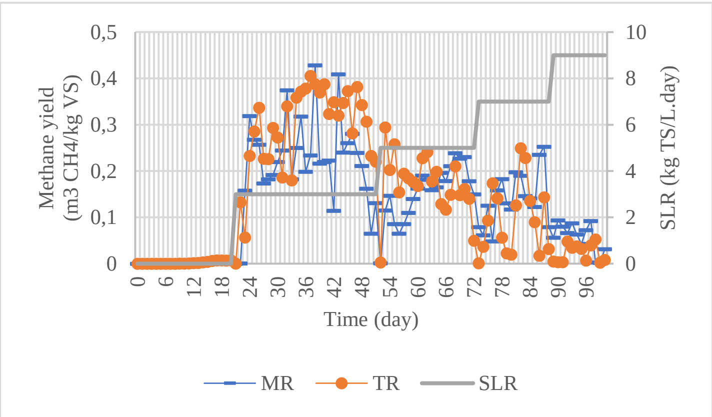
The effect of an increased SLR on the process performance was evaluated and optimized. Daily methane composition, methane production rate, and methane yield from treating CM using dry digestion under mesophilic (MR) and thermophilic (TR) conditions are revealed in Figure 2.



(a)



(b)



(c)

Figure 2. The profile of (a) methane composition; (b) methane production rate and; (c) methane yield under various SLRs and either mesophilic and thermophilic conditions.

As illustrated in figure 2(a), in general, the trend of the methane composition for both digesters increased in parallel to the increasing SLR. However, their introduction to the higher SLRs (7 kg TS/L.day and 9 kg TS/ L.day) yielded decreasing tendencies. Methane gas was identified on day 7 during the start-up phase, proving that the lag phase inhibition occurred in both digesters. However, day 8 was linked with the detection of methane compositions, which increased to almost 40 % in both T_R and M_R digesters. Following the stable condition during the start-up phase, a new SLR was introduced to each digester starting from 3 kg TS/ L.day to 9 kg TS/ L.day. A slight decrease was thus observed in the methane composition, and production rate (figure 2(b)) and yield (figure 2(c)) for each incremental shift in SLR. This happened during a short period after the new load. It is attributable to the acclimatization period for biomass towards the increment of organic loading concentration [4]. Then, downtrend methane composition and production rate observations were noted for SLRs 7 kg TS/ L.day and 9 kg TS/ L.day for all digesters, indicating their decreased performance. However, when the digester T_R reaches the thermophilic state during phases 3 and 4, the trend for methane production rate differed significantly compared to the remaining two digesters. A fluctuating tendency could be seen during these phases, which may be associated with the transformation due to anaerobic microorganism adaptation in the digesters [27]. It was observed that both digesters revealed that the methane yields had significantly decreased as the SLR increased. It was reduced from 0.39 $m^3CH_4/kgVS$ (Phase 1) to 0.04 $m^3CH_4/kgVS$ (Phase 4) for the T_R digester, and from 0.25 $m^3CH_4/kgVS$ (Phase 1) to 0.08 $m^3CH_4/kgVS$ (Phase 4) for the M_R digester. The highest methane yield (% CH_4 content) values were achieved during Phase 1, whereby 0.39 $m^3CH_4/kgVS$ (65 %) was obtained for the T_R digester and 0.25 $m^3CH_4/kgVS$ (60 %) for the M_R digester. This allowed the conclusion that the highest methane yield was achieved during Phase 1 for the T_R digester. Thus, it is representative of the higher activity of the thermophilic anaerobic microorganisms in terms of their degradation and shifts to the renewable energy of CM in comparison with the mesophilic anaerobic microorganisms [27]. The results from this current study disclosed much higher renewable energy recoveries compared to the previous studies, yielding 0.03 $m^3CH_4/kgVS$ [29], 0.38 m^3 $CH_4/kgVS$ [30], 0.18 $m^3CH_4/kgVS$ [23] and 0.31 $m^3CH_4/kgVS$ [31].

The reactor performance in dry thermophilic and mesophilic conditions achieved in this study was compared with other previous study were summarized in Table 3. In general, the results showed higher reactor performance in terms of methane composition, methane production rate, and methane yield in thermophilic conditions compared to mesophilic condition. The results from this current study disclosed much higher renewable energy recovery compared to previous studies achieved by [31].

Table 3. Reactor performance in dry thermophilic and mesophilic used in other studies in terms of methane composition, production and yield.

A	B	C	D	Reactor performance			Reference
				E	F	G	
DTCSTR	CM	3	29	65	0.23	0.39	This study
MTCSTR	CM	3	29	61	0.15	0.25	
DTCSTR	FW	9.2	25	62	n.s	0.47	[26]
MTCSTR	FW	9.2	25	67	n.s	0.43	
DTB	BL	n.s	36	73.7	n.s	0.31	[31]
DMB	BL	n.s	36	73.5	n.s	0.22	

A: System; B: Substrate: C: SLR (kgTS/L.day); D: HRT (d); E: Methane composition (%); F: Methane production rate (m^3/m^3.d); G: Methane yield ($m^3CH_4/kgVS$) DTCSTR: Dry thermophilic continuous stir tank reactor; MTCSTR: Dry mesophilic continuous stir tank reactor; DTB: Dry thermophilic batch experiment; DMB: Dry meso batch experiment; CM: cow manure; FW: food waste; BL: beer lees; n.s: not stated.

There are several factors affecting the higher methane yield production, such as pre-treatment process and the composition in the substrate. The CM used in this study was pre-treated by grinding the sample into smaller sized particles (1 mm) compared to beer less (1.5 - 2mm). Particle size is one of the factors that greatly affect enzymatic hydrolysis. Smaller particle size leads to an increase in microbial and enzyme substrate contact, resulting in higher methane yield generation as stated by [32]. Besides, the composition in the substrate also contributes to methane yield production. The higher methane yield produced in this study is because the inhibitory factors are eliminated before AD takes place, which in this case, is lignin. Lignin was degraded during the pre-treatment process. Beer less is a by-product from the filtration stage consisting primarily of husk, leaf bud, protein, hemicellulose, fat, ash, and a little amount of undissolved starch.

These fat, ash, and a little amount of undissolved starch have been reported to cause inhibition of acetoclastic and methanogenic bacteria [33]. However, the methane yield produced in FW digestion is higher compared to this study due to higher SLR applied and different types of substrate used, where FW contained higher organic content compared to CM [18].

3.2. Effect of SLR on the volatile solid reduction.

Figure 3 shows the profile of VS reduction throughout the four SLRs tested. In general, the trend for both M_R and T_R digesters in VS removal is similar. The VS reduction during steady-state was recorded for each phase, namely during start-up and Phases 1, 2, 3 and 4. The T_R marked a reduction of about 38.5 %, 67 %, 48 %, 24 %, and 14 %, respectively, while values of 38.1 %, 62 %, 48 %, 24 % and 16 % were obtained for the M_R digester, respectively.

During the start-up phase, a low VS reduction was observed for both digesters. Another study previously revealed the same situation whereby slow degradation (< 50 % VS reduction) was recorded for 14 days during the lag phase period [34]. These results were following methane production. Therefore, the results obtained supported the previous study, which disclosed that low biodegradation of organic matter during start-up processes to be due to the acclimatization phase, thus reducing their conversion to biogas [4].

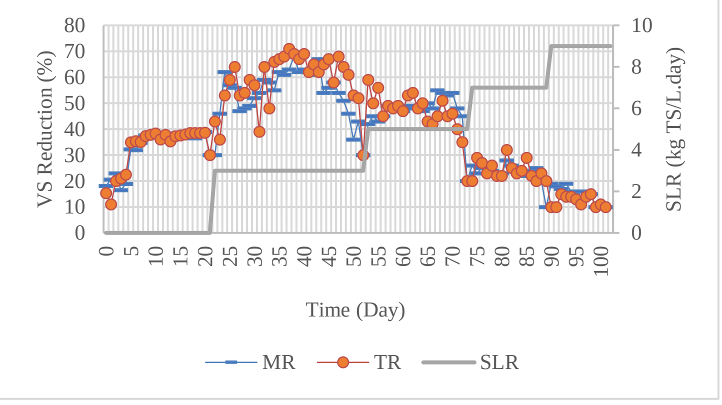


Figure 3. The profile of VS reduction for mesophilic and thermophilic digesters.

After the start-up phase was observed to be stable, new SLR was introduced in stages. After the incremental shift in SLR, a slight decrease was observed for a short time, which was attributable to the acclimatization period for biomass after a new SLR introduction into the reactor. Based on the observation, the SLR of 3 kg TS/L.day indicated the highest VS reduction and recorded as the effective stage for microbial activities. A previous study done by [35] also found that higher VS can be degraded to produce methane in thermophilic condition compared to mesophilic condition. This is due to higher organic carbon mineralization int biogas [35]. Thereafter, a slight decrease could be observed in VS reduction in the case of SLR of 5 kg TS/L.day until 9 kg TS/ L.day. The results were in accordance with the methane composition and production rate and yield, indicative of an upset reactor condition.

3.3. Effect of SLR on the intermediate product.

As an intermediary product of anaerobic digestion, volatile fatty acids (VFAs) accumulation through mesophilic and thermophilic AD processes was analyzed accordingly. The results obtained are shown in Figure 4.

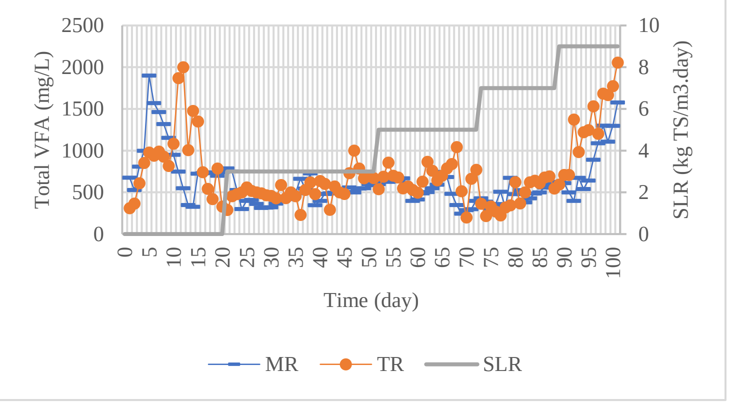


Figure 4. The profile of total VFA in both digesters during operation

During the start-up process, the total volatile fatty acid (TVFA) accumulated and reached its maximum values of 2000 mg/L and 1900 mg/L in the T_R and M_R digesters, respectively. Both values approached the maximum limit of 900 mg/L, thus highlighting the significant methanogen inhibition occurring at this phase [36]. This result was following methane production, whereby no methane was produced until day 12. Then, starting on day 13 onwards, a declining trend under the maximum limit for inhibition occurred (900 mg/L), demonstrating the achievement of stable operation. However, the trend in both digesters showed a significant increment during Phase 3 and Phase 4. The TVFA level was 23.99 % higher in the TR digester than in the MR digester. The same situation happened in previous works, whereby the TVFA accumulation was found to be higher in thermophilic digester compared to mesophilic digester [27]. Besides, the decrease in pH was observed due to the accumulation of TVFA, while the alkalinity in both digesters was reduced to the range of 5-6 g/L CaCO3. Consequently, methane production in both digesters also decreased, reaching almost zero methane production at day 100 of digestion and resulted in a reactor failure. This is happening due to the inhibited growth of anaerobic microorganisms [27].

4. CONCLUSIONS

The continuous dry anaerobic digestion of CM at 20% TS can be successfully performed at SLR of 3-5 kg TS/ L.day. Both digesters were most effective during Phase 1 (SLR: 3 kg TS/ L.day) compared to the remaining phases. The maximum methane yield and composition were observed to be 0.39 m3 CH4/kg VS (65 %CH4) for thermophilic conditions and 0.25 m3 CH4/kg VS (60 %CH4) for mesophilic condition, respectively. The VS reduction in thermophilic digester also marked a higher reduction compared to the mesophilic digester. The intermediary products indicated no inhibition occurred during the optimum SLR. In conclusion, this study showed that the performance and efficiency of biogas conversion can yield higher methane production by controlling the operating conditions during the AD process. The outcomes of this research are expected to provide a better understanding of methane gas production enhancement in the development of AD technology in the future.

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