Two-phase anaerobic digestion of unscreened dairy manure

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Abstract

Concentrated animal feeding operations along with a corresponding absence of suitable manure disposal methods have been shown to cause significant environmental and public health problems, including odors and nutrient enrichment and pathogen contamination of surface and ground waters. Anaerobic digestion (AD) of manure can offer substantial benefits, both economic and intangible, to animal feeding operators and surrounding communities, such as on-site energy generation, production of stable, liquid fertilizer and high quality solid soil amendment, reduction in odors, and reduction in ground and surface water contaminations. The two-phase AD has several advantages over conventional one-phase processes, such as selection and enrichment of different bacteria in each phase, increased stability of the process, and higher organic loading rates (OLR) and shorter hydraulic retention times (HRT). Even though several aspects of two-phase configuration might be very significant for efficient AD of dairy manure, its application has been limited to screened dairy manure only. Therefore, this study investigated possible exploitation of the advantages of two-phase AD for unscreened dairy manure. The results indicated that the use of a two-phase reactor at a SRT/HRT of 10 days (2 days acidogenic and 8 days methanogenic) for AD of dairy manure resulted in 50 and 67% higher biogas production at OLRs of 5 and 6 g VS/L day, respectively, relative to a conventional one-phase configuration with SRT/HRT of 20 days. Furthermore, the phased configuration could tolerate an elevated OLR of 12.6 g VS/L day, which was not achievable with a conventional one-phase configuration.

Keywords: Dairy; Manure; Anaerobic; Digestion; Phase; Separation

1. Introduction

Acidogenic and methanogenic microorganisms present in a mixed anaerobic culture diverge, not only in terms of their nutritional and pH requirements, but also with respect to their physiology, growth and nutrient uptake kinetics and in their ability to tolerate environmental stresses. Therefore, conditions that are favorable to the growth of acid-forming bacteria (short HRT, low pH) may be inhibitory to methane-forming bacteria. Furthermore, in a one-phase digester, the pH and organic loading rate are adjusted to suit the slow-growing methanogenic organisms at the expense of the relatively fast-growing acidogens and the process efficiency as a whole [1]. The different growth rates and pH optima for acidogenic and methanogenic organisms, and thus different requirements regarding reactor conditions, have led to the development of two-phase AD processes [1–6]. The two-phase configuration has several advantages over conventional one-phase processes. Firstly, it allows the selection and enrichment of different bacteria in each phase; in the first phase, complex pollutants are degraded by acidogenic bacteria into VFA, which are subsequently converted to CH4 and CO2 by acetogenic and methanogenic bacteria in the second phase. Secondly, it increases the stability of the process by controlling the acidification-phase in order to prevent overloading and the build-up of toxic material. Thirdly, the first stage may protect methanogens from overloads, preventing pH shock to the methanogenic population; in addition, low pH, a high organic loading rate and a short hydraulic retention time are all factors which favor the establishment of the acidogenic phase, and preclude the establishment of methanogens. Finally, the process can be smaller and more cost efficient [3–5,7–9].
Applications of two-phase AD have occurred in the biogasification of: wastewater treatment sludge [10–12], organic fractions of municipal solid wastes [13–17], industrial wastes and sludge [18], olive mill solid waste and olive pomace [19–21], grass [22], coffee pulp juice [23], food waste [24–26], cane–molasses alcohol stillage [27], spent tea leaves [28], brewery wastewater [29], dairy wastewater [30–32], abattoir wastes [33] as well as some studies focusing on improving reactor design, control and operational parameters [3,8,34–37].

Conventional one-phase slurry digestion is not an effective system for wastes containing high solids (>10%), since they require the manure to be capable of being pumped which in itself necessitates a concentration below 10% solids. This, in turn, results in a significant increase in fluid and digester volume which results in increased capital and operating costs. Although most animal wastes are produced as slurry, the housing methods, bedding, and collection methods used produce a material of much higher solids content. For example, cattle housed in sheds and bedded on straw produce a farmyard manure of much higher solids content. For example, cattle housed in bedding, and collection methods used produce a material wastes are produced as slurry, the housing methods, increased capital and operating costs. Although most animal increase in fluid and digester volume which results in decreased capital and operating costs. Although most animal wastes are produced as slurry, the housing methods, bedding, and collection methods used produce a material of much higher solids content. For example, cattle housed in sheds and bedded on straw produce a farmyard manure of approximately 26% solids [38]. The significance of high solid content animal manure in relation to the performance of AD in terms of reactor volumes, pumping, handling, mixing, and clogging are emphasized in several studies [38–40]. The associated investment costs for large-size reactors, as well as in the heating, handling, dewatering, and disposal of the digested residue decrease the benefits of conventional slurry anaerobic digestion (AD) of high solids containing wastes.

One relevant feature of the two-phase approach is that when a high solids containing waste is introduced to the first phase it is liquefied along with acidification. This translates into less liquid addition and, thus, less energy requirements for heating, storing, and spreading for two-phase AD systems. The results of several studies [13–17,19–21,24–26,41] have clearly demonstrated the applicability and efficiency of two-phase AD for high solids containing wastes.

Even though several aspects of two-phase configuration including liquifaction might be very significant for efficient AD of dairy manure, its application has been limited to screened dairy manure only [42–46]. Burke [47] also pointed out the fact that phased digestion has not been applied to dairy waste. In recognition of this fact and in support of its needed application to high solids waste, the objective of this study was to exploit the advantages of two-phase AD for unscreened dairy manure. To this purpose, one-phase conventional (R1) and two-phase (R2) reactor configurations were run at a range of OLRs and influent VS concentrations. R1 served as a conventional one-phase anaerobic digester with a SRT/HRT of 20 days. The total SRT/HRT of the two-phase configuration, meanwhile, was 10 days which consisted of an acidogenic first phase with a SRT/HRT of 2 days and a methanogenic second phase with a SRT/HRT of 8 days.

### Table 1

<table>
<thead>
<tr>
<th>Composition of raw dairy manure* [49]</th>
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</thead>
<tbody>
<tr>
<td>Dry matter (%)</td>
</tr>
<tr>
<td>Composition (% of dry matter)</td>
</tr>
<tr>
<td>Neutral detergent fiber (NDF)</td>
</tr>
<tr>
<td>Acid detergent fiber (ADF)</td>
</tr>
<tr>
<td>Acid detergent lignin (ADL)</td>
</tr>
<tr>
<td>Hemimicellulose (NDF–ADF)</td>
</tr>
<tr>
<td>Cellulose (ADF–ADL)</td>
</tr>
<tr>
<td>Lignin (ADL)</td>
</tr>
<tr>
<td>Total carbon</td>
</tr>
<tr>
<td>Total nitrogen</td>
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* Data is expressed as mean ± S.D. of three replicates.

### 2. Materials and methods

#### 2.1. Dairy manure and anaerobic seed cultures

Fresh manure was collected from the Dairy Center at Washington State University in Pullman, WA, and stored at 4 °C prior to use. The composition of the raw dairy manure is presented in Table 1 [48].

The mixed anaerobic culture used as seed was obtained from the anaerobic lagoon of the Dairy Center at Washington State University in Pullman, WA, and stored at 4 °C prior to use. The mixed anaerobic culture was filtered through a screen of 0.0469 in. (1.19 mm) mesh size and concentrated by settling before being used as inoculum. The volatile suspended solids (VSS) concentration of the concentrated seed cultures used was 4520 ± 180 mg/L.

#### 2.2. Experimental set-up

The experimental set-up used is depicted in Fig. 1. The one-phase conventional configuration (R1) was run as the control for the two-phase configuration (R2). The effective volumes of R1, R2-1, and R2-2 were 2.0, 0.4, and 1.6 L, respectively. The two-phase configuration contained R2-1 and R2-2 as the first (acidogenic) and second (methanogenic) phases. The SRT/HRT values of R2-1, R2-2, and the overall two-phase configuration were 2, 8, and 10 days, respectively. All the reactors were fed daily. The gas production in R1 and R2-2 were monitored by a wet-tip
gas-meter (Speece Tip, Nashville, TN), while a water replacement device \[49\] was used to monitor the gas production in R2-1. R1 and R2-2 were maintained at 36 °C \(+\pm 2^\circ\) in a temperature-controlled water bath and were shaken manually once a day after gas production determination. R2-1 was incubated in an incubator shaker (New Brunswick Scientific, Edison, NJ) at 35 ± 1 °C and 180 rpm. R1, R2-1, and R2-2 were seeded with 500, 100, and 400 mL of mixed anaerobic seed culture. Before the onset of daily feeding, R2-1 to R2-2, R2-1 were operated for 26 days (Fig. 4d) at an SRT/HRT of 2 days to achieve an active acidifying culture. Operation of the two-phase configuration was started on Day 27. The operational parameters (loading rates, influent concentrations, etc.) used for all the reactors are depicted in Figs. 2 and 4.

The performance of the reactors was monitored by measuring biogas production and chemical oxygen demand (COD), volatile solids (VS), ammonia nitrogen (NH\(_3\)-N), total kjeldahl nitrogen (TKN), total phosphorus (TP), and pH.

2.3. Analytical methods

COD, VS, NH\(_3\)-N, TKN, TP, and pH analysis were performed at the WSU Water Quality Lab as described in Standard Methods \[50\]. The content of CH\(_4\) in the biogas was determined as follows. A known volume of the headspace gas (V\(_1\)) produced in a serum bottle used in the biochemical methane production (BMP) experiments was syringed out and injected into another serum bottle which contained 20 g/L KOH solution. This serum bottle was shaken manually for 3–4 min so that all of the CO\(_2\) and H\(_2\)S were absorbed in the concentrated KOH solution. The volume of the remaining gas (V\(_2\)), which was 99.9% CH\(_4\), in the serum bottle was determined by means of a syringe. The ratio of V\(_2\)/V\(_1\) provided the content of CH\(_4\) in the headspace \[51\]. Methane content of biogas was determined in six duplicate samples and found to be 63.5 ± 3.7%.

3. Results and discussion

3.1. Conventional one-phase configuration (R1)

The operation was started with an organic loading rate (OLR) of 1.0 g VS/L day (0.96 g COD/L day). The biogas production rate and biogas yield at this OLR averaged 0.356 ± 0.072 L/day and 0.176 ± 0.035 L biogas/g VS added, respectively (Fig. 2c). The OLR was increased to 2.0 g VS/L day (1.91 g COD/L day) on Day 27. The corresponding influent VS concentration was 9.13% (Fig. 2b). At this OLR, the observed biogas production rate and biogas yield were 2.375 ± 0.536 L/day and 0.235 ± 0.053 L biogas/g VS added, respectively (Fig. 2c). On Day 76, the OLR and influent VS concentration were increased to 6.3 g VS/L day (6.02 g COD/L day) (Fig. 2a and b) and 11.5%, respectively. It must be noted at this point that the OLR value adopted at this phase of operation is considerably higher than the values reported in studies on AD of dairy and cattle manure \[42,52–54\]. Even though this final increase in the OLR resulted in a little increase in the biogas production from 2.375 ± 0.536 to 2.725 ± 0.236 L/day, the biogas yield decreased from the previous level of 0.235 ± 0.053 to 0.214 ± 0.019 L biogas/g VS added (Fig. 2c). Thus, when a decrease in biogas yield was noted against an increase in OLR no further increase in the OLR was attempted. The 96-day operation of R1 between OLRs of 1–6.3 g VS/L day (0.96–6.02 g COD/L day) (Fig. 2a and b) and influent VS concentrations of 1.83–11.5% was thought to be sufficient to represent a conventional one-phase anaerobic digester treating dairy manure.

After obtaining the base-line performance data for a conventional one-phase anaerobic digester, the SRT/HRT of R1 was reduced to 10 days on Day 97.
this was to observe the response of mixed anaerobic cultures in R1 which was acclimated to dairy manure for 96 days of operation, to low SRT/HRT operation. When the SRT/HRT was reduced to 10 days, the corresponding OLR and influent VS concentration were also reduced to 3.15 g VS/L day (3.01 g COD/L day) and 5.75%, respectively (Fig. 2 b). After acclimating to and operating at higher OLR and influent VS values, R1 performed noticeably better. The biogas production rate and biogas yield increased to 3.000 \( \text{C}_6 \) 0.222 L/day and 0.470 \( \text{C}_6 \) 0.035 L biogas/g VS added, respectively, for the first 13 days of operation at SRT/HRT of 10 days (Fig. 2 c). However, starting with Day 110, an abrupt decline in both biogas production rate and yield were observed. Then, biogas production dropped to 0.2–0.3 L/day only in 6 days, indicating wash-out conditions. This is also obvious from Fig. 3b.

### 3.2. Two-phase configuration (R2)

The first phase reactor (acidifying reactor or R2-1) was operated for 26 days (Fig. 4a) at an SRT/HRT of 2 days and OLR of 1.0 g VS/L day (1.19 g COD/L day) to achieve an active acidifying culture. On Day 27 the effluent of the first phase (R2-1) was fed to the second phase (methanogenic reactor or R2-2). After the onset of this second feeding the two-phase configuration was operated at the same OLR (1.0 g VS/L day or 1.19 g COD/L day) and influent VS concentration (0.91%) for 21 additional days (Fig. 4a and b). The observed biogas production rate and biogas yield during this operation (Days 27–48) were 0.154 ± 0.051 L/day and 0.076 ± 0.025 L biogas/g VS added, respectively (Fig. 4c). The next OLR level was 2.0 g VS/L day (2.39 g COD/L day) which was applied to the two-phase system between Days 48–61 (Fig. 4a and b). The corresponding influent VS concentration was 1.83% during this phase of operation. Parallel to this increase in the OLR, the biogas production rate and biogas yield also increased to 0.419 ± 0.122 L/day and 0.103 ± 0.030 L biogas/g VS added, respectively (Fig. 4c). The pH values of the feed, R2-1 and R2-2 were 7.7, 6.7, and 7.3, respectively. These pH values indicated that the first phase reactor (R2-1) was achieving a certain acidification of the fed dairy manure but the corresponding pH value (6.7) was higher than that of acidified food and solid wastes (4.5–7.5 and 5.0–6.0) in two-phase AD configurations reported in the literature [26]. However, further decreases in the pH value of the first phase reactor were observed at higher loading rates as will be discussed below. Furthermore, the increase of pH observed in the methanogenic reactor (R2-2) can be explained by both the volatile fatty acids (VFA) converted to CH4 and CO2 by methanogens as well as the alkalinity generated by the anaerobic biodegradation of nitrogenous organic compounds [55] contained in the dairy manure used in this study [48].

In the next operational phase, an OLR of 5.0 g VS/L day (5.97 g COD/L day) was employed between Days 62–90 (Fig. 4a and b). The resulting influent VS concentration was 4.57%. At this OLR, the observed biogas production rate and biogas yield increased to 1.782 ± 0.493 L/day and 0.176 ± 0.049 L biogas/g VS added, respectively (Fig. 4c). An important observation at this stage of operation is with respect to the pH values of the first phase reactor (R2-1). As seen in Fig. 4e, the pH values of R2-1 went down to around 6.0 from an earlier value of around 6.7 for an increase of OLR from 2.0 to 5.0 g VS/L day (2.39 to 5.97 g COD/L day). This decrease in pH in R2-1 indicated a higher extent of acidification in the first phase reactor. On Day 90, the OLR and influent VS concentration were increased to 6.3 g VS/L day (7.53 g COD/L day) and 5.75%, respectively. This increase in the OLR resulted in a little increase in the biogas production from 1.782 ± 0.493 to 2.272 ± 0.243 L/day. However, the biogas yield remained more or less the same (0.178 ± 0.019 L biogas/g VS added) relative to the previous phase (0.178 ± 0.049 L biogas/g VS added).

Even though the biogas yield stayed more or less the same between OLRs of 5.0–6.3 g VS/L day (5.97–7.53 g COD/L day), the OLR was increased one last time to an exceptionally high value of 12.6 g VS/L day (15.06 g COD/L day) to test the performance of the two-phase
configuration under extreme conditions (Fig. 4a and b). The corresponding influent VS concentration was 11.5%.
The first observation was the reduction in pH in the R2-1 from a previous level of 6.0 to a level near 5.8 while the
pH value of the methanogenic stage (R2-2) indicated constant, normal anaerobic operating conditions (Fig. 4e).
Additionally, a gradual increase in the biogas production was observed. However, this increase was not pro-
portional with the increase in the OLR which was reflected in the low biogas yield values (Fig. 4c). The average biogas
production rate and biogas yield values observed for this last phase of operations (Days 111–134) were 3.822
/C6 0.332 L/
day and 0.150/C6 0.013 L biogas/g VS added, respectively (Fig. 4c).

3.3. COD, VS, NH3-N, TKN, and TP removal efficiencies

The observed COD, VS, NH3-N, TKN, and TP removal efficiencies for R1 and R2 are given in Tables 2 and 3,
respectively. The observed COD removal efficiency was more or less within the typical performance level of
anaerobic digesters treating dairy manure [56], while the observed VS removal was lower [42], similar to [38], and
higher [52] than some of the studies in the literature. For similar COD loading rates, the COD removals were
slightly better in R1 (Tables 2 and 3). This is probably due to lower corresponding influent COD values (Figs. 3
and 4). However, VS reductions were higher in R2, probably through better hydrolysis in the acidification
phase (R2-1). The negative values observed for ammonia nitrogen and TKN removal corresponds to an increase in
concentration for these nutrients in the reactor. This is an observation also reported by other researchers [56,57] and
can be attributed to the anaerobic biocconversion of proteins contained in manure into amino acids and then
to ammonia. The low treatment efficiencies observed for TKN and TP are also expected since anaerobic digesters
are known to reduce negligible amounts of nutrients [58].

Table 2
COD, VS, NH3-N, TKN, and TP removal efficiencies for R1

<table>
<thead>
<tr>
<th>COD loading rate (g/L day)</th>
<th>VS in the feed (%)</th>
<th>SRT/HRT (days)</th>
<th>Percent removals</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>COD</td>
</tr>
<tr>
<td>0.96</td>
<td>1.83</td>
<td>20</td>
<td>45–66</td>
</tr>
<tr>
<td>1.91</td>
<td>3.65</td>
<td>20</td>
<td>69–77</td>
</tr>
<tr>
<td>6.02</td>
<td>11.5</td>
<td>20</td>
<td>55–71</td>
</tr>
<tr>
<td>3.01</td>
<td>5.75</td>
<td>10</td>
<td>24–50</td>
</tr>
</tbody>
</table>

Table 3
COD, VS, NH3-N, TKN, and TP removal efficiencies for R2

<table>
<thead>
<tr>
<th>COD loading rate (g/L day)</th>
<th>VS in the feed (%)</th>
<th>SRT/HRT (days)</th>
<th>Percent removals</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>COD</td>
</tr>
<tr>
<td>1.19</td>
<td>0.91</td>
<td>10</td>
<td>30–43</td>
</tr>
<tr>
<td>2.39</td>
<td>1.83</td>
<td>10</td>
<td>59</td>
</tr>
<tr>
<td>5.97</td>
<td>4.57</td>
<td>10</td>
<td>42–62</td>
</tr>
<tr>
<td>7.53</td>
<td>5.75</td>
<td>10</td>
<td>48–54</td>
</tr>
<tr>
<td>15.06</td>
<td>11.5</td>
<td>10</td>
<td>54–71</td>
</tr>
</tbody>
</table>

Fig. 4. (a–d) SRT/HRT, loading rates used, biogas production and yields, and pH values observed during the operation of R2.
3.4. Performance evaluation of two-phase versus one-phase configuration

Fig. 6 depicts biogas productions for one- and two-phase configurations for different OLRs and influent VS concentrations. As seen in Fig. 6a, biogas production in R2 increased with the increase in OLR. Because the SRT/HRT ratio of R2 to R1 is 0.5 (20 versus 10 days, respectively), the biogas production in R2 must be more than 50% that of R1 to realize any beneficial effect of phase separation on AD of dairy manure. Therefore, the biogas production in R2 was normalized over this value and plotted in Fig. 6b. Fig. 6b clearly shows that there is no advantage of the two-phase over conventional one-phase configuration at low OLRs. However, at high OLRs, the normalized biogas production was significantly high, namely 0.750 and 0.834 for OLRs of 5 and 6 g VS/L day, respectively (Fig. 6b). These values correspond to 50 and 67% higher biogas production or volume reduction for two-phase AD of dairy manure at OLRs of 5 and 6 g VS/L day, respectively. The advantage of a two-phase configuration is also clear when the biogas production with respect to varying influent VS concentrations are considered (Fig. 6c). The benefit of the two-phase system becomes more apparent as the influent VS concentration increases. This is logical since increased influent VS concentrations corresponds to more insoluble fiber content of the manure which is hydrolyzed in the first (acidification) phase of the two-phase configuration. Furthermore, it must also be noted that the two-phase configuration could perform fairly well at an elevated OLR of 12.6 g VS/L day (Figs. 4–6) which was not possible for conventional one-phase configuration.

4. Conclusions

This study investigated for the first time the possible exploitation of the advantages of two-phase AD for unscreened dairy manure which could result in significant environmental and public health problems. The results indicated that the use of a two-phase reactor at a SRT/HRT of 10 days (2 days acidogenic and 8 days methanogenic) for AD of dairy manure:

- Resulted in 50 and 67% higher biogas production or volume reduction at OLRs of 5 and 6 g VS/L day,
respectively, relative to a conventional one-phase configuration with SRT/HRT of 20 days.

- Made an elevated OLR of 12.6 g VS/L day possible which was not achievable for conventional one-phase configuration.
- Translates into significant cost savings due to both superior performance and reduced volume requirements.

References


