Examining a Hybrid plug-flow Pilot reactor for Anaerobic Digestion of Farm-Based Biodegradable Solids

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ABSTRACT: Plug-flow digesters with periodic loading mechanism are more enthusiastic than fully mechanized digestion plants for the majority of small or medium scale farms according to the costs and operational complexities. A dual-compartment plug-flow reactor equipped with a passive heating system was designed and experimentally operated by purpose of demonstrating a simple and low cost technology for handling the biodegradable agricultural wastes. The reactor was successfully started up with pig feces as feedstock under a quasi-continuous loading and semi-dry condition with an average total solids content of 12.8% inside the digester and an average organic loading rate of 2.06kg-VS/(m³.d). The start-up phase was followed by co-digestion of pig feces and pre-treated cotton stalk. Even though the digester actually worked at a temperature range 12 to 30% below the optimal mesophilic level, acceptable rates of methane generation and VS destruction were observed. The biogas and methane yield were measured for single digestion stage as 0.332 and 0.202 m³/(kg-added VS) and for co-digestion stage as 0.482 and 0.325 m³/(kg-added VS) respectively. The cumulative biogas production data demonstrated a reasonable correlation (R² over 0.99) with a simplified consecutive kinetic model.

Key words: Biogas, Plug-flow, Start-up, Agricultural wastes, Pig feces, Cotton stalk, Kinetic model

INTRODUCTION

Anaerobic digestion of farm-based biodegradable solids has not only been widely studied in the literature within bench and pilot scale but also practically applied in full scale in numerous countries for the purpose of biomass-to-energy conversion as well as a waste management alternative for farms (Balasubramanian et al., 1992; Chynoweth et al., 1987; Field et al., 1985; Ward et al., 2008). As evidence, the share of agricultural resources in total biogas production in European Union (EU25) countries was increased from 27.1% in 2006 to 35.7% in 2007 and a total biogas generation equal to 36.5 billion m³ is anticipated as a target for 2020 (AEBIOM, 2009). The number of farm-based medium and large-scale biogas generation plants (MLBGP) in China has been increased from approximately 1500 in 2004 (Zhang et al., 2009) to around 4700 by the beginning of 2010; while China’s target for 2020 is to generate 2 billion m³ out of total 44 billion m³ biogas from agricultural sources (GTZ, 2010; He, 2010). Covered anaerobic lagoons, plug-flow reactors and completely mixed reactors are the most popular anaerobic digestion systems that are applied in full scale biogas generation plants utilizing agricultural wastes among Europe, Northern America and many other countries (AEBIOM, 2009; Bracmort & Burns, 2008; USEPA, 2010; USDA, 2007). The majority of farm-based anaerobic digestion plants in the United States (76 units out of 151) employ plug-flow reactors (USEPA, 2010). Several technologies of anaerobic digestion including anaerobic contact process, completely mixed reactor, upflow anaerobic sludge blanket (UASB), upflow anaerobic hybrid blanket, anaerobic filter and plug-flow reactor are popular in China for handling livestock wastes and crop residues (He, 2010). However, the majority of the Chinese on-farm biogas plants visited by the authors, have been working with digester influents containing below 8% total solids (TS) content whilst Chinese farmers often separated the larger solid constituents e.g. feces and straw from agricultural waste streams in advance and sold them as a fertilizer neglecting their significant potential of methane production as well as their potential pollution load in the terms of organic compounds, pathogens, odors and parasites.

Plug-flow anaerobic digesters have their own advantages including simpler structure, lower capital
cost, low operational energy demand, prevention of short circuiting and assured retention time, satisfactory pathogen destruction, and ability to accept high solids content (Smith et al., 2005; Martin et al., 2003; Lansing et al., 2008). They have proven their efficacy in single digestion of livestock wastes as well as co-digestion with other biological wastes through numerous cases among which, organic loading rate (OLR) varied between 1 and 6 kg COD/m³.d, hydraulic retention time (HRT) from 20 to 50 days, and biogas production from 0.4 to 0.8 m³/m³.d averaged around 0.6 m³/m³.d with specific yield of 0.3 to 0.8 m³/kg VS added (Martin et al., 2003; Cantrell et al., 2008; Lansing et al., 2008; Lansing et al., 2010). Nevertheless, some drawbacks accompany them such as lower mass transfer due to lack of mixing, lower efficiency in low solid content, thermal stratification and solids sedimentation problems (Chynoweth, 1987; Monnet, 2003; Myers et al., 2006; Cantrell et al., 2008).

This article describes the attempt for development of a modified plug-flow anaerobic digester equipped with externally partial mixing by purpose of working with farm-based biodegradable solids at a TS between 10 to 16 percent in order to further scale-up and development of this technology aiming to facilitate China’s long-term bioenergy targets.

MATERIALS & METHODS

The configuration of pilot-scale anaerobic digestion system has been schematically illustrated in Fig. 1.

The pilot digester was designed in the form of a cylindrical chamber with a 2.5 height: diameter ratio containing a smaller tube assembled inside as a coaxial heat exchanger. The digester was equipped with four ball valves by 50mm I.D. as inlet, outlet, and two sampling ports at different levels and polar angles to each other. Two baffles were improvised to divide the reactor chamber into two compartments until one sixth of the digester’s height before the bottom therefore; they prevent any short circuiting and enforce the plug-flow regime of digester contents upon the following sequence: inlet-port A- port B- outlet. Fig. 2. shows the digester cross section and flow sequence.

The main body of the digester was fabricated from mild steel in Iran and final assembling works were carried out in Bioenergy and Biomaterials Laboratory of Zhejiang University, China. The heat exchanger was filled with heat transfer fluid instead of water. The thermal fluid was selected from XCELTERM®-LV brand supplied by Shanghai Yancui Import & Export Ltd. The heat exchanger was equipped with an 110W electrical heating element. The adopted heat transfer fluid presents some 60% saving in heating energy comparing water according to their specific heat difference (1.6 versus 4.186 J/g.K). The thermal oil’s temperature was being controlled by an electrical thermostat which was connected to the heating element. The completed digester system can bee seen in Fig. 3.

Matured swine sludge (3 months aged) from Tong Ren, Haining swine farm biogas plant was initially fed

![Fig. 1. Schematic configuration of pilot plant system](image-url)
to the digester on the first two days of start-up. High-TS digestate sludge from another swine farm biogas plant in Lin’an, Zhejiang was utilized in the third stage and thereafter, mixed with fresh feedstock during the next couple of feeding stages. The air inside the digester was replaced by nitrogen gas injection. Pig feces were supplied from the research swine farm in the Huajiachi campus of Zhejiang University. Cotton stalks were collected from cultivation farms in the same campus. They were ground for size reduction to less than 3mm. The ground cotton stalks were mixed with the diluted process liquid from the digestion process and boiled at 100°C for around 15 minutes for pre-treatment prior to feeding. The characteristics of the inocula and feedstock have been summarized in Table 1.

The loading of the digester was carried out in two stages with a 32 day gap in between in order to investigate the biogas production trend. The first phase was allocated to single digestion of swine manure and the second phase included combined digestion of pig feces and pre-treated cotton stalk. It should be mentioned that every batch of fresh feedstock was being mixed with a part of the withdrawn content from the digester’s sampling ports before loading to the inlet. This procedure actually provided an external mixing for the digester therefore, modified it to a hybrid plug-flow reactor.

Sampling was performed for all materials during the whole experimentation period. Total solids (TS)
Pilot reactor for Anaerobic Digestion

were measured for every sample in triplicates on the wet basis throughout drying the samples at 75±3 °C in order to decrease the effects of thermochemical degradation on dry matter and volatile solids (VS) (Greenhill, 1960; Mayland, 1968; Brahmakshatriya, 1971). Ash content was determined for the oven dried samples by oxidizing them in a muffle furnace at 575±25 °C following the method recommended by NREL (Sluiter et al., 2005).

Ammonium nitrogen (NH₄-N) was calculated by reaction of sample with an alkaline solution including sodium hypochlorite and sodium hydroxide and measurement of released gaseous nitrogen volume, immediately after sampling.

Total carbonaceous alkalinity and total volatile fatty acids (TVFA) for samples were evaluated using titration by 0.05M sulfuric acid (Jenkins et al., 1983; Ripley et al., 1986; Lahav & Morgan, 2004).

Cumulative biogas volume was measured by an analog wet gas volume recorder, model BSD-0.5 made by Shanghai Keluo Lab Instruments Ltd. The recorded biogas volume was then normalized for standard temperature and pressure (STP) conditions according to practical temperature and pressure.

Methane, oxygen, and H₂S content of biogas were being measured using a portable gas detector (Polytector® G450) provided and calibrated by GfG Co. Ltd. The water vapor content was being estimated on the base of gas temperature using the psychrometric charts. The content of biogas components were being used for calculation of the real time specific gravity and subsequently the partial mass of biogas.

The biogas production trend was modeled using a simplification of consecutive reactions that occur towards anaerobic degradation of substrates. The basic equation for determining the concentration of final product in a consecutive reaction within batch or plug-flow reactor has been mentioned in the literature as follows (Levenspiel, 1999).

\[
A \xrightarrow{k_1} M \xrightarrow{k_2} F \quad \Rightarrow \quad C_F = C_{A0} - C_A - C_M
\]

Where C represents the concentration, A, M and F are initial substrate, intermediate and final products respectively, and \(k_1\) and \(k_2\) are kinetic constants in [1/time]. Assuming biogas as the final product, the following equation will apply well for prediction of its concentration in batch or plug-flow reactors over the retention time (Levenspiel, 1999).

Table 1. The characteristics of Inocula and feedstock

<table>
<thead>
<tr>
<th>Parameter</th>
<th>TS</th>
<th>VS</th>
<th>TAC*</th>
<th>TVFA**</th>
<th>Elemental analysis (%-dry basis)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>% wet basis</td>
<td>% dry basis</td>
<td>mg/kg wet</td>
<td>mg/kg wet</td>
<td>C</td>
</tr>
<tr>
<td>Mature swine sludge</td>
<td>9.23±3.13</td>
<td>71.98±0.58</td>
<td>16201</td>
<td>15935</td>
<td>38.7</td>
</tr>
<tr>
<td>Thick digestate</td>
<td>15.26±0.7</td>
<td>49.41±0.59</td>
<td>48480</td>
<td>18085</td>
<td>27.1</td>
</tr>
<tr>
<td>Cotton stalks</td>
<td>95.84±1.3</td>
<td>93.74±1.85</td>
<td>n.a</td>
<td>n.a</td>
<td>41.7</td>
</tr>
<tr>
<td>Swine wastes</td>
<td>19.99±3.74</td>
<td>75.92±1.75</td>
<td>12692 ± 929</td>
<td>21551 ± 3710</td>
<td>40.8</td>
</tr>
</tbody>
</table>

*) Total alkalinity (as CaCO₃)  **) Total volatile fatty acids (as CH₃COOH)
by the following equation.

The resultant cumulative biogas volume was calculated in order to perform the least squares algorithm.

The optimal fitted kinetic constants (k₁ and k₂) and ultimate yield were determined on the laboratory results’ basis in this study using a computational program in order to perform the least squares algorithm.

In case of multiple feeding during a time sequence, the resultant cumulative biogas volume was calculated by the following equation.

\[ C_f = C_{0f}[1 - \exp(-k_1t) - (k_1/(k_2-k_1))\exp(-k_2t)] \]

Re-arranging the equation (3) on a mass basis will consequent the equation (4) that predicts the cumulative volume of biogas (Gₜ) at time (t) as follows:

\[ G_i = (Y_g \cdot m_b)/(k_2-k_1) \left[ k_2\left[1-\exp(-k_2t)\right]-k_1 \right]/\left[1-\exp(-k_2t)\right] \]

Where, \( Y_g \) is the ultimate biogas yield as unit volume of gas per unit mass of initial substrate, and \( m_b \) is the biodegradable mass.

The optimal fitted kinetic constants (k₁ and k₂) and ultimate yield were determined on the laboratory results’ basis in this study using a computational program in order to perform the least squares algorithm.

In case of multiple feeding during a time sequence, the resultant cumulative biogas volume was calculated by the following equation.

\[ V_i = \sum_{i=1}^{n} G_i(t) = \sum_{i=1}^{n}(Y_g \cdot m_{b,j})/(k_2-k_1) \left[ k_2\left[1-\exp(-k_2T)\right]-k_1 \right]/\left[1-\exp(-k_2T)\right] \]

Where, \( n \) is the number of feedings, \( t \) is the absolute time after start-up, (i) represents the feeding batch, and \( t_i \) is time at which, the batch (i) has been fed to the digester.

RESULTS & DISCUSSION

Fig. 4 shows the mass balance chart of the digester in which, the variations in the components of input and output masses have been illustrated. During the 1st stage that lasted the content of inocula was gradually increased till the 25th day. The 2nd stage of feeding was carried out between day 57th to day 85th through which, the mixtures of pig feces and pre-treated cotton stalk were loaded to the digester.

The ambient temperature of the laboratory was often low during the start-up period and simultaneously, some troubles occurred in the passive heating system of the digester. These conditions culminated in lower internal temperatures than anticipated optimal mesophilic range. The plot of internal temperature in various parts of digester (Fig. 5.a) showed a thermal stratification inside the digester reflecting the insufficient efficacy of the passive heating system as there was neither continuous mixing nor circulation of warm heat transfer fluid inside the digester. However, in a full-scale system it can be substitute with a heating circuit by application of heat transfer fluid instead of water and high thermal conductive copper pipes instead of conventional steel pipes. This combination will need lower energy and eliminates the problem of fouling in the pipes. The risk of pipe corrosion inside the digester can be resolved by cathodic protection.

The actual VS contents in the feedstock and in the different locations of the digester were calculated as a percent of whole fresh mass. The resultant VS values at the inlet were calculated by taking into account the weighted average of the VS in feedstock and in the recycled sludge from middle part of the digester hence, they are considerably lower than their corresponding feedstock VS. The ratio of feedstock-VS to recycled-VS was fluctuating in the range of 0.8 to 1.45. The results have been illustrated in Fig. 5.b. The VS at port “A” that represents the first compartment of the digester was increased as far as the organic loading proceeded and then followed a descending trend after the end of feeding stage. Port “B” showed a smoother curve of VS with lower values than the curve in port “A”. The VS values at outlet were slightly lower than the port “B”. Considering the partial retention times between inlet, port “A”, port “B” and outlet, VS reduction from inlet towards port “A” varied from 7.2% at early start-up to 24.8% at the end of the first stage and from 24.68% at the beginning of the 2nd stage to 36.4% at its end, while from port “A” towards port “B” it was in the range of 16.8% to 23.2%. The overall VS destruction between inlet and outlet was estimated as 40.0%. The Fig. 5.b suggests that the majority of VS destruction occurred within the partial volume between inlet and port “B” or in other words within the 55% of effective volume of the reactor. Measurements of ammonium nitrogen in pig feces showed its variation in the range of 2065 to 4557 with an average of 3408 mg per kg-fresh matter. The average content of NH₄-N was being decreased to an average of 2290± 197 mg/kg after partial retention time between inlet and port “A”.

The first gas flow was recorded 6 days later than the initiation of start-up on December 5th, 2009. The methane content was initially below the detecting level nevertheless, it was increased to 35%\(^\prime\) within 5 days after the first gas production recording. The chart of cumulative biogas volume and methane content has been presented in Fig. 6. It should be mentioned that biogas leakage in the system was detected twice and fixed afterwards, once between the day 21 and 24, and again during the day 94 till 105 just after the second feeding stage. A comparison between Fig.4 and Fig.6 reveals that the methane content was maximized a few days after feeding stages and then tended to decrease to around 50%\(^\prime\), with a rather rapid rate. Maximum biogas production rate occurred during the co-digestion stage between day 79 and day 85 and was equal to 44.2 dm\(^3\)/d.
Fig. 4. Cumulative mass of inputs to and outputs from the digester during four months of test

Fig. 5. Internal parameters of the digester: a) temperature variations b) volatile solids’ trends c) pH variations d) total alkalinity and total volatile fatty acids
Bench-scale batch experiments’ results for biogas yield estimation from the applied substrates in this study have been summarized in Table 2. The produced biogas from raw pig feces was quite negligible. The plot of cumulative biogas production from inoculated pig feces has been established in Fig. 7. The net biogas yield of feedstock in an inoculated mixture was calculated from the equation (6).

\[
Y_g^{\text{feedstock}} = \left( V_t - Y_g^{\text{inocula}} m_{\text{inocula}} \right) / m_{\text{feedstock}} 
\]

Where, \( Y_g^{\text{inocula}} \) is the mean biogas yield of control assay, \( m_{\text{feedstock}} \) and \( m_{\text{inocula}} \) are the mass of feedstock and inoculum respectively.

The practical biogas production trend for batch experiment of inoculated pig feces was compared with the model by the use of Eq. (3) and assuming the values in Table 3 for \( Y_g \) and VS-mass of pig feces and inoculum respectively. The best fitted values for \( k_1, k_2 \) and \( Y_g \) for the above mentioned equation were numerically calculated and illustrated on Fig. 7 where the coefficient of determination shows a good correlation (\( R^2=0.9969 \)). The similar attempt for modeling of cumulative biogas volume in the digester was carried out using the equation (5) in which, VS-mass data were entered and the optimal fitted values of kinetic constants and biogas yield were numerically calculated. During the 1st stage of loading the most fitted kinetic constants were obtained as \( k_1=0.0835/d \) and \( k_2=0.1455/d \) with a correlation factor of 0.9967 as shown in Fig. 8. A glance to the graphs in Fig. 8 suggests the influence of biogas leakages (which were discussed before in Fig. 6) on creating a differentiation between the predicted and practical biogas volume. The kinetic constants imply that creation of intermediate products (e.g. volatile fatty acids) in the process had a faster rate than generation of methane as \( k_1 \) was significantly higher than \( k_2 \).

The input mass flow rates of VS to the digester were classified into components including inoculum-VS, pig manure-VS, and cotton stalk-VS. Similarly, the output VS masses from digester were classified into discharged sludge-VS and biogas corresponding-VS. The plots of variations in the above mentioned components of VS during the start-up and experimental operation of the digester have been illustrated in Fig. 9 in which, the plot of cumulative biogas volume has been shown as well for a better understanding of compatibility between the trends of VS and biogas. The decreasing trends on the cumulative total VS plot in Fig. 9 correspond to the destructed VS masses that have been released in the form of biogas as well as the discharged VS from the digester. Biogas and methane yield during the 1st phase of start-up (till day 57) were obtained equal to 0.332 \( L/g-VS_{\text{added}} \) and 0.202 \( L/g-VS_{\text{added}} \) respectively. The similar parameters after co-digestion feeding during the 2nd phase of start-up (from day 57 till day 95) were equal to 0.325 for biogas and 0.325 for methane. The biogas yield between the day 57 and day 117 was obtained equal to 0.570 and the biogas yield per removed VS between day 57 and day 95 was calculated as 0.872. During the first stage of operation the recorded biogas volume was equal to 44% of ultimate biogas potential of the digester’s contents.
Table 2. Results of biogas potential batch tests

<table>
<thead>
<tr>
<th>Test set</th>
<th>Mean Fresh mass (g)</th>
<th>Mean VS mass (g)</th>
<th>Ultimate biogas volume (mL)</th>
<th>Net biogas yield (mL/g-VS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inoculum</td>
<td>Inoculum</td>
<td>Feedstock</td>
<td>Inoculum</td>
<td>Feedstock</td>
</tr>
<tr>
<td>Inoculum</td>
<td>149.06</td>
<td>n.a.</td>
<td>11.34</td>
<td>n.a.</td>
</tr>
<tr>
<td>Inoculated pig feces</td>
<td>163.64</td>
<td>153.57</td>
<td>11.68</td>
<td>20.68</td>
</tr>
<tr>
<td>Inoculated cotton stalk</td>
<td>188.5</td>
<td>36.10</td>
<td>7.55</td>
<td>3.28</td>
</tr>
</tbody>
</table>

![Graph showing cumulative biogas volume vs. predicted curve for the batch digestion of pig feces.](image)

Fig. 7. Practical curve of cumulative biogas volume vs. predicted curve for the batch digestion of pig feces

Table 3. Comparison of biogas and methane yield in current study with some other plug-flow reactors

<table>
<thead>
<tr>
<th>Technology</th>
<th>Feedstock</th>
<th>Operational temp. (°C)</th>
<th>Biogas yield (m³ per kg-added VS)</th>
<th>Methane yield (m³ per kg-added VS)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>HormozMehr® (current study)</td>
<td>Single digestion of pig feces</td>
<td>24 to 29</td>
<td>0.332</td>
<td>0.202</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Co-digestion of pig feces and cotton stalk</td>
<td>26 to 31</td>
<td>0.482 (0.872 m³/kg-removed VS)</td>
<td>0.325</td>
<td>a</td>
</tr>
<tr>
<td>Un-heated plug-flow reactor</td>
<td>Co-digestion of swine manure and used grease</td>
<td>24 to 26</td>
<td>0.17 to 0.46</td>
<td>0.012 to 0.31</td>
<td>a</td>
</tr>
<tr>
<td></td>
<td>Mono-digestion of swine manure</td>
<td>24 to 26</td>
<td>0.42</td>
<td>0.29</td>
<td>a</td>
</tr>
<tr>
<td>Pilot reactor</td>
<td>Co-digestion of corn stalk and vermicompost</td>
<td>35 ±1</td>
<td>0.410 m³ per kg-added TS</td>
<td>0.259 m³ per kg-added TS</td>
<td>b</td>
</tr>
<tr>
<td></td>
<td>Mono-digestion of corn stalk</td>
<td>35 ±1</td>
<td>0.335</td>
<td>0.217</td>
<td>b</td>
</tr>
<tr>
<td>Inclined-plug-flow</td>
<td>Fruit and vegetable wastes</td>
<td>35 to 38</td>
<td>1.07</td>
<td>0.68</td>
<td>c</td>
</tr>
<tr>
<td>Large-scale digester</td>
<td>Dairy manure</td>
<td>Mesophilic</td>
<td>1.493 m³ per kg-removed VS</td>
<td>0.835 m³ per kg-removed VS</td>
<td>d</td>
</tr>
<tr>
<td>DRANCO-FARM full scale</td>
<td>Corn (whole plant)</td>
<td>Thermophilic</td>
<td>0.792 m³ per kg-added VS</td>
<td></td>
<td>e</td>
</tr>
</tbody>
</table>

References:

a) Lansing et al., (2010)
b) Chen et al., (2010)
c) Sharma et al., (2000)
e) De Baer, (2010)
This index was increased to 62% by the end of day 116 corresponding to the co-digestion practice. The average OLR during the first phase and second stage of loading was equal to 2.4 and 1.57 g-VS/(dm³.d) respectively. A comparative statement of the current study and some referenced experiences with farm-based biodegradable feedstock in plug-flow digesters has been summarized in Table 3 as follows. The biogas and methane yield of pig feces were acceptable comparing to the similar experiences.

CONCLUSION
The passively-heated hybrid plug-flow reactor in this study demonstrated a successful start-up and a simple and low-cost technology for handling the biodegradable solids on the farms. Nevertheless, the heating system requires modifications to avoid thermal stratification inside the digester. The performance of high-TS stabilized digestate from swine farm was found suitable for inoculation of fresh feedstock and stabilization of biogas production and methane content within a pretty short time. Combined digestion of pig feces with pre-treated cotton stalk demonstrated higher biogas yield and slightly faster reaction rate and verified its efficacy in accelerating the digestion process and offers an alternative for recycling this type of agro-residues. External mixing of fresh feedstock with partially digested contents of digester before loading to the inlet had a significant influence on the process as it increased the contact between microorganisms and fresh biodegradable substances and enhanced the mass transfer.

ACKNOWLEDGEMENT
This research was partly supported by the fund of National Key Technology R&D Program No.
2008BADC4B10 as well as by Robert Bosch Foundation within the framework of China Applied Technologies for Environment-Biogas (CATE-B); a technical collaboration between Zhejiang University and Lübeck University of Applied Science, Germany. The authors should express their gratitude to Mr. Akbar Hormozjani, Mr. Majid Biglari, Mr. Mahmoud Adl and Ms. Laleh Abbasi for their contributions and supports during the fabrication and preparation of pilot reactor.

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