Process Parameters of Dry Co-anaerobic Digestion of Kitchen Waste and Cow Manure on Laboratory Scale

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Two dry anaerobic digestions of kitchen waste and cow manure were conducted in a lab-scale experiment for investigating the start-up performances under mesophilic and thermophilic conditions. According to cumulative biogas production (CBP), biogas production rate (BPR), pH value and VFAs variation, the start-up performances were separated into 3 stages- adaption stage, fluctuation stage and stabilization stage. The effective start-up of the system proceeded for approximately 140 days (55 days for adaption and fluctuation stage and 85 days to reach suitable conditions and stabilize the main operational parameters at hydraulic retention times (HRT) for 20 days). During these start-up performances, CBPs are 1070.5 and 1289.1 L respectively, and the average concentrations of CH$_4$ in biogas are 52.9% and 56.1% respectively. The final NH$_4$$^+$-N concentrations are 1897 and 1999 mg/L under mesophilic and thermophilic conditions, respectively. Thermophilic digestion was more efficient than mesophilic.

Key words: Kitchen waste; Cow manure; Anaerobic digestion; Start-up.
leachate and its digested residues are facile to be treated by composting process or being used as fertilizer\textsuperscript{11}. So far, few has been reported on the study of dry co-anaerobic digestion of kitchen wastes with cow manure and the explanation of solid wastes anaerobic digestion performance in start-up period. Hence, this study were aimed at investigating the start-up performance of dry anaerobic digestion with the emphasis on the co-digestion of kitchen wastes and cow manure under mesophilic or thermophilic conditions in a lab-scale batch experimental process with 4 completely mixed one phase anaerobic digesters.

**MATERIALS AND METHODS**

**Experimental apparatus**

The experiments were conducted in 4 laboratory-scale continuously stirred tank reactors (CSTR) with a total volume of 30 L and active liquid volume of 21 L (Fig. 1), mainly consisting of a vertical cylinder tank. Each group of two reactors was maintained at 37 and 55 °C separately. Shaking stick was used to agitate mixture at a speed of 20 rpm and for 10 min 5 times per day. pH index was monitored with a pH meter. Cumulative biogas and methane content were measured with a wet gas meter and GC.

Every day during the experiment, 500 ml of leachate was discharged from outlet and fresh kitchen waste and cow manure (1:1 v/v) were mixed and feed into reactors. At the beginning, 1 L of inocula, 1.25 kg of fresh kitchen waste/cow manure mixture (1:1 m/m) and pure water were added until 21 L. Adjusting the feeding quality according to the pH value and biogas production. A lower pH value would be increased with the addition of 1% of NaOH aqueous solution. The feeding quality increased when the system was stabilized.

**Feedstock and Inocula preparation**

The fresh samples of kitchen waste without garden waste were collected from Shenyang source separation pilot families. And fresh manure was collected from ShenBei development area farm. The samples were fully mixed by 1:1 (m/m) and blended in order to maintain the same VS content and then conserved at 4 °C. Active inocula at 37 °C were purchased from Shenyang North Wastewater Treatment Plant. The inocula were transported with containers of 25 L through delivery service. Although the temperature decreased into ambient temperature during delivery, the activity was maintained. In order to readapt the inocula, easily degradable organic matter still present in the inocula should be degraded for a period and dissolved methane be removed. Then the inocula was stored in an anaerobic headspace for three days in an incubator at 37 and 55 °C. Bio-chemical indexes of the kitchen waste, cow manure and inocula are shown in Table 1.

**Analytical methods**

The parameters for the characterization of kitchen waste, cow manure and inocula were pH, density, total solids (TS), volatile solids (VS) and total organic carbon (TOC). All analytical determinations were performed according to “Standard Methods” (APHA, 1995) after pretreatment, such as drying, grinding and dilution. The TOC analysis was analyzed by Liaoning Analysis and Measurement Center. TS and VS analysis were conducted by glass filter method. Samples for TS were dried in an oven at 105–110 °C, and the samples were calcined into an ash waste at 550±5 °C in a furnace for VS. The pH was determined by a digital pH meter.

Biogas production was measured by a wet gas meter. The contents of H\textsubscript{2}, CH\textsubscript{4} and CO\textsubscript{2} in biogas were determined by a SHIMADZU GC17A gas chromatograph equipped with a thermal conductivity detector (TCD) and a stainless column of 2 m packed with Porapak Q (50/80 mesh). Operational temperatures at the injection port, column oven and detector were 100, 70 and 150 °C, respectively. Nitrogen was used as the carrier gas at a flow rate of 30 mL/min.

**RESULTS AND DISCUSSION**

**Start-up of CSTR**

Start-up and stabilization of the reactor was carried out under mesophilic and thermophilic conditions (37 and 55°C) with a sequence that consists of 3 stages of which the organic loading rate was modified three times with different hydraulic retention times (HRT). It can be observed from Fig. 2 that the initial TS concentration was relatively low (5%) in order to check if the system evolved appropriately during the first 20 days, which belongs to adaption stage and HRT is of
200 days. This phenomenon could be ascribed to the adaptation period for the microorganisms in inoculum to the waste and the differences between the waste used in experiment and that used in the modified CSTR system. According to the pH and biogas production, from day 20 to 55, which was in fluctuation stage and HRT was of 40 days, by changing the feeding amount into 1.25 kg/every 2 days, TS concentration was increased from 1.77% (mesophilic) and 1.87% (thermophilic) to 4.15% and 4.5% respectively. Moreover, from day 55 to 150 in stabilization stage and the HRT was of 20 days, TS concentration was increased from 4.99% (mesophilic) and 4.58 % (thermophilic) to 15.87% and 15.23% respectively after the feed amount was adjusted into 1.25 kg/every 2 days.

The difference of TS concentrations represents the fraction of unconsumed organic materials and, as can be seen, it remains practically constant in region 2-5 and 5-15% for the HRT values of 40 and 20 days. This result indicates a stable system and that the microbial population had adapted to the experimental ambience. As a consequence, it could be stated that the value of parameter TS is a representative of the recalcitrant fraction of waste, indicating that the assumed TS and HRT values are appropriate for the system.

**Biogas production**

Analysis of data about generated biogas shows that stage 1 in Fig. 2 can be considered as an adaptation period. During this stage solubilization of the components proceeded through hydrolysis of the waste and this leads to colonization. As a result, the average methane yield during stage 1, expressed as cumulative biogas production (CBP) and biogas production rate (BPR), is practically negligible. Stage 2 can be considered as fluctuation stage, where BPR was enhanced with TS concentration. At the end of this stage, BPR reaches 30.7 and 44.21 L/d under mesophilic and thermophilic conditions respectively. Finally, in stages 3 the yield of biogas remains essentially constant, with BPR in the range of 82-106 L/d, entering into a stable state. In summary, the overall average BPR in this study was 36.9 (mesophilic) and 44.5 L/d (thermophilic).

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**Table 1. Bio-chemical indexes of kitchen waste, cow manure and inocula**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>Kitchen waste</th>
<th>Cow manure</th>
<th>Inocula</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>SU</td>
<td>6.12</td>
<td>6.50</td>
<td>7.44</td>
</tr>
<tr>
<td>Density</td>
<td>Kg/L</td>
<td>0.67</td>
<td>0.73</td>
<td>-</td>
</tr>
<tr>
<td>Total organic carbon</td>
<td>% (of TS)</td>
<td>43.96</td>
<td>41.88</td>
<td>-</td>
</tr>
<tr>
<td>Total solid</td>
<td>%</td>
<td>23.9</td>
<td>18.33</td>
<td>9.33</td>
</tr>
<tr>
<td>Volatile solid</td>
<td>% (of TS)</td>
<td>70.50</td>
<td>62.76</td>
<td>37.77</td>
</tr>
<tr>
<td>C/N ratio</td>
<td></td>
<td>18.32</td>
<td>12.39</td>
<td>-</td>
</tr>
</tbody>
</table>

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**Fig. 1. Schematic diagram of experimental set-up**

and CBP was 299.1 (mesophilic) and 377.4 L/d (thermophilic). The consumed VS amount corresponds to reported values.\textsuperscript{12-15}

**Evolution of pH, VFAs and NH\textsubscript{4}\textsuperscript{+}-N**

The evolution of pH, VFAs and ammonia nitrogen of dry anaerobic mesophilic and thermophilic digestions of kitchen waste and cow manure is illustrated in Fig. 3. The pH decreased from initial value 6.5 to 5.6 under mesophilic condition and to 5.3 under thermophilic condition.

![Fig. 2](image1.png)

**Evolution of cumulative biogas production (CBP), biogas production rate (BPR) and TS for Dry anaerobic mesophilic and thermophilic Digestions**

![Fig. 3](image2.png)

**Evolution of total VFAs\textsuperscript{a} and NH\textsubscript{4}\textsuperscript{+}-N and pH for of Dry anaerobic Mesophilic and Thermophilic Digestions**

![Fig. 4](image3.png)

**Evolution of gas composition of CH\textsubscript{4}, CO\textsubscript{2} and H\textsubscript{2} for anaerobic Mesophilic and Thermophilic Digestions**
in two weeks. Then 300 mL of hydroxide sodium solution (3 N) was added into all reactors at the end of day 15 and 25 to adjust the pH into 6.8 and 6.9 respectively. Biodegradable organic solid wastes were gradually hydrolysed and degraded, resulting in a final dry weight loss of 48% and 50% for mesophilic and thermophilic digestions, respectively.

The curves of temporal evolution of total VFAs exhibit three stages: initially, a decrease was observed from day 0 to day 15 (HRT=200), from 1800 to 1187 (mesophilic) and 985 mg/L (thermophilic). Then, from day 15 (HRT=200) to day 55 (HRT=40), total VFAs increased quickly from 1187 to 8865 mg/L (mesophilic), and from 985 to 8065 mg/L (thermophilic), in accordance with corresponding pH variation. At the end (HRT=20), total VFAs reached 17895 (mesophilic digestion) and 16895 mg/L (thermophilic digestion) from day 55 to 140 after a rapid increase and a gradual stabilization process.

The concentration of volatile fatty acids in the reactor was determined by the generation rate and consumption rate. During the first stage, hydrolysis and acidogenesis converted easily biodegradable fractions of organic waste into volatile fatty acids (such as propionate and acetate). However, the methanogens were still in adaptation period. During the next stage, with the feeding increasing, the generation rate of total VFAs was higher than consumption rate, even though hydrolysis and acidogenesis were still proceeding. In the final stage, the balance between hydrolysis/acidogenesis and methanogenesis was formed. The volatile fatty acids produced by easily biodegradable organic mass were immediately digested to generate methane, but refractory organics would be accumulated in reactors, which was the reason why the concentration of total VFAs increasing with the addition of feed.

The hydrolysis of amino acids and proteins caused the accumulation of ammonia from 388 mg/L to 1897 (mesophilic digestion) and 1999 mg/L (thermophilic digestion). Afterwards, an increase of NH$_4^+$-N concentration was observed since the generation rate was higher than consumption rate, although NH$_4^+$-N was assimilated as nitrogen source for the growth of methanogens. It maintained basically constant until HRT=20, and then the concentrations of NH$_4^+$-N started to grow gradually due to the growth of methanogens possibly.

**Biogas composition**

As far as the daily generation of biogas concerned, in the first 5 days of stage 1 there is a significant level of production due to hydrolysis of the main components within the reactor. The main gases produced in this period are characteristics of the hydrolytic process and the concentration of CO$_2$ was 69.6% and 67.9% under mesophilic and thermophilic condition respectively. This stage involves the breakdown of complex molecules that are readily biodegraded (particularly carbohydrates) into simpler molecules. For this reason, stage 1 is considered as an adaptation phase in the above mentioned discussion.

In stage 2, yield of biogas, especially methane, was greatly increased attributed to the degradation of organic materials accumulated in the previous stage, and the average CBP and BPR were higher than previous stages. In the final days of stage 2, corresponding to a HRT of 40 days, the percentage of CH$_4$ reaches at around 48.6% and 55.3% under mesophilic and thermophilic conditions, respectively. And in stage 3 the percentage of CH$_4$ reaches at around 63.3% and 68.8%, respectively. It proves to be a typical system of which the CH$_4$ production corresponds to the activity of CO$_2$/H$_2$-utilizing microorganisms. The average concentration of CH$_4$ in biogas was 52.9% and 56.1% under mesophilic and thermophilic conditions, respectively, in other reported ranges [12-17].

**CONCLUSION**

A start-up and stabilization process in a CSTR for mesophilic and thermophilic dry anaerobic digestion of kitchen waste and cow manure has been successfully operated. Effective start-up of the system lasts for approximately 140 days (55 days of adaption and fluctuation stage and the other 85 days to reach suitable conditions to stabilize the main operational parameters at HRT of 20 days). These results represent a significant advance for the start-up of similar reactors compared with other reported start-ups of at least 250 days.

According to CBP, BPR, pH value and VFAs variation, the start-up performances of dry
co-anaerobic mesophilic and thermophilic digestions of kitchen waste and cow manure consists of 3 stages- adaption stage, fluctuation stage and stabilization stage. Among these start-up performances, CBP was 1070.5 and 1289.1 L under mesophilic and thermophilic conditions, respectively, and the average content of CH$_4$ in biogas was 52.9% and 56.1% respectively. Final NH$_4^+$-N concentration was 1897 and 1999 mg/L respectively. In summary, thermophilic digestion is more efficient than mesophilic digestion for the degradation of kitchen waste and cow manure.

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