Optimization of Anaerobic Digestion of Sewage Sludge Using Thermophilic Anaerobic Pre-Treatment

Lu, Jingquan

Publication date: 2007

Document Version
Publisher's PDF, also known as Version of record

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Citation (APA):

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Comparison of the two-phase anaerobic digestion (73°C/55°C) with the single-phase anaerobic digestion (55°C) in treating sewage sludge

Jingquan Lu and Birgitte Kiær Ahring*

BioScience and Technology, Building 227, Biocentrum, Technical University of Denmark, Kgs. Lyngby-2800, Denmark

*Corresponding author.
Phone: +45 4525 6183
Fax: +45 4588 3276
Email: bka@biocentrum.dtu.dk
ABSTRACT

The two-phase anaerobic digestion (73°C/55°C) has advantages over the single-phase anaerobic digestion (55°C) in treating sewage sludge. Results of this study show that the two-phase process could totally eliminate pathogens and achieve satisfactory hygienic effect. If RT was set for 15 days, more VS can be degraded and more biogas can be produced by the two-phase process than the single-phase process. When RT of the two-phase process was set for 9 days, of which 2 days was used for the pre-treatment reactor and 7 days was used for the methane reactor, the VS reduction rate was as high as 60.61%, which was still comparable to that of the single-phase process when RT was set for 15 days. When sodium nitrate and glucose were used to disturb the single-phase process and the two-phase process during the steady stage, it was shown that the two-phase process was able to buffer the perturbation cause by either toxic substance or overload of easily degradable substrate, while the single-phase was totally broken down by the perturbation.

Key words—comparison, two-phase, single-phase, anaerobic digestion, sewage sludge

INTRODUCTION

Anaerobic digestion (AD) is a unique biological process in treating sewage sludge. Through this process, the major portion of organic material in the sludge is degraded, and at the same time valuable biogas is produced. AD is usually conducted under mesophilic or thermophilic conditions at temperatures of 30-40°C or 50-60°C, respectively (Buhr and Andrews, 1977; Ahring, 1995). Most of the anaerobic
digesters in Europe were started and operated in mesophilic manner (De Baere, 2000). However, recognizing the advantage of the thermophilic process that derives from higher metabolic activities of the thermophiles makes the shift to the thermophilic operational temperature attractive. The latter allows the shortening of the retention time (RT), treating larger volume of sludge in existing capacities or building of new reactors with smaller volume (Hamer, et al., 1985; Buhr and Andrews, 1977).

Conventionally, AD is conducted in a single-phase reactor that must be operated at conditions conducive to growth of all the microorganisms involved in the whole process if the waste organic material should be stabilized. However, the physiology, nutrient needs, growth kinetics and sensitivity to environmental conditions of hydrolytic-fermentative bacteria and methanogens are different, so it is not possible to select a single set of reactor operating conditions that can maximize the growth of both groups of the microorganisms (Demirel & Yenigün, 2002; Solera, et al., 2002). Conditions that are favorable to the growth of the hydrolytic-fermentative bacteria such as short hydraulic retention time, acidic pH and increased temperature, are inhibitory to the methanogens. Consequently, single-phase reactors are usually operated at relatively long HRTs (typically 15 days for thermophilic AD), neutral or slightly basic pH and temperatures just below the optimal temperature, 60°C, for aceticlastic methanogens, normally at 55°C. But single-phase AD process is still subjected to instability due to change of temperatures and inadvertent organic, hydraulic or toxic overloads caused by short-term variations in waste flows or characteristics (Henry, et al., 1987). Studies carried out on the stability of biogas plants in Denmark also showed that cases with high and fluctuation of VFA level
could usually be linked to specific events such as temperature instability or abrupt changes in substrate composition. (Angelidaki, et al., 2005).

To optimize AD process, two-phase system, in which the whole AD process is artificially separated into acid-phase and methane-phase, has been studied since 1970s (Pohland & Ghosh, 1971). The most obvious advantage of the two-phase process is the possible selection and enrichment for hydrolytic-fermentative bacteria and methanogens in each reactor by independent control of the reactor operating conditions. Thus, the acid phase can be optimized for hydrolytic-fermentative bacteria and the methane phase can be optimized for methanogens. Consequently, higher microbial population levels (Zhang & Noike, 1990) and increased activities (Skiadas, et al., 2005) are achieved. Because the organic material in sewage sludge exists in the form of particulates and thus makes hydrolysis of the particulates the obstacle of the proceeding of the whole AD process, many efforts have also been exerted to pre-treat the sludge for enhancing the hydrolysis step (Müller, 2001; Müller, et al., 2004). In our previous study (Lu & Ahring, 2005), it was confirmed that thermophilic anaerobic pre-treatment at 73 °C for a RT of 2 days could enhance not only hydrolysis but also acidogenesis; and therefore, this pre-treatment can function as the acid-phase of the two-phase AD process. The obvious advantage of the two-phase process is the improved pasteurization. It has been demonstrated that this pre-treatment method could warrant the pre-treated sludge a highly hygienic property so that the most heat resistant microorganism indicator, Faecal Streptococci (FS), could be totally eliminated.
Nowadays, reuse of stabilized sludge to recycle plant nutrients and extracting energy from waste to substitute the depleting fossil fuels are encouraged by many sludge management directives (e.g., EU, 2000). It will make AD process more attractive if high efficient pasteurization, organic material degradation and bioenergy production can be incorporated into one unit. The purpose of this study was to assess the improvements of the two-phase system (73°C/55°C) over the single-phase system (55°C) in the aspects of pathogen reduction effect, organic material degradation rate, methane yield, and process stability.

METHODS AND MATERIAL

Sewage sludge collection and storage

In order to keep all the sludge to be used in the entire experimental period to be identical and thus eliminate or reduce the variation of the experimental results caused by the change in composition of influent, enough of raw sludge (mixture of primary sludge and waste activated sludge at a ratio of 71% to 29% based on the VS content) was taken in one collection from Lundtofte WWTP, Lyngby, Denmark. The sludge was immediately dispensed into plastic bags with 1 l in each and then stored at –20°C. Once a day, a certain amount of the frozen sludge was thawed at ambient temperature and fed into the influent bottle of the reactors.

Startup and steady stage operation of the reactors

The reactor in the single-phase system, R1, and the methane reactor of the two-phase system, R2-2, were run not only at the identical temperature, i.e., 55°C, but also with identical RT, i.e., 15 days. A pre-treatment reactor, R2-1, running at 73°C for a RT of 2 days, was put before R2-2 in the two-phase system. The active volume of R1 and
R2-1 and R2-2 was 3.0 l, 0.5 l and 3.0 l, respectively. Three peristaltic pumps were used to drive the sludge from the influent bottle into R1, R2-1 and from Reactor 2-1 into R2-2, respectively. The reactors were fed 4 times a day. When R1 and R2-2 were fed, the same amount of effluents was driven out of the reactors at the same time. Feeding of R2-1 was just after the feeding of R2-2. During feeding, the influent bottles were stirred and the reactors were mixed. A computer installed with Lab-view software was used for the automatic control of the stirrers of the influent bottles mixers of the reactors and the pumps.

The strategy described by Ahring (2003) was followed for the start-up of the reactors. The innocula used was taken from a laboratory-scale thermophilic anaerobic reactor treating primary sludge at 55 °C for a RT of 15 days. R1 and R2-2 were started up first. To keep the performance of these two reactors to be as identical as possible, the biomass in these two reactors was exchanged for several times. When both of these reactors had reached their steady stages, R2-1 was started up. When R2-1 reached the steady stage, its effluent was added with gradually increased portion into the influent of R2-2 before its effluent tubing was connected to R2-2 so as to avoid shock VFA load to R2-2.

Liquid sample was taken just before raw sludge was pumped into the reactors and the effluents of the reactors were pumped out of the reactors. Throughout the experimental period, FS number, pH, concentration of VFA, solids concentration, and biogas production and composition of the two systems were measured.
Ultimate methane potential of the organic residue in the two-phase effluent

200 ml of the effluent from R2-2 of the two-phase system was used when the reactor was running for a RT of 15 days, among which, 100 ml was first centrifuged and then the solid fraction was washed twice with sterile water by centrifugation and subsequent re-suspension to a final volume of 100 ml again. The other 100 ml digestate was treated by adding sodium hydroxide to a concentration of 40 meq and heated at 121°C for 30 minutes (Kim, et al., 2003). Finally, batch experiment was used with the inocula from R2-2 to test the CH₄ potential of the organic residue in the digestate. Potential of raw sludge was also tested at the same time.

Reduction of RT of R1 and R2-2

When enough data at the steady stage used to compare the single-phase and the two-phase process had been obtained, the RT of R2-1 was kept constant at 2 days while the RT of R2-2 was gradually reduced to 13, 11, 9, and 7 days. For comparison, the RT of R1 was set at 15, 13, 11 and 9 days, respectively, so that the two-phase system was comparable with the single-phase system. The above-mentioned monitoring parameters were also followed.

Perturbation tests

Two groups of simplified single-phase and two-phase reactor set-ups were used to make parallel perturbation tests. In each of the single-phase process, the reactor with a working volume of 1.35 l, was run at 55°C for a RT of 9 days, while in each of the two-phase system, the pre-treatment reactor with a working volume of 0.3 l, was run at 73°C for a RT of 2 days and the methane reactor with a working volume of 1.05 l, was run at 55°C for a RT of 7 days, so that both the working volume and RT in the
single and two-phase process were kept comparable as well. The inocula used for starting up these reactors were taken from R1, R2-1 and R2-2, respectively.

Group A was used to test the capability of the single-phase process and the two-phase process in buffering the sudden perturbation of toxic substance. During this test, sodium nitrate was added into the influent sludge up to a final concentration of 10mM. Group B was used to test the capability of the single-phase process and the two-phase process in buffering the shock overloading of easily degradable substrates. Glucose was put into the influent sludge to double the organic loading rate (OLR).

VFA and biogas was followed before and after the perturbations were executed.

**Analytical methods**

FS was used as the indicator microorganism to evaluate the pathogen reduction effects of the AD process, because pathogens or parasite eggs can be killed or inactivated long time before FS is eliminated (Bendixen, H. J., 1999). Although FS in the influent sludge was detected, the concentration of viable cells could change during the storage at −20°C for a period. For this reason, we put the pure culture, *Enterococcus faecalis, Strain 25v-2*, a subgroup of *Faecal Streptococci* (Dalgaard et al, 2003) provided by Danish Institute for Fisheries Research, into the influent sludge to make sure that high number of this indicator organism could present in the influent sludge during the experimental period.

For the numeration of FS number in 1 ml of sludge, the method prepared by Nordic Committee on Food Analysis (1992) was followed. 0.1 ml of the sample or its decimal dilutions was sowed on the *Enterococcus* selective agar, the composition of which
was described previously (See Paper I). The characteristic red colonies were counted after incubation at 44°C for 48 hours.

COD, TS, VS were determined according to standards methods (APHA, 1992). For the analysis of VFAs, sample was acidified by 17% orthophosphoric acid and then centrifuged at 10,000 rpm for 10 min. The liquid fraction was filtered and then analyzed by gas chromatograph (Hewlett Packard, Palo Alto, Calif., USA) as described previously (See Paper I).

Biogas flow was quantified by liquid displacement technique using paraffin oil as liquid in a U-tube with a working volume of 10 ml for R1 and R2-1. For R2-1, it was quantified by liquid displacement technique using 8% NaCl/HCl solution as liquid in a cylinder with a working volume of 100 ml to provide enough buffering capacity when effluent was pumped out. Concentration of CH₄ in the biogas was monitored according to the methods used by Sørensen et al. (1991).

RESULTS AND DISCUSSION

Comparison of two-phase and single-phase process at RT of 15 days

The RT of R2-1 was set at 2 days for the compromise of obtaining satisfied pathogen reduction effect, hydrolysis effect and acidogenesis effect (Paper I). As the RT for thermal AD is usually set at 15 days, the RT of R1 and R2-2 was set at 15 days during start-up period and in the beginning period of the experiment. Figure 1a, b and c show the performance of the reactors of R2-1, R2-2 and R1, respectively.
It can be seen from Figure 1 that all of the reactors run stably after undergoing a start-up period. R1 functioned well for acidogenesis because, in comparison with the influent, high VFA was generated in it. Before R2-1 was connected to R2-2, the performance of R2-2 and R1 was quite similar, while obvious improvements such as lower VFA concentration and reduced variation of the biogas production could be observed in R2-2 after it has been connected to R2-1.

The performance of the single-phase process and the two-phase process during a one-month steady stage period was summarized in Table 2. The VFA concentration in R2-2 was only 42% of that in R1, which were stabilized at 270 mg-COD/l and 645 mg-COD/l, respectively. This indicated that the activities of acetogens and methanogens were higher in R2-2 than those in R1, and thus VFA in R2-2 could be degraded more completely. These increased activities that had been confirmed in our previous study (Skiadas, et al., 2005) were caused by the fact that higher concentration of substrates such as butyrate, propionate and acetate were provided due to the acidification effect by R2-1, so relative more microbes had been enriched. The variation of CH$_4$ production of the two-phase process was only 71% of that in the single-phase process, indicating that the methane reactor in the two-phase process was more stable than that in the single-phase process. The improvement in reactor stability could be contributed to two facts. One is that the organic material in the influent provided by R2-1 was more homogeneous and more easily degradable than the raw sludge, and toxic substances, if it existed, might have been detoxified in R2-1. The other fact was that higher microbial population might have stronger capacity to cope with the impulse caused by the variations such substrate concentration and composition. No *Faecal Streptococci* was detected in the effluent of the two-phase system while the
concentration of *Faecal Streptococci* in the single-phase system was 350 CFU/ml. The success of pathogen reduction effect achieved by the two-phase system owed to the thermal effect of R2-1, because *Faecal Streptococci* was not detectable in its effluent before the subsequent treatment in R2-2.

**Determination of sludge biogas potential**

It had been expected that the two-phase process could, to a greater extent, increase the degradation rate of organic material. However, the degradation rate of the two-phase process was increased only by 4.48% in comparison with the single-phase process. However, it was observed that the CH4 production in R2-2 during the period of one circle of feeding was much faster in the beginning than in the end, as shown in Figure 2. This phenomenon was not so prominent for R1. It seemed that after pre-treatment two fractions of organic material were formed, and for the easily degradable fraction, it could be degraded within a short period, for the difficultly degradable fraction, the degree of degradation was low, indicating by the slower increase in biogas production as indicated in Figure 2. It only needed about 50% of the total feeding circle period for the two-phase process to obtain the same amount of CH4 from the single-phase process in the total circle period. For the two-phase process, the CH4 production in the latter half of the circle period accounted for about only 11.66% of the total CH4 produced.

In sewage sludge, organic material is composed of several fractions, one of which is non-biodegradable (Hamer, et al., 1985). After being treated by the two-phase process, around 64.97% of the total organic material was degraded, and the CH4 potential of the effluent from the two-phase process was rather low, unless being
treated by alkali-thermal method. Figure 3 shows the comparison of the biogas potential of the raw sewage sludge, the effluent sludge from the two-phase process before alkali-thermal treatment.

**Reduction of RT of R1 and R2-2**

The low biogas potential of the effluent from the two-phase process indicates that the content of easily degradable organic material in the effluent of the two-phase process was low, so it seems that significantly higher degradation level of the two-phase process over the single-phase process could not be obtained or difficult to be obtained, i.e., the advantages of the two-phase process was not manifested in this aspect. It seems that the increased microbial activities were not brought into full play, which was implied by the slower increase in CH$_4$ production in the latter half period of the feeding circle. So, effort in the following study was exerted to test the possibility of reducing the RT.

The RTs of 15 days, 13 days, 11 days, 9 days and 7 days were tested. As shown in Figure 4, with the decrease in RT, CH$_4$ production increased and VS reduction decreased, but the CH$_4$ production rate and the VS reduction rate of the two-phase process were always higher than those of the single-phase process. When RT was set for 9 days, the VS reduction rate of the two-phase process was 60.61%, which was still comparable to that of the single-phase process when running for the RT of 15 days. So, two-phase process can be regarded as a high rate process, which used 60% of the RT of the single-phase process and obtained comparable VS degradation rate. This means that when treating the same amount of sewage sludge in the wastewater treatment plant, if the two-phase process was adopted, the total reactor working
volume of the two-phase process can be reduced to 60% of that of the single-phase process to be used, and still comparable VS reduction rate can be achieved. When RT was further decreased to 7 days, the VS degradation rate of the two-phase process was decreased to 54.60%, which was lower than that of the single-phase process when it was run for the RT of 15 days.

**Perturbation tests**

As discussed previously, the two-phase process was more stable than the single-phase process. Ammonia, nitrite, nitrate, sulfite and etc. are toxic to methanogens and may cause instability of the AD process (Balderston Payne, 1976; Angelidaki, et al., 2005). In this study, sodium nitrate was added in the influent sludge when the single-phase process and the two-phase process were run for RT of 9 days. It can be seen from Figure 5a that the dosage of NaNO₃ caused severe instability to R1. This was indicated by the sudden drop of CH₄ production and increase of VFA accumulation. This instability lasted about 20 days. By the same dosage, stability of the two-phase process also suffered the negative effect. Reduced production of VFA in R2-1, and slight VFA accumulation and CH₄ drop in R2-2 were noticed. However, this slight instability in the two-phase lasted only for about 5 days. In the single-phase process, methanogens were directly exposed to the toxic substance added, so they were immediately inhibited. The consequence of the inhibition might cause the accumulation of VFA and drop of pH, so the condition for methanogens might become even worse. This is why the instability lasted for such a long period. In the two-phase process, however, the toxic substance first contacted with the biomass in the pre-treatment reactor. The acidogens were known to be robust and in the pre-treatment reactor de-nitrification reaction might be carried out as shown in the
following equation: \[ C_6H_{12}O_6 + 4NO_3^- \rightarrow 6CO_2 + 5H_2O + 2N_2 + \text{energy} \]. So the toxicity of nitrate was eased off before it would have affected the methanogens in the methane reactor. Due to the high methanogenic activity in the methane reactor, the accumulated VFA was quickly turned over. So, no further damage to the process stability was caused.

In the AD system, a precious balance between the hydrolyzing-fermenting bacteria and the methanogens are needed (Ahring, 2003). A sudden inadvertent overloading of easily degradable substrate may cause a sudden increase of VFA and results in the break down of the stable stage of the AD process (Holfman-Bang, 2003). In this study, when glucose was added to the single-phase process and the two-phase process, different results were found, as shown in Figure 5b. For the single-phase process, CH\(_4\) production increased in the beginning and then decreased. This can be explained by the fact that accumulation of VFA led to the drop of pH and then caused inhibition on the methanogens. On the contrary, the two-phase process could turn over almost all of the added glucose producing more CH\(_4\). No severe disturbance was observed. This again illustrated the enhanced methanogenic activity due to phase separation.

**CONCLUSIONS**

From this study, it can be concluded that two-phase process has a lot of advantages over the conventional single-phase process.

The effluent from two-phase process is hygienically satisfied and could be used as fertilizer in the farmland without any fear of spreading diseases, while the effluent from the single-phase process has to undergo special sanitation treatment before
application on farmland or has to go into other final disposal streams such as landfill, incineration, and etc that might be costly.

The two-phase process is a high rate process. If RT is set for the same period, more VS can be degraded and more biogas can be produced by the two-phase process than the single-phase process. To achieve the same biogas production rate and VS reduction rate, the two-phase process needed shorter RT than the single-phase system. When RT of the two-phase process was set for 9 days, among which 2 days was used for the pre-treatment reactor and 7 days for the methane reactor, the VS reduction rate was as high as 60.61%, which was still comparable to that of the single-phase process when RT was set for 15 days.

Process stability can be greatly enhanced by the phase separation. Perturbation test using pulse dosage of sodium nitrate and glucose demonstrated that two-phase process could buffer the shock load of toxic substance and easily degradable substrate, while the single-phase was broken down by the fluctuations coming from the influent.

Even though the two-phase may cost more energy and capital in construction and operation of the control system, but considering the smaller volume of reactor needed, the value of the application of its effluent on farmland and the elimination of the cost on effluent disposal, the two-phase system is still attractive.
ACKNOWLEDGEMENT

The authors wish to thank the Danish Technical Research Council (STVF) for the financial support of this work under the Framework program 26-01-0119 “Optimisation of biogas processes”.

REFERENCE


### Table 1 Characteristics of the influent sludge

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>Mean (SD(^a), N(^b))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solids</td>
<td></td>
<td></td>
</tr>
<tr>
<td>TS</td>
<td>g/l</td>
<td>54.54 (0.26, 7)</td>
</tr>
<tr>
<td>VS</td>
<td>g/l</td>
<td>38.40 (1.35, 5)</td>
</tr>
<tr>
<td>Dissolved COD</td>
<td>g/l</td>
<td>3.35 (0.21, 9)</td>
</tr>
<tr>
<td>VFA</td>
<td>g/l</td>
<td>1.17 (0.05, 3)</td>
</tr>
<tr>
<td>pH</td>
<td></td>
<td>6.69 (0.17, 30)</td>
</tr>
<tr>
<td>FS</td>
<td>CFU ×10(^4) / ml</td>
<td>8.3 (0.8, 26)</td>
</tr>
</tbody>
</table>

\(^a\) Standard deviation; \(^b\) Number of measurements.

### Table 2 Comparison of the performance of the single- and the two-phase AD systems

<table>
<thead>
<tr>
<th>System</th>
<th>Single-phase</th>
<th>Two-phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reactor</td>
<td>R1</td>
<td>R2-1</td>
</tr>
<tr>
<td>pH</td>
<td>7.66(^a) (0.07(^b), 15(^c))</td>
<td>5.68 (0.06, 15)</td>
</tr>
<tr>
<td>VFA (mg-COD/l)</td>
<td>645 (126, 15)</td>
<td>7972 (174, 15)</td>
</tr>
<tr>
<td>FS (CFU / ml)</td>
<td>350 (28, 5)</td>
<td>0</td>
</tr>
<tr>
<td>CH(_4) in biogas (%)</td>
<td>61.6 (2.1, 10)</td>
<td>3.3 (0.6, 15)</td>
</tr>
<tr>
<td>Methane production (ml/d)</td>
<td>2333 (205, 15)</td>
<td>68 (15, 15)</td>
</tr>
<tr>
<td>VS removal rate (%)</td>
<td>60.49 (2.4, 5)</td>
<td>Not measured</td>
</tr>
</tbody>
</table>

\(^a\) Mean value; \(^b\) Standard deviation; \(^c\) Number of measurements.
FIGURE LEGENDS

Figure 1. Performance of R2-1, R2-2 and R1 when RT of the single-phase process and the two-phase process was set at 15 days (Influent pH and t-VFA are indicated in a. CH₄ produced in R2-1 is not shown due to low production).

Figure 2. Accumulative biogas production in the single and two-phase process during one circle of feeding. The biogas production of the two-phase is about 9% higher than that of the single-phase system, but it needs about only 53% of the RT of the single-phase to reach the same biogas production.

Figure 3. Biogas potential from the raw sludge, the effluent of the two-phase system before and after NaOH-thermal treatment.

Figure 4. Methane production and VS reduction of the single-phase and the two-phase process running for different RTs.

Figure 5. Performance comparison of the single-phase process and the two-phase process after perturbation by toxic substance (a) and easily degradable substrate (b).
Figure 1

Figure 2
Figure 3

Figure 4
Figure 5
April 10, 2006

Editorial Receiving Office,

Re: Effects of temperature and retention time on thermophilic anaerobic pre-treatment of sewage sludge

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Attached is our paper for possible publication in Water Science and Technology. Thank you for your attention, and I am looking forward to receiving your positive reply.

Sincerely yours,

Jingquan Lu

Bioprocess Science and Technology
BioCentrum-DTU, Building 227, st. Technical University of Denmark DK-2800 Kgs. Lyngby Denmark
Tel.: +45 4525 6187
Fax: + 45 4588 3276
E-mail: jql@biocentrum.dtu.dk
www.biocentrum.dtu.dk