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

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Effects of temperature and hydraulic retention time on thermophilic anaerobic pretreatment of sewage sludge

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Abstract
In this study, we investigated by running continuous stirred tank reactor the effects of different thermophilic temperatures (55, 60, 65, 70, 75 and 80°C) for different hydraulic retention times (0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 days, respectively) on the pretreatment of sewage sludge. We found that hydrolysis was mainly determined by temperature instead of hydraulic retention time, i.e. the higher the temperature was, the more dissolved COD produced, while extension of hydraulic retention time did not significantly increase dissolved COD products. Acidogenesis had the highest products when temperature was at 70°C or 75°C and for a hydraulic retention time around 2 days, under the conditions of which higher acidogenic activity was maintained and methogenic activity was inhibited. We also determined the minimum guaranteed retention time for each temperature when faecal streptococci were totally inactivated.

Introduction
Anaerobic digestion (AD) is a matured and widely used technology to treat sewage sludge. By anaerobic digestion, organic compounds such as carbohydrates, proteins, lipids and etc, are converted to methane, carbon dioxide, water and etc. through the sequential four steps of hydrolysis, acidogenesis, acetogenesis and methanogenesis. At the same time, nutrients such as N and P are kept in the residue of the effluent. So, anaerobic digestion is a way of solving environmental problem with simultaneous production of energy in the form of biogas and nutrients enriched effluent that can be recycled back to the farmland.

Studies have shown that hydrolysis is the rate-limiting step during the whole AD process in treating sewage sludge, because organic matters in it are in the form of particulates (Tiehm, et al., 2001; Wang, et al., 1999; Li & Noike, 1992). One way to enhance hydrolysis is to add a pretreatment step before AD. Different pretreatment methods, such as thermal (heat), mechanical (mixing, ultra-sonic), chemical (acid or base, ozone) and biological methods (aerobic composting, methophilic or thermophilic anaerobic) have been studied at laboratory scale and some of which have already been implemented for industry production. It will be of advantage if the acidogenesis process can be enhanced by the pretreatment so that more substrates available for acetogens and methogens in the subsequent second step reactor can be produced. Also, it will be very interesting if pasteurization can be incorporated into this pretreatment step, so that the hygienic requirement on AD effluent for reuse on farmland can be satisfied. Based on these considerations, it will be very interesting to focus pretreatment on thermophilic anaerobic method. The reasons for this are (1) it is reported that high temperature above 60°C can improve hydrolysis (Li & Noike, 1992), (2) thermophilic bacteria that can hydrolyze and/or degrade organic compounds are widely exist and they have optimal temperature in the range from 60 to 75°C (Kristjansson, 1992), (3) when AD is run at thermophilic temperatures, pasteurization can be improved (Nielsen & Petersen, 2000).

Both temperature and hydraulic retention time (HRT) are important parameter for AD. The main advantages of increased temperature are generally higher reaction rates, higher solubility of most chemicals, and increased fluidity and diffusion rates (Kristjansson, 1992). For continuous stirred tank reactor (CSTR), HRT is not only a good operational parameter that is easy to control, but also a macro-conceptual time for the organic material to stay in the reactor. In bio-reaction engineering studies, the reverse of HRT is defined as dilution rate, for which if it is bigger than the growth rate of microbial cells in the reactor, the microbe will be washed out, and otherwise the microbe will be accumulated in the reactor. Either of these situations may result in the breakdown of the biological process happening in the reactor. Literatures that can be referred to regarding to the study on anaerobic
pretreatment for hydrolysis and acidogenesis of sewage sludge are normally limited to ambient temperature (Banister & Pretorius, 1998), methanophilic temperature or thermophilic temperature lower than 55°C (Ghosh et al., 1995; Henry et al., 1997; Ferreiro & Soto, 2003). Under these temperature conditions, hydrolytic rate and acidogenic rate are normally low.

The purpose of this study is to investigate the effects of temperature and hydraulic retention time on the pretreatment of sewage sludge. We confined our study in the range of from 55°C to 80°C for temperature and in the range of 0.5 day to 3.0 days for hydraulic retention time. In this study, achievements of pretreatment are evaluated by specific productivity of hydrolysis, specific productivity of acidogenesis and pathogen reduction effect.

**Materials and methods**

**Raw sludge**

In order to keep all sludge used in the entire experimental period to be identical, the raw sludge (mixture of primary sludge and waste activated sludge) used in this study was dispensed into plastic bags with 1 litter in each and then stored at -20°C immediately after it was sampled from Lundtofte WWTP, Lyngby, Denmark. Once a day, a certain amount of the frozen sludge was melted at ambient temperature and fed into the influent bottle of the reactors. Table 1 shows the characteristics of the raw sludge before frozen.

Table 1  Characteristics of the raw sludge used in this study

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>Mean (SD, N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS</td>
<td>g/l</td>
<td>45.15 (0.26, 7)</td>
</tr>
<tr>
<td>TSS</td>
<td>g/l</td>
<td>41.84 (1.57, 5)</td>
</tr>
<tr>
<td>VS</td>
<td>g/l</td>
<td>33.60 (0.27, 7)</td>
</tr>
<tr>
<td>VSS</td>
<td>g/l</td>
<td>31.30 (1.96, 5)</td>
</tr>
<tr>
<td>Total COD</td>
<td>g/l</td>
<td>46.20 (1.31, 5)</td>
</tr>
<tr>
<td>Dissolved COD</td>
<td>g/l</td>
<td>3.35 (0.21, 9)</td>
</tr>
<tr>
<td>Acetatic</td>
<td>g-COD/l</td>
<td>1.10 (0.01, 6)</td>
</tr>
<tr>
<td>Propionate</td>
<td>g-COD/l</td>
<td>0.56 (0.02, 6)</td>
</tr>
<tr>
<td>Butyrate</td>
<td>g-COD/l</td>
<td>0.26 (0.01, 6)</td>
</tr>
<tr>
<td>Iso-butyrate</td>
<td>g-COD/l</td>
<td>0.10 (0.02, 6)</td>
</tr>
<tr>
<td>Lipids</td>
<td>g/l</td>
<td>6.22 (0.18, 3)</td>
</tr>
<tr>
<td>Crude protein</td>
<td>g/l</td>
<td>10.60 (0.12)</td>
</tr>
<tr>
<td>pH</td>
<td>----</td>
<td>6.92 (0.17, 30)</td>
</tr>
<tr>
<td>FS</td>
<td>CFU ×10⁸/ml</td>
<td>3.5 (0.8, 26)</td>
</tr>
</tbody>
</table>

a. Standard deviation; b. Number of measurements.

**Experimental set-up**

Six 1000-ml CSTRs with a working volume of 667 ml for each, marked as R₁, R₂, R₃, R₄, R₅, R₆, were run in two series: in Series I, R₂ and R₃ were run at 55°C, 60°C and 65°C respectively, and in Series II, R₄, R₅ and R₆ were run at 70°C, 75°C and 80°C respectively. The process temperature for each reactor was guaranteed by applying recirculation of hot water from a temperature-controlled water bath through the tubing coiled around side wall of the reactor. All of the reactors were inoculated with effluent from a one-step thermophilic anaerobic CSTR, which had been fed by mixed raw sludge and running in the steady stage at 55°C and a HRT of 15 days in our lab for more one year. According to the HRT at which the reactors were operated, a certain amount of pre-treated sludge was pumped from the reactor into the effluent bottle and then the same amount of raw sludge was pumped into the reactor from the influent bottle. For the sake of avoiding a shock loading and/or keeping a certain MGRT, the frequency of withdraw/feed was changed at each HRT. The reactors were mixed intermittently for 30 second in each 30 minutes by a magnetic stirrer set at 300rpm. Both the stirrer for reactor and the
stirrer for the influent bottle were turned on during the period when effluent withdrawing and influent feeding were proceeding so as to keep the homogeneity of effluent and influent. A computer installed with Lab-view Software was used for the automatic control of each series of the reactors. Figure 1 shows one of the six experimental reactor set-ups.

**Figure 1. One of the six reactor set-ups**

Reactor operation, monitoring and sampling
For each series of the reactor set-ups, HRT started with 3.0 days and then continued in the stepwise with 2.5, 2.0, 1.5, 1.0, 0.5 days. For each HRT, the reactors were run at least for a period of 5 HRTs until they had reached steady stage, characterized by stable gas production and composition, stable pH value, stable d-COD concentration, stable concentration and composition of VFAs in the effluent. When steady stage was observed, three successive sets of samples of gas and the liquid of each of the reactors were taken and analyzed. Sampling was carried out in reasonably regular intervals and was just at the same time when effluent was pumping out of the reactor. The average analyzing results and standard deviations of each of the three sets of samples were used for evaluating the effects of temperature and HRT on pre-treatment.

Analytical methods
COD, TS, VS, TSS, VSS, lipids and k-N were determined according to standards methods (APHA, 1992). For determination of d-COD, the sample was centrifuged at 13000 rmp for 20 min and then filtered with a 0.45 μm. The filtrate was used for analyzing.

For the analysis of VFA, sample was acidified by 17% orthophoshyric acid and then centrifuged at 10000 for 10 min. The liquid fraction was filtered and then analyzed by a HP model 5890-II gas chromatograph equipped with a flame ionization detector and a silica capillary column. Nitrogen was used as carrier gas with a flow of 18 ml/min. The injection temperature was 175°C, and the temperature of the detector was 200°C. The initial temperature of the oven as 115°C and was increased by 5°C per min up to 130°C. With these parameters, the retention times were about 1.7, 2.3, 2.5 and 3.1mins for acetate, propionate, iso-butyrate and butyrate respectively. The data obtained from the detector were processed by software, HP-Chemstation.

Pathogen reducing effect (PRE) can be expressed by using faecal streptococci (FS) as indicator organism. This is based on the fact that most pathogenic bacteria and viruses are inactivated and parasite eggs lose their viability long time before FS are eliminated (Bendixen 1999). For the numeration of FS number in 1 ml of sludge, the method prepared by Nordic Committee on Food Analysis (1992) was followed. By changing feeding strategy, minimum guaranteed retention time (MGRT) of sludge in the reactor was obtained. Samples for analysis were taken for each MGRT.

Results and discussion
After 6-month experiment, all of the reactor performance for each temperature and each hydraulic
retention time were tested. We found that at 55, 60, 65°C, it was difficult to eliminate methanogenic activities which were indicated by the high methane production, as shown in Figure 2. At 55°C and HRT of 3 days, the biogas production can be as high as 1847mg d-COD/g-VSS. When temperature was at 75 and 80°C, H2 was detectable with a concentration of 10.3% and 14.4% respectively.

From this observation, we know that for a two-phase AD, phase separation can not distinctively isolate hydrolysis from the four sequential AD steps, especially at low thermophilic temperatures.

Net specific hydrolysis production in the pre-treated sludge is shown in Figure 3. It can be seen that hydrolysis is rather dependent on temperature than on HRT. The higher the temperature is, the more specific d-COD is produced. However, for the HRT of 0.5 days, around 80% of d-COD was produced; increasing HRT resulted in slower increase of specific d-COD production. Hydrolysis might catalyzed by both heat and hydrolytic enzymes, or the enzymes had higher activities at higher temperature. But it must be a fast process with a saturation concentration inhibiting hydrolysis going ahead. This can be seen by looking at Figure 2, if we take the COD removed by methanogens.
VFA production is not like d-COD production. It is also a fast process indicating by high production at low retention. However, for the same HRT, it was not always increased by temperature. When temperature was set at 70°C, VFA had the highest specific production rate for all of the HRTs, as it is shown in Figure 4.

![Specific VFA production vs. HRT for different temperatures](image)

Figure 4. Specific VFA production vs. HRT for different temperatures

For the pathogen reduction effect, it was both the temperature and the minimum guarantee retention time that control the effect. Table 2 lists the minimum guaranteed retention time need for inactivating FS until undetectable.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>55</th>
<th>60</th>
<th>65</th>
<th>70</th>
<th>75</th>
<th>80</th>
</tr>
</thead>
<tbody>
<tr>
<td>MGRT (hour)</td>
<td>18</td>
<td>18</td>
<td>12</td>
<td>2</td>
<td>2</td>
<td>1</td>
</tr>
</tbody>
</table>

**Conclusions**

This study demonstrated that thermophilic anaerobic pre-treatment was a good method for pre-treating sewage sludge. Like the other pre-treatment method, thermophilic anaerobic pre-treatment can to a great extent enhance the hydrolysis of organic particulates in the sludge. Besides this, a large fraction of the hydrolysis products could be converted to VFAs that are the precursors of methane. However, the most valuable virtue of this pre-treatment is the pathogen reduction effect, which can make it possible for the AD effluent to be recycled to the farmland as fertilizer and soil conditioner.

**Nomenclature**

COD—Total chemical oxygen demand, g/l
d-COD—dissolved chemical oxygen demand, g/l
t-COD—total chemical oxygen demand, g/l
CSTR—Continuous stirred tank reactor
FS—Faecal streptococci, $CFU \times 10^4 / ml$
MGRT—Minimum guaranteed retention time, hour
PRE—Pathogen reduction effect
VFA—Volatile fatty acids, g-COD/l
VS—Volatile solids, g/l
VSS—Volatile suspended solids, g/l
TS—Total solids, g/l
TSS—Total suspended solids, g/l
WWTP—Waste water treatment plant

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References


