Pre-treatment of wastewater sludge before anaerobic digestion - hygienisation, ultrasonic treatment and enzyme dosing

Davidsson, Åsa; la Cour Jansen, Jes

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Abstract

Pre-treatment of sludge before anaerobic digestion can increase methane production and degradation of organic matter. There are various pre-treatment methods for this purpose. Anaerobic digestion tests were performed for comparison of three pre-treatment methods (hygienisation, ultrasonic treatment and enzyme dosing) used separately or in combination on biosludge and mixed sludge. COD solubilisation and methane potentials from the differently pretreated sludges were used for comparison. Pilot-scale digestion was further used for evaluation of hygienised/untreated mixed sludge in semi-continuous operation.

The results show that pre-treatment of biosludge leads to increased methane potential, especially hygienisation and ultrasonic treatment. Combining enzyme dosing with hygienisation or ultrasonication implies additional increase in methane potential while hygienisation combined with ultrasonication does not.

Increased COD solubilisation seen after pre-treatment does not always bring about an increase in methane potential. On the other hand, pre-treatment methods like ultrasonication can lead to higher methane production although the COD solubilisation is low.

Key words – wastewater, sludge, anaerobic digestion, pre-treatment, hygienisation, ultrasonic, enzyme, hydrolysis
Introduction

Anaerobic digestion of waste sludge at municipal wastewater treatment plants is widely used. More than 2/3 of the generated municipal sewage sludges in Sweden are treated by anaerobic digestion. Anaerobic digestion reduces the sludge amount by degrading organic material while methane gas is generated. The methane gas can be used for production of heat, electricity or vehicle fuel and thereby replace fossil fuels. Organic matter in the sludge is normally degraded by up to 50% in anaerobic digestion leaving a significant part of the organics to final disposal. Sludge disposal in Sweden poses a problem at the moment. Landfilling of organic waste is forbidden, recycling in form of fertilising agricultural land with sludge is highly questioned and available incineration capacity cannot take care of all the waste sludge. Therefore an increased degradation of the sludge organics is desired. Anaerobic digestion is often limited by the first step, the hydrolysis, i.e. conversion of complex organic matter (particulate and soluble polymers) into soluble products (Shimizu et al., 1993). Hydrolysis can be promoted by pre-treatment of the sludge in form of biological, physical or chemical methods. Various methods have been used on primary sludge and/or waste activated sludge to reduce particle size and increase solubilisation, e.g in Park et al. (2005), Wang et al. (2005), Kim et al. (2003), Chiu et al. (1997) and Del Borghi et al. (1999).

This paper presents results from anaerobic digestion tests where three promising pre-treatment methods (hygienisation, ultrasonication and enzyme dosing) have been used separately or in combination on biosludge and mixed primary and biosludge.

A separate hygienisation step in connection to anaerobic digestion for controlled kill-off of patogens in sludge (which is applied at Swedish biogas plants treating other waste than sludge) is a demand from the food industry for increased acceptance of sludge as fertiliser on farmland. Thermal treatment (70°C for 1 h) is often suggested to kill off pathogens and if applied before digestion it could improve hydrolysis and thereby methane production.

Ultrasonic treatment breaks up flocs and/or bacterial cells in the sludge and has been shown to improve anaerobic digestion in waste activated sludge e.g in Tiehm et al. (2001) and Kim et al. (2003). A full-scale installation is found at the wastewater treatment plant in Kävlinge, Sweden.

Enzyme dosing for enhanced hydrolysis has been tested in previous work on biological surplus sludge and on mixed primary and biological surplus sludge (Wawrzynzcyk et al., 2003 and Jansen et al. 2004a). Increased methane production was seen in both cases.

COD solubilisation and methane potentials from the differently pretreated sludges are used for comparison of the pre-treatment methods and combinations of methods. Pilot-scale digestion was further used for evaluation of hygienised/untreated mixed sludge in semi-continuous operation.

Materials and Methods

Sludges and sludge pre-treatment

Sludges from two different municipal wastewater treatment plants were collected. Biological surplus sludge before and after ultrasonic treatment (Sonix™ 12 kW, 0.05 kWh/kg TS, Max. 50 kHz) was collected at Kävlinge wastewater treatment plant, Sweden. Mixed primary and biological surplus sludge (50:50) was collected at Sjölunda wastewater treatment plant, Malmö, Sweden. Part of these sludge types were further hygienised by heating them to 70°C and keeping the temperature for 1 hour. Main properties for the sludge types are found in Table 1. Enzyme mixes were further added to some of the biosludges during set-up of digestion experiments. In total, nine combinations of pre-treated sludges (see Table 2) were digested in triplicate.

Methane potential tests

The methane potential of the sludges with and without pre-treatment was tested in triplicate by the laboratory-scale anaerobic batch tests described in Hansen et al.

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Table 1. Used sludge types and their main properties after pre-treatment.

<table>
<thead>
<tr>
<th>Sludge type</th>
<th>Pre-treatment</th>
<th>pH</th>
<th>NH₄-N mg/l</th>
<th>COD mg/l</th>
<th>COD₅₉ₐ₉ₐ mg/l</th>
<th>TS %</th>
<th>VS %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biosludge</td>
<td>None</td>
<td>6.48</td>
<td>45</td>
<td>40070</td>
<td>1210</td>
<td>3.3</td>
<td>2.7</td>
</tr>
<tr>
<td>Biosludge</td>
<td>Ultrasonic</td>
<td>6.41</td>
<td>73</td>
<td>40000</td>
<td>1580</td>
<td>3.3</td>
<td>2.7</td>
</tr>
<tr>
<td>Biosludge</td>
<td>Hygienisation</td>
<td>6.02</td>
<td>90</td>
<td>40200</td>
<td>8745</td>
<td>3.4</td>
<td>2.8</td>
</tr>
<tr>
<td>Biosludge</td>
<td>Ultrasonic + hygienisation</td>
<td>5.90</td>
<td>144</td>
<td>43900</td>
<td>9125</td>
<td>3.5</td>
<td>2.8</td>
</tr>
<tr>
<td>Mixed primary + biosludge</td>
<td>None</td>
<td>6.79</td>
<td>87</td>
<td>33000</td>
<td>1755</td>
<td>2.9</td>
<td>2.0</td>
</tr>
<tr>
<td>Mixed primary + biosludge</td>
<td>Hygienisation</td>
<td>6.63</td>
<td>84</td>
<td>34100</td>
<td>3995</td>
<td>2.8</td>
<td>1.9</td>
</tr>
</tbody>
</table>
The tests were performed in 2-litre-reactors (Figure 1) containing an amount of test substrate representing 40% of the total volatile solids as well as ~400 ml of inoculum. The reactors were kept at mesophilic temperature (35ºC) and methane production was monitored by a gas chromatograph until the gas production ceased and the accumulated gas production remained at a fixed level. The method provides an easy-to-operate and fast means of measuring methane potentials in the sludge. The size of the reactors allows simultaneous tests of many reactors although the volume is large compared to many other laboratory anaerobic digestion methods. (Hansen et al., 2004). Too small amounts of substrate can be crucial for the representativity of the test. Reference substrate in form of cellulose was used to test the function of the inoculum. Cellulose was chosen because it was expected to digest slowly and give about the same potential as the tested sludges.

Continuous pilot-scale digestion tests

The continuous pilot-scale digestion tests can be used to evaluate operation and determine the specific gas production/methane yield under varying parameters as substrate type, retention time, organic loading, temperature etc. The pilot-scale equipment used resembles a full-scale biogas plant including heating, feeding once a day, stirring and gas collection. Each set of test equipment (Figure 2) included a cylindrical 35-litre-digester connected to a 77-litre-gas-collection-tank (Jansen et al., 2004b). The digesters were kept at mesophilic temperature, 35ºC. A top-mounted mechanical stirrer ensured a totally mixed tank. Feeding and residue removal was carried out manually once every day. The hydraulic retention time was chosen to be 13 days to have a reasonable high organic loading rate. The fed sludge had a TS-content of ~4% and this gave an organic loading rate of 2.3 kgVS/m³·day.

Table 2. Tested combinations of pretreated sludges.

<table>
<thead>
<tr>
<th>Sludge/Pre-treatment</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Bio</td>
<td></td>
</tr>
<tr>
<td>Bio/Ultrasonic</td>
<td></td>
</tr>
<tr>
<td>Bio/Hyg</td>
<td></td>
</tr>
<tr>
<td>Bio/Ultrasonic/Hyg</td>
<td></td>
</tr>
<tr>
<td>Bio/Enz</td>
<td></td>
</tr>
<tr>
<td>Bio/Ultrasonic/Enz</td>
<td></td>
</tr>
<tr>
<td>Bio/Hyg/Enz</td>
<td></td>
</tr>
<tr>
<td>Mixed</td>
<td></td>
</tr>
<tr>
<td>Mixed/Hyg</td>
<td></td>
</tr>
</tbody>
</table>
The enzymes added were divided into two mixtures, mix A and mix B. Mix A consists of four polysaccharide degrading enzymes and a lipase. Mix B contains protease, for complete hydrolysis of protein and glyco-proteins, and was separately added to avoid hydrolysis of enzymes in mix A during preparation and storage. The mix A-enzymes are immersed in an emulsifier combined with a surface-active substance.

A dose relative to 1 (also referred to as 100 %) corresponds to 0.06 % (w/w) of each enzyme final concentration per 1% (w/w) of the sludge TS. All used reagents are of analytical purity. Lipase, protease and glycosidic enzymes were a gift from Novozymes A/S, Denmark. Fatty alcohol ethoxylate (FAE) and xanthan gum were a gift from MB-Sveda, Malmö, Sweden. Details of the development of the procedure can be found in Wawrzynzcyk et al., (2003).

**Analytical methods**

In the methane potential measurements, VS-content, pH, ammonium and COD were measured before and after the test using standard methods (APHA, 1995). The methane production was measured in 0.2 ml samples, taken out from the reactors by a pressure tight gas syringe. The methane was measured on a gas chromatograph (Agilent 6850 series) equipped with a flame ionisation detector (FID) and a 30m/0.32mm/0.25μm column.

Analyses of produced gas and digested residues were carried out every day in the continuous tests. Gas composition (CH$_4$ and H$_2$S) was analysed by a Gas surveyor 431 Portable Gas Detector, GMI Gas measurement Instruments Ltd, Scotland, UK. For the digested residue, temperature and pH were controlled daily. In addition, HCO$_3$-, VFA, TS, VS, P-tot, N-tot and NH$_4$-N were analysed once a week. Standard methods for those analyses where applied (APHA, 1995).

**Results and Discussion**

**COD solubilisation**

Solubilisation of COD could be used as a measure of the pre-treatment effect. Solubilisation of COD for the different pre-treatments are found in Table 3. The results show a very low solubilisation of COD for the ultrasonic treatment. This implies that the treatment time is too low to destroy cells, but still can be enough to divide flocs. Hygienisation on the other hand solubilises much COD, especially for the biosludge. The effect on COD solubilisation from the enzymes was not measured, since the enzymes were added directly to the digester. However, previous experiments, where sludges were pre-treated with the same enzymes, showed a significant increased COD solubilisation (Wawrzynzcyk et al., 2003 and Jansen et al. 2004a).

**Digestion results**

**Biosludge**

Figure 3 shows the average methane potentials during the test period for biosludge and pre-treated biosludge and the final potentials are found in Table 4. It can be
seen that the highest methane potentials are found for enzyme added ultra-sonicated and enzyme added hygienised sludge. However enzyme addition to untreated biosludge only gives a small effect on the methane production. Methane potential for ultrasonicated sludge is significant higher than for untreated biosludge although the COD solubilisation was very low. On the other hand, hygienisation of biosludge resulted in a high COD solubilisation, but the methane potential increase compared to untreated biosludge is not in proportion to the COD solubilisation. The methane potential for hygienised ultra-sonicated biosludge is lower than for both hygienised biosludge and for ultra-sonicated biosludge. That is, there is no additional effect when combining ultrasonic treatment and hygienisation.

The results show that a high COD solubilisation from a pre-treatment does not necessarily lead to an increased methane production. On the other hand pre-treatment methods like ultra-sonication can lead to increased methane production although the COD solubilisation is low as was also seen in Tiehm et al. (2001).

Mixed sludge

Figure 4 shows the average methane potentials for mixed sludge with or without hygienisation. It can be seen that the hygienisation leads to a significant increase (17 %) in methane potential. Similar results could be seen in the semi-continuous pilot-scale digestion of corresponding sludges, where hygienisation resulted in 20 % higher methane yield (see Figure 5 and Table 5). The results show that hygienisation is a more effective way to increase methane production for mixed primary and biosludge than for biosludge alone.
Table 5. Methane yield and VS reduction from pilot-scale continuous anaerobic digestion of hygienised/untreated mixed sludge (primary and biological surplus sludge).

<table>
<thead>
<tr>
<th></th>
<th>Methane yield (Nml CH₄/g VS₀)</th>
<th>VSₐₙₐₙ (Nml CH₄/g VS₀)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hygienised mixed sludge</td>
<td>270</td>
<td>58</td>
</tr>
<tr>
<td>Mixed sludge</td>
<td>224</td>
<td>56</td>
</tr>
</tbody>
</table>

Conclusions

Pre-treatment of sludge by hygienisation or ultrasonication increases the methane potential in biological surplus sludge.

Combination of enzyme dosing with hygienisation or ultrasonication implies additional effects on methane potential, while combination of hygienisation and ultrasonication does not affect the methane potential.

Hygienisation of sludge at 70ºC for 1 hour before anaerobic digestion leads to a significant increase in methane production both for biosludge and mixed sludge (10–20%). This was seen in both batch and continuous digestion.

It can also be concluded that a high COD solubilisation from a pre-treatment does not necessarily lead to an increased methane production. On the other hand pre-treatment methods like ultrasonication can lead to higher methane production although the COD solubilisation is low.

Acknowledgements

The wastewater treatment plants in Kävlinge and Malmö are acknowledged for helping out with sludge samples. VA-verket, Malmö is also acknowledged for providing digestion equipment and assistance in operation with the continuous digestion experiments.

References


