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PRE-TREATMENT OF WASTEWATER SLUDGE BEFORE ANAEROBIC DIGESTION – HYGIENISATION, ULTRASONIC TREATMENT AND ENZYME DOSING

Förbehandling av avloppsslam innan rötning –
Hygienisering, ultraljudsbehandling och enzymtillsats

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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

Sammanfattning

Förbehandling av slam innan rötning kan öka metanproduktionen och nedbrytningen av organiskt material. Det finns flera metoder för detta ändamål. Anaeroba röt försök har gjorts för att jämföra tre förbehandlingsmetoder (hygienisering, ultraljudsbehandling och enzymtillsats) som använts separat eller kombinerats på bioslam och blandslam. Ökning av löst COD och metanpotential för de olika förbehandlade slammen har mätts. Vidare har semikontinuerliga röt försök i pilotskala utförts för utvärdering av hygienisering av blandslam.

Resultaten visar att förbehandling av bioslam ger en ökning av metanpotentialen, speciellt hygienisering och ultraljudsbehandling. Kombinerad enzymtillsats med hygienisering eller ultraljud ger ytterligare en ökning av metanpotentialen hos blandslammet. Däremot fås ingen ökning genom att hygienisera ultraljudsbehandlat bioslam.

Ökning av löst COD efter förbehandling behöver inte innebära en motsvarande ökning i metanpotentialen, men förbehandlingsmetoder som ultraljudsbehandling kan leda till en ökning av metanpotentialen trots att löst COD inte ökar.

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 pathogens 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 (Wawrzynczyk *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 Sjölanda 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.*

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

Sludge type	Pre-treatment	pH	NH ₄ -N mg/l	COD mg/l	COD _{sol} mg/l	TS %	VS %
Biosludge	None	6.48	45	40700	1210	3.3	2.7
Biosludge	Ultrasonic	6.41	73	40000	1580	3.3	2.7
Biosludge	Hygienisation	6.02	90	40200	8745	3.4	2.8
Biosludge	Ultrasonic + hygienisation	5.90	144	43900	9125	3.5	2.8
Mixed primary + biosludge	None	6.79	87	33000	1755	2.9	2.0
Mixed primary + biosludge	Hygienisation	6.63	84	34100	3995	2.8	1.9



Figure 1. Reactors for determination of methane potential (2-litre glass bottles with septum corks).

(2004). 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 (35°C) 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.

Table 2. Tested combinations of pretreated sludges.

Sludge/Pre-treatment
Bio
Bio/Ultrasonic
Bio/Hyg
Bio/Ultrasonic/Hyg
Bio/Enz
Bio/Ultrasonic/Enz
Bio/Hyg/Enz
Mixed
Mixed/Hyg



Figure 2. The systems for pilot-scale continuous anaerobic digestion.

(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, 35°C. 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 3. COD solubilisation for different treatments $\left(\frac{COD_{sol}^{treated}-COD_{sol}^{untreated}}{COD_{tot}^{untreated}}\right)$.

Sludge treatment	COD solubilisation %
Ultrasonication of biosludge	1
Hygienisation of biosludge	19
Hygienisation of ultrasonicated biosludge	19
Hygienisation and ultrasonication of biosludge	19
Hygienisation of mixed sludge	7

Enzymes used

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 Wawrzynczyk *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

Table 4. Final average methane potentials (after 41 days).

Sludge/Pre-treatment	Methane potential Nml CH ₄ /g VS _{in}	Standard deviation %
Bio	313	5
Bio/Ultrasonic	358	5
Bio/Hyg	345	4
Bio/Ultrasonic/Hyg	324	3
Bio/Enz	322	1
Bio/Ultrasonic/Enz	370	4
Bio/Hyg/Enz	357	2
Mixed	353	4
Mixed/Hyg	410	1

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 (CH₄ and H₂S) 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, HCO₃, VFA, TS, VS, P-tot, N-tot and NH₄-N were analysed once a week. Standard methods for those analyses were 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 (Wawrzynczyk *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

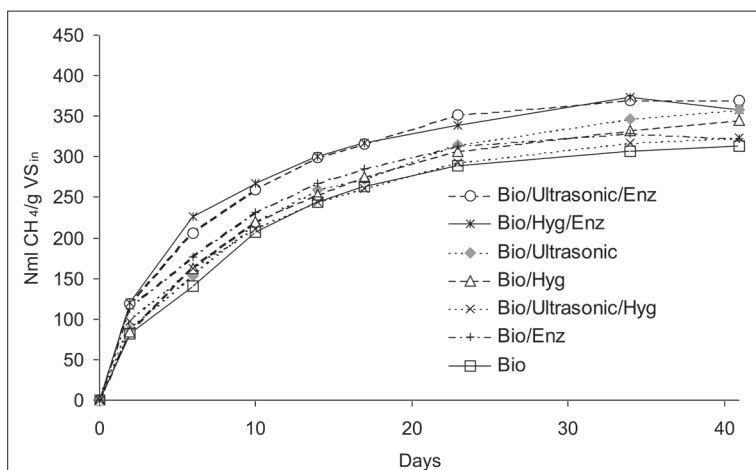


Figure 3. Methane potentials (average of triplicate reactors) during the test period for biosludge and pre-treated biosludge. The standard deviations did not exceed 5%.

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

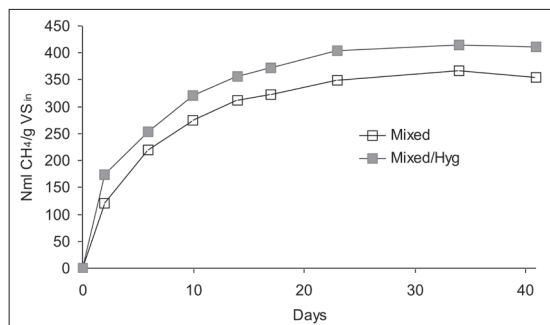


Figure 4. Methane potentials (average of triplicate reactors) during the test period for mixed sludge and hygienised mixed sludge.

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.

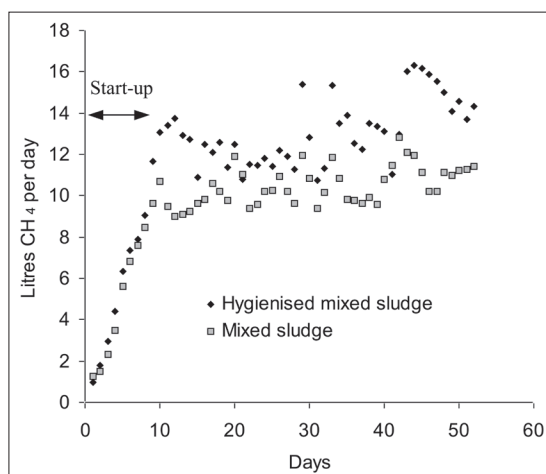


Figure 5. Daily methane production during continuous pilot-scale digestion of mixed sludge with or without hygienisation.

Table 5. Methane yield and VS reduction from pilot-scale continuous anaerobic digestion of hygienised/untreated mixed sludge (primary and biological surplus sludge).

	Methane yield (Nml CH ₄ /g VS _{in})	VS _{red} %
Hygienised mixed sludge	270	58
Mixed sludge	224	56

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 70°C 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.

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