Grubbens Deflaker model GLD 200 (from Cellwood Machinery AB)

Pre-treatment of biomass for anaerobic digestion

Verification Report
J.no. 1003

Version 4, June 6th 2012

1. Table of Contents

2. Introduction

Environmental technology verification (ETV) is an independent (third party) assessment of the performance of a technology or a product for a specified application, under defined conditions and quality assurance.

DANETV is a Danish center for verification of environmental technology.

This verification report includes protocol describing the framework for the verification of the technology

2.1. Name of product

The product name is Grubbens Deflaker model GLD 200

2.2. Name and contact of vendor

Producer (vendor)
Cellwood Machinery AB, Box 65 SE- 571 21 Nässjö, Sweden

Contact: Olof Lekander , phone: +46 (0) 386 76093, e-mail: olof.lekander@cellwood.se

Danish distributor
Al 2- Agro Krøgebækvej 25, DK 8682 Hovborg
Contact:Preben Nissen, phone: +45 3169 6501, e-mail: pbn@al-2.dk

2.3. Name of centre/verification responsible

Danish Technological Institute, Verification Centre, Life Science Division, Kongsvang Allé 29, DK-8000, Aarhus C.
Verification responsible: Arne Grønkjær Hansen, phone: +45 72202142
E-mail: agha@teknologisk.dk

Internal reviewer: Lars D. M. Ottosen (LDMO), phone: + 45 72202194
E-mail:ldmo@teknologisk.dk

2.4. Verification and test organization

The verification conducted by Danish Technological Institute

The organization of test and verification is shown in Figure 1.

The verification was planned and conducted to satisfy the requirements of the ETV scheme currently being established by the European Union (EU ETV).

Verification and tests were performed by Danish Technological Institute under DANETV under contract with Cellwood Machine AB

The day to day operations of the verification and tests was will be coordinated and supervised by TI personnel, with the participation of the vendors Danish distributor Preben Nissen form Al-2 Agro.

The testing was conducted at Bånlev Biogas, Trige.

TI Testcentre, performed all samplings of biomass for further laboratory testing.
AL2- agro personnel installed and operated the Deflaker and assisted with all necessary tasks for verification as described in the contract.

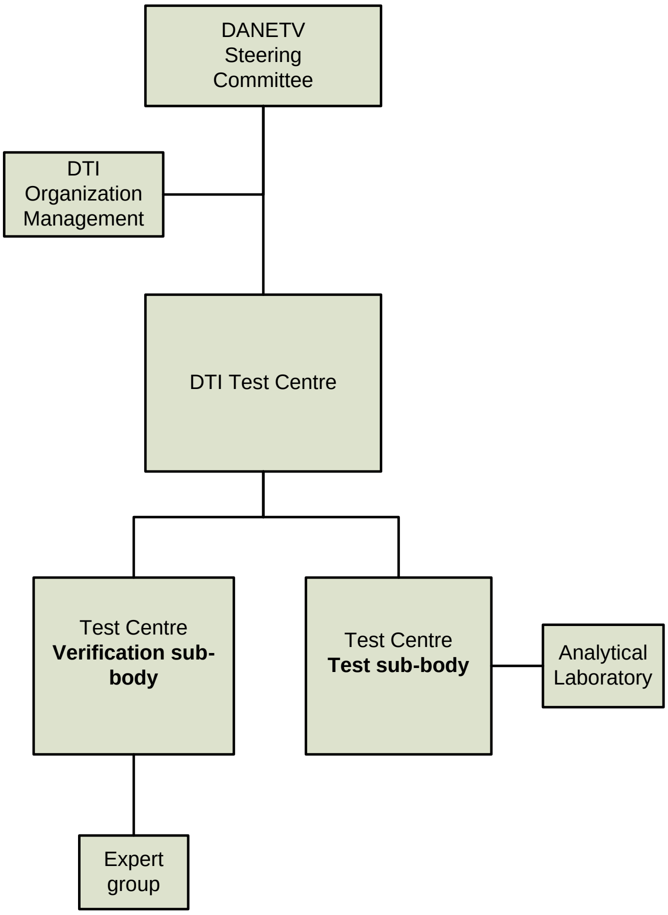

Figure 1 Verification organization

Table 1. Responsible personnel in test organization

Unit in test organization	Responsible
DTI Dan ETV steering committee member	Arne Grønkjær Hansen
DTI organization management Life science division	Bo Frølund
DTI Life science division Test Centre, Verification subbody	Mikael Poulsen
DTI Life science division Test Centre, Test subbody	Bjørn Malmgren-Hansen

2.5. Expert group

The expert group assigned to this verification and responsible for review of the verification plan and report documents includes:

Thorkild Qvist Frandsen (TQF), Agrotech, phone: +45 87438468, e-mail: tqf@agrotech.dk.
Kasper Stefanek (KPS), Agrotech, phone: +45 87438468, e-mail: kps@agrotech.dk

Verification process
Verification and tests were conducted in two separate steps, as required by DANETV. The steps in the verification are shown in Figure 2.
Verification and tests were performed by Danish Technological Institute DANETV verification and test centre. The verification sub-body is responsible for preparation and compilation of the Verification protocol and the Verification report. The Test sub-body is responsible for the test plan and the test report.

The day to day operations of the tests and verification was coordinated and supervised by DTI, with participation of the vendor, Cellwood Machinery and the Danish distributor AL2-Agro.
DTI collected samples of treated and non-treated biomass from the Cellwood Deflaker GLD 200 installed at a test facility.
The laboratory testing was conducted in the DTI Laboratories, Århus, Denmark.
Cellwood provided a full scale setup of the Grubben Deflaker and all needed instructions including user manuals containing operating instructions. In addition Cellwood participated in the development of the protocol and test plans in collaboration with DTI.
A part of the verification organization is the external expert group who supports DTI and reviews all plan and report documents during the verification process.

The steps in the verification are shown in Figure 2.

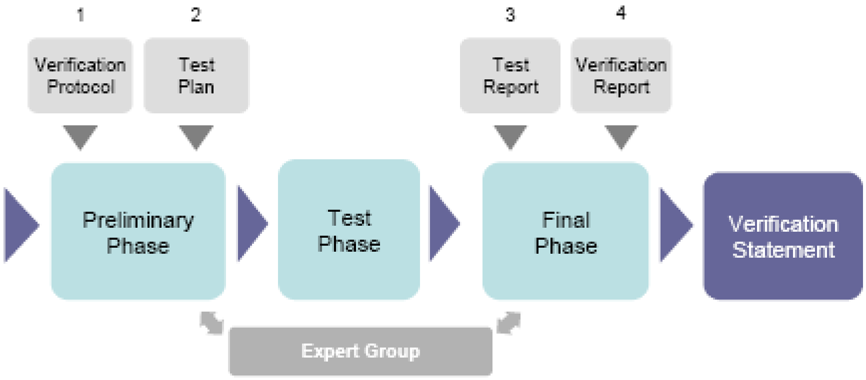

Figure 2 ***Verification steps***

A DANETV verification statement is issued after completion of the verification

3. Description of the technology

The process is a mechanical process for pre-treatment of biomass to improve bio gasification. The Deflaker is used for pre-treatment of biomass- originally developed for the paper industry. The process is using a wet pulp of fibres mixed with liquid (water or manure).

4. Description of the product

The product verified is a “Deflaker” used to pre-treat biomass fibres suspended in liquid prior to anaerobic digestion to achieve increased/faster biogas production resulting in increased methane yield.
In the pretreatment the fibre structure is disintegrated.
The principle is shown in Figure 3.

The system consists of two discs with teeth- One disk rotates and the other is stationary-(stator disc). The fibre pulp is pumped into the centre of the stationary disc passing the teeths which rip up the fibre structure and the fibrepulp is hurled to the outlet. Grubbens Deflaker types GLD 200 have fixed axial rotor discs, and the gap can therefore not be adjusted on this model.

The discs are made of hardened, acid-resistant steel with Brinell hardness of about 400 HB.

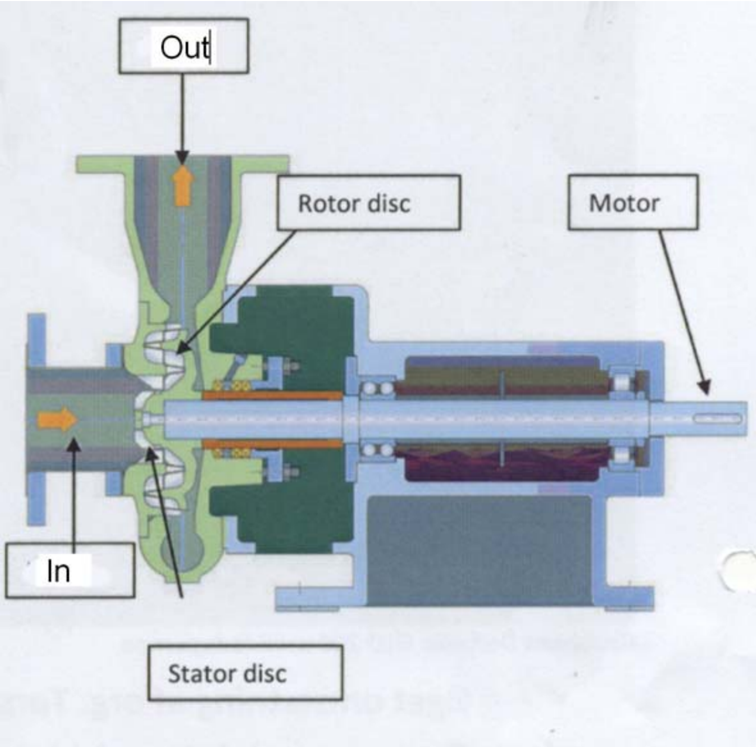

Figure 3 Schematic design of the Grubben Deflaker GLD 200


Figure 4 Discs

5. Application and performance parameter definitions

5.1. Matrix/matrices

The matrix is the type of material the product is intended for.
Matrix: Biomass for anaerobic digestion
Application: Mechanical pre-treatment of biomass

5.2. Target(s)

A target is defined as the property affected by the product

The targets of the product are:

- methane yield

5.3. Effects

The effects are described as the way the target is affected

The effects of this application are

- Increased methane yield from treated and digested biomass compared to untreated digested biomass.

5.4. Performance parameters for verification

The ranges of performance relevant for the application, as derived in Appendix 3, are presented in Table 2. These ranges were used for planning the verification and testing only.

The performance parameters relevant for this protocol are:
For added biomass and treated biomass the following parameters must be measured:

- Biogas potential
- Dry matter
- Volatile solids

For characterisation purposes the following parameters were measured:

- Total nitrogen content
- Ammonia content
- Total phosphor and potassium

Other performance-parameters which were measured included:

- The amount of added biomass for a complete run
- The amount of added additives (water) for a complete run
- All other added amounts or removed amounts either through weighing or from calculation
- Electricity consumption

5.5. Additional parameters

Besides the performance parameters to be obtained by testing, compilation of parameters described in user manual/documentation, occupational health & safety issues of the product were required as part of the verification.

6. Existing data

6.1. Summary of existing data

The Cellwood Grubbens Deflaker has been tested on sludge from municipal water. The results showed a higher velocity of the initial gas production and an increased methane production (33%) In addition the specific energy consumption using sludge 0.75 kWh/m^3 (0.02 kWh/kg DS).(8) The promising results on sludge and the fact that the biomass of agricultural origin has a higher dry matter content indicate that there is a potential for the Deflaker as pretreatment technology on biogasplants.

6.2. Quality of existing data

A number of unpublished experiments at Århus University have been performed testing the performance of the Deflaker using manure fibres from pig and dairy farms. The tests indicate an improvement in biogas potential in some of these some tests. Further a reduction in particle size was measured. The most promising data are manure from cows and pressed manure fibres from growing-finishing pigs.

The standard deviation on data from the tests on manure fibre at Århus University was relative high and therefore these could not be used in the verification.

6.3. Accepted existing data?

Existing data were evaluated to be "not suitable for verification" because of large variations

7. Test plan requirements

7.1. Test design

The test design was based on taking representative samples of fed untreated biomass and samples of treated biomass.

The effects of the process was tested measuring methane yield from treated biomass compared to untreated biomass using batch digestion experiments

The typical incubation period in anaerobic digesters at biogas plants is approximately 15-30 days and this is simulated in the tests and used when results are evaluated.

Further the electricity consumption of the Deflaker was logged.

The detailed test design is given in the test report.

Measuring biogas potential

Biogas yield was measured during anaerobic digestion of added biomass and pretreated biomass. The biogas potential was measured according to the method for measuring biogas potential described in Appendix5.

The result, a calculation of (l CH_4 /VS of added biomass) for treated and non-treated biomass is presented as a function of time for methanophilic biogasification.

The dry matter TS and volatile solids content of the samples were analyzed prior to biogas yield testing

Performing tests
At least 3 independent samples were taken of input and output streams for measuring biogas yield in order to calculate standard deviations.

To reduce sampling error a number of representative samples were taken during feeding and unloading of treated biomass. Subsamples were taken and mixed to representative samples. This was done to evaluate the standard deviations of parameters which depend on fibre composition such as moisture content and ash content (volatiles).

During the test period the operational stability and deviations from normal operational functioning were observed and registered, and the observations reported in the test report.

Evaluating accumulated methane production
To be able to conclude on whether there is a difference in produced accumulated methane/g VS of two separate tests it is required that the calculated standard deviations do not overlap at the point (day of production) of comparison.

7.2. Reference analysis

For batch tests of biogas potential a reference component in the medium was be tested as described in ref.5.

7.3. Data management

Data storage, transfer and control must be done in accordance with the requirements of the “ETV centre quality manual “DTI Water and Chemistry Technology” enabling full control and retrieval of documents and records.

7.4. Quality assurance

The quality assurance of the tests must include control of the reference system, control of the test system and control of the data quality and integrity.
The test plan and the test report have been subject to review by the expert group as part of the review of the verification protocol and verification report, see Figure 2.

7.5. Test report

The test report follows the template of the TI verification centre quality manual /2/ with data and records from the tests presented.

8. Evaluation

8.1. Performance parameter summary

Target and measured values of tested parameters.

Parameters	Target	Measured value	Method/comment
Overall performance			
Capacity		(approximately 20 m3/h)	Based on set flow.
Chemicals		None	
Energy			
Electricity consumption		35 kw	Based on measurement during test on cow manure (9%DM) and maize silage 9.3% in water
Treatment effects			
Increase in Methane production % (maize silage)		9.5	Methane potential (mesophilic 35˚C) after 30 days active methane production at 12.3 gVS/l
Increase in Methane production % (cow manure)		No increase	Methane potential (mesophilic 35˚C) after 30 days active methane production at 25 g VS/l and 10 g VS/l

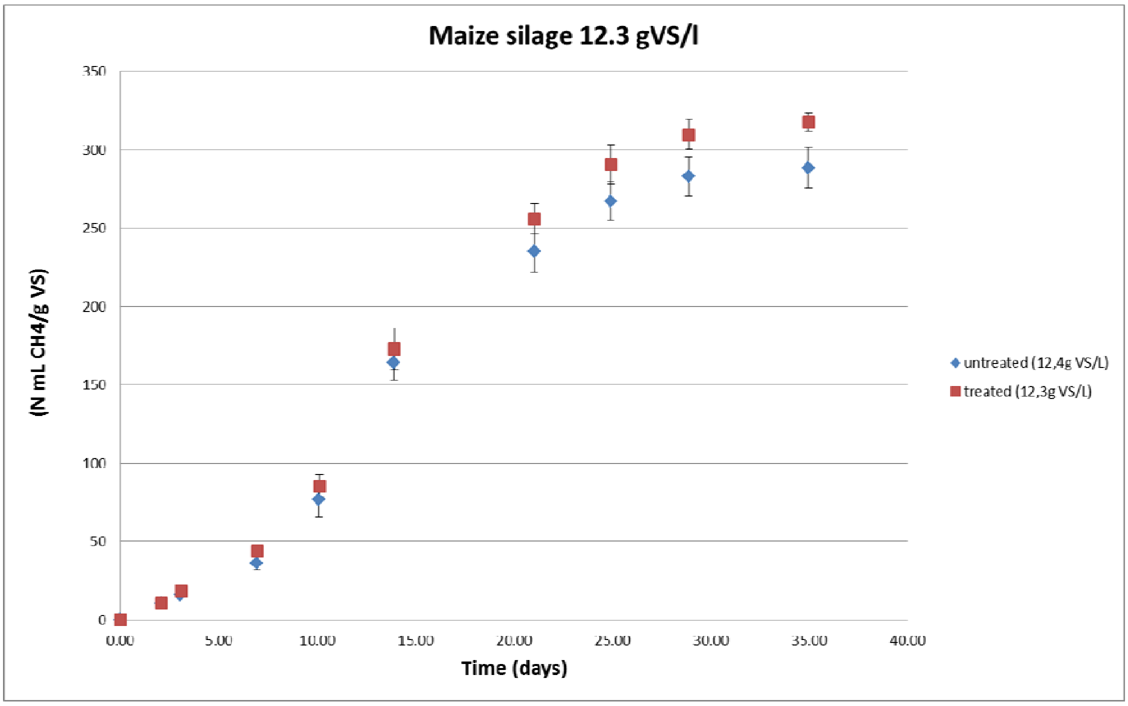

Figure 5 Example of curve showing increased accumulated methane production from treated maize silage. using 5 replicates 12.3gVS/l and method 1 (appendix 5).

8.2. Conclusion on performance

It is concluded for Cellwood Deflaker GL200 system:

- there is a positive effect on methane production when treating maize silage of approximately 10% after 30 days active mesophilic biogasification when adding 12 gVS/l
- there is no effect on methane production when treating cow manure after 30 days active mesophilic biogasification when adding 25 gVS/l and 10 gVS/l
- the power consumption when treating maize silage was approximately 0.02 kwh/kg added maize silage.

8.3. Compilation of additional parameters

8.3.1. User manual

The description in the user manual regarding handling of the Deflaker are regarded as sufficient and includes instructions for:

- Operation of the system
- Prevention of and dealing with incidents
- Occupational health and safety measures
- Service and maintenance
- Surveillance of the installation

8.3.2. Occupational health and environment

The safety instructions in the user manual regarding handling of the Deflaker GLD 200 is regarded as sufficient. It is concluded that the application of the Deflaker system at biogas plants does not result in increased environmental risks provided that all described precautions and maintenance measures are taken.

Recommendations for verification statement
It is recommended to issue a verification statement exclusively on the DEFLAKER GLD 200 for pre-treatment of maize silage for biogas production and emphasize that this specific product is expected to increase biogas yield in the range 0-10% at full scale biogas plants. Following liability exclusions should be included in the verifications statement.

9. Liability exclusion

ETV verifications are based on an evaluation of technology performance under specific, predetermined operational conditions and parameters and the appropriate quality assurance procedures. DTI makes no expressed or implied warranties as to the performance of the technology and do not certify that the technology will always operate as verified.
The end user is solely responsible for complying with any applicable regulatory requirements.

10. Quality assurance

The test protocol, test plan, test report and verification report were reviewed by internal and external experts according to the Quality plan for the verification.

Table 2 QA plan for the verification

Reviewers	TI	Experts
Plan document with application definition, verification protocol and test plan	NHN	TQF, LDMO
Report document with test report and verification report	NHN	TQF, LDMO

Reviews were done using the TI review report template.

Appendix 1 Terms and definitions used in the verification protocol

Terms and definitions used in the protocol are explained in table 1.

Table 1 Terms and definitions used by the DANETV test centres

Word	DANETV	Comments on the DANETV approach
Analytical laboratory	Independent analytical laboratory used to analyse test samples	The test centre may use an analytical laboratory as subcontractor
Application	The use of a product specified with respect to matrix, target, effect and limitations	The application must be defined with a precision that allows the user of a product verification to judge whether his needs are comparable to the verification conditions
DANETV	Danish centre for verification of environmental technologies	
(DANETV) test centre	Preliminary name for the verification bodies in DANETV with a verification and a test sub-body	Name will be changed, when the final nomenclature in the EU ETV has been set.
Effect	The way the target is affected	The effect could be concentration reduction, decrease in treatment period, pH increase etc.
(Environmental) product	Ready to market or prototype stage product, process, system or service based upon an environmental technology	The product is the item produced and sold and thus the item that a vendor submit for verification
Environmental technology	The practical application of knowledge in the environmental area	The term technology is covering a variety of products, processes, systems and services.
Evaluation	Evaluation of test data for a technology product for performance and data quality	None
Experts	Independent persons qualified on a technology in verification	These experts may be technical experts, QA experts for other ETV systems or regulatory experts

Word	**DANETV**	**Comments on the DANETV approach**
Matrix	The type of material that the product is intended for	Matrices could be soil, drinking water, ground water etc.
Method	Generic document that provides rules, guidelines or characteristics for tests or analysis	An in-house method may be used in the absence of a standard, if prepared in compliance with the format and contents required for standards.
Performance claim	The effects foreseen by the vendor on the target (s) in the matrix of intended use	None
Performance parameters	Parameters that can be documented quantitatively in tests and that provide the relevant information on the performance of an environmental technology product	The performance parameters must be established considering the application(s) of the product, the requirements of society (regulations), customers (needs) and vendor claims
Procedure	Detailed description of the use of a standard or a method within one body	The procedure specifies implementing a standard or a method in terms of e.g.: equipment used
Producer	The party producing the product	None
Standard	Generic document established by consensus and approved by a recognized standardization body that provides rules, guidelines or characteristics for tests or analysis	None
Target	The property that is affected by the product	Targets could be *e.g.*. contaminant concentration
Test centre, test sub-body	Sub-body of the test centre that plans and performs test	None
Test centre, verification sub-body	Sub-body of the test centre that plans and performs the verification	None
Test/testing	Determination of the performance of a product for parameters defined for the application	None

Word	DANETV	Comments on the DANETV approach
Vendor	The party delivering the product to the customer	Can be the producer
Verification	Evaluation of product performance parameters for a specified application under defined conditions and adequate quality assurance	None

Appendix 2 References (verification protocols, requirement documents, standards, methods, existing data)

1. DANETV. Centre Quality Manual, DTI 2009.

2. European Parliament and Council. Directive 2006/42/EC of the 17th May 2006 on machinery and amending Directive 95/16/EC (recast).

3. European Council: Directive 89/655/EEC of 30 November 1989 concerning the minimum safety and health requirements for the use of work equipment by workers at work (amended 2007/30/EC).

4. ISO 12100-2:2003: Safety of machinery - Basic concepts, general principles for design - Part 2: Technical principles.

5. Measurement protocol for biogas potential measurements for ETV tests at DANETV. (Method 1 and 2)

6. Unpublished results from test on initial tests on deflaked manure fiberes and straw, Århus University, (Maibritt Hjort,Research centre Foulum) ….
7. Cellwood machinery (2008) Information about the deflaker.http://www.cellwood.se/Templates/ArticleProduct.aspx?PageID=2888cd67-ae71-4893-b424-0dcc18d5397c [assessed 23 September 2008]
8. Sundin, Anna Maria (2008), DISINTEGRATION OF SLUDGE - A WAY OF OPTIMIZING ANAEROBIC DIGESTION. 13th European Biosolids & Organic Resources Conference & Workshop,Käppala Association, Lidingö, Sweden. (http://www.kappala.se/admin/bildbank/uploads/Dokument/Processutveckling/Sundin,_A-M._Disintegration_of_Sludge__A_way_of_Optimizing_Anaerobic_Digestion.pdf)

Appendix 3 Application and performance parameter definitions

This appendix defines the application and the relevant performance parameters application as input for verification and test of an environmental technology following the DANETV method.

A3.1 Applications

A3.1.1 Matrix/matrices

A3.1.2 Target(s)

A3.1.3 Effects

The effects claimed by the vendor are presented in Table 2:

Table 2 Performance parameters and vendor claims

Performance parameter	Vendor claim of performance
Biogas potential	Upto 20 % increase of methane production per kg VS depending on fibre type after 30 days of active mesophilic production
Electricity consumption	< 0,03 kWh/added kg DM

A3.1.4 Exclusions

A3.2 General performance requirements

A3.2.1 Regulatory requirements

A3.2.2 Application based needs

A3.3 State of the art performance

A3.4 Performance parameter definitions

Appendix 4 Test report
see separate test report

Appendix 5 In-house analytical methods
Measurement protocol for methane potential measurements for ETV tests at DANETV:

***Method 1:* Measurement protocol for biogas potential measurements for verification tests (ETV, CBMI)**

First version:12-5-09 revision v6 16/5-11

B. Malmgren-Hansen and Lars Ditlev Mørck Ottosen, Danish Technological Institute
Revised by Thorkild Quist Frandsen/Kasper Stefanek, Agrotech Henrik B.Møller,DJF

The protocol v5 16/5-09 was developed as part of the CBMI project subproject 05 Test, certification and declaration, www.cbmi.dk . The present version is slightly modified

Purpose
The purpose is to make a common work protocol for performing batch fermentation on biomass used for mesophilic or thermophilic biogasification.

The protocol is based on methods used at DJF, Agrotech and DTI. DTU methods have also been evaluated.

Description of test
The test is a modified version of ISO 11734 [1)]

The test is based on performing batch biogasification with degassed inoculum from a biogas plant and added media with recording of produced gas amounts and content of methane.

The biogasification is performed for
- test material
- inoculum (blank test)
- reference material
- varying concentrations of added test material (inhibition test)

The test on test material and blanks are performed as at least a triplicate test. In case of high inhomogeneity the number of replicates can be increased.

Conditioning of test material
Samples must be representative of the biomass to be tested and with a homogeneous structure allowing for taking representative subsamples. Procedures for correct conditioning of biomass (test material) and subsampling must be described elsewhere as it will depend on the structure of the biomass.

Handling and storing of samples

Test material (fibre samples/liquid) samples are taken in e.g. 1 litre PE bottles, filled only 80% allowing for freezing.

If testing cannot be performed immediately, the samples are frozen.

Materials

- Infusion bottles which can withstand a pressure of 2 bar (volume ½-1 litre)
- Butyl rubber stoppers+ Al Crimps
- Measurement device for measuring volume of produced gas (volume measurement or pressure measurement)
- Reference substrate

Conditions
Incubation at 35 +/- 1 ˚C (mesophilic) or 52 +/- 1˚ C (thermophilic)
The incubation temperature must be verified in the thermostating equipment within at least +/- 1˚C using calibrated temperature measurement devices.
When infusion bottles are removed for gas volume measurement, the period of storage outside the incubation chamber should be minimized (<1 hr).

Inoculum
Manure from biogas plant degassed 2 weeks at temperature of interest (mesophilic or thermophilic).
The NH4-N content shall be below 4 g/l unless a special test condition is chosen. pH must be between 6.5 and 8.5. For mesophilic biogasification, inoculum from thermophilic reactors may be used, as the mesophilic culture exists in such media, however, at a lower concentration.

Trial period
The test period may be up to 90 days. However the test period may be shortened if the period of interest is lower. In normal operations of biogas plants the period of mesophilic operation is approx 30 days and thermophilic operation approx. 20 days. In this case 45 days of test is sufficient. See also figure 1 and 2 later.
Sufficient measurement points on the curve (10-15) should be made to calculate the biogas potential at least after 20 or 30 days and after the total number of days in the test.
If a lag phase in methane production is observed, the days in the lag phase should be added to the test period.

When running comparisons of products/process treatments etc. the same manure batch should be used as inoculum to decrease uncertainty from blank subtraction.

Biogas potential test in infusion bottles
Inoculum of known volume/weight and test samples were added to the infusion bottles.

There must be 40-60% free space in bottles allowing for accumulation of gas.

Addition of inoculum:
Preferred conditions:

- 500 ml infusion bottles : 200 ml inoculum (measured with 0.1% accuracy)
- 1000 ml infusion bottles : 400 ml inoculum (measured with 0.1% accuracy)

Addition of test material (biomass):

DM and VS was measured/known on test material before addition.

Test materials was added within a range that gives sufficient sensitivity and no inhibition. The exact concentration must be estimated in an inhibition experiment.

Typical concentrations of test material was in the range
1 -30 g VS per liter inoculum.

The added amount was measured with 0.1% accuracy.

The test samples were flushed with N_2- 4 minutes before testing.

Tests were made in triplicate

Blanks
Tests were performed on inoculum (triplicate) for each new batch of inoculum.

The blanks were flushed with N2 for 4 minutes before testing.

Reference
A test compound (like sodium benzoate/cellulose powder or known fiber mix) should be run in inoculum (double or triplicate) for each new batch of inoculum.

The reference samples were flushed with N2 for 4 minutes before testing.

Inhibition
Inhibition from different substances may occur.
For NH4-N, inhibition may occur at levels of approx. 4g/l in the inoculum/test material mixture.

If no inhibition occurs, the same volume methane/g VS should be obtained for different values of added VS after complete fermentation.

To verify whether inhibition is present at test conditions, tests are performed with at least two concentrations of added VS etc. 100% and 30-50% and followed for at least 45 days.

Produced gas
Volume is calculated as pressure increase (ISO 11734) in headspace or measured directly with a volume collection tube (syringe or water filled gas collecting cylinder).

Efforts must be made to ensure no loss of process gas (ensuring gastight connections by pressure test).

CH4/CO2
Measured by GC for each measurement point during test (Method description in Biomass and Energy v26, 2004, p.487). GC must be calibrated using reference gas each day.

pH
pH is measured on inoculum batch before test.
pH is measured in test samples after finished biogasification as control of inhibitory acidification. (The measurement may be reduced to 1 pH measurement of triplicates showing same biogas production curves).

Result
For each measurement point, the ml methane amount is calculated.

Blank tests are subtracted.

A sum curve of produced (net) nml methane/gVS as function of time is calculated and plotted using correction for T,P.

All raw data on produced gas volume and methane should be available upon request.

Typical methane production curves
In Figure 1 is shown a typical curve for accumulated methane production at mesophilic biogasification of fibres separated from the slurry. Figure 2 shows the production rate for methane. In this test there is a lag phase during the first 10 days with the major production of methane from day 15 to 30. The lag phase is a delay in production while microorganisms are adapting to the biomass. It may be observed for some biomasses.

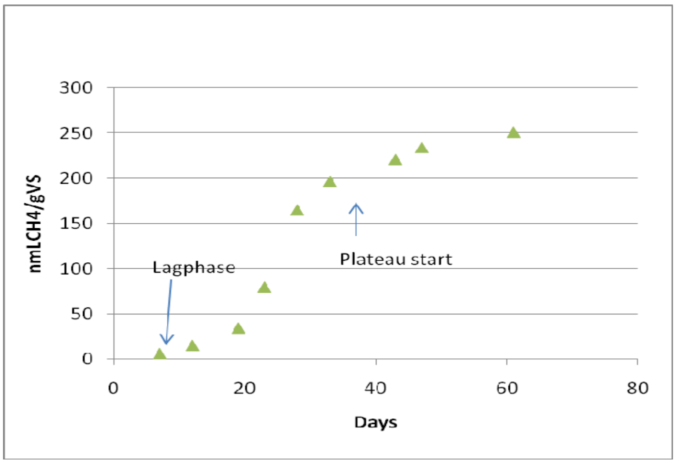

Figure 1. Accumulated methane production

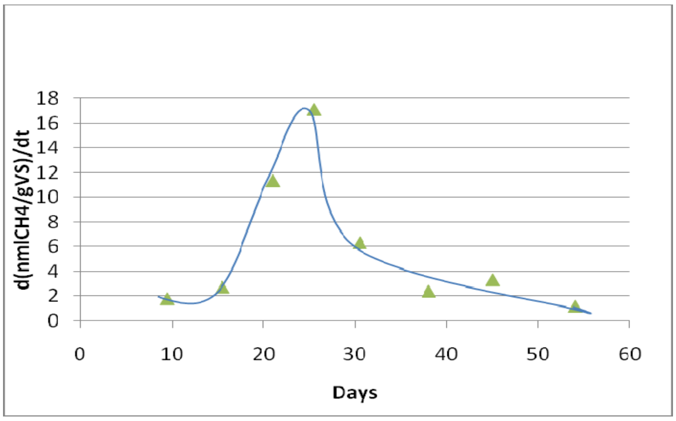

Figure 2. Methane production rate

1) The test is a modification of ISO 11734 including a simple inhibition test like required in Angelidaki Water sci. & Tech p.927, 2009. Additional nutrient medium is omitted – standard fermentet manure is used as reference (including sufficient nutrients and bacteria adapted to anaerobic fermentation at such circumstances).

Method 2: Measurement using the bioprocess control system

Purpose

The purpose is to make a common work protocol for performing methane potential tests with biomass using mesophilic or thermophilic anaerobic fermentation
The protocol is based on an automated biogas measurement method from bioprocess control.
The method has been intercalibrated with method 1 on separated fibres from swine.

Description test

The test is a modified version of ISO 11734 [1)]

The test is based on performing batch fermentation with degassed inoculum from a biogas plant and added media with recording of produced bioggas and methane volume

The anaerobic fermentation is performed for

- test material
- inoculum (blank test)
- reference material
- varying concentrations of added test material (inhibition test)

The test on biomass for test and blanks are performed as at least a triplicate test. In case of high inhomogeneity the number of replicates can be increased.

Conditioning of test material

Samples must be representative of the biomass to be tested and with a homogeneous structure allowing for taking representative subsamples. Procedures for correct conditioning of biomass (test material) and subsampling must be described as it will depend on the structure of the biomass.

Handling and storing of samples

Test material (fibre samples/liquid) samples are taken in e.g. 1 litre PE bottles, filled only 80% allowing for freezing.

If testing cannot be performed immediately, the samples are frozen.

Materials

- Complete measurement setup from bioprocess control

Conditions

Incubation at 35 +/- 1 ˚C (mesophilic) or 52 +/- 1˚ C (thermophilic)
The incubation temperature must be verified in the thermostating equipment within at least +/- 1˚C using calibrated temperature measurement devices.

Inoculum

Manure from biogas plant degassed 2 weeks at temperature of interest (mesophilic or thermophilic).

The NH4-N content shall be below 4 g/l unless a special test condition is chosen. pH must be between 6.5 and 8.5. For mesophilic fermentation, inoculum from thermophilic reactors may be used, as the mesophilic culture exists in such media, however, at a lower concentration.

Trial period
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However the test period may be shortened if the period of interest is lower. In normal operations of biogas plants the period of mesophilic operation is approx. 30 days and thermophilic operation approx. 20 days. In this case 45 days of test is sufficient. See also figure 1 and 2 later.
Sufficient measurement points on the curve (10-15) should be made to calculate the biogas potential at least after 20 or 30 days and after the total number of days in the test.
If a lag phase in methane production is observed, the days in the lag phase should be added to the test period.

When running comparisons of products/process treatments etc. the same manure batch should be used as inoculum to decrease uncertainty from blank subtraction.

Biogas potential test in infusion bottles
Inoculum of known volume/weight and test samples are added to the infusion bottles. The bottles are placed in a heating box. Produced biogas is cleaned for CO_2 in a acid trap and the volume of total gas and cleaned gas is measured automatically. The system is developed for on-line measurements of low methane flows produced from the anaerobic digestion in laboratory scale tests.

Laboratory setup for measurement of biogas and methane production of different substrates.

Addition of inoculum:
Preferred conditions:

- 500 ml infusion bottles : 300-400 ml inoculum (measured with 0.1% accuracy)

Addition of test material (biomass):

DM and VS shall be measured/known on test material before addition.

Test materials are added within a range that gives sufficient sensitivity and no inhibition.
The exact concentration must be estimated in an inhibition experiment.

Typical concentrations of test material are expected to be in the range
1 -30 g VS per liter inoculum.

The added amount is measured with 0.1% accuracy.

The test samples are flushed with N_2- 2-4 minutes before testing (using an N_2 amount of 10 times the free volume of filled flasks).

Tests are made at least in triplicate.

Blanks
Tests are performed on inoculum (triplicate) for each new batch of inoculum.

The blanks are flushed with N_2- 2-4 minutes before testing (using an N2 amount of 10 times the free volume of filled flasks).

Reference
A test compound (like sodium benzoate/cellulose powder) or reference fibres with known biogas potential should be run in inoculum (double or triplicate) for each new batch of inoculum.

The reference samples are flushed- 2-4 minutes before testing (using an N2 amount of 10 times the free volume of filled flasks).

Inhibition
Inhibition from different substances may occur.
For NH4-N, inhibition may occur at levels of approx. 4g/l in the inoculum/test material mixture.

If no inhibition occurs, the same amount of ml methane/g VS should be obtained for different added amounts of VS after complete fermentation.

To verify whether inhibition is present at test conditions, tests should be performed with at least two concentrations of added VS etc. 100% and 30-50% and followed for at least 30 days of active biogas production.

Produced amount of Methane
The bioprocess control system removes Carbon dioxide by washing with NaOH and measures Methane directly

pH
pH is measured on inoculum batch before test.
pH is measured in test samples after end of fermentation as control of inhibitory acidification. (The measurement may be reduced to 1 pH measurement of triplicates showing same biogas production curves).

Result
For each measurement point, the volume of methane (in ml) is calculated.

Blank tests are subtracted.

A sum curve of produced (net) nml methane/gVS as function of time is calculated and plotted using correction for T,P.

All raw data on produced volume of methane should be available upon request.

Typical biogas production curves
In Figure 1 is shown a typical curve for accumulated methane production at mesophilic biogasification of fibres separated from the slurry. Figure 2 shows the production rate for methane. In this test there is a lag phase during the first 10 days with the major production of methane from day 15 to 30. The lag phase is a delay in production while microorganisms are adapting to the biomass. It may be observed for some biomasses.

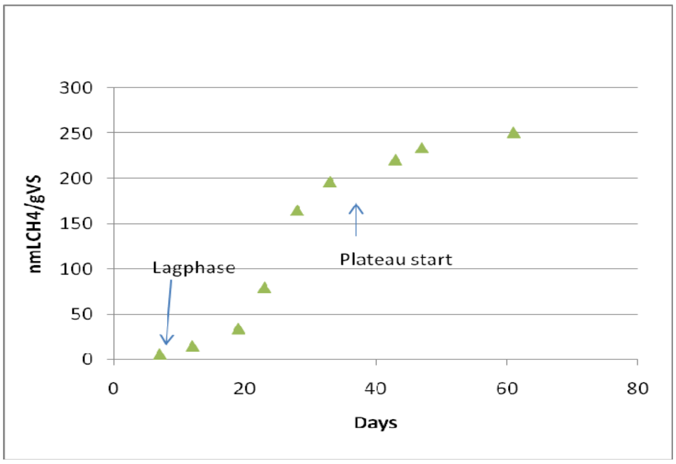

Figure 1. Accumulated methane production

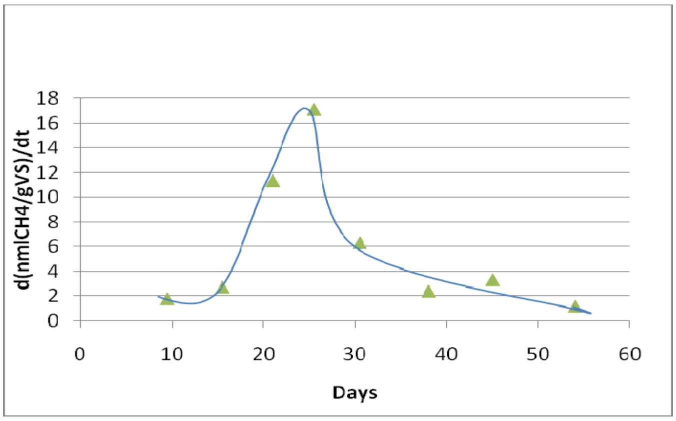

Figure 2. Methane production rate

Appendix 6 Amendment and deviation report for verification

The amendments in the review report has been implemented in this verification report.