

# Reader

des Förderprogramms für energetische Biomassenutzung



## 2nd conference on Monitoring & process control of anaerobic digestion plants

March 17 - 18, 2015  
in Leipzig, Germany

**Special workshop**

Foam formation in anaerobic digestion plants  
19<sup>th</sup> of March, 2015

Supported by



Federal Ministry  
for Economic Affairs  
and Energy

Project management



Programme support



Partners



# Imprint

## **Publisher**

Jan Liebetrau, Daniela Thrän, Diana Pfeiffer

## **Contact**

DBFZ Deutsches Biomasseforschungszentrum gemeinnützige GmbH  
Torgauer Straße 116  
D-04347 Leipzig  
Telefon: +49 (0)341 2434-554  
Telefax: +49 (0)341 2434-133  
E-Mail: [diana.pfeiffer@dbfz.de](mailto:diana.pfeiffer@dbfz.de)  
[www.energetische-biomassenutzung.de](http://www.energetische-biomassenutzung.de)

## **General Management**

Scientific Managing Director  
Prof. Dr. mont. Michael Nelles  
Administrative Managing Director  
Dipl.-Kfm. (FH) LL.M. Daniel Mayer

## **Editorial stuff**

Programme support of the funding programme "Biomass energy use"  
[www.energetische-biomassenutzung.de](http://www.energetische-biomassenutzung.de)

## **Photos**

Unless otherwise specified, DBFZ gGmbH retains the rights to images inside this publication.

## **Layout**

Angela Gröber

## **Print**

se druck, Leipzig

## **Funding**

Created with funds from the Federal Ministry for Economic Affairs and Energy  
(BMWi)

**ISSN 2366-4169**

## Contents

<b>Background</b>	<b>5</b>
<b>Programme</b>	<b>6</b>
<b>1<sup>st</sup> day</b>	
<b>Session A: SOLUTIONS FOR MONITORING &amp; PROCESS CONTROL - An overview</b>	
Overview and fundamentals of process monitoring in biogas plants <i>(Bernhard Drosig)</i>	13
On-line monitoring and process control of AD plants <i>(Jens Bo Holm-Nielsen)</i>	14
Biogas Plant mprocess monitoring in China: Several constraints and solutions <i>(Renjie Dong)</i>	15
<b>Session B: LABORATORY METHODS for MONITORING &amp; PROCESS CONTROL</b>	
Biomethane potential test – parameter, results and evaluation <i>(Hans Oechsner)</i>	16
Reliability of analytical measurements in anaerobic reactors: Case study for volatile fatty acids (VFAs) <i>(Francisco Raposo)</i>	17
NIR for determination of biogas yields: state of the art <i>(Alastair Ward)</i>	18
<b>Session C: MEASUREMENT of EMISSIONS on AD plants</b>	
Monitoring of fugitive methane emissions from an Austrian biogas plant <i>(Marlies Hrad)</i>	19
Residual energy potentials of digestates <i>(Michael Tauber)</i>	20
Methane emissions from biogas plants – methods and equipment, results, operational states <i>(Torsten Reinelt)</i>	21
<b>Session D: SOLUTIONS for PROCESS MONITORING in FULL SCALE APPLICATION</b>	
Power on demand: Optimized feeding by predictive control systems <i>(Jens Bischoff)</i>	23
Process and machinery monitoring in Thöni biogas plants <i>(Urban Zell)</i>	24
<b>Session E: EFFICIENCY CONTROL on AD plants</b>	
Evaluation of digestate conditions for minimization of energy demand of mixers? <i>(Kay Rostalski)</i>	25
Prediction of biogas yield potential based on the chemical composition: Possibilities and limitations <i>(Vasilis Dandikas)</i>	27

## 2<sup>nd</sup> day

### Session A: MEASUREMENT TECHNOLOGY, NEW APPROACHES for PROCESS CONTROL

NIRS in the biogas process: Restrictions of calibration transfer between similar processes ( <i>Michael Schmidt &amp; Fabian Jacobi</i> )	30
MIR spectroscopy for monitoring of AD processes - Prospects and challenges ( <i>Christian Wolf</i> )	31
Estimation of the foaming propensity of substrates in anaerobic digestion ( <i>Lucie Moeller</i> )	32

### Session B: NEW DEVELOPMENTS in SENSOR TECHNOLOGY

In-line acetate measurement in anaerobic digesters – development of a bioelectrochemical sensor platform ( <i>Jörg Kretzschmar</i> )	33
Application of laser absorption spectroscopy ( $^{13}\text{C-CH}_4$ and $^{13}\text{C-CO}_2$ ) for online monitoring in biogas plants ( <i>Daniela Polag</i> )	34
Development of process optimisation tools for both efficient biogas research and operation of full-scale plants ( <i>Mihaela Nistor</i> )	35

### Session C: SIMULATION, MODELLING & CONTROL of AD plants

Models as keys to optimization, monitoring and control of AD processes ( <i>Finn Aakre Haugen</i> )	36
Development of process optimisation tools for both efficient biogas research and operation of full-scale plants ( <i>Eric Mauky</i> )	37

### Session D: PROCESS CONTROL of ADs by MICROBIOLOGICAL MEASUREMENT METHODS

Monitoring the process microbiology in full-scale biogas plants – Identification of process-relevant microorganisms usable as validation standards or as indicators for process disturbances ( <i>Susanne Theuerl</i> )	38
Transferability of isotopic data to methanogenic pathways - an applicable process tool? ( <i>Tobias May</i> )	40
Process control by calorimetry - advantages and challenges ( <i>Thomas Maskow</i> )	41

### Poster session

Electronic nose technology for reactor state and biogas quality assessment in anaerobic digestion ( <i>G. Adam</i> )	43
Modelling of biogas production with ASPEN Plus for Model-based control ( <i>J. A. Arzarte</i> )	45
Pyrosequencing analysis of bacterial community structure in anaerobic co-digestion process for biohydrogen production ( <i>J. G. Romero</i> )	46
Online monitoring of volatile fatty acids in biogas with use of chemoresistive sensors ( <i>H. Hieber</i> )	47
Multivariate process control charts based on reputed process indicators as an alternative to univariate control charts for the monitoring of anaerobic reactors submitted to acidosis ( <i>S. Lemaigre</i> )	48
Optimization of anaerobic percolation processes and method development for substrate characterization ( <i>H. Wedwitschka</i> )	50
Hydrothermal lysis: A method for controlling the output of biogas and digestate ( <i>A. Cenian</i> )	51

### Exhibition & Sponsors

Bioprocess Control AB	53
Boreal Europe BV	54
DBFZ – Deutsches Biomasseforschungszentrum gGmbH	55
The Hessen State Laboratory	56
Helmholtz Centre for Environmental Research - UFZ	57
BINDER GmbH	58
Umwelt- und Ingenieurtechnik GmbH	59

## Background

Anaerobic digestion is a complex process of subsequent and interacting degradation steps. A precise control of this complex biological process is crucial to make the biogas production process more efficient, reliable and profitable. Novel process monitoring and controlling tools are necessary to improve performance of anaerobic digestion. The conference „Monitoring & process control of anaerobic digestion plants“ focuses on the requirements of measurement tools, new developments as well as best practice and the evaluation and presentation of practically implemented applications of monitoring and control devices.

Furthermore as a special event a workshop on “Foam formation in anaerobic digestion plants” is part of the programme. Foaming is one of the most common disorders in the process of anaerobic digestion in biogas plants. Although this phenomenon affects relatively high number of biogas plants, research is still in its infancy. The goal of the workshop is therefore to bring together the existing knowledge and to set priorities for further research on the foam formation in the biogas reactor.

The conference is organized by the programme „Biomass energy use“ (funded by the Federal Ministry of Economic Affairs), Deutsches Biomasseforschungszentrum gGmbH (DBFZ, German Biomass Research Centre), the Helmholtz Centre for Environmental Research – UFZ and the Hessian State Laboratory (LHL).

### Topics of the conference at a glance:

- **Laboratory measurements:** Reliability and validity.
- **Monitoring of plant operation**
- **Safety concepts and emissions:** Optimal monitoring and control technology.
- **Simulation and Control**
- **New sensor concepts**
- **Monitoring of the efficiency** of anaerobic digestion plants
- **Spectroscopy:** Opportunities and constraints.
- **Microbiological analysis:** Potential for process characterization, supervision and control?
- **Foam formation** in anaerobic digestion plants

We wish you a most successful, enriching and instructive discussion, but also an enjoyable conference, and a very pleasant stay in Leipzig.

### The organizers

## 1<sup>st</sup> day - 2015-03-17

### 10:00 – Conference opening

Room 1A

Michael Nelles (Scientific managing director, DBFZ/University of Rostock)  
Jan Liebetrau (DBFZ)

### Session A: SOLUTIONS FOR MONITORING & PROCESS CONTROL - An overview

Chair: Jan Liebetrau (DBFZ)

#### 10:15 - 10:45

- Overview and fundamentals of process monitoring in biogas plants  
Bernhard Drosig (BOKU Universität für Bodenkultur Wien/ Vienna)

#### 10:45 - 11:15

- On-line monitoring and process control of AD plants  
Jens Bo Holm-Nielsen (Aalborg University)

#### 11:15 - 11:45

- Biogas Plant Process Monitoring in China: Several Constraints and Solutions  
Renjie Dong (China Agricultural University)

### 11:45 – Lunch break

Foyer

### Session B: LABORATORY METHODS for MONITORING & PROCESS CONTROL

Chair: Fabian Jacobi (Landesanstalt hessisches Landeslabor, LHL)

#### 12:45 - 13:25

Room 1A

- Biomethane potential test – parameter, results and evaluation  
Hans Oechsner (University of Hohenheim)

#### 13:25 - 13:55

- Reliability of analytical measurements in anaerobic reactors: Case study for volatile fatty acids (VFAs)  
Francisco Raposo (CSIC-Instituto de la Grasa)

#### 13:55 - 14:25

- NIR for determination of biogas yields: state of the art  
Alastair Ward (University of Aarhus)

### 14:25 – Short afternoon break

Foyer

### Session C: MEASUREMENT of EMISSIONS on AD plants

Chair: Tanja Westerkamp (DBFZ)

#### 14:50 - 15:20

Room 1A

- Monitoring of fugitive methane emissions from an Austrian biogas plant  
Marlies Hrad (BOKU - Universität für Bodenkultur Wien)

## 1<sup>st</sup> day - 2015-03-17

### 15:20 - 15:40

- Residual energy potentials of digestates  
Michael Tauber (JOANNEUM RESEARCH Forschungsgesellschaft mbH)

### 15:40 - 16:00

- Methane emissions from biogas plants – methods and equipment, results,  
Torsten Reinelt (DBFZ)

### 16:00 – Afternoon break

Foyer

### Session D: SOLUTIONS for PROCESS MONITORING in FULL SCALE APPLICATION

Chair: Renjie Dong (China Agricultural University)

### 16:30 - 17:00

Room 1A

- Power on demand: Optimized feeding by predictive control systems  
Jens Bischoff (EnviTec Biogas AG)

### 17:00 - 17:30

- Process and machinery monitoring in Thöni biogas plants  
Urban Zell (Thöni Industriebetriebe GmbH)

### Session E: EFFICIENCY CONTROL on AD plants

Chair: Renjie Dong (China Agricultural University)

### 17:30 - 18:00

Room 1A

- Evaluation of digestate conditions for minimization of energy demand of mixers?  
Kay Rostalski (KSB)

### 18:00 - 18:30

- Prediction of biogas yield potential based on the chemical composition: Possibilities and limitations  
Vasilis Dandikas (Bayerische Landesanstalt für Landwirtschaft, LfL)

### 18:30 End of the first conference day

### 19:30 Networking dinner at the TELEGRAPH - Café & Restaurant

Dittrichring 18-20, 04109 Leipzig

The TELEGRAPH is situated in the centre of Leipzig (corner Dittrichring/Mathäikirchhof).  
You can reach the TELEGRAPH by tram #9 (station Thomaskirche) and by tram #1 (station Gottschedstraße).  
Parking places: around the TELEGRAPH or in the car parks Marktgalerie and Dresdner Bank/Zentralstraße close by.



## 2nd day - 2015-03-18

**09:00 – Opening of the 2<sup>nd</sup> conference day**

Jan Liebetrau (DBFZ)

**Room 1A**

### Session A: MEASUREMENT TECHNOLOGY. NEW APPROACHES for PROCESS CONTROL

Chair: Alastair Ward (Universität Aarhus)

**09:10 - 09:40**

- NIRS in the biogas process: Restrictions of calibration transfer between similar processes  
Michael Schmidt & Fabian Jacobi (Landesanstalt hessisches Landeslabor, LHL)

**Room 1A**

**09:40 - 10:10**

- MIR spectroscopy for monitoring of AD processes - Prospects and challenges  
Christian Wolf (Cologne University of Applied Sciences)

**10:10 - 10:40**

- Estimation of the foaming propensity of substrates in anaerobic digestion  
Lucie Moeller (Helmholtz Centre for Environmental Research - UFZ)

**10:40 – Morning break**

**Foyer**

### Session B: NEW DEVELOPMENTS in SENSOR TECHNOLOGY

Chair: Michael Mertig (KSI Meinsberg/TU Dresden)

**11:00 - 11:30**

- In-line acetate measurement in anaerobic digesters – development of a bioelectrochemical sensor platform  
Jörg Kretzschmar (DBFZ)

**Room 1A**

**11:30 - 12:00**

- Application of laser absorption spectroscopy ( $^{13}\text{C-CH}_4$  and  $^{13}\text{C-CO}_2$ ) for online monitoring in biogas plants  
Daniela Polag (Ruprecht-Karls-Universität of Heidelberg)

**12:00 - 12:30**

- Development of process optimisation tools for both efficient biogas research and operation of full-scale plants  
Mihaela Nistor (Bioprocess control Sweden Ab)

**12:30 – Lunch break**

**Foyer**



## 2nd day - 2015-03-18

### Session C: SIMULATION, MODELLING & CONTROL of AD plants

Chair: Christian Wolf (Cologne University of Applied Sciences)

**13:30 - 14:00**

**Room 1A**

- Models as keys to optimization, monitoring and control of AD processes  
Finn Aakre Haugen (Telemark University College, Norway)

**14:00 - 14:30**

- Model predictive control for demand-driven biogas production  
Eric Mauky (DBFZ)

**14:30 – Afternoon break**

**Foyer**

### Session D: PROCESS CONTROL of ADs by MICROBIOLOGICAL MEASUREMENT METHODS

Chair: Sabine Kleinsteuber (Helmholtz Centre for Environmental Research - UFZ)

**15:00 - 15:30**

**Room 1A**

- Monitoring the process microbiology in full-scale biogas plants – Identification of process-relevant microorganisms usable as validation standards or as indicators for process disturbances  
Susanne Theuerl (ATB Potsdam)

**15:30 - 16:00**

- Transferability of isotopic data to methanogenic pathways - an applicable process tool?  
Tobias May (Johannes Gutenberg-Universität Mainz)

**16:00 - 16.30**

- Process control by calorimetry - advantages and challenges  
Thomas Maskow (Helmholtz Umweltforschungszentrum - UFZ)

**16:30 – End of the 2nd conference day**

All abstracts & presentations are available on our website:

<https://www.energetische-biomassenutzung.de/en/events/conferences/process-control-2015/welcome.html>

## 2015-03-19

### Special workshop on „Foam formation in anaerobic digestion plants“

#### Spezialworkshop zur „Schaumbildung in Biogasanlagen“

**The workshop will be held in German. / Der Workshop findet in deutscher Sprache statt.**

Foaming is one of the most common disorders in the process of anaerobic digestion in biogas plants. Although this phenomenon affects relatively high number of biogas plants, research is still in its infancy. The goal of the workshop is therefore to bring together the existing knowledge and to set priorities for further research on the foam formation in the biogas reactor.

Schaumbildung gehört zu den häufigsten Störungen im Prozess der anaeroben Vergärung in Biogasanlagen. Obwohl dieses Phänomen eine relativ hohe Anzahl von Biogasanlagen heimsucht, steckt seine Erforschung noch in Kinderschuhen. Das Ziel des Workshops ist es, das bisher vorhandene Wissen zusammenzutragen und Schwerpunkte für die weitere Untersuchung der Schaumbildung im Biogasreaktor zu setzen.

Veranstalter: Dr. Lucie Moeller (Umwelt- und Biotechnologisches Zentrum, AG Bioprozesstechnik am UFZ)

Moderation: Prof. Dr. Andreas Zehnsdorf (Helmholtz-Zentrum für Umweltforschung GmbH - UFZ)

#### 10:00 Uhr – Grußworte

Roland Müller (Helmholtz-Zentrum für Umweltforschung GmbH - UFZ)

**UFZ-Gebäude 1.0 | Zimmer 254**

#### 10:10 Uhr – Vorstellungsrunde

#### 10:30 - 11:00 Uhr

- Der Traum vom kontrollierten Schaum  
Lucie Moeller (Helmholtz-Zentrum für Umweltforschung GmbH - UFZ)

#### 11:00 - 11:30 Uhr

- Prozessstörungen: Vermeidung oder Gegenmaßnahme? (Tandemvortrag)  
Anne Kleyböcker / Tobias Lienen (Helmholtz-Zentrum Potsdam - Deutsches GeoForschungsZentrum GFZ)

#### 11:30 - 11:50 Uhr – Kaffeepause

#### 11:50 - 12:10 Uhr

- Alptraum Schaum: Ursachen, Erscheinungsformen und Bekämpfungsstrategien in der Biogas-Praxis  
Harald Lindorfer (Schaumann BioEnergy GmbH)

#### 12:10 - 12:30 Uhr

- Tierische Nebenprodukte als Monosubstrat: eine komplexe Herausforderung  
Florian Rüscher Pfund (zhaw IBT Umweltbiotechnologie, Wädenswil)

#### 12:30 - 12:50 Uhr

- Chronische Schaumbildung auf der NawaRo-Biogasanlage der Agrarenergie Andelbach GmbH & Co KG  
Hans-Joachim Nägele (Universität Hohenheim, Landesanstalt für Agrartechnik und Bioenergie)

#### 12:50 Uhr – Mittagspause

**13:50 - 14:05 Uhr**

- Schaum in Fermentern: Schadenauswirkungen / -konsequenzen mit den Augen eines Sachverständigen  
Wolfgang A. Stachowitz (DAS-IB GmbH)

**14:05 - 14:20 Uhr**

- Erfahrungen mit Schaumbildung auf der BGA Schwabach  
Andreas Eichhorn (Bioenergie-Bayern GmbH & Co.KG)

**14:20 - 14:35 Uhr**

- Ursachen für die Schaumbildung in Biogasanlagen und praxistaugliche Gegenmaßnahmen  
Dorothea Telschow (Biogas - Additive.de GmbH & Co. KG)

**14:50 - 15:20 Uhr – Kaffeepause**

**11:50 - 12:10 Uhr**

- Transparente Prozessüberwachung in Biogasanlagen  
Jürgen Wiese (GKU Gesellschaft für kommunale Umwelttechnik mbH)

**15:40 - 16:00 Uhr**

- Effizienz von Biogasanlagen  
Jaqueline Daniel-Gromke (DBFZ Deutsches Biomasseforschungszentrum gGmbH)

**16:00 - 17:00 Uhr**

- Auswertung und Formulierung neuer Forschungsfragen  
im Plenum

**17:00 Uhr – Ende der Veranstaltung**

## Abstracts

### **Conference on**

Monitoring & process control  
of anaerobic digestion plants

1<sup>st</sup> day - 2015-03-17

## Overview and fundamentals of process monitoring in biogas plants



Bernhard Drosig (BOKU - University of Natural Resources and Life Sciences, Vienna)  
Contact: [bernhard.drosig@boku.ac.at](mailto:bernhard.drosig@boku.ac.at)

**Keywords:** biogas plants, biogas process monitoring, monitoring parameters

Biogas plants are biological systems involving various interacting microorganisms that anaerobically degrade organic matter. The degradation involves four consecutive biological processes: hydrolysis, acidogenesis, acetogenesis and methanogenesis. If one of these processes is negatively affected in any way, there is an immediate influence on the other processes and the biogas process can become unstable. Process monitoring can help to understand what happens in a biogas plant and help to maintain a stable process. In many cases, a strongly inhibited microorganism population or a total crash of the whole plant can have severe financial consequences for the biogas plant operator. In general, process monitoring can help to: (i) give an overall picture of the biogas process, (ii) identify upcoming instabilities in anaerobic digesters before a crash happens and (iii) accompany a successful start-up or re-start of a plant.

Typical biogas monitoring parameters can be divided into three categories. First, there are parameters which characterise the process. These are feedstock quantity and composition, biogas production and composition, fermentation temperature, total solids concentration, ammonia nitrogen concentration and pH. Other parameters can indicate in advance if a process instability is upcoming. These are: volatile fatty acids, alkalinity ratio, hydrogen concentration, redox potential or other complex monitoring parameters. Last but not least, there are variable process parameters which to some extent can be varied by the plant operator before a process imbalance occurs, such as organic loading rate or hydraulic retention time.

The microbial community within each digester is able to adapt to changes to a certain extent, therefore, it is often not possible to state definitive stability limits for monitoring parameters. In practice, every biogas plant develops its own unique process conditions. As a result there is no single value for each process parameter that can be referenced to all plants. Nevertheless, it is possible to give a certain recommended range for most monitoring parameters. For each plant it is important that values of relevant process parameters, such as temperature and pH, are established during stable operation. By recording these process parameters over the life of the plant, any change from "normal" can be identified quickly. In practice, many process imbalances can be avoided by good operation practice, where adequate training of the operating staff is very important. General recommendations to keep up a stable process are: continuous feeding rate, consistent feedstock mix (e.g. manure and biowaste), gradual and careful change of feedstock mixes when required, avoid temperature changes, keep intervals and intensity of stirring or agitating constant and continuous process monitoring and control.

### Further informations:

Drosig, Bernhard (2013): Process Monitoring in biogas plants. IEA Bioenergy – ISBN 978-1-910154-02-1

URL: [http://www.iea-biogas.net/files/daten-redaktion/download/Technical%20Brochures/Technical%20Brochure%20process\\_monitoring.pdf](http://www.iea-biogas.net/files/daten-redaktion/download/Technical%20Brochures/Technical%20Brochure%20process_monitoring.pdf) (2015-02-10).

## On-line monitoring and process control of AD plants



Jens Bo Holm-Nielsen (Aalborg University)  
Contact: jhn@et.aau.dk

**Keywords:** AD-process control, on-line technologies, monitoring, control, on-line monitoring, Near InfraRed spectroscopy (NIR), anaerobic digestion (AD), volatile fatty acids (VFA), Process Analytical Technologies (PAT), representative sampling, Theory of Sampling (TOS), biogas production, biorefinery concepts, biofuels analytics and sampling

Biorefineries are evolutionary concepts of integrated biomass conversion processes which open an industrial paradigm change, shifting from a centralized way of production into a decentralised situation, adapting the distributed character of feedstocks. The technology is based on flexible more or less non-sterile fermentation processes to form basic and intermediate organic chemicals and fuels, which can be integrated into producing a cascade of relevant products. Nowadays decentralised biorefineries, including the last two decades of biogas developments, are the platform and inspiration for tomorrow's projects.

Various pre-treatment steps for the next generation of biorefineries are under consideration including wet milling, thermal hydrolysis, enzymatic pretreatment. Advanced Process Analytical Technologies (PAT's) also have to be integrated into the biorefinery concepts in the future in order to ensure optimal process monitoring, optimization and control for the purpose of increasing the yields and optimizing the biorefinery product portfolio.

Focus has been on biogas plant implementation of Process Analytical Technologies (PAT) to develop chemometric multivariate calibration and prediction models for on-line monitoring and control of the anaerobic digestion process in a re-current loop modus.

Most studies reported in literature have investigated near infrared spectroscopy (NIR) in laboratory-scale or minor pilot biogas plants; not many studies have examined on-line process monitoring of full-scale plants. In all realistic scales it is necessary to obtain a fairly constant level of volatile fatty acids (VFA) concentration which leads to a stable biogas production. Uncontrolled VFA contents have a significant negative impact on biogas production, and VFA concentrations should not exceed 5-6000 mg/l. On-line control and management of VFA concentration levels are critical in order to be able to speed up or slow down the anaerobic digestion processes which produce the desired sustainable bioenergy for combined heat and power production. Biogas contains the key as a controllable and manageable base load energy supplier compared to wind or solar energy.

By calibrating NIR-spectra to laboratory VFA reference concentrations at the experimental locality, it was possible to develop calibration models by Partial Least Squares (PLS) regression, notably acceptable to very good prediction models for total VFA as well as for all essential individual acids. The average statistics assessing prediction performance, accuracy (slope-value) and precision: correlation ( $r^2$ ), were both 0.92.

Biogas and biorefinery plants have the capability of using multiple heterogenous biomass substrates for production of various value-added products. Moreover, by-products generated in one part of the biorefinery can serve as substrates in another process module. This is apparent for instance when integrating production of liquid biofuels with biogas. The digested biogas substrate can be separated into a liquid fraction, which is an efficient organic fertiliser rich in Nitrogen and Potassium nutrients and a solid lignocellulosic Phosphorous fibre fraction suitable for harsh treatment, enzymatic hydrolysis, and subsequent fermentation, yielding biofuels. The term waste product is therefore non-existing in the context of biorefining and bioenergy systems.

### Further information:

[www.vbn.dk](http://www.vbn.dk) - search Jens Bo Holm-Nielsen  
[www.et.aau.dk](http://www.et.aau.dk)  
[www.esbjerg.aau.dk](http://www.esbjerg.aau.dk)

## Biogas plant process monitoring in China: Several constraints and solutions



Renjie Dong, Shubiao Wu, Jianbin Guo, Wei Qiao (China Agricultural university, Beijing)  
Contact: rjdong@cau.edu.cn

**Keywords:** Biogas Plant, Monitoring, Constraint, China

“China Agricultural Statistics 2012” (Ministry of Agriculture, China) shows as of the end of 2012, China has had 91.9 thousand biogas plants, including 338 biogas plants for the treatment of industrial waste, and 5246 large, 9767 medium and 76588 small scale biogas plants taking agricultural residues as feedstocks, with a total volume of 14.334 million m<sup>3</sup> and annual biogas production of total of 1.98 billion m<sup>3</sup>. Compared with Germany, in China, the average single volume is smaller, and the annual biogas production per unit plant volume is lower. This would indicate that the recent fast development of biogas plants in China is more embodied in the increase in the number (Figure 1).

Many reasons lead to the above results, but mainly are:

- (1) the managers and on-site technicians are not well trained,
- (2) as the raw material matrix is more complex than in Germany, the pretreatment of raw materials could not meet the optimal requirements as the anaerobic fermentation feedstock,
- (3) the fermentation process parameters control is inaccurate and not timely, including feeding time and quality, fermentation temperature, stirring, the biological desulfurization oxygen, etc.
- (4) the fermenter rancidity problem happens quite commonly which stops the biogas plants.

A reasonable automatic control and process monitoring of biogas plants, based on the clear know-how, is the important guarantee to and an urgent demand for the improvement of the current biogas plants and for further healthy development of biogas in China.

The automatic control technology has been used on Chinese biogas plants in different extent. At present, the automatic control is mostly based on relay logic control with high complexity and low reliability. This does not meet the requirements of convenient management and professional automatic control.

Recently in China, many studies have been conducted by IT technicians on the technology development of programmable control system based on a Programmable Logical Controller, to automatically monitor and control the fermentation system, feeding system, biogas purification system and methane storage system. Some control systems have been implemented onto biogas plants.

However the main constraints of the auto-monitoring/controlling application in biogas plants development are the misunderstandings of each components of the biogas plant system:

- (1) the amount, organic matter content, and frequency of feeding per day,
- (2) the temperature of the points in the materials, and the allowed variation of the temperatures, based on site-specific optimization of overall biogas plants thermal insulation,
- (3) the speed and frequency of stirring,
- (4) the pH and other biological/chemical parameters based on in-dept understanding of anaerobic process.

For the last point of the above mentioned 4 points, to explore the testing method of volatile fatty acid concentration and bicarbonate alkalinity of the anaerobic fermentation process, based on Nordmann titration method and LabVIEW, China Agricultural University research team has innovated an integrated equipment (Figure 2), to achieve pH automatic calibration, automatic titration and rehydration, to generate real-time titration curve and data processing. It could store lots of test results. The device allows for precise determination of VFA and TIC concentration, and estimation of the actual acetic acid concentration. The ratio of VFA/TIC can realize the early warning of the anaerobic fermentation system.

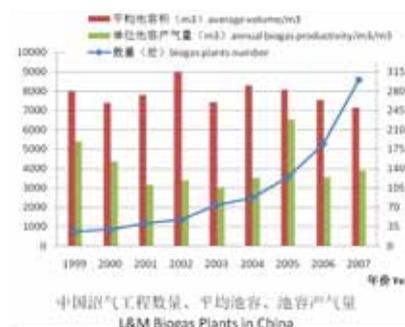


Figure 1: The large and medium scale biogas plants in China



Figure 2: The VFA/TIC Automatic Titrator

## Biomethane potential test – parameter, results and evaluation



Hans Oechsner (State Institute of Agricultural Engineering and Bioenergy, University of Hohenheim)  
Contact: hans.oechsner@uni-hohenheim.de

**Keywords:** Residual methane potential, emission potential, evaluation of biogas process

Today, the conversion of organic materials to biogas is a common resource for electric energy and gaseous fuel supply. For an efficient and environmental friendly biogas process, it is necessary to ensure a maximum substrate utilization which includes a high methane yield and a good degradation rate of the feeded substrate. This efficiency of the biogas process is especially important by using of energy crops as feedstuff, because their production causes costs and demands a lot of different input to grow them.

The currently used biogas plants are very different designed, but they have to be optimal adapted to the used substrate. In Germany, mostly CSTR-Systems are used, sometimes arranged in series with different hydraulic retention time (HRT) and organic loading rate (OLR). In some cases, fibrous substrate is pretreated. New systems have to have a minimum HRT of 150 days, to reduce uncontrolled methane emissions. Some plants have a very long retention time and also the storage tank for digestate is covered gas tight.

For the control of the efficiency and stability of the biogas system, mostly the concentration of volatile fatty acids in the digesters are measured and also the buffer capacity. A very good parameter for evaluation of the quality of the anaerobic system is the residual methane potential. It is defined in the VDI-guideline 4630. With the residual methane potential can be measured, which biogas- or methane volume can be lost with the digestate, leaving the last covered digester. This is a very good way, to get information about the efficiency of the process and to decide, if it is economically worthwhile, to optimize the system.

The residual methane potential can be analyzed at a psychrophilic temperature (20 °C), to get information about the environmental relevant, uncontrolled methane losses over the digestate. To get more information about the maximal potential of methane losses, the digestion will be operated at a temperature of 37 °C. For a good result, it is very important, to take a representative sample of the last gas tight covered digester or storage tank. This sample will be put with three replicates into the laboratory batch digester and will be incubated over the explained temperatures for duration of 60 days. Parallel some data of the biogas plant like volume of digesters, mass and quality of used substrates, produced biogas, produced electricity have to be collected. The specific methane yield of the digestate will be multiplied by the daily digestate mass to get the potential. The potential will be set in relation to the average methane volume which is already produced in the digesters during the last 6 months. This quotient in % gives information about the efficiency of the process and about the environmental risk.

In a full scale study, the State Institute of Agricultural Engineering and Bioenergy analyzed samples from each digester and digestate storage from 25 biogas systems in a winter and in a summer-test. Also chemical parameters of the samples were analyzed. The results will be shown in the presentation. The enclosed figure 1 shows the residual emission potential. It documented, that the most biogas plants have a very low emission potential. Only in cases of too short HRT, high OLR, suboptimal substrate structure, the emission risk was higher.

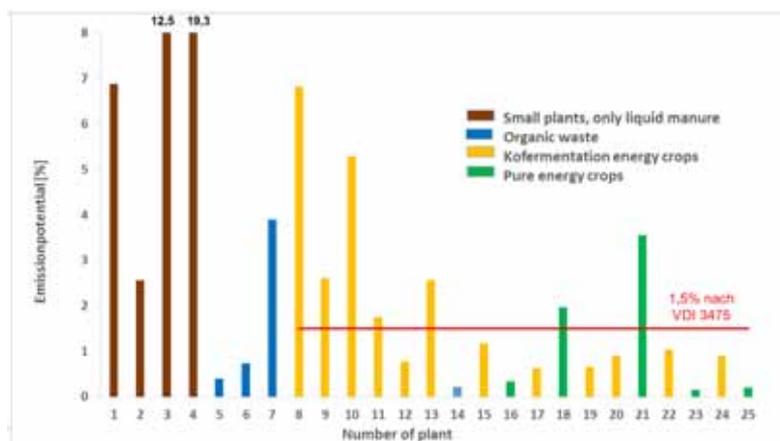


Figure 1: Emission potential of 25 biogas plants, analyzed at a digestion temperature of 20°C with different digester design and different substrates.

### Further information:

Ruile, S.; Schmitz, S.; Mönch-Tegeeder, M.; Oechsner, H. (2015): Degradation efficiency of agricultural biogas plants - A full-scale study. In: *Bioresource Technology*, 178, pp. 341-349

## Reliability of analytical measurements in anaerobic reactors: Case study for volatile fatty acids (VFAs)



Francisco Raposo (University of Sevilla)  
Contact: [fraposo@cica.es](mailto:fraposo@cica.es)

**Keywords:** analytical performance, chromatography, interlaboratory study, volatile fatty acids

### Introduction

The VFAs concentration can be considered as the most useful indicator for monitoring and control of anaerobic reactors. There are a large number of analytical methods available for their quantification, some of them giving the total organic acidity content (TVFAs). However, TVFAs does not provide sufficient information about the overall status of the biochemical process. Then, individual components have been considered of particular and high interest for an early warning indicator for process failure. By this way, chromatographic techniques such as GC and HPLC are capable of separating the individual components and provide appropriately the quantitative determination of VFAs profile. But, what we know about the quality of results dealing to VFAs measurement by chromatography techniques? This question is difficult to answer because to date information about the external performance of laboratories for these analyses has not been published.

### Objective

To try to increase the knowledge about VFAs measurements, an international interlaboratory study was carried out among laboratories providing services in the anaerobic digestion research field. The mean goal was to assess the degree of measurement equivalence thought the analytical performance obtained from international peer laboratories.

### Materials & methods

Two different samples (reference materials-RM) were prepared to analyze the organic acids, from C2 to C7. RM A can be considered as a blind calibration standard for checking the calibration curves at low concentration level. RM B was conceived as a synthetic material to simulate a sample from an anaerobic reactor with most of analytical parameters at medium-high concentration levels.

A total of 25 laboratories, using both chromatographic techniques with a broad spectrum of experimental conditions and different sample preparation procedures, reported their analytical results.

The statistical treatment of analytical performance was carried out based on the z-score values.

For the interpretation of results:  $|z\text{-score}| \leq 2$ , is acceptable;  $2 < |z\text{-score}| \leq 3$ , is doubtful;  $|z\text{-score}| > 3$ , is unacceptable.

In addition to the individual z-score values for each individual VFA, the overall evaluation in a single figure is also important. By this way, a 75 - 90 % can be considered as an appropriate overall pass rate (OPR). Therefore, at the individual laboratory level, a 75 % OPR might be seen as a minimum target that should be achieved for routine analysis by laboratories involved in the analysis and control of VFAs.

### Results

The results demonstrated that several laboratories involved in the monitoring of VFAs had problems in providing good results. For a total of 311 determinations, 137 (44 %) were considered acceptable, 148 (48 %) unacceptable and 26 (8 %) doubtful. Relating to the degree of analytical difficulty of different compounds, none of individual VFAs achieved the satisfactory trueness criterion (75 % of z-scores considered acceptable). Despite of the high prevalence in anaerobic reactors, the major analytical problem was found in C2 (acetic acid) measurement, with only 27.7 % of z-score values considered as acceptable. The main causes of poor analytical performance for this interlaboratory study were human errors and inadequate calibration procedures.

### Further information:

The full results of this interlaboratory study were published in the journal Trends in: *Analytical Chemistry* (TrAC), 51, pp 127-243 (2013).

## NIR for determination of biogas yields: state of the art



Alastair James Ward (Aarhus University)  
Contact: [alastair.ward@eng.au.dk](mailto:alastair.ward@eng.au.dk)

**Keywords:** NIR, biogas yield, BMP

The use of near infrared spectroscopy (NIR) for determination of biogas and/or methane yields has received increasing interest recently. The accepted method of measuring the potential yield, the Biochemical Methane Potential test (BMP), involves the digestion of test substrates in an inoculum sourced from an active biogas reactor. The details of the method vary, despite attempts at standardisation. However, all the various methods are time consuming, with digestion times from 30 to 100 days and they are also rather labour intensive. The use of NIR to predict gas yields can potentially cut this time to a matter of minutes or even be implemented as an online measurement, but there are many obstacles to overcome before the method can completely replace the BMP assay.

The choice of variability within samples upon which NIR models have been built is a subject for debate: Lesteur et al (2011) used a very broad range of materials from lignocellulosics to municipal solid wastes, whereas Raju et al (2011) limited the sample set to meadow grasses only and Jacobi et al (2012) used maize silage as a single substrate. Using a broad range of substrates could make a seemingly better model (in terms of R<sup>2</sup> in particular) than a single substrate due to the fact that the range of gas yields can be very small in the latter, but the use of a number of models based on single or grouped substrates may provide more useful information to biogas plant operators.

Using fresh (wet) material for measurement poses the problem of reduced representativity. This applies to both the NIR and the BMP measurements as it can be very difficult to ensure a representative sample in the small amounts typically used for the BMP assay and also when presenting samples to the spectrometer. Many studies have used substrates that have been dried and ground to a fine powder before measurement. This greatly improves substrate homogeneity for better NIR measurement but is also time consuming and to attain true pairwise comparison the BMP test must also be conducted with substrates treated in an identical manner, and such treatments may affect the overall yield.

However, what could be the greatest challenge is the reference method itself: the use of inoculum from different reactors or even from the same reactor, even with consistent substrate feeding but collected at a different time, can lead to differences in both the ultimate methane yield (B<sub>0</sub>) and the kinetics of digestion. Also, the use of inoculum not adapted to the substrate can affect the data considerably although this factor is more easily avoided. So to really reap the benefits of NIR for yield prediction we need to look more closely at our reference method first.

Further information:

[http://pure.au.dk/portal/da/persons/id\(f9a4b6c0-e9da-4440-8e10-e4b3b598bb79\).html](http://pure.au.dk/portal/da/persons/id(f9a4b6c0-e9da-4440-8e10-e4b3b598bb79).html)

## Monitoring of fugitive methane emissions from an Austrian biogas plant



**Marlies Hrad, Martin Piringer, Marion Huber-Humer** (University of Natural Resources and Life Sciences Vienna, Austria)  
Contact: marlies.hrad@boku.ac.at

**Keywords:** fugitive emission, methane, open-path tunable diode laser, inverse dispersion modelling, anaerobic digestion

The preceding discussion has described biogas and the use of its energy content as a strategy to substantially mitigate anthropogenic greenhouse gases (GHG). However, the positive environmental benefit depends on the level of methane ( $\text{CH}_4$ ) losses during production. Robust and reliable monitoring techniques must be developed to quantify the fugitive emissions in order to reflect and improve the plant-specific process efficiency. One promising method is the inverse dispersion technique that uses path-integrated concentration measurements (e.g. Open-Path Tunable-Diode-Laser-Spectroscopy [OP-TDLS]) downwind from the source (and wind and stability information) to infer the emission rate from the whole plant or component emissions. Examples of their application include the determination of fugitive emissions from dairy and swine farms [1, 2], landfills [3], agricultural biodigesters [4, 5] and land-application of slurry [6].

Within the research project "KLIMONEFF" the inverse dispersion technique using an OP-TDLS system and an dispersion model (LASAT) [7] was applied on an Austrian biogas plant (2 x 526 kW electric energy, processing energy crops and pig manure) over the period of more than one year to examine its suitability to determine  $\text{CH}_4$  emissions from the whole plant and component emissions (e.g. open digestate storage tanks, silage storage). In addition, the sensitivity of the inverse dispersion technique to assumptions of source geometry at different meteorological conditions and varying measurement locations using synthetic experiments in a real-world biodigester setting was investigated.

Whole plant emissions from the study site were determined between October 2011 – March 2013, coinciding with the periods before and after digestate were removed from open storage tanks. During filled tanks an average emission rate of 7.2 kg  $\text{CH}_4$ /h (approx. 4 % of the  $\text{CH}_4$  production) was determined, while 5.4 kg  $\text{CH}_4$ /h of emissions (approx. 3 % of the  $\text{CH}_4$  production) were quantified after the tanks had been emptied. There is a good congruency between the average  $\text{CH}_4$  emissions inferred by direct measurements at the open digestate storage tanks (approx. 1.2 % of the  $\text{CH}_4$  production) using the multi-source reconstruction applied in [5] and the difference between whole plant emissions determined by full and emptied tanks (approx. 1 % of the  $\text{CH}_4$  production).

The applied method combining an inverse dispersion technique with OP-TDLS proved to be a suitable tool to monitor and quantify whole plant and component emissions from biogas plants. It could be observed that besides the operation mode (e.g. filling level and agitation of the open tank for digestate storage, maintenance), also the meteorological conditions such as wind speed and solar radiation (e.g. heat flux) can in principle affect the emission rate.

The minimum distance of the measurement from the source area, for which the homogenous assumption is valid, is dependent on the stability of the atmosphere and furthermore on the height of the potential sources above the ground. In case of the study site, this distance is about 50 m. However, it should not exceed 150 m in order to meaningfully measure concentration rise.

### Further information:

- [1] Flesch, T., Harper, L., Powell, J.M., Wilson, J., Inverse-dispersion calculation of ammonia emissions from Wisconsin dairy farms, *American Society of Agricultural and Biological Engineers* 52 (2009), pp. 253-265.
- [2] VanderZaag, A.C., Flesch, T.K., Desjardins, R.L., Baldé, H., Wright, T., Measuring methane emissions from two dairy farms: Seasonal and manure-management effects, *Agricultural and Forest Meteorology* 194 (2014), pp. 259-267.
- [3] Zhu, H., Letzel, M. O., Reiser, M., Kranert, M., Bächling, W., Flassak, T., A new approach to estimation of methane emission rates from landfills, *Waste Management* 33 (2013), pp. 2713-2719.
- [4] Flesch, T.K., Desjardins, R.L., Worth, D., Fugitive methane emissions from an agricultural biodigester, *Biomass and Bioenergy* 35 (2011), p. 3927-3935.
- [5] Hrad, M., Piringer, M., Kamarad, L., Baumann-Stanzer, K., Huber-Humer, M., Multisource emission retrieval within a biogas plant based on inverse dispersion calculations—a real-life example, *Environmental Monitoring and Assessment* (2014), pp. 1-12.
- [6] Carozzi, M., Ferrara, R.M., Rana, G., Acutis, M., Evaluation of mitigation strategies to reduce ammonia losses from slurry fertilisation on arable lands, *Sci Total Environ* 449 (2013), pp. 126-133.
- [7] Janicke Consulting, Dispersion Model LASAT Version 3.2 Reference book, (2011).

## Residual energy potentials of digestates



Michael Tauber, Ludek Kamarád, Günther Bochmann, Roland Kirchmayer (JOANNEUM RESEARCH Forschungsgesellschaft mbH, Graz)  
Contact: michael.tauber@joanneum.at

**Keywords:** Digestate, Residual energy potential, methane emissions, measures

In this study digestates from 16 agricultural and 6 waste treating biogas plants were analysed regarding to the residual biological methane potential (RBMP) as well as other chemical parameters such as total solids, volatile solids, total nitrogen (TKN), ammonia concentrations (NH<sub>4</sub>-N) and volatile fatty acids (VFA). Temperature profiles have been recorded. Samples taken more often from one spot show variations of the parameters corresponding to the actual status which is influenced by certain factors, such as:

- (a) homogeneity of the fermenter,
- (b) organic load,
- (c) process operation,
- (d) weather / rain,
- (e) homogeneity of the sample itself.

Waste treating biogas plants show residual biological methane potentials (RBMP) two times higher than agricultural biogas plants. Filling levels of the storage tanks and the temperature of the digestates have an influence on the climate relevant methane emissions. The VFA content correlates with the RBMP only in case of the waste treating biogas plants. The methane losses emitted by digestates leaving the closed system are in the range of 1 – 2 % based on the annually produced methane amounts.

Measures to minimize the potential methane emissions are: digestates in the opened manure tank should be as cold as possible and of a low volatile solid content, closed digestate tanks (gas tight) are recommended if the tanks are well insulated.

### Further information:

This presentation is based on parts of the final report "KLIMONEFF - Klimagasmonitoring zur Optimierung der Energiebilanz und Verfahrenseffizienz bei Biogasanlagen", (<http://www.klimaaktiv.at/erneuerbare/biogas/klimoneff.html>) which was supported by FFG – Klima+Energie Fonds.

<http://www.joanneum.at/resources>

## Methane emissions from biogas plants - methods and equipment, results, operational states



Torsten Reinelt (Deutsches Biomasseforschungszentrum gemeinnützige GmbH)  
Contact: Torsten.Reinelt@dbfz.de

**Keywords:** Operational methane emission, leakage

The ecological aim of biogas production is the reduction of greenhouse gases in the energy sector. The life cycle assessment from the biogas technology is strongly affected by methane emissions. Methane actually has a Global Warming Potential of 28 (Myhre et al., 2013). Typical agricultural biogas plants have a few methane emission sources including diffuse sources like leakages as well as time-variant sources like operational methane emissions from pressure relief vents (PRV). This paper describes the state of technology to monitor these methane emission sources.

In the German biogas sector have been established different methods and systems for the verification of impermeability to gas and biogas leakage detection. These terms have to be distinguished. The confirmation of impermeability to gas is regulated by the German TRBS (Technical Rule for operational safety) and additional rules from the gas industry, e.g. DVGW (German association for gas and water) G 465-4 and G469 A4 (SVK Biogas, 2013). There is described the use of gas analyzers and foaming agents for the correct evaluation of gas leakages. Areas exposed to explosion hazards like biogas plants have to be periodically checked every three years according to the German Industrial Safety Regulation. Furthermore the use of alternative systems has

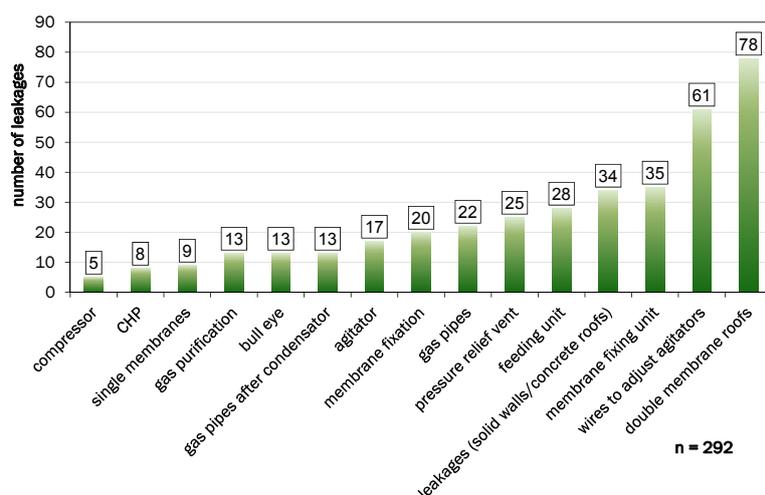


Figure 1: Number and location of detected leakages (modified from Clemens, 2014)

risen including infrared (IR) cameras and hand held methane lasers allowing remote sensing of leakages from plant components which are difficult to access. These systems use the absorption maxima of methane in the IR spectrum. An IR-camera visualizes the methane emission as grey or false color cloud. The detailed metering principle of this system is explained in Schreier (2011). There are a few experiences about the occurrence of leakages on gas-bearing plant components from biogas plants. By means of ten investigated biogas plants Schreier (2011) showed that biogas losses from leakages are relevant. Eight plants had an overall number of 22 leakages and seven of them were evaluated as serious leakages. Clemens (2014) investigated a lot of single biogas plants and evaluated the data concerning the occurrence of leakages. The results are shown in Figure 1.

Additionally to unknown leakages operational methane emissions have to be considered, too. This refers especially to time-variant emissions from the PRVs of the biogas storages. Each gastight tank or biogas storage has to be equipped with at least one PRV for positive and negative pressure each to avoid damages on the tanks, but any triggering results in the emission of raw biogas. The time-variant characteristic of the methane emissions from a PRV needs a continuous monitoring of triggering to determine the cause and to initiate counteractions. Mechanical PRVs use generally a high opening pressure (> 10 hPa). Then occurring triggering events should be indicated by a pressure loss. Hydraulic PRVs use normally a lower pressure level (< 10 hPa), because they are often installed on foil roof digesters. In practice operation the methane emission from a PRV is unknown. So far at five plants in Germany the triggering of PRVs was determined by using a photo sensor and counting the single impulses. At three plants the PRVs have shown triggering during the measurement period (297 to 484 days). Two of them have been activated during three to six days and the 3rd only once during 70 days (Lehner et al., 2010). An alternative system to count the single triggering events is an inductive proximity sensor, which responds to a heaving guiding tube (Köberle, 2015). However, this data do not provide a clue to the emitted methane volume.

A method applied by the DBFZ (Reinelt et al., 2015) determines the emitting biogas volume flow with an explosion protected vane anemometer and temperature sensor which are implemented in the exhaust pipe of the PRV. The biogas volume flow is converted to standard conditions (0 °C, 1 013.25 hPa, dry) and the methane content in the biogas is taken from a stationary biogas analyzer or is directly measured at the investigated tank. In addition to the technical refitting of the investigated PRV, a large amount of operational data has to be collected (Operating pressure, CHP load, etc.). The first investigations on full scale plants showed that the permanent monitoring of a PRV is the most suitable option to quantify the operational methane emissions from PRVs. In Figure 1 is exemplified the biogas volume flow, biogas temperature as well as air temperature and air pressure from an entire day.

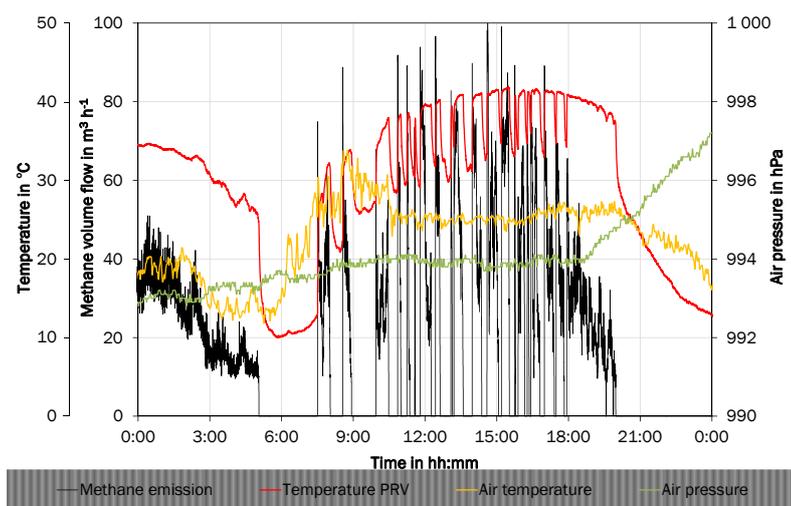


Figure 2: Diurnal variations of an investigated PRV and the atmospheric conditions (modified from Westerkamp et al., 2014)

Figure 2 shows clearly the time-variant characteristic of the methane emissions from a PRV. After each triggering event the temperature inside the exhaust pipe of the PRV decreases gradually. Especially if positive and negative pressure is compensated by the same exhaust pipe (e.g. at a surge tank), the registered temperature from the emitted biogas validates the data from the vane anemometer. By integration of the response curve (black line) the overall emitted methane volume from a triggering event is determined. The most common operational state causing methane emissions from PRVs is the scheduled or non-scheduled outage of the CHP (combined heat and power) unit. Scheduled reasons are typically maintenance periods and non-scheduled reasons include defects, overheating in the summer or changes in the biogas production (Westerkamp et al., 2014).

If triggering events can be related to specific operational states (e.g. outage of the CHP, maintenance periods of the process technology), the methane emission potential will be possibly estimated from documented operational data (e.g. time periods of CHP outages or run-time of the biogas flare) without additional measurements at the PRV.

## Literature:

- Clemens, J. (2014): Erfahrungen bei der Untersuchung von Biogasanlagen auf Gasdichtheit (Diffuse emissions from biogas plants – practical experience). Report language: German. In: *Gwf Gas|Erdgas* Vol. 3 (2014), pp. 128 – 130. Link: [http://www.bonalytic.de/cps/bonalytic/ds\\_doc/GE\\_03\\_2014\\_Clemens.pdf](http://www.bonalytic.de/cps/bonalytic/ds_doc/GE_03_2014_Clemens.pdf) (Accessed: 2015-02-16).
- Myhre, G., Shindell, D., Bréon, F.-M., Collins, W., Fuglestedt, J., Huang, J., Koch, D., Lamarque, J.-F., Lee, D., Mendoza, B., Nakajima, T., Robock, A., Stephens, G., Takemura, T., Zhang, H. (2013): Anthropogenic and Natural Radiative Forcing, in: *Climate Change 2013: The Physical Science Basis. Contribution of Working Group I to the Fifth Assessment Report of the Intergovernmental Panel on Climate Change*. [Stocker, T.F., D. Qin, G.-K. Plattner, M. Tignor, S.K. Allen, J. Boschung, A. Nauels, Y. Xia, V. Bex and P.M. Midgley (eds.)]. Cambridge University Press, Cambridge, United Kingdom and New York, NY, USA.
- Köberle, E. (2015): Link: <http://www.biogaskontor.de/index.php?id=28> (Accessed: 2015-02-16).
- Lehner, A., Effenberger, M., Gronauer, A. (2010): Optimierung der Verfahrenstechnik landwirtschaftlicher Biogasanlagen (Optimizing of the process technology from agricultural biogas plants). *Schriftenreihe Bayerische Landesanstalt für Landwirtschaft*. S. 75 – 76. Link: [http://www.lfl.bayern.de/mam/cms07/publikationen/daten/schriftenreihe/p\\_38044.pdf](http://www.lfl.bayern.de/mam/cms07/publikationen/daten/schriftenreihe/p_38044.pdf) Report language: German. (Accessed: 2015-02-16).
- Reinelt, T., Liebetrau, J., Nelles, M. (2015): Operational methane emissions from pressure relief vents on two agricultural biogas plants. Submitted to the "International Conference on Solid Waste 2015 – Hong Kong" from 19 – 23 May 2015.
- Sachverständigenkreis (SVK) Biogas (2013): Information zur Überprüfung der Gasdichtheit und Leckagemittlung insbesondere mittels Messgeräten an Biogasanlagen (Information to the use of analyzer for survey of impermeability to gas and leakage detection at biogas plants). Report language: German. Link: [http://www.das-ib.de/mitteilungen/SVK\\_Dichtheit%20-%20Leckage%20Pr%C3%BCfungen\\_16VII2013.pdf](http://www.das-ib.de/mitteilungen/SVK_Dichtheit%20-%20Leckage%20Pr%C3%BCfungen_16VII2013.pdf) (Accessed: 2015-02-16)
- Schreier, W. (2011): Untersuchung von Gasleckagen bei Biogasanlagen (Investigation of gas leakages from biogas plants). Report language: German. Report No.: B66711 IR\_BGA Sachsen. (ed.): Sächsisches Landesamt für Umwelt, Landwirtschaft und Geologie. Link: <https://publikationen.sachsen.de/bdb/artikel/15246> (Accessed: 2015-02-16).
- Westerkamp, T., Reinelt, T., Oehmichen, K., Ponitka, J., Naumann, K. (2014): KlimaCH<sub>4</sub> – Klimaeffekte von Biomethan (Climate effects of the biomethane economy, DBFZ Report Nr. 20). Report language: German. DBFZ, Leipzig – ISSN 2197-4632

## Power on demand: Optimized feeding by predictive control systems



Jens Bischoff (EnviTec Biogas AG)  
Contact: j.bischoff@envitec-biogas.de

**Keywords:** Feedcontrol, Redox measurement

Due to rising substrate prices and higher official regulations it has become more and more difficult to earn money with biogas. One way to improve the economic efficiency is the delivery of power on demand in the German electricity market which causes technical adaptations of the whole biogas plants. To reduce the associated investment costs (e.g. for a larger gas storage) and also to maximise the utilisation of these installations the required biogas production has to be predicted and controlled effectively.

In 2009, EnviTec has developed a flexible system called Feedcontrol which adjusts automatically the feeding amounts according to the pressure in the gas storage. Later on, as a consequence of the mentioned changes in the market, EnviTec has conducted additional research how to predict the required biogas production on base of data from the gas storage, the substrate quality and the load curve of the CHP.

First of all, a "GasHmeter" (measurement of the hydrostatic pressure in a corrugated oil-filled hose, fixed on the roof) has been tested to indicate the state of the gas storage. The results show that this height measurement brings much more suitable data than other systems such as cable or pressure sensors.

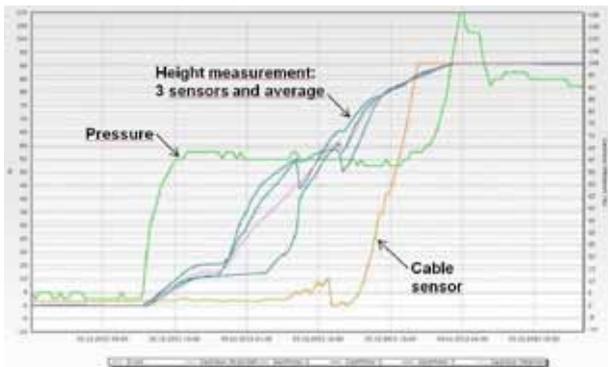


Figure 1: Gas storage measurement

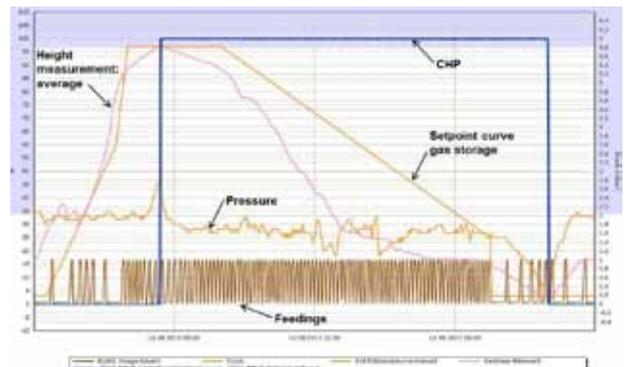


Figure 2: Regulated feeding and power on demand

Afterwards, both feeding quality and load curve have also been integrated into the Feedcontrol system.

Practical tests have proven that this invention is able to optimise the biogas production and to minimise the needed amounts of substrate. No overloading or surplus gas production has been observed.

To avoid bad consequences for the economic efficiency by overloading and biological disturbances EnviTec has made tests to control the biological process in the digester via the measurement of the redox potential (ORP). Therefore, different ORP-electrodes have been installed and long-term tests were executed in several biogas plants. The data have shown that the ORP-measurement is suitable to indicate the state of the biology. The optimum level seems to be between -500 and -540 mV under these circumstances.

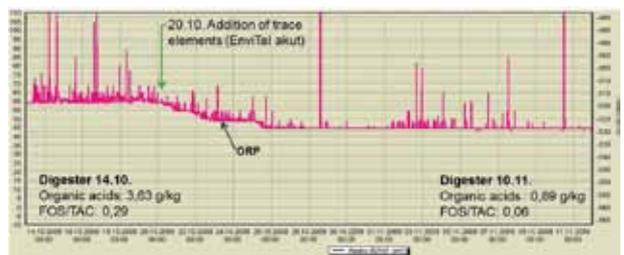


Figure 3: Redox measurement at a biogas plant with trace element deficiencies

Furthermore this system seems to show biological disturbances much earlier than other techniques. E.g. the shift in pH as a consequence of acidification occurs several days or even weeks later.

Further research has to be done to simplify the interpretation as the tests have demonstrated that the long-term monitoring is sometimes difficult and requires time and know-how. Therefore EnviTec tries to develop a new evaluation procedure to interpret the data automatically which allows the operator to see directly the status of the biology.

Further information:  
[www.envitec-biogas.de](http://www.envitec-biogas.de)

## Process and Machinery Monitoring in Thöni Biogas Plants



Urban Zell (Thöni Industriebetriebe GmbH)  
Contact: [urban.zell@thoeni.com](mailto:urban.zell@thoeni.com)

**Keywords:** Process monitoring, machinery, safety, analysis

The Thöni group is introduced by the speaker with special focus on the environmental and energy engineering part (Umwelt Energietechnik UET) of the group.

The types of biogas plant offered by Thöni are introduced with special focus on the application for the plant types. The safety process and monitoring system is briefly explained as well as the machinery monitoring. Furthermore the speaker talks about the feeding system of the biogas plants as well as the accounting system for input and output values.

After that the gas analysis system and the interface to the rest of the plant are introduced.

Further information:

[www.thoeni.com](http://www.thoeni.com)

## Minimising mixing expenditure in biogas plants



Kay Rostalski (KSB Service GmbH)

Contact: [kay.rostalski@ksb.com](mailto:kay.rostalski@ksb.com)

**Keywords:** mixing, flow acceleration, turbulence, hydraulic system, AD plants

With regard to biogas plants, the term “mixing” refers to a number of sometimes very complex processes. To prevent the formation of floating sludge, for example, high turbulence needs to be generated to saturate structural matter with liquid. Turbulence, however, should be localised in order to minimise mixing expenditure. One good way to effectively and significantly reduce the necessary extent of turbulence is to precomminute the substrate. Other measures designed to wet the substrate prior to its introduction also have positive effects on the mixing effort by working against buoyancy. At the same time, the wetted structural matter must be kept so well agitated that it cannot accumulate at the surface. For this, the substrate in the reactor needs to be distributed as evenly as possible throughout the entire volume of the reactor by means of global bulk flow. This objective requires a different type of mixing function, namely flow acceleration.

Other mixing functions in addition to those just mentioned include, for example, inducing local shear velocities to help separate bubbles of gas from the liquid as a prerequisite for optimal bioconversion. Another good example is the prevention of sedimentation, which also requires a certain degree of turbulence.

Simply said, a distinction can be drawn between two different kinds of mixing processes that take place in biogas plants and can be more or less markedly pronounced, depending on the task at hand:

1. Flow acceleration
2. Turbulence generation

From this, it follows that no single type of hydraulic system will be able to fulfil all of the diverse mixing functions required, or, if so, then only as an inferior technical compromise.

In sum, however, we may conclude that any and all mixing functions require that a certain local velocity, or change in velocity, be induced. How well a given mixing task is fulfilled depends on the employed or required type of hydraulic system, the amount of force being generated in the axial direction (axial thrust), and the corresponding operating conditions. Rule of thumb: thrust is indispensable for good mixing. In addition, it must be possible for that thrust to propagate into all parts of the tank in which flow is supposed to take place.

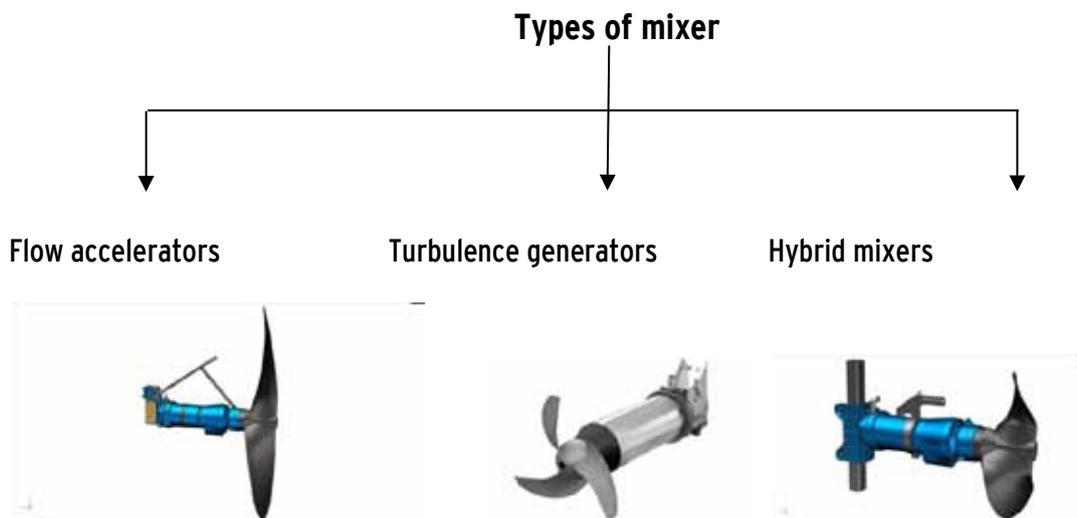
Qualitative assessment of the mean bulk-flow velocity as a function of flow behaviour is a technically challenging endeavour. In many cases, especially when the substrate contains a large amount of structural matter, it is practically impossible, because the sensors are liable to become blocked by stringy material very quickly. In actual practice, magnetic-inductive measuring systems have proven capable of yielding useful results. The utility value of measuring a directional base flow in axial direction must be regarded as limited with respect to the fulfilment of all mixing functions. Digesters and post-digesters with a mean bulk-flow velocity of 8 – 15cm/s, though, usually function quite well.

Nevertheless, importance must be attached to ensuring locally distributed velocities or changes in velocity. Frequently, biogas plants with high volume loads exhibit a pseudoplastic, i.e., shear-thinning, flow behaviour characterised by a distinct limit where the fluid starts to flow. In other words, the fluid begins to flow on exposure to high shear rates, because, from a certain point on, the application of a certain minimum amount of energy overcomes its resistance to flow. This is what enables fulfilment of one or several mixing tasks in the first place. Conversely, it frequently also limits successful mixing to a local scale, sometimes causing submersible mixers in biogas plants to recirculate the thinned material back to their own propeller. One way to avoid that problem is to ensure “correspondence”. This means that, when several mixers are working together, each one makes use of the thinned material from its neighbour(s). Properly positioned and correctly spaced, they prevent backflow and create favourable conditions for an optimal process. Large-volume flow accelerators can play a supporting role in this process.

In sum, then, as far as biogas plants are concerned, the term mixing refers to the inducement of velocity or of changes in velocity, not merely within a localised area, but in all parts of the tank, with as little energy expenditure as possible. The idea is to generate only as much turbulence as necessary, because turbulence always involves major local consumption of energy. The less turbulence, the less energy consumed. On the other hand, directional velocity is no good substitute for real turbulence. During the start-up phase, for example, floating sludge is difficult to contend with - despite the presence of adequate mean velocities throughout the reactor - because the fluid still lacks viscosity and displays a strong tendency to float.

Inversely, an optimal mixing process should be accommodated to the flow behaviour and provide large volume flows with a global bulk flow and sufficient turbulence.

This axiom should always be kept in mind for selecting and combining mixers for any relevant task.



Turbulence generators are mixers with small propellers running at a relatively high speed (300 rpm and above) and a low ratio of thrust and power input. As such, they are of only limited suitability for use in biogas plants, as they tend to be extremely prone to recirculation behaviour, which frequently prevents them from imposing the locally generated gain in velocity on the remainder of the tank. Also, in terms of energy efficiency, they are significantly inferior to both flow accelerators and hybrid mixers.

Flow accelerators are characterised by large, low-speed propellers, high volume flow and very high thrust despite very low power consumption. This makes them especially energy-efficient and able to achieve very good correspondence coupled with fairly adequate turbulence. Consequently, combining a flow accelerator with a hybrid mixer often appears as the option of choice for typical biogas applications.

Hybrid mixers are characterised by hydraulic properties that combine the advantages of a flow accelerator with the turbulence-inducing properties of turbulence generators and can cope with even very critical flow behaviour. In addition to good energy efficiency, they also have impressive depth of penetration and a low tendency to recirculation. Consequently, they are often given preference for plants showing highly critical flow behaviour.

Bottom line: no thrust, no velocity. Flow accelerators, with their very good thrust/power ratios, fulfil that requirement with very little energy expenditure. Both the comminution of structural matter and appropriately tailored loading have very positive effects in that connection. In addition, shear-rate propagation throughout the tank must be ensured in order to achieve a “globally” successful mixing effect.

The generation of directional bulk-flow velocity alone does not suffice to simultaneously achieve all mixing functions. In many cases, the combined effect of appropriate mixer positioning, the combined deployment of different hydraulic systems, and an appropriate level of turbulence will help to minimise mixing expenditure.

## Prediction of biogas yield potential based on the chemical composition: Possibilities and limitations



V. Dandikas<sup>a</sup>, H. Heuwinkel<sup>b</sup>, F. Lichti<sup>a</sup>, J. E. Drewes<sup>c</sup> and K. Koch<sup>c</sup>

a) Institute for Agricultural Engineering and Animal Husbandry, Bavarian State Research Center for Agriculture

b) Department of Agriculture and Food Economy, Hochschule Weihenstephan-Triesdorf

c) Chair of Urban Water Systems Engineering, Technische Universität München

Contact: vasilis.dandikas@lfl.bayern.de

**Keywords:** biogas yield, empirical model, energy crops, regression analysis

The number of agricultural biogas plants increased all over Europe in recent years. In Germany, most of the agricultural biogas plants are running with maize silage as sole substrate or in a mixture with other energy crops or manure. The quality of the feedstock has been identified as an important parameter for maintaining the efficiency of biogas plants. Therefore, a flexible operation regarding the use of different substrates is needed. Determine biogas and biomethane potential (BMP) is crucial for assessing the substrate's quality; however the tests are very expensive in terms of time and costs. The aim of this study was to develop an empirical model based on the chemical composition of different energy plants in order to predict the BMP quickly and reliably.

Forty-one unsystematically and sixty-one systematically collected samples (typically feedstock of agricultural biogas plants) were analyzed, in order to assess the influence of the chemical composition on the biogas yield. Standardized anaerobic batch tests and fodder analysis were carried out to determine the biogas / methane yield and the chemical composition of the samples.

The results have shown that the lignin content and the hemi-cellulose content can be used for biogas yield prediction across energy crops species, with an estimation accuracy of 8 % for the calibration and 10 % for the validation of the model. A Plant-group or plant species specific model could increase the accuracy of the prediction, but the analytical accuracy should be considered.

Round robin tests were carried out to evaluate the accuracy and precision of the fodder analyses. Although, the relative standard deviation for the chemical parameters between laboratories was high enough to render the results between the laboratories incomparable, the repeatability of all laboratories was within the tolerance range. The differentiation on the chemical compounds of different sample could be reflected. Hence, for model development, the data should be collected from the same laboratory in order to minimize the relative estimation error.

However, statistically, the proportion of a chemical compound will be related to its effect on methane yield and not necessarily to its properties. It has to be considered that any change in the content of one compound will always change the content of the others due to the fact that they are recorded in percentage of total solids.

On the one hand, the more variables are used in the model, the higher the accuracy for the calibration but the model will be less robust for an external dataset. On the other hand, the less the variables are used, the higher the possibility to make the model robust, but with a lower accuracy. Finally, for model calibration, the variables should be as independent as possible and external factors -such as laboratory, analytical methods, analytical accuracy and sampling conditions- should be considered.

### Further information:

Dandikas, V.; Heuwinkel, H.; Lichti, F.; Drewes, J.E.; Koch, K. (2014): Correlation between biogas yield and chemical composition of energy crops. *Bioresour. Technol.* 174, 316–320.



## **Conference on**

Monitoring & process control  
of anaerobic digestion plants

2<sup>nd</sup> day - 2015-03-18

## NIRS in the biogas process: Restrictions of calibration transfer between similar processes



Michael Schmidt, H. Fabian Jacobi<sup>a</sup>  
a) Landesbetrieb Hessisches Landeslabor

Contact: fabian.jacobi@lhl.hessen.de

**Keywords:** Near infrared spectroscopy, biogas, anaerobic digestion

Based on data of wet chemical laboratory analysis, a rising number of attempts to calibrate near infrared reflexion measuring systems for the use on active biogas slurry were performed and can be found in the literature. The goal was to utilize the non-invasive optical probes for online-monitoring and regulation of substrate feeding to ensure a stable and efficient biogas yield at low-maintenance costs. For this task most projects focussed on a single to a few industrial or agricultural biogas plants to establish a functional long-term monitoring for this particular plant, substrate(s) and process.

Due to having multiple absorption frequencies for each component in the near infrared range, allocation of spectra to specific concentrations is possible, but requires a comprehensive statistic variability to minimize the influence of disturbance variables. Hence ambitious pursuits to apply such (restricted) calibration sets to new biogas plants have failed so far without further determination of the disturbing factors.

The NIR spectra acquired at the LHL Eichhof laboratory were based on multiple test series performed in stirred digesters of 20 to 200 L-volume. Among other substrate feeds, maize silage, maize sludge (shredded maize silage under the addition of water) and starch as a single component were fed.

Frequent chemical analysis (i.e. gas chromatography for volatile acids, polarimetric measurement of starch remains and dry matter determination) and associated NIR measurement of the samples were performed in the in-house laboratory immediately after sampling.

The NIR-spectra were acquired using a Carl Zeiss Corona 45 NIR and used to establish calibrations modelled with WinISI-4 Software. The goal was to narrow and categorise disturbance variables to receive a universally valid calibration for multiple components of biogas slurry.

Calibration of parameters such as dry matter, pH, concentrations of starch, fat, raw fibre, ammonium, raw protein and volatile fatty acids resulted in promising calibrations, as long as a single process or sample source was used. Unfortunately the applications of such calibrations onto similar feeding- and process-set-ups did not show satisfying results. Moreover, also replicated conditions already resulted in unsatisfactory comparability, i.e. calibrations from the original trial did not perform well on replicated trials (e.g.: 2 maize silage feeds - acetate-value: 0 – 2.6 g/L; mean: 1.2 g/L; standard error of prediction: 0.8 g/L; standard error of lab method: 0.2 g/L).

Simplification of the feeding strategy by limiting the substrates to different well defined substances (e.g. vegetable cream substitute, starch, casein and sodium salts) showed infrequent improvement within the overall precision of the NIR-data, but depended on the individual case (e.g: 2 starch feeds – starch-value: 0 – 1.8 g/L; mean: 0.5 g/L; standard error of prediction: 0.2 g/L; standard error of lab method: 0.1 g/L).

Therefore multiple standard addition series of substrates in slurries were supplemented, precluding any changes of the slurry matrix by metabolism effects. Similar transfer interferences could be observed in these basic designs for calibration sets (e.g: starch-value: 0 – 6.0 g/L; mean: 2.6 g/L; standard error of prediction: 1.7 g/L).

By analysing regression lines from standard addition series of different dry matter dilutions, an overarching effect was noted: Rising dry matter resulted in rising false substrate signals with a dimming effect of the signal strength for high substrate levels (see figure 1.). This effect could also be repeated using non-organic clay composites.

Similar particle density and particle size distribution within watery samples seems to be the determining factor for applying a NIR calibration to a new set of samples. As the digester experiments have shown so far, the diversity of the matrix between similar slurries already seems to exceed the acceptable range for reproducible NIR calibrations.

To conclude: A universally usable slurry calibration for NIRS does not seem to be achievable without additional adjustments to the measurement setup and NIR-based monitoring in the field of biogas plants is yet limited to operate under restricted conditions.

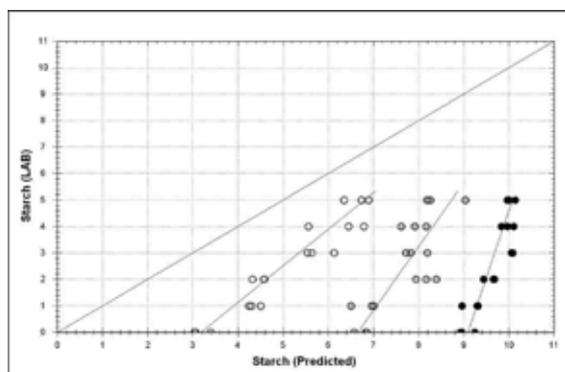


Figure 1: Regression lines of predicted starch contents in slurries with different dry matter (DM) contents. Calibrations were performed in water; shown values are predictions, based on the calibration of starch in water. Colour and corresponding DM (in %) from left to right: white (0.38), grey (0.94), black (1.5). The angle bisector represents the calibration with 0 % DM (data points not shown).

## MIR spectroscopy for monitoring of AD processes - Prospects and challenges



Christian Wolf and R. Eccleston (Cologne University of Applied Sciences)  
Contact: christian.wolf@fh-koeln.de

**Keywords:** Anaerobic Digestion, middle-infrared, MEMS, online sensor

Monitoring of Anaerobic Digestion (AD) processes is crucial to the development and implementation of optimization and control strategies (Kujawski and Steinmetz 2009, Madsen et al. 2011). In particular, renewable energy production in full-scale AD plants suffers from a backlog in online instrumentation and control, which needs to be cleared in order to make AD plants financially viable and competitive compared to other renewables. Unfortunately, commonly used online instrumentation on AD plants only comprises biogas composition ( $\text{CH}_4$ ,  $\text{CO}_2$ ,  $\text{H}_2$ ,  $\text{H}_2\text{S}$ ), produced energy (kWh) and in some cases basic biochemical process parameters such as pH-value and Oxidized Redox Potential (ORP) (Wolf et al. 2013). In order to properly monitor and assess process stability in time, this has proven to be insufficient as changes in organic acid concentrations and carbon buffer capacity cannot be detected. Therefore, new inline process measurement systems specifically targeting organic acids, carbon buffer as well as total solids (TS) and ammonium ( $\text{NH}_4$ ) using spectroscopic probes in the near-infrared (NIR) and middle-infrared range (MIR) are currently being developed.

The primary goal of the research presented in this paper is to develop a financially feasible and sufficiently accurate and robust MIR inline sensor for AD plants that allows real-time measurement of the total organic acid concentration and carbon buffer capacity. Furthermore, challenges and prospects of the MIR technology are discussed.

### Materials and Methods

Every spectroscopic measurement system consists of a probe, a spectrometer and a data processing and analysis unit. In this case a diamond-tipped ATR probe manufactured by the Berlin-based company art photonics<sup>1</sup> was used. This probe design has two main advantages. (1) Due to the diamond optics, the probe is very robust and can be used at high TS concentrations (>20 %) and high pressures (up to 180 bar) and (2) it uses a special poly-crystalline fibre (PIR), which allows higher transmission rates.

Initial experiments were conducted with the Nicolet is5 process spectrometer from Thermo Scientific<sup>2</sup> in the laboratory and later at a full-scale industrial AD plant. Based on those results, new miniaturized MEMS-based (microelectronic-mechanical system) spectrometers in the ranges of 6.8-8  $\mu\text{m}$  and 8-10  $\mu\text{m}$  manufactured by the Finnish company Spectral Engines<sup>3</sup> are currently being developed and tested within the research project MEMS-Biopro<sup>4</sup> funded by the BMWi. This allows not only the reduction of spectrometer costs from €25,000 to approximately €100 but also allows the integration of the spectrometer into the probe which reduces the fibre length significantly improving the S/N ratio.

### Results

The obtained results show that total organic acids concentration and carbon buffer capacity can be detected at wavenumbers 1,416  $\text{cm}^{-1}$ , 1,544  $\text{cm}^{-1}$  and 1,364  $\text{cm}^{-1}$ , 1,620  $\text{cm}^{-1}$  respectively (Figure 1). Furthermore, the desired process variables cause distinctive peaks in the MIR spectrum, which makes data analysis easier and increases accuracy. Nevertheless, until now the S/N ratio using the Nicolet is5 does not satisfy the necessary requirements, which is mainly due to fibre length (3 m) and spectrometer sensitivity. First measurements with the MEMS-based MIR sensor indicate that sensitivity can be significantly increased, thus allowing a detection of organic acids and carbon buffer in lower concentrations. Further laboratory and full-scale tests with the new MIR sensor system will be conducted in 2015.

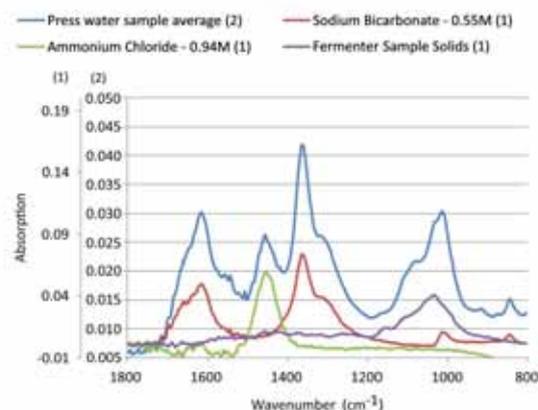


Figure 1: MIR spectrum of AD sludge and press water in comparison to spectral data from ammonium and sodium bicarbonate.

- 1) <http://www.artphotonics.de>
- 2) <http://www.thermoscientific.de/product/nicolet-is-5-ft-ir-spectrometer.html>
- 3) <http://www.spectralengines.com/>
- 4) Funding reference number: KF21 3781 ORE3

### Literature:

- Kujawski, O. ; Steinmetz, H. (2009): Development of instrumentation systems as a base for control of digestion process stability in full-scale agricultural and industrial biogas plants. *Water Science & Technology*, 60 (8), 2055–2063.
- Madsen, M.; Holm-Nielsen, J.B. and Esbensen, K.H. (2011): Monitoring of anaerobic digestion processes: A review perspective. *Renewable and Sustainable Energy Reviews*, 15 (6), 3141–3155.
- Wolf, C.; Gaida, D.; Stuhlsatz, A.; Ludwig, T.; McLoone, S. and Bongards, M. (2013): Predicting organic acid concentration from UV/vis spectrometry measurements - a comparison of machine learning techniques. *Transactions of the Institute of Measurement and Control*, 35 (1), 5-15.

## Estimation of the foaming propensity of substrates in anaerobic digestion



Lucie Moeller, Andreas Zehndorf (Helmholtz Centre for Environmental Research)  
Contact: lucie.moeller@ufz.de

**Keywords:** foam formation, process disturbances, anaerobic digestion, LEIPZIG F0AM TESTER

Foam formation in the course of anaerobic digestion often represents a serious problem for biogas plant operators because the foam can plug gas pipes, lift the biogas roofs and lead to losses in biogas yield. Research into foaming causes had been focused on anaerobic digesters of municipal wastewater sludge until recently (Ganidi et al., 2009). Foam formation in other anaerobic digestion systems for biogas production has only recently begun to attract research attention.

Surveys by Moeller & Görsch (2015) and Kougias et al. (2014) showed the high percentage of biogas plants that suffered from foam formation: 12 out of 15 waste treating biogas plants in Germany (Moeller & Görsch, 2015) and 15 out of 16 full-scale biogas plants in Denmark had experienced foaming in fermenters or substrate storage/pre-digesters (Kougias et al., 2014). Unfortunately, the majority of biogas plant operators are unable to identify the causes of foaming in their biogas reactor. The occurrence of foam is mostly related to the chemical composition of substrates fed to the reactor. The digestate consistency is also a crucial part of the foam formation process. For this reason, no general recommendations concerning substrates can be given in order to prevent foam formation in biogas plants. The safest way to avoid foaming is to test the foaming tendency of substrates on-site.

A possible solution is offered by an innovative foaming test LEIPZIG F0AM TESTER. With the help of this tool, biogas plant operators have the possibility to evaluate the foaming disposition of new substrates prior to use in order to adjust the composition of substrate mixes. This method was also used for the development of foam avoiding strategies in the laboratory scale. Both the effects of grain milling stage as well as of N-containing compounds in sugar beet AD have been studied. Real-world solutions were developed that were successfully applied in full-scale anaerobic digestion plants.

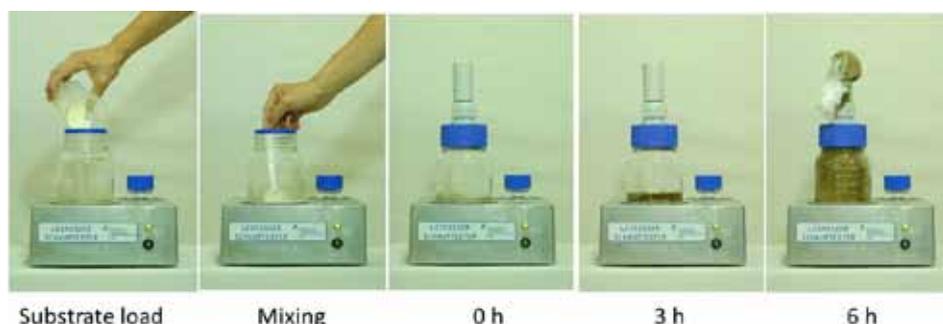


Figure 1: Foaming test by use of LEIPZIG F0AM TESTER (experimental set-up: 10 g protein powder in 490 g active digestate, 37 °C)

### Literature:

- Ganidi, N.; Tyrrel, S.; Cartmell, E. (2009): Anaerobic digestion foaming causes – a review. *Bioresour. Technol.* 100, 5546–5554.  
Kougias, P.G.; Boe, K.; O-Thong, S.; Kristensen, L.A.; Angelidaki, I. (2014): Anaerobic digestion foaming in full-scale biogas plants: a survey on causes and solutions. In: *Water Sci. Technol.* 69 (4), 889–895.  
Moeller, L.; Görsch, K. (2015): Foam formation in full-scale biogas plants processing biogenic waste. In: *Energy, Sustainability and Society* 2015, 5-1. doi:10.1186/s13705-014-0031-7.

### Further information:

- Moeller, L.; Lehnig, M.; Schenk, J.; Zehndorf, A. (2015): Foam formation in biogas plants caused by anaerobic digestion of sugar beet. In: *Bioresource Technology* 2015, 178, 270-277. doi:10.1016/j.biortech.2014.09.098.  
Moeller, L.; Görsch, K.; Neuhaus, J.; Zehndorf, A.; Müller, R.A. (2012): Comparative Review of Foam Formation in Biogas Plants and Ruminant Bloat. In: *Energy, Sustainability and Society* 2012, 2:12, 1-9.

## In-line acetate measurement in anaerobic digesters – development of a bioelectrochemical sensor platform



Jörg Kretzschmar<sup>1</sup>, Jan Liebetrau<sup>1</sup>, Falk Harnisch<sup>2</sup>

1) DBFZ Deutsches Biomasseforschungszentrum gemeinnützige GmbH, Biochemical Conversion Departement

2) Department of Environmental Microbiology, Helmholtz Centre for Environmental Research - UFZ

Contact: joerg.kretzschmar@dbfz.de

**Keywords:** biosensor, biogas, three electrode arrangement, microbial electrochemistry

Despite its wide distribution and existing practical experiences, the anaerobic digestion process is still low automated compared to other industrial processes. Especially in the case of biogas production for energy purposes the power production has to meet the requirements of the energy market. This implicates a flexible but stable biogas production which e.g. can be achieved by variable substrate feeding. This however bares the risk of process acidification and breakdown. Online control of highly process-relevant organic acids and adaption of the feeding rate could prevent process disturbances caused by fermenter overfeed. Nowadays organic acids are measured via expensive, time consuming and offline methods like gas or liquid chromatography. One opportunity to overcome this deficit is the development of cost efficient sensors for online determination of volatile fatty acids (VFA). Here bioelectrochemical sensors based on microbial fuel cells were already proposed for VFA quantification in anaerobic digestion. Following and optimizing the presented approach is based on a membraneless microbial sensor with a three electrode transducer for in-line measurement of acetic acid and other volatile fatty acids. The sensing element of such a sensor is a mixed species microbial anode consisting among others predominantly of *Geobacter spp.*. These bacteria oxidise acetate and are able to use the solid anode as terminal electron acceptor. The current produced can be measured as sensor signal. The proof of principle of the sensor concept has already been provided within AD experiments in 500 mL flasks with dried and grounded maize silage as substrate.

Within the present study the performance of the microbial anode as sensing element was examined in non sterile, continuous stirred flow cells (100 mL, 1 mL min<sup>-1</sup>) using artificial wastewater as growth medium. Sodium acetate (NaOAc) served as sole carbon source and analyte respectively with concentrations ranging from 0 to 15 mM. Anodes and cathodes were made from graphite rods (ø 0.5 cm, CP-Handels-GmbH, Germany). Ag/AgCl reference electrodes were used throughout all experiments (SE 11, Sensortechnik Meinsberg, Germany, +0.197 V vs. SHE). Electrochemical measurements were performed with a multipotentiostat (PARSTAT MC, AMETEK Inc., USA) using the following electrochemical techniques: Chronoamperometry (CA) at 0.2 V vs. Ag/AgCl for the whole time of the experiment interrupted every 24h by cyclic voltammetry (CV). CV vertex potentials were set to 0.3 and -0.5 V vs. Ag/AgCl with a scan rate of 1 mV s<sup>-1</sup>. With the described experimental sensor setup we could determine a measurement range between 0.5 and 5 mM NaOAc with an absolute measurement resolution of > 0.25 mM and < 1 mM NaOAc. Most important for practical implementation in AD processes is the increase of the sensor measurement range. In case of incidents acetic acid concentrations can range up to 60 mM acetic acid and more. For process control within safety limits an upper limit of the sensor of 20 mM acetic acid (~1.2 g/L) could be sufficient. Furthermore the cross sensitivity for process relevant organic acids like butyric or propionic acid and their influence towards the sensor signal is of great interest and therefore one part of future research. Assuming a successful development such a sensor is one important step toward process automation of AD processes and others.

## Application of laser absorption spectroscopy ( $^{13}\text{C}\text{-CH}_4$ and $^{13}\text{C}\text{-CO}_2$ ) for online monitoring in biogas plants



**Daniela Polag**, Liane Müller, Tobias May, Fabian Jacobi, Stephan Laukenmann, Helmut König, Frank Keppler  
(Institute of Geosciences, Ruprecht-Karls-University of Heidelberg)

Contact: [daniela.polag@geow.uni-heidelberg.de](mailto:daniela.polag@geow.uni-heidelberg.de)

**Keywords:** stable isotopes, laser absorption spectroscopy, anaerobic digestion, methano

Energy production by biomass decomposition within biogas plants plays an important role in the renewable energy sector of Germany. In recent years, the number of biogas plants has greatly increased and this upward trend appears to continue. For example, in Germany during the last 10 years the number of biogas plants has grown from 1000 to 7500. Efficient operation and stability of biogas plants requires a continuous monitoring of the digester content. Traditional laboratory analysis of digester sludge is often complex and time-consuming and shows a delayed response to disruptions within the fermentation process. As a new approach, we apply laser absorption spectrometers (LGR, Los Gatos Research) for real-time monitoring of the stable carbon isotopes of methane and  $\text{CO}_2$  ( $\delta^{13}\text{C}_{\text{CH}_4}$  and  $\delta^{13}\text{C}_{\text{CO}_2}$ ) in a pilot-scale biogas digester located at Deutsches Biomasseforschungszentrum in Leipzig, Germany. The general aim is use  $\delta^{13}\text{C}_{\text{CH}_4}$  and  $\delta^{13}\text{C}_{\text{CO}_2}$  as new process indicators to characterize the actual process state of the anaerobic digesters and to indicate possible process changes at an early stage. Therefore, we connected the laser absorption spectrometer to the gas outlet of a  $190\text{m}^3$  maize-digester and carried out high-resolution monitoring of the stable isotopes under variable organic loading rates ( $2\text{-}13\text{ kg oTS/m}^3\text{d}$ ). We compared the isotopic values with different fermenter parameters such as gas production rate, gas composition, VFA composition, and pH. Further, fermenter sludge samples were taken and were analyzed for microbiological composition (quantitative qPCR-analysis). These samples were also used as starter cultures for batch experiments with various C-sources to deduce the specific isotopic signature depending on the actual methanogenic pathway.

The  $\delta^{13}\text{C}_{\text{CH}_4}$ -values reacted promptly to abrupt changes in the process state such as feeding events and also indicated a long-term variability depending on the environmental conditions. During a phase of high organic loading  $\delta^{13}\text{C}_{\text{CH}_4}$ -values showed a strong increase (enrichment) of about 10‰. The strong isotopic enrichment occurs 5-10 days prior to a significant change in the other parameter values.

Thus, for process control, it might be possible to estimate 'critical' isotopic values with respect to fermenter conditions. As a new parameter tool the stable isotopes even might be coupled to automatisisation routines (i.e., amount and intervals of digester feeding) in future time.

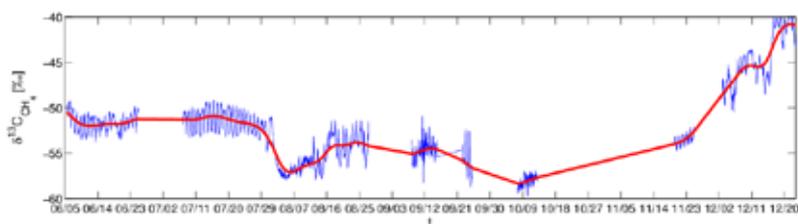


Figure 1: Monitoring of  $\delta^{13}\text{C}_{\text{CH}_4}$  at a pilot-scale maize-digester with  $190\text{m}^3$  at DBFZ

### Further information:

<http://www.geow.uni-heidelberg.de/forschungsgruppen/keppler/>  
List of publications with reference to biogas research:

- D. Polag, L.C. Krapf, S. Laukenmann, H. Heuwinkel, J. Lelieveld, F. Keppler (2014). Continuous measurements of stable carbon isotopes in anaerobic digesters. *Engineering in Life Sciences*, 4, 153-160.
- D. Polag, S. Laukenmann, H. Heuwinkel, M. Greule, F. Keppler (2013). Evidence of anaerobic syntrophic acetate oxidation in biogas batch reactors using stable carbon isotopes labelling experiments. *Isotopes in Environmental and Health Studies*, 49, 365-77
- S. Laukenmann, D. Polag, H. Heuwinkel, M. Greule, A. Gronauer, J. Lelieveld, F. Keppler (2010). Identification of methanogenic pathways in anaerobic digesters using stable carbon isotopes. *Engineering in Life Sciences*, 10, 509-514.
- F. Keppler, S. Laukenmann, J. Rinne, H. Heuwinkel, M. Greule, M. Whiticar, J. Lelieveld (2010). Measurements of  $^{13}\text{C}/^{12}\text{C}$  Methane from Anaerobic Digesters: Comparison of Optical Spectrometry with Continuous-Flow Isotope Ratio Mass Spectrometry. *Environmental Science & Technology*, 44, 5067-5073.

## Development of process optimisation tools for both efficient biogas research and operation of full-scale plants



J. Liu<sup>1,2</sup> and M. Nistor<sup>1</sup>  
1) Bioprocess Control Sweden AB  
2) Department of Biotechnology, Lund University

Contact: [mn@bioprocesscontrol.com](mailto:mn@bioprocesscontrol.com)

**Keywords:** instrumentation, process optimization, biogas plant, anaerobic digestion

Production of biogas through anaerobic fermentation from abundant biomass can provide important economical and environmental benefits to the region, as well as reduces the use of fossil fuels. However, this is feasible only if biomethanisation for energy recovery and resource utilization is highly efficient and cost-competitive. Today a majority of commercial biogas plants around the world are unfortunately not fully utilised and operated, with utilisation rates standing far below the potential capacity. This has in turn had a serious negative impact on the profitability of many biogas producers. Plant operators are thus in need of solutions to increase their plant utilisation rates and profitability. It is also generally accepted that with the correct instrumentation and automation tools, along with adequate operator knowledge, these utilisation rates can be significantly increased.

As a university spin-off from over 16 years biogas research activities in instrumentation control and automation within anaerobic digestion field, Bioprocess Control was founded on the idea of optimising the production of biogas. The company has over the past 9 years, developed numerous laboratory technologies for analysing the energy content of different biomass feedstocks, as well as technologies for simulating biogas processes at lab-scale. The company has also been working on database and process diagnosis of full-scale data as well as new sensor technologies and control algorithms for optimising biogas plants. The technical team has attained an in-depth and broad understanding of the needs of both researchers and full-scale plant operators for the further growth and maturation of biogas industry and business.

The current presentation provides a technologies overview of latest development of technologies and their applications in both biogas research and industrial scale production including standardization and early predication of methane/biogas potential analysis, Cloud based simulation tool for getting better understanding of the fermentation dynamic, and process diagnosis based on plant-wide simulation of biogas plants. Examples of test and simulation results are presented to demonstrate the benefit and performance of these technologies tools.

## Models as keys to optimization, monitoring and control of AD processes



**Finn Aakre Haugen** (Telemark University College, Porsgrunn, Norway)  
Contact: finn.haugen@hit.no

**Keywords:** mathematical models, anaerobic digestion, optimization, Kalman filter, control

The presentation demonstrates with a practical application how mathematical models can be a key to optimization, monitoring and control of AD processes. The application stems from a research project at Telemark University College involving theoretical and experimental work based on a real pilot UASB type AD reactor fed with animal waste (cow manure) at Foss dairy farm, Skien, Telemark, Norway.

Two models are developed:

(1) A mechanistic dynamic AD model, named the modified Hill model, has been adapted to the pilot reactor using steady-state and dynamic data from on-line sensors and laboratory analysis.

(2) A dynamic model for the reactor temperature based on an energy balance of the liquid is adapted to the pilot reactor.

These two models have been used in various ways in the project:

- Optimization: Optimal design and operation, regarding e.g. reactor volume and temperature, of the planned full-scale reactor are determined using simple brute-force optimization based on steady state simulations of the modified Hill AD model combined with models of the reactor temperature and heat exchanger temperatures based on energy balances. Various optimization criteria are considered.
- Design of temperature control system using thermal process simulations: Both simulations and practical experiments show that the produced  $\text{CH}_4$  gas flow depends on the reactor temperature, indicating a need for feedback temperature control. Both on-off control and industry-standard PI control are shown appropriate as controllers. The Skogestad PI controller tuning method for “integrator with time-delay” process dynamics with parameters estimated from the process step response is identified as the favoured tuning method. A novel closed loop tuning method, named Relaxed Ziegler-Nichols tuning method, which is based on a combination of the Skogestad method and the original Ziegler-Nichols closed-loop tuning method, compares favourably with both the Ziegler-Nichols method and the Tyreus-Luyben tuning method.
- Design of  $\text{CH}_4$  flow control using AD process simulations: The produced power is proportional to the produced  $\text{CH}_4$  gas flow, at fixed conditions. Therefore, a constant power production may be obtained by controlling the  $\text{CH}_4$  flow control to a setpoint. Conditions for safe reactor operation are found using steady-state responses of dynamic simulations, taking into account an upper limit of the reactor VFA concentration recommended in the literature. Both simulations and practical experiments indicate that both an on-off controller and the industrial-standard PI controller are viable for  $\text{CH}_4$  gas flow control. The Skogestad method is (again) identified as the favoured controller tuning method.
- Implementation of state estimator of AD process: A state estimator, or a soft sensor, is implemented in the form of the Unscented Kalman Filter (UKF) algorithm based on the modified Hill model and using continuous measurement of  $\text{CH}_4$  gas flow. The UKF estimates continuously the four model state variables of the modified Hill model, and an augmented state variable which is the concentration of volatile solids of the influent. The estimates are verified against laboratory analyses. (Contrary to the common Extended Kalman Filter, the UKF avoids model linearization.)
- Model-based  $\text{CH}_4$  control: Various model-based control systems have been designed using the modified Hill model and the UKF: A model-based predictive controller (MPC) is designed for controlling the methane gas flow to a setpoint which may be varied, e.g. due to changing produced power demands. Simulations indicate that, with a known setpoint profile, the setpoint tracking with MPC is considerably better than with PI control. For unknown disturbance changes, the disturbance compensation is not much better with MPC than with PI control, as expected. The MPC is applied successfully to the real pilot reactor with known  $\text{CH}_4$  gas flow setpoint profile. An MPC aiming at retaining the reactor at an operating point with maximum allowable VFA concentration, is compared with PI control based on feedback from VFA estimated with the UKF, on a simulator (no practical experiments). Here, MPC and PI control have similar performance, but PI control being much simpler, of course.

Further information:

<http://fossbiolab.no>; <http://hit.no/ansatte/vis/finn.haugen>

## Development of process optimisation tools for both efficient biogas research and operation of full-scale plants



Eric Mauky, Hans-Fabian Jacobi, Sören Weinrich, Jan Liebetrau, Michael Nelles (DBFZ)  
Contact: eric.mauky@dbfz.de

**Keywords:** demand-driven; model predictive control; flexible; full-scale; ADM1

For future energy supply systems with high proportions of renewable energy sources biogas plants are a promising option (Szarka et al. 2013). They have the potential to supply demand-driven electricity to compensate the divergence between energy demand and supply by uncontrolled sources like wind and solar power. So far biogas plants have been designed to produce a stable and constant energy output. Common flexibility concepts for existing biogas plants are often technical-oriented and imply mostly high investments – for example the expansion of gas storage capacities and building multi-stage concepts (ReBi or Gicon). An additional option for flexibilization – besides the expansion of Combined Heat- and Power-Units and storage capacity – is the direct adaption of gas production to times, when electrical power is required to be produced.

The major aim of the presented study was to proof the general flexibility of the anaerobic processes under full scale conditions for an energy output according to the grid demand and to examine the effect of discontinuous feeding on daily and long term process stability. Former laboratory-scale-experiments in Continuous Stirred Tank Reactors (CSTR) show a long-time stable process even under highly dynamic operation (Mauky et al., 2015). To the authors knowledge no data has been published showing the effects of biological flexibilization at a full-scale biogas plant. Improving plant operation and thus efficiency requires more and more intelligent control and optimization systems.

A model predictive control (MPC) was developed to predict feeding sequences in order to fulfill a demand-driven utilization timetable and react effectively to alterations. MPCs use an internal dynamic model of the process, a history of past control moves and an optimization cost function over the receding prediction horizon to calculate the optimal control moves. The MPC is based on a simplified version of the ADM1. Benefits of such control concepts are the possibility of reacting predictively to future changes in demand and looming disturbances at advanced biogas plants and reduced investment costs in extra gas storage capacity. The experiments were carried out at a research biogas plant and at a full-scale biogas plant.

The experiments showed a high flexibility in the gas production and high process resilience towards pulse feeding. The prediction of biogas production by simplified dynamic models gave reliable results compared to real measured data. Depending on the used substrates the necessary gas storage demand could be reduced compared to the common constant feeding operation. The flexibilization of existing biogas plants can contribute substantially to balance demand and production within the future energy system.

### Further information:

Szarka, N.; Scholwin, F.; Trommler, M.; Fabian Jacobi, H.; Eichhorn, M.; Ortwein, A.; Thrän, D. (2013): A novel role for bioenergy: A flexible, demand-oriented power supply. *Energy* 61, 18–26.

Mauky, E.; Jacobi, H.F.; Liebetrau, J.; Nelles, M. (2015): Flexible biogas production for demand-driven energy supply – Feeding strategies and types of substrates. *Bioresour. Technol.* 178, 262-269.

## Monitoring the process microbiology in full-scale biogas plant - identification of process-relevant microorganism usable as validation standards or as indicators for process disturbances



Susanne Theuerl (Leibniz-Institute for Agricultural Engineering Potsdam-Bornim e.V. - ATB)  
Contact: stheuerl@atb-potsdam.de

**Keywords:** process microbiology, ecosystem functionality and community organisation, resilience, microbial indicators for disturbances

Anaerobic digestion of biomass into energy-rich biogas is carried out by a diverse community of microorganisms belonging to different functional guilds whereby the majority of the species involved and their impact on the process efficiency are still rather unknown.

Each biogas plant (BGP) contains its own microbial community, specifically adapted to the occurring conditions (e.g. reactor design, process performance and supplied feedstock). In regard to this, one of the most discussed questions is: What is the right composition of a “healthy”, well-balanced, microbial community, which is resilient in case of disturbances and enabled to produce a constant, sufficient amount of biogas? To answer this question a broad range of methods is available to investigate the process microbiology (Fig. 1). Ideally, a combination of different methods should be used in order to link community structure information to its role in its respective habitat by correlating the microbiological data with the prevalent process parameters. With this background, a comprehensive analysis of nine full-scale BGPs was carried out. For a microbiological inventory the community profiling method TRFLP (terminal restriction fragment length polymorphism) in combination with a cloning-sequencing approach was used and in special cases combined with metaproteome analyses. To determine the impact of specific process parameters on the microbial community and to identify representative (groups of) species that may exert a “key” function various ecology indices were applied.

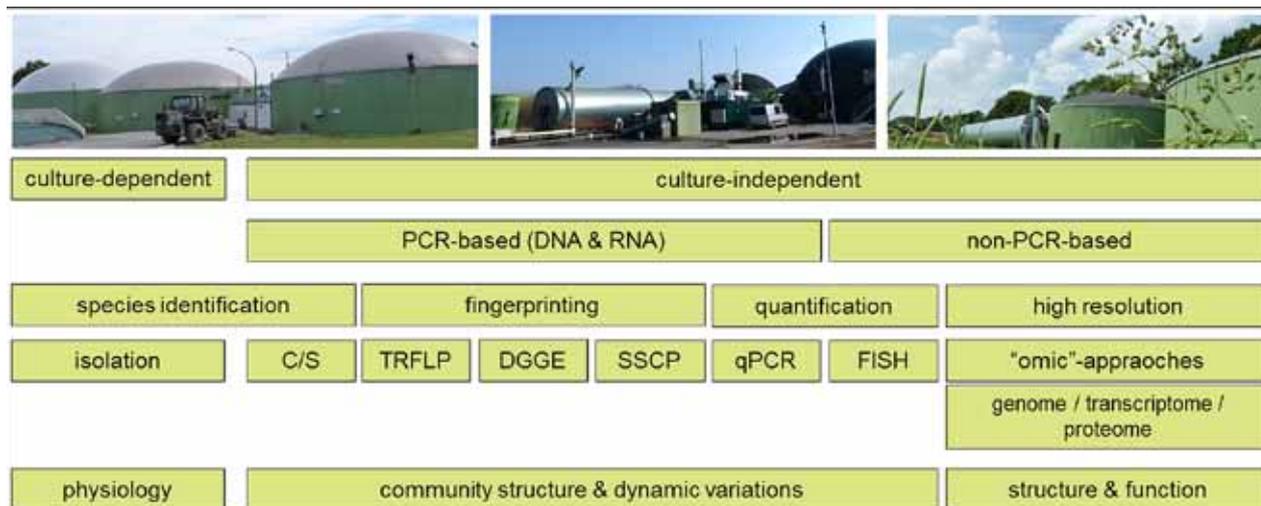


Fig. 1: Methods to investigate the process microbiology in anaerobic digesters (modified after Talbot et al. 2008 Water Res 42: 513-537, Su et al. 2012 Appl. Microbiol. Biotechnol. 93: 993-1003 and Vanwonterghem et al. 2014 Curr. Opin. Biotechnol. 27: 55-64). C/S = cloning/sequencing, TRFLP = terminal restriction fragment length polymorphism, DGGE = denaturing gradient gel electrophoresis, SSCP = single strand conformation polymorphism, qPCR = quantitative real-time polymerase chain reaction, FISH = fluorescence in situ hybridisation.

The comparison of the microbial communities revealed that each community consisted of an elevated amount of some dominant species (the generalists) whereby the majority of the species (approximately 2/3) were present in lower abundances. It was further shown that the microbial communities are highly sensitive and in most cases resilient in terms of compensating sudden stress situations. For example one of the investigated BGPs showed small variations in terms of the process temperature, the pH value as well as the ammonium ( $\text{NH}_4^+\text{-N}$ ) respectively the free ammonia nitrogen ( $\text{NH}_3$ ) content. The results showed that at higher temperature ( $45^\circ\text{C}$ ) and a rather high  $\text{NH}_4^+\text{-N}$  content ( $2.9\text{ g L}^{-1}$ ) but lower pH (7.7) which buffered the release of the cell-toxic  $\text{NH}_3$  ( $281\text{ mg L}^{-1}$ ) members of the versatile family Methansarcinaceae maintained the biogas production by the acetoclastic pathway whereby a slight pH increase (8.0) and hence a higher  $\text{NH}_3$  content ( $> 400\text{ mg L}^{-1}$ ) resulted in a system adjustment to the hydrogenotrophic methanogenesis caused by other members of the family Methanosarcinaceae in combination with members from the genus Methanobacterium. Consequently these findings revealed that the occurring microbial, in this case the archaeal community was able to compensate process fluctuation by maintaining the overall system functionality. In contrast, the comparative analyses also revealed that not all process instabilities can be compensated. During the monitoring, two of the investigated BGPs showed serious disturbances:

(1) a floating layer formation mainly caused by an insufficient digestate mixing in combination with high fibre content feedstocks, and

(2) the anaerobic digestions of low quality maize silage which was used as sole feedstock. In both cases, the disturbance was characterized by high acid concentration ( $\geq 10\text{ g L}^{-1}$  respectively  $\geq 15\text{ g L}^{-1}$ ) in combination with a diverse acid spectrum.

In the BGP with the floating layer formation, members from the bacterial family Porphyromonadaceae (phylum Bacteroidetes) were found with high abundance almost two weeks before a strong drop in the biogas yield was recorded. Members of this family are well known as secondary degraders responsible for production of different types of acids, in this special case of acetate, propionate and butyrate. Once the daily feeding was strongly reduced, the slow growing syntrophic acid oxidizer became predominant and the system started to recover. In case of the anaerobic digestion of low quality maize silage up to 80 % of the bacterial community were related to acid producers while no acid converters were detected. This explains the diverse and extremely high acid concentration of up to  $17\text{ g L}^{-1}$ . As a consequence, the pH value was low with 6.5 and unfavourable for the methanogens which were strongly inhibited in their activity.

To conclude, the comparative analysis delivered interesting insights into the system ecology especially into the complex microbial network responsible for the production of biogas. Moreover, in order to identify benchmarks for the stability, efficiency and adaptability of the microbial community unpredictable random process failures and further the evaluation of potential process key players can provide useful information considering future microbiological control and management concepts.

## Transferability of isotopic data to methanogenic pathways - an applicable process tool?



**May, Tobias;** Polag, Daniela; Keppler, Frank; Müller, Liane; Jacobi, Fabian; König, Helmut  
Contact: mayto@uni-mainz.de

**Keywords:** carbon isotopes, real-time PCR, isolation of pure cultures, metabolic studies, process control

Anaerobic digestion as the only renewable energy supply with base load capability has become an increased scientific interest in the last decades, since the complex microbiological degradation and the carbon fluxes to the final biogas (CH<sub>4</sub> and CO<sub>2</sub>) do remain a number of unresolved issues. There is still an insufficient knowledge of the specific degradation steps, released metabolic compounds and microbiological interactions which have an influence on the efficiency and stability of the biogas formation process. Furthermore, technical or microbiological devices which allow a real-time process control were rarely.

With a focus on the methanogenesis as the last step of the anaerobic decomposition in a biogas plant, we investigate the occurrence of the three methanogenesis pathways (hydrogenotrophic, acetoclastic or methylotrophic) in the course of time under certain process conditions (i.e. after feeding procedures, substrate changes, disruptions). Changes in the quantity of a methanogenic pathway may provide an early recognition of process failures. To draw conclusions about the present methanogenic pathways and the diversity of the correspondent microbiota, we used a combination of microbiological methods and stable isotope techniques. For this purpose, we isolated and cultured methanogenic archaea as well as bacteria from a mesophilic full-scale biogas reactor. The application of growth media including specific carbon sources (i.e. H<sub>2</sub> and CO<sub>2</sub>, formate, acetate and methanol) enabled the detection of a clearly distinguishable, pathway-depending carbon-isotope fractionation in the evolving CH<sub>4</sub> and CO<sub>2</sub>, thus allowing quantitative information about the prevailing metabolic processes. In parallel, batch fermenters containing the same, selective media were inoculated with small amounts of reactor sludge and revealed almost identical δ<sup>13</sup>C values as obtained from the pure culture experiments. For <sup>2</sup>H- and <sup>13</sup>C- analysis of CH<sub>4</sub> and CO<sub>2</sub>, gas samples were taken for 21 days from the headspace and were measured by a gas chromatograph, coupled to an isotope ratio mass spectrometer [1].

In an additional approach, we used a combination of <sup>2</sup>H- and <sup>13</sup>C-labelled substrates in pure and mixed cultures as well as in samples from anaerobic digesters to retrace and determine the specific metabolic pathways and typical carbon flows. Here, it was figured out that hydrogen has a distorting effect on the isotopic signature of the acetoclastic pathway leading to a mixed isotopic ratio that ranged between the acetoclastic and the hydrogenotrophic pathway. Even the equation of the degradation of formic acid seemed strongly effected by the hydrogen partial pressure. For this, metabolic statements were rendered more difficult. Additionally, the release and fate of methanol, the usually ignored methanogenic substrate, was traced in each process stage by pure culture isolates and broad metabolic analyses (HPLC, GC). Here, highest CH<sub>4</sub> yields were obtained in methanol fed batch fermenters. It became evident that methylotrophic methanogenesis is basically possible in biogas plants and has to receive consideration in isotopic studies.

Besides isotopic studies, real-time PCR analyses of the methanogenic community were carried out with weekly withdrawn reactor samples of a 90-day period at different loading scenarios [2]. This revealed unexpected and rapid changes in the microbial diversity from an acetoclastic dominated structure to a progressively hydrogenotrophic microbiota which again can induce a distinct impact on online isotopic studies of anaerobic digesters.

In a final step, the data obtained from these experiments will be compared with a long-term application of a laser ablation spectroscope (δ<sup>13</sup>C<sub>CH<sub>4</sub></sub> and δ<sup>13</sup>C<sub>CO<sub>2</sub></sub>) connected to the headspace gas outlet of a biogas plants. As part of this project, we try to extend the knowledge and the great potential of isotopic studies as a tool for real-time process controls in anaerobic reactors.

### Further information:

[1] Polag, D.; Heuwinkel, H.; Laukenmann, S.; Greule, M.; Keppler F (2013): Evidence of anaerobic syntrophic acetate oxidation in biogas batch reactors by analysis of <sup>13</sup>C carbon isotopes. *Isotopes Environ Health Stud.* 49:365-377.

[2] May, T.; Singenstreu, M.; Ebling, J.; Stantscheff, R.; Müller, L.; Jacobi, F.; König, H. (2015): Design and application of a synthetic DNA standard for real-time PCR analyses of microbial communities in anaerobic digesters. Submitted.

## Process control by calorimetry – advantages and challenges



Thomas Maskow, Sven Paufler, Heike Sträuber (UFZ Helmholtz Centre for Environmental Research)  
Contact: Thomas.maskow@ufz.de

**Keywords:** biothermodynamics, calorimetry, anaerobic bioprocesses, process control, acidogenesis, ABE fermentation, *Clostridium acetobutylicum*

Due to the intended turn from the crude oil-based to a bio-based economy many microbial catalysed conversions are discussed to synthesize industrial relevant products from readily available feeding materials. Commonly, in anaerobic biological processes more of the substrate's energy is transferred to the product as it is in aerobic bioconversions. This makes anaerobic bacteria interesting for the production of gaseous and liquid biofuels. Real time monitoring tools are essential to effectively control such industrial applications to increase productivity, product quality, and process stability. Given that heat flux measurement reflects changes in stoichiometry and kinetic immediately, calorimetry has the potential to become a great tool for monitoring. Forming the enthalpy balance of a reactor allows the determination of the metabolic heat production rate in an easy way. Yet the higher energy content, of the products of anaerobic bioconversions also means that less energy is dissipated throughout the conversion process. These tiny heat production rates are challenging the calorimetric devices and thermokinetic data interpretation.

For exploring the information content of calorimetric signals of anaerobic bioprocesses the heat production rate of *Clostridium acetobutylicum* as test system was monitored using calorimetric devices of different scales. Microcalorimetry, flow-through calorimetry, and high resolution reaction calorimetry were applied. The test system was chosen due to the large body of literature data of acetone-butanol-ethanol (ABE) fermentation. The heat production rates were analysed and related to conventionally derived growth kinetics and product formation during different phases of metabolism (i.e. acidogenesis and solventogenesis).

All applied calorimetric methods were common, that different metabolic phases can easily be retraced by the calorimetric signal. This underlines the particular suitability of calorimetry for the analysis and control of anaerobic bioprocesses. However, the advanced thermokinetic modelling of the heat trace proved to be challenging. Small errors in chemical analysis of intermediates and products lead to large differences in predicted enthalpies easily changing the whole conclusion of a model. This means that the calorimetric measurement provides more accurate process information than the chemical reference analysis. The challenge now is to combine the high resolution on-line signal of heat production with other on-line (e.g. gas production and analysis, pH) and off-line signals (concentration of biomass, substrate, products etc.) to yield more detailed and more accurate information regarding the anaerobic process. Advantages and problems of a calorimetric approach analysing and controlling anaerobic bioconversions processes at different scales will be discussed and progress in developing monitoring tools will be presented in detail.

### Further information:

Maskow, T.; Paufler, S. (2014): What does calorimetry and thermodynamics of living cells tell us? *Methods* (DOI 10.1016/j.ymeth.2014.10.035)

Abstracts

**Poster-Session**

## Electronic nose technology for reactor state and biogas quality assessment in anaerobic digestion



Gilles Adam<sup>1</sup>, Sébastien Lemaigre<sup>2</sup>, Xavier Goux<sup>2</sup>, Anne-Claude Romain<sup>1</sup>, Philippe Delfosse<sup>2</sup>  
 1) University of Liège, Arlon Campus Environnement, Belgium  
 2) EVA Environment and Agro-biotechnologies Department, Belvaux, Luxembourg

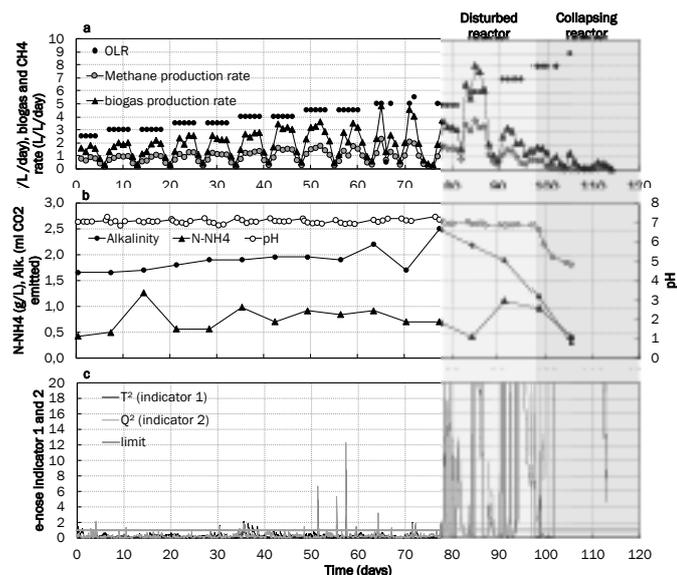
Contact: Gilles.Adam@ulg.ac.be

**Keywords:** gas sensor array, biogas quality, process monitoring

Electronic noses (e-noses) have been documented as potential tools to be employed in the anaerobic digestion (AD) domain, especially for process monitoring (Madsen et al., 2011; Pind et al., 2003). E-noses could also be used for biogas quality assessment and for safety purposes (biogas leak detection, biogas combustion, etc.) These instruments obviously present a potential in AD field but have also clear limitations. This work presents the application of a same e-nose evaluated on a pilot-scale continuously stirred tank reactor (100 L) for two purposes: i) reactor stability monitoring and, ii) biogas quality assessment (methane and hydrogen sulphide content).

Electronic nose is a generic term that refers to a biologically inspired system composed of an array of non-specific but complementary gas sensors. Most widely employed sensors in e-noses are low cost and unspecific metal oxide semiconductor (MOX) sensors. Those sensors were employed in this study. In a previous work, we demonstrated that an e-nose based on MOX gas sensors could detect process disturbances on lab-scale semi-continuous anaerobic reactors through multivariate statistical process control techniques (MSPC) (Adam et al., 2013). The work presented here uses the same approach and compares data of an on-line electronic nose exposed to the pilot-scale reactor headspace (Figure 1c) with biogas and methane production rate (Figure 1a), sludge pH, total ammonia nitrogen (N-NH<sub>4</sub>) and alkalinity (Figure 1b). A self-adapting model was used to correct signal drift. Figure 1a,b and c shows that the e-nose indicators were good predictors of reactor stability.

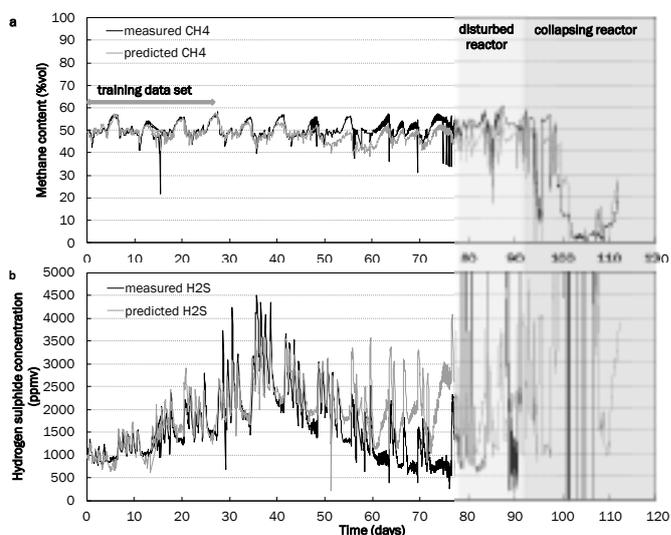
In addition, the same instrument was employed to assess CH<sub>4</sub> and H<sub>2</sub>S content in raw biogas by partial least squares (PLS) modelling. Data of reactor steady state period were used to train (30 first days, training by cross validation) and test (next 30 days) the model. Finally, the models were evaluated on the complete data set. For Methane prediction, cross-validation R<sup>2</sup> and root mean square error of prediction (RMSEP) were respectively of 0.73 and 1.7 % with 3 retained components. For H<sub>2</sub>S predic-



Figures 1a,b and c: Time serie of reactor state monitoring. a. OLR, biogas and methane production rate; b. pH, N-NH<sub>4</sub> and alkalinity; c. e-nose indicators. (reference)

tion cross-validation  $R^2$  and RMSEP were respectively of 0.78 and 275 ppmv (4 components). Time series of predicted versus measured  $\text{CH}_4$  and  $\text{H}_2\text{S}$  concentrations are presented in Figures 2a and 2b. For both variables, models degraded over time but initially presented good agreement with measured variables.

Results showed that the same simple e-nose was able, focusing on raw biogas, to assess reactor stability and predict  $\text{CH}_4$  and  $\text{H}_2\text{S}$  content. Even though, models were drifting over time and for  $\text{H}_2\text{S}$ , model was lacking of precision. It was possible to update the model for reactor state assessment, but for  $\text{CH}_4$  and  $\text{H}_2\text{S}$  predictions, an appropriate data pre-treatment must be investigated in order to reduce drift and model degradation.



Figures 2a and b. Time series of measured and e-nose predicted concentrations of a) methane and b) hydrogen sulphide.

#### Further information:

- Adam, G.; Lemaigre, S.; Romain, A.-C.; Nicolas, J.; Delfosse, P. (2013): Evaluation of an electronic nose for the early detection of organic overload of anaerobic digesters. *Bioprocess Biosyst. Eng.* 36, 23–33. doi:10.1007/s00449-012-0757-6
- Madsen, M.; Holm-Nielsen, J.B.; Esbensen, K.H. (2011): Monitoring of anaerobic digestion processes: A review perspective. *Renew. Sustain. Energy Rev.* 15, 3141–3155. doi:10.1016/j.rser.2011.04.026
- Pind, P.; Angelidaki, I.; Ahring, B.; Stamatelatos, K.; Lyberatos, G. (2003): Monitoring and Control of Anaerobic Reactors Biomethanation II, in: Ahring, B., Ahring, B., Angelidaki, I., Dolfing, J., Euegaard, L., Gavala, H., Haagensen, F., Mogensen, A., Lyberatos, G., Pind, P., Schmidt, J., Skiadas, I., Stamatelatos, K. (Eds.), Springer Berlin / Heidelberg, pp. 135–182. doi:10.1007/3-540-45838-7\_4

## Modelling of biogas production with ASPEN Plus for Model-based control



J. A. Arzate<sup>1</sup>, E. Kielhorn<sup>1</sup>, A. Bockisch<sup>1</sup>, H.-J. Nägele<sup>2</sup>, H. Oechsner<sup>2</sup>, K. Bailly<sup>3</sup>, E.M. N. Cruz Bournazou<sup>1</sup>, P. Neubauer<sup>1</sup>, S. Junne<sup>1</sup>

1) Technische Universität Berlin, Department of Biotechnology, Chair of Bioprocess Engineering

2) State Institute of Agricultural Engineering and Bioenergy, University of Hohenheim

3) SOTAsolutions GmbH, Berlin

Contact: j.arzate@mailbox.tu-berlin.de

**Keywords:** ASPEN, modelling, mechanism recognition, multiposition monitoring

The Anaerobic Digestion model No. 1 (ADM1) (Bastone, 2002) and other models (Angelidaki I, 1999) have opened the gate to the application of the software ASPEN Plus, which is nowadays widely used in chemical production processes either to predict the performance of a new process or to find failures in a current operation. The first application of ASPEN plus in an anaerobic digestion (AD) process was to evaluate feedstock like municipal solid waste (MSW), and co-digestion of slaughterhouse waste, food waste and cow manure in steady state conditions (Rajendran et al., 2014).

The present work aims at evaluating the biogas process using maize silage as single substrate as case study. The four stages of the AD, hydrolysis, acidogenesis, acetogenesis and methanogenesis, are simulated in ASPEN plus, the model applied for thermodynamic balancing is ElectNRTL. The first stage of the process consists of 13 stoichiometric reactions, in which the organic compounds from the feedstock crack down from carbohydrates, lipids, and proteins into sugars, volatile fatty acids and amino acids, respectively. The following Gibbs reactor is suggested to generate the ions in the medium through seven equilibrium reactions. Acidogenesis, acetogenesis and methanogenesis stages were simulated through 25 reactions, 4 reactions, and 2 reactions, respectively, in a CSTR at 40°C and atmospheric pressure. The results were compared with the biogas cost calculator (KTBL - <http://daten.ktbl.de/biogas/startseite.do>) in order to prove the relevance of the simulations. A good correlation could be achieved in all cases.

The simulation tool is further used to identify process disturbances, e.g. at flexible feedstock utilization. If the yield was differing from the one that is predicted with the model, a mechanism recognition procedure will allow for an early detection of failures. If the simulation was adapted to different process stages (optimal operation point and several non-optimal operation points), the mechanism recognition allows to identify the kind of disturbance by comparing the actual state with the previously simulated reference states.

This concept is further evolved in the project FlexFeed, funded by the German Federal Ministry for Economic Affairs and Energy. The impact of flexible feedstock load is investigated with the usually applied monitoring infrastructure and additional, recently developed multiposition sensors in the liquid phase. An optimal choice of the sensor location becomes feasible when many areas of the liquid phase are screened. The gained data of these trials is integrated into the described modelling approach for early process failure detection.

The choice of the feedstock mixture and the resulting amount of biogas produced is made based on a neural network approach, for which historical data of operation is used to calibrate the model. Hence, a combination of experience-based prediction for operational choices and stoichiometry-based simulation for the detection of disturbances is applied to decrease process risks when fluctuating loading rates and feedstock mixtures are applied.

### References

- Kolver, E.; Roche, J. R.; Miller, D.; Densley, R. (2001): Maize silage for dairy cows. In: Proceedings of the conference - New Zealand grassland association. pp. 195-202.
- Angelidaki I, E. L., Ahring, BK. (1999): A comprehensive model of anaerobic bioconversion of complex substrates to Biogas. In: *Biotechnology and Bioengineering* 63. S. 363-372.
- Bastone D.J.; K. J., Angelidaki I, Kalyuzhnyi SV, Pavlostathis S.G.(2002): The IWA Anaerobic Digestion Model No 1 (ADM1). In: *Water Sci Technol. Water Sci Technol.* 45: 65-73.
- Rajendran, K.; Kankanala H. R.; Lundin, M.; Taherzadeh M. J. (2014): A novel process simulation model (PSM) for anaerobic digestion using Aspen Plus. *Bioresource Technology*(0).

## Pyrosequencing analysis of bacterial community structure in anaerobic co-digestion process for biohydrogen production



Jacob Gómez Romero, Elvia Inés García Peña

Contact: bocaj23@hotmail.com

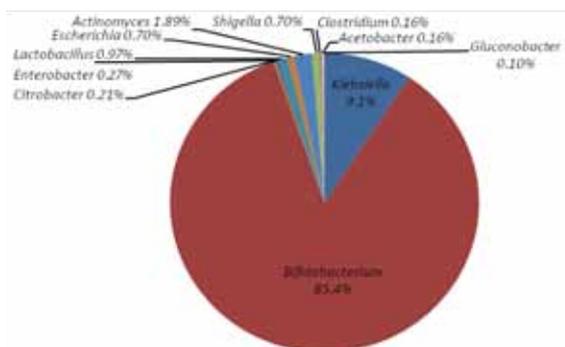
**Keywords:** Biohydrogen, Pyrosequencing analysis, Co-digestion

Hydrogen is considered as a fuel for the “future” because it has the highest energy density of 3042 cal/m<sup>3</sup>. Especially the production of biological hydrogen is promising as it can be obtained from a variety of organic feedstocks. Anaerobic co-digestion has been attracting strong interest due to its potential to improve the buffer capacity, the nutrient balance, the carbon/nitrogen (C/N) ratio and the macro and micronutrients concentration. A balanced C/N ratio allows enhancing the buffer capacity of the system. Additionally, the co-digestion process also reduces the possibility of inhibitory effects, which, in turn, increases the yield of biohydrogen production. On the other hand, the knowledge of the process in biohydrogen reactors has to be improved especially for industrial biohydrogen production applications from complex feedstocks. For instance, the analysis of the microbial community structure is essential for determining how the activity is affected inside the bioreactors. Thus, the knowledge about the dynamics of the microbial community structure and activity is essential for a successful planning of the biogas process, monitoring its parameters and for reaching the main goal of process stability and maximum biohydrogen yield.

In this context, the co-digestion process of crude cheese whey (CCW) with fruit vegetable waste (FVW) for biohydrogen production was investigated in batch and continuous systems, in stirred 1.8 L bioreactors at 37 °C. Five different C/N ratios (7, 17, 21, 31, and 46) were tested in batch systems. In the continuous system, eight different conditions of hydraulic retention time (from 60 to 10 h) and organic loading rate (from 21.96 to 155.87 g COD/L d) were evaluated. In batch results, the highest specific biohydrogen production rate of 10.68 mmol H<sub>2</sub>/Lh and biohydrogen yield of 449.84 mL H<sub>2</sub>/g COD were determined at a C/N ratio of 21. In the continuous co-digestion system, the optimum hydraulic retention time and organic loading rate were 17.5 h and 80.02 g COD/L d, respectively. Under these conditions, the highest volumetric production hydrogen rate (VPHR) and hydrogen yield were 11.02 mmol H<sub>2</sub>/L h and 800 mL H<sub>2</sub>/COD, respectively (table 1). Pyrosequencing analysis was done to determine the microbial community structure. The results of the analysis showed that the main microbial population at the initial stage of the co-digestion consisted of Bifidobacterium, with a predominance of 85.4 %. Hydrogen producing bacteria such as *Klebsiella* (9.1 %), *Lactobacillus* (0.97 %), *Citrobacter* (0.21 %), *Enterobacter* (0.27 %), and *Clostridium* (0.18 %) were less abundant at this culture period (figure 1). The microbial community structure correlated with the lactate, acetate, and butyrate concentration profiles obtained. The results demonstrated that the co-digestion of CCW with FVW improves the biohydrogen production due to a better nutrient balance and improvement of the system’s buffering capacity. Hence, the results obtained in this study increase the knowledge about the influence of mixing different substrates on the microbial community structure involved in the anaerobic co-digestion degradation of real feedstocks and provide valuable information to optimize the fermentation process. Finally, the characterization of microbial community structures should be also considered in the engineering context, as the understanding of the microbial community structure can provide information to analyze the anaerobic process under dynamic conditions and optimize the biohydrogen production process.

**Table 1: Results of volumetric production hydrogen rate (VPHR) and biohydrogen yield of co-digestion process at batch and continuous systems.**

Batch system		
Ratio C/N	VPHR (mmol L <sup>-1</sup> h <sup>-1</sup> )	Biohydrogen yield (mL H <sub>2</sub> /g COD)
7	1.37±0.16	144.49
17	6.21±0.07	325.80
21	10.68±1.5	449.84
31	9.40±0.23	341.04
46	10.45±0.32	330.13
Continuous system		
HRT (h)/OLR (g COD L <sup>-1</sup> d <sup>-1</sup> )	VPHR (mmol L <sup>-1</sup> h <sup>-1</sup> )	Biohydrogen yield (mL H <sub>2</sub> /g COD)
60/21.9	2.1±1.0	185.0
48/29.2	2.8±0.7	217.5
38/41.1	3.5±1.2	197.4
28/54.0	3.8±1.5	300.9
18.7/76.4	5.6±1.0	790.3
15/97.6	5.9±0.9	488.4
10/155.6	7.7±2.3	372.8
17.5/80.2	8.6±1.3	813.3



**Figure 1. Pyrosequencing of 16S rRNA gene analysis on predominance of bacterial communities at co-digestion process.**

## Online monitoring of volatile fatty acids in biogas with use of chemoresistive sensors

Hartmann Hieber, Christian Hubert (ICR Jena, Weimar)

Contact: hieber@icrjena.de

**Keywords:** Array of chemoresistive sensors, partial selectivity, dynamics of reduction and reoxidation, reversibility and drift kinetics, scalar parameter from the rate vectors, stability during long term application under harsh environment

The concentration of volatile fatty acids (VFA) can be used as an indicator for monitoring of the anaerobic process. Depending of the mixing in the digester the VFA profile indicates the actual process status and can be used as an early warning indicator for process failure. The calibration of the sensor array in the laboratory is carried out with acetic, propionic, n- and iso-butyric acid in the concentration range from  $<1$  up to  $>100$  vol-ppm. The dynamic sensor signal functions  $v + m t + w \exp(-r t)$  are evaluated for the transient amplitude  $w > 0$  for reduction and  $w < 0$  for oxidation of the chemoresistors. The rate coefficients  $m$  and  $-wr$  depend on the chemical activity of the carbon oxide and aliphatic hydrocarbon radicals. A strictly monotonous relationship between the rate coefficients, the transient amplitudes, and the concentration of VFA is found in the laboratory.

The data stock from measurements at the digester includes  $>107$  measurement sequences over periods of months. The data analysis tries to figure out the relationship between the observed time series and the process manipulations e.g. by feeding the digester. It is observed that the  $m$  and  $-wr$  are fully reversible during short periods of stable digester operation. Biogas samples with added VFA in different concentrations show that the cross sensitivity of the sensors to chemically active components in the biogas does not affect the applicability of the laboratory calibration for evaluating the biogas data. The maximum values of the dynamical sensor signal functions  $m$  and  $-wr$  yield the sensitivity coefficients  $S_k$  of the sensors indexed with  $k$ . From the  $S_k$  a probability density function  $p(S_k, t)$  is calculated for the fact that a certain combination of VFA exceeds a given limit,  $t$  is the time. The stochastic analysis for the 1<sup>st</sup> passage time  $t^*$  reacts sensitively on the treatment of the digester.  $t^*$  is assumed to indicate a critical event as if the digester runs into sour condition.

# Multivariate process control charts based on reputed process indicators as an alternative to univariate control charts for the monitoring of anaerobic reactors submitted to acidosis



Sébastien Lemaigre<sup>2</sup>, Gilles Adam<sup>1</sup>, Xavier Goux<sup>2</sup>, Philippe Delfosse<sup>2</sup>

1) Environmental Research and Innovation Department (ERIN), Luxembourg Institute of Science and Technology (LIST)  
 2) University of Liège, Arlon Campus Environnement, Belgium

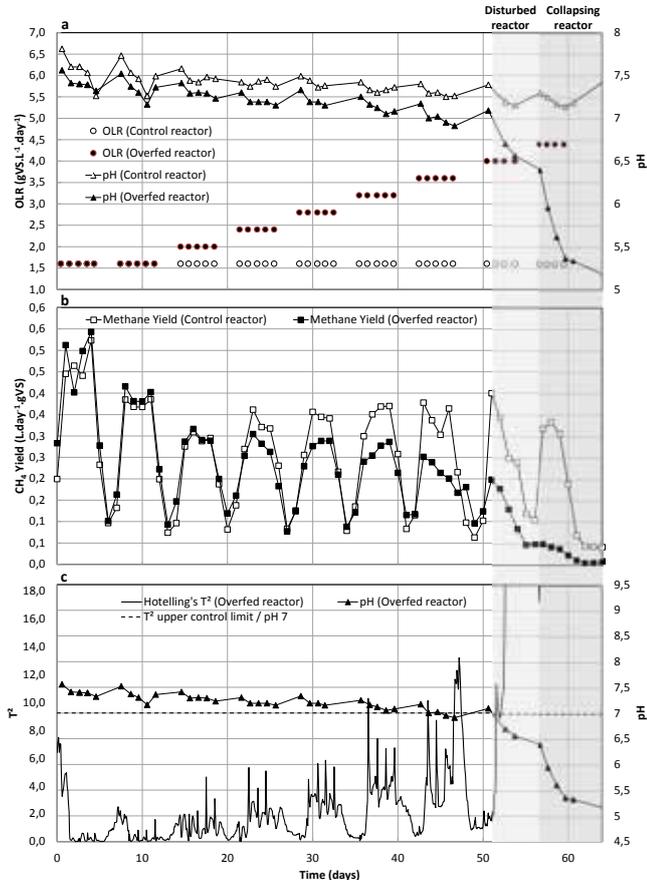
Contact: sebastien.lemaigre@list.lu

**Keywords:** Anaerobic digestion, monitoring, Hotelling's T<sup>2</sup>, multivariate statistical process control

The anaerobic digestion (AD) process (syn. biomethanation) has a high potential to contribute to sustainable energy production because it is one of the most advanced options available to convert especially wet biomass into multipurpose fuels (CH<sub>4</sub> and H<sub>2</sub>), valuable products to serve the green chemistry/biorefinery sectors, and fertilizers readily available to agriculture. Nevertheless, a major limitation to further development of the sector is the difficulty to keep the anaerobic flora of the digesters in optimal conditions of activity. Numerous methods have been assessed to properly monitor the process but none appears to be ideal (Madsen et al., 2011). Most of them consist of measuring individual parameters judged to reflect the capacity of the flora to convert biomass into biomethane with an optimal yield without taking into account the potential correlation between the measured signals (Lay et al., 1998, Boe et al., 2010). Nevertheless, the monitoring of biological process as biomethanation is often characterized by a high degree of correlation between the measured variables and univariate monitoring methods are limited for reflecting such interactions. Interesting alternatives to handle these complex datasets reside in multivariate approaches and were recently promisingly applied to monitoring of a real-scale biomethanation unit using an electronic nose (Adam

et al., 2015). Among these methods, control charts based on the Hotelling's T<sup>2</sup> are probably the most popular for monitoring industrial processes. If these control charts can be constructed on the basis of the raw value of the individual measured parameters, another option consists in performing principal components analysis (PCA) on the dataset prior to compute the Hotelling's T<sup>2</sup> statistic using the scores obtained on a restricted number of principal components. The benefits are double:  
 (1) Retaining in the model only the amount of information that is necessary for a correct interpretation of the process status in eliminating the meaningless data (i.e. the noise);  
 (2) Allowing the construction of contribution charts to enable the process manager to identify which variables were responsible for the alarm and to emit the correct diagnostic about the related situation.

This study aimed to (1) assess the potential of Hotelling's T<sup>2</sup> charts built from individual parameters commonly recognized as process status indicators to interpret the behaviour of lab-scale continuously stirred anaerobic reactors (CSTR) submitted to an increasing organic loading rate (OLR) until intoxication due to volatile fatty acid accumulation (i.e. acidosis); (2) and evaluate if this multivariate approach provides an added value compared to classical univariate monitoring methods (individual Shewhart's X-bar charts). In this framework, an overfeeding campaign was performed with 4 CSTR (100 L) and consisted to (1) submit a triplicate of reactors to increasing OLR, (2) maintain 1 control reactor in cautious feeding conditions. Experiments were conducted in the mesophilic temperature range (37 °C) using sludge from the anaerobic digester of a wastewater treatment plant



Figures 1a, b and c: Time series of reactor state monitoring for the control reactor and 1 overfed reactor. a. OLR and pH; b. Methane yield; c. Relationship between T<sup>2</sup> and pH for the selected overfed reactor.

as inoculum and dried sugar beet pulp as unique feeding substrate. After inoculation, the 4 reactors were identically fed (daily, during working days) with a low OLR ( $1,5 \text{ gVS.L}^{-1}.\text{day}^{-1}$ ) allowing them to reach a steady digestion rate with an hydraulic retention time of 30 days ( $\text{HRT} \sim 30$  days). Then, 1 reactor was defined as a control and maintained in unchanged conditions whereas the OLR of the 3 other reactors was increased weekly with an increment of  $0,5 \text{ gVS.L}^{-1}.\text{day}^{-1}$  until process failure, diagnosed by a brutal pH drop and an interruption in the  $\text{CH}_4$  production (Figure 1a, figure 1b). During all the experiment duration, hourly biogas production,  $\text{CH}_4$ ,  $\text{CO}_2$  and  $\text{H}_2$  concentrations were continuously measured in the gas phase. Total solids (TS), volatile solids (VS), total inorganic carbon (TIC) and total ammonia nitrogen (TAN) were measured in the liquid phase. On one hand, Hotelling's  $T^2$  control charts were constructed for the overfed reactors on the basis of this dataset (after principal components analysis), using the measurements performed on the control reactor to define the in-control status of the process and to build the monitoring model. For each detected event, contribution charts were computed to properly visualize the influence of each individual variable. On the other hand, univariate X-bar charts were computed for each individual parameter, using the data set of the control reactor to define their control limits.

The results showed that:

(1) Univariate control charts delivered a high rate of unjustified alarms whereas the process indicators (pH and  $\text{CH}_4$  yield) did not authorize to predict a dysfunction. In addition, such a large number of individual control chart did not appear as a convenient method to evaluate the progression of the reactor condition from steady state to acidosis,

(2) The unique signal delivered by Hotelling's control charts progressed in good accordance with the sludge pH and reached its critical level immediately prior process collapsing (Figure 1c),

(3)  $\text{CH}_4$  and  $\text{CO}_2$  were the first parameters to cause an increase of the  $T^2$  above its upper control limit prior to acidosis (Figure 2a).  $\text{CH}_4$ ,  $\text{CO}_2$  and  $\text{H}_2$  concentrations in the gas phase brought the major overall contribution in the detection of abnormal events (Figure 2b).

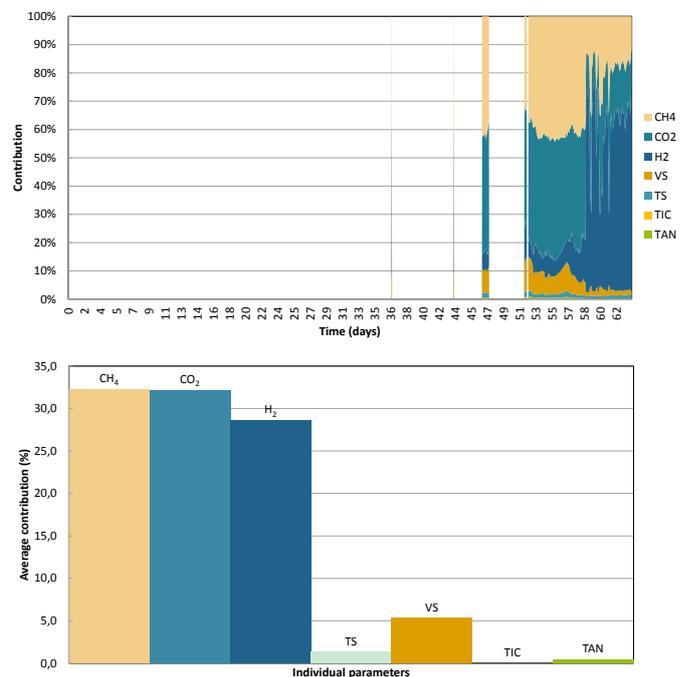


Figure 2: Contribution of the individual parameters in the computation of  $T^2$  values exceeding upper control limit for the selected overfed reactor. a. According to measurement date; b. Averaged contribution for a typical out-of control situation.

## References

- Adam, G.; Lemaigre, S.; Goux, X.; Delfosse, P.; Romain, A. C. (2015): Upscaling of an electronic nose for completely stirred tank reactor stability monitoring from pilot-scale to real-scale agricultural co-digestion biogas plant. In: *Bioresource technology*, 178, 285-296.
- Boe, K.; Batstone, D. J.; Steyer, J. P.; Angelidaki, I. (2010): State indicators for monitoring the anaerobic digestion process. In: *Water research*, 44 (20), 5973-5980.
- Lay, J. J.; Li, Y. Y.; Noike, T. (1998): The influence of pH and ammonia concentration on the methane production in high-solids digestion processes. In: *Water Environment Research*, 70 (5), 1075-1082
- Madsen, M.; Holm-Nielsen, J. B.; Esbensen, K. H. (2011): Monitoring of anaerobic digestion processes: A review perspective. In: *Renewable and Sustainable Energy Reviews*, 15 (6), 3141-3155

## Optimization of anaerobic percolation processes and method development for substrate characterization



Harald Wedwitschka, Earl Jenson, Jan Liebetrau  
 Contact: harald.wedwitschka@dbfz.de

**Keywords:** substrate characterisation, anaerobic percolation processes

Anaerobic treatment of organic residues and wastes can be accomplished with several technologies. Dependent on the substrate characteristics the technology promising the largest energy output under the condition of minimal energy input and high reliability should be selected. The garage digester system features a low sensitivity to disturbing material in the substrate, a low technical effort and a low energy input. Therefore those systems can be utilized for any kind of wastes. However, one crucial factor of the treatment is the percolation of liquid through the substrate heap. The percolate serves as transportation medium, it drains intermediates out of the process and transports microorganism in. The even distribution of percolate and consequently the permeability and the structure of the substrate are crucial factors for the process. However – so far there are no scientific methods available to relate the substrate characteristics and the performance in such an anaerobic treatment.

Figure 1: Material characterisation of different feedstocks, carrier materials and digestates

### Feedstocks

Dry cattle manure

Cattle slurry

Corn silage

Bio waste

Carrier Materials

Wheat straw

Cornstover

Woodchips

Tireshred

Grit materials



Regarding this background, it was the aim to develop a substrate characterisation method which allows to quickly quantify material suitability with regards to permeability and structure.

By developing quantification methods for various feed stocks anaerobic digestion process options will be expanded and process performance will be predictable. In order to find such a physical characterization method for different substrates, analysis with potential substrates, digestates and structure or matrix building materials have been performed. The effect of the structure on the digestion process has been verified by means of dry digestion trials at pilot plant scale. Studies have been

performed on the relation of the substrate characteristics and the performance in an anaerobic treatment. A variety of materials (feedstocks, carrier materials and digestates) were collected for material characterisation tests and for digestion trials in pilot scale. The material characterization and assessment tests were generally based on standard methods found in ASTM or DIN standards with some adaptations. The tests included bulk density, wet density, compacted wet density, waterholding capacity, permeability and compactibility, pore space, kinematic viscosity of the percolate during a trial and (BMP) biomethane potential tests.

In total 50 different sample materials were characterized and the test results have been analysed statistically. The outcomes of material characterisation and the results of 15 dry digestion trials were studied for correlating patterns. The results validate that substrate permeability and structure integrity is eminent for anaerobic percolation processes and showed that material structure decreases during digestion leading to a reduced permeability of the substrate. A lab test procedure has been developed with which the permeability and volumetric size reduction of material mixtures can be dynamically measured under compaction. The methodology can be used for the conditioning of substrate mixtures with focus on a good permeability and structure. Gas potential analyses have shown that substrates without added structure materials and without percolation achieved very low gas yields. With the help of the material characterization tests adapted mixtures of substrates and carrier materials have been prepared. It could be shown that a material mixture of substrate, straw and wood chips achieved better structural resistance and permeability compared to the sole substrate. Based on the measurement results, substrate mixtures were optimized for pilot scale fermentation experiments. The optimized material mixtures achieved up to 80 % of the maximum gas yield in digestion trials.

## Hydrothermal lysis: A method for controlling the output of biogas and digestate



Adam Cenian<sup>1</sup>, Tadeusz Zimiński<sup>1</sup>, Jacek Dach<sup>2</sup>, Andrzej Lewicki<sup>2</sup>

1) Instytut Maszyn Przepływowych PAN im. Roberta Szwalskiego, Gdańsk, Poland

2) Instytut Inżynierii Biosystemów, Uniwersytet Przyrodniczy w Poznaniu, Poznań, Poland

Contact<sup>1</sup>: cenian@imp.gda.pl

Contact<sup>2</sup>: jdach@up.poznan.pl

**Keywords:** hydrothermal lysis, biogas, digestate

Problems associated with disposal and treatment of sewage sludge and some organic-waste digestates remain a challenge for the waste industry. Especially sewage sludge utilisation generates serious problems (e.g. waste water pollution, disposal problems, high treatment costs). Therefore, investigations on the development of a technology reducing the digestate/sewage sludge amount and increasing the biogas output are done in various research institutions. For instance, Hydrothermal Carbonization (HTC) technology is beginning to be used for the treatment of sewage sludge in plants capable of treating the sewage output of small towns. One practical example is the current implementation of the HTC technology in a demonstration plant of the Municipal Water Management and Waste Disposal Company in Halle, Germany.

In this abstract the preliminary results of the investigation of Hydrothermal Lysis (HTL) is presented. HTL is applied as a pre-treatment step for sewage sludge before its repeated fermentation. In summary, an increased production of biogas and a substantial decrease of sewage sludge were achieved.

The main concept of the method proposed comprises firstly, an HTL application for the pre-treatment of sewage sludge or other digestate and secondly, the repeated use of the resulted hydrolysate as a substrate for anaerobic fermentation. Before the hydrolysate can be successfully used as fermentation substrate a procedure for removal of nutrients (especially N-NH<sub>4</sub> and phosphorus), which inhibit the fermentation process if occurring in high concentrations, must be applied.

The sewage sludge used in these studies was collected from water cleaning facilities applying anaerobic digestion with pre-treatment (CAMBI procedure - Bydgoszcz facility, 22 days of fermentation) or without (Gdańsk facility, 28 days of fermentation). In the case of sewage sludge from the facility without pre-treatment application 500g sludge (21,43 % d.m. including 65,17 % o.d.m.) was dissolved in 250 g water before HTL was applied. The reason for that was to avoid local overheating and carbonisation. In the case of sewage sludge from CAMBI technology 400 g of sludge (27,4 % d.m. including 59,5 % o.d.m.) was dissolved in 400 g of water. The HTL procedure was performed in a 1000 mL closed reactor which allows heating the sludge up to 100 – 200 °C under pressure of 3 – 20 bar. In the experiments the sludge was heated up to 100 °C before the heating system was turned-off.

Although, the heating was turned-off, the temperature of the sludge increased further due to heat transfer from the reactor walls as well as from exothermal reactions of the heated sludge. Depending on the sludge origin the maximum temperature range reached was 120 – 200 °C. The temperature of the sludge (about 145 – 160 °C) was held 4 hours by regulating the heating set-up with a simple electronic control system.

The hydrolysate generated (showing tendency to separate into liquid and solid parts) was dewatered in a centrifuge. The chemical oxygen demand (COD) of the sedimentation fluid from the hydrolysate (containing 12 – 18 % d.m.) was 42000 – 86000 mg O<sub>2</sub>/dm<sup>3</sup>. The resulting dewatered solid part constituted 30 – 40 % of the initial volume of the sewage sludge. For further fermentation the total hydrolysate or only the fluid part, respectively can be used. An increasing biogas output of up to 30 % can be achieved for some water cleaning facilities; the produced biogas contains 64 – 75 % of methane. If biogenic inhibitors are not removed the biogas production is hindered in the following repeated fermentation process.

In conclusion, the application of the HTL technology is a promising way to straightforward control of the amount of digestate / sewage sludge (by up to 60 %) and biogas (up to 30 %) production.

# **2<sup>nd</sup> Conference on** Monitoring & process control of anaerobic digestion plants

Exhibition



## **Bioprocess Control AB**

Scheelevägen 22, 223 63 Lund, Sweden

Associate Prof. Jing Liu  
tel. +46 46 163951  
E-mail: [jl@bioprocesscontrol.com](mailto:jl@bioprocesscontrol.com)

Bioprocess Control is a technology and market leader in the area of advanced instrumentation and control technologies for research and commercial applications in the biogas industry. The company brings to market more than 16 years of industry leading research in the area of instrumentation, control and automation of anaerobic digestion processes. Today Bioprocess Control has products exports to more than 40 countries and regions.

### Potfolio:

Bioprocess Control's flagship product the Automatic Methane Potential Test System or AMPTS has quickly become the preferred analytical instrument around the world for conducting biochemical methane potential (BMP) tests. The company also offers a portfolio of smart instruments for substrate analysis, process simulation, gas flow measurement, as well as a series of bioreactors. This comprehensive and broad product portfolio allows the company to offer technology solutions that can both stabilise and unleash the true potential of a biogas plant.

Further information:

[www.bioprocesscontrol.com](http://www.bioprocesscontrol.com)



## Boreal Europe BV

Distributieweg 62, 2645EJ, Delfgauw, The Netherlands

Michael Sosef  
tel. +31653590747  
E-mail: michael.sosef@boreal-laser.nl

Manufacturer of "Open Path" measuring equipment. The technology we use is "Tuneable Laser Absorption Spectroscopy" in the near infrared region. We supply portable systems for research and temporary campaigns and "fixed" systems for permanent monitoring. Either safety or emissions.

Further information:

[www.boreal-laser.nl](http://www.boreal-laser.nl)



## DBFZ - Deutsches Biomasseforschungszentrum gGmbH



The work of the DBFZ is centered on politically relevant issues, such as how the limited availability of biomass resources can contribute in the most efficient and sustainable manner to existing, as well as future energy system. The DBFZ monitors and evaluates the most promising fields of application for bioenergy in theory and practice, supported through various collaborative research projects, carried at both national and international level, with partners and stakeholders ranging from industry, academia and various scientific research associations. The project orientated research provides scientifically-based results to support informed decision making governmental and non-governmental organizations, and adjacent industrial sectors in the energy, agriculture and forestry, while also identifying areas for further research. The scientists of the DBFZ are represented as experts in bioenergy research due to their excellent technical expertise and their presence in numerous national and international committees.

### Biochemical Conversion Department

#### Research focus areas:

- Characterisation and design of anaerobic processes
- Process monitoring and simulation
- Biogas technology
- System optimisation
- UFZ Working Group „MicAS“

#### Equipment:

- Biogas lab
- Biogas pilot plant
- Emissions measurement systems

#### Further information:

DBFZ Deutsches Biomasseforschungszentrum gGmbH  
Torgauer Straße 116 | D-04347 Leipzig | Germany  
Tel.: +49 341 2434 716  
E-Mail: [jan.liebetrau@dbfz.de](mailto:jan.liebetrau@dbfz.de)

[www.dbfz.de](http://www.dbfz.de)

**Landesbetrieb  
Hessisches  
Landeslabor**



## The Hessen State Laboratory

We are point of contact for Hessian consumers at our locations in Gießen, Kassel, Bad Hersfeld, Frankfurt and Wiesbaden. Our scientific staff carries out tests and analyses, gives expert opinions as well as provides advisory services for you on-site. We cooperate closely with state veterinary and consumer protection offices and thus monitor consumables from along the food production chain... from field to plate.

### **Pooled competences**

Scientists from a variety of fields, lab personnel and administrative staff work at LHL. We are at your service throughout Hessen concerning preventative consumer protection!

Our range of services includes

- Testing and analysis
- Providing advice
- Training

### **Agricultural testing and analysis**

We examine and analyse

- Soil
- Fertilizer
- Fodder and plant products
- Agricultural products

### **Environmental and trace analytics**

In our laboratories, we examine

- Fodder
- Seeds
- Fertilizer
- Agricultural products
- Foods for residues and contaminants.

In environmental analytics, we examine and analyze

- Water
- Waste
- Deposits of toxic waste.

We monitor industrial inducers and check laboratories for their licence status.

### **Biogas production and monitoring**

At our laboratory in Bad Hersfeld we are specialized on the anaerobic process in biogas plants. On behalf of our customers we investigate all parameters relevant for the monitoring of the anaerobic biological process and the evaluation of the substrates quality. For practical and scientific purposes we seek for ways to improve anaerobic digestion and monitoring of anaerobic digestion.

### Further information:

LHL - Headquarters / The Hessen State Laboratory  
Schubertstraße 60, Haus 13  
35392 Gießen  
Tel.: +49 (0641) 4800 - 555  
E-Mail: [poststelle@lhl.hessen.de](mailto:poststelle@lhl.hessen.de)



## Helmholtz Centre for Environmental Research

As an international competence centre for the environmental sciences, the Helmholtz Centre for Environmental Research (UFZ) investigates the complex interactions between mankind and nature under the influence of global change. In close cooperation with decision-makers and stakeholders, scientists at the UFZ develop system solutions to improve the management of complex environmental systems and to tackle environmental issues. The Helmholtz Centre for Environmental Research - UFZ was established in 1991 and has more than 1,100 employees in Leipzig, Halle/S. and Magdeburg.

### Portfolio

- LEIPZIG FOAM TESTER – Test set for measuring foaming propensity of substrates for biogas plants
- Stable isotope fingerprinting of biogas as a tool for process monitoring of anaerobic digesters (Nikolausz et al. 2013, Lv et al. 2014a,b)
- High-resolution single cell analysis by flow cytometry as a tool for process monitoring of anaerobic digesters (Koch et al. 2013a-d, Koch et al. 2014a,b) incl. bioinformatics tool for data analysis and interpretation (Schumann et al. 2014a,b)
- Biocalorimetry as a tool for monitoring and controlling anaerobic microbial processes (Maskow 2013, Paufler et al. 2013)
- Electrochemical sensors for process monitoring and control of anaerobic digesters

### References

- Moeller, L.; Krieg, F.; Beyer, D.; Köster, Y.; Müller, R.A.; Zehndorf, A. (2014): LEIPZIGER SCHAUMTESTER - Testset und dessen Verwendung zur Bestimmung der Schaumneigung von Substrat in Biogasanlagen. In: Thrän, D.; Pfeiffer, D. (ed.): Festschrift Konferenz „Fünf Jahre Förderprogramm „Energetische Biomassenutzung“: Wege zur effizienten Bioenergie“, 2014, pp. 170-175.
- Moeller, L.; Görsch, K. (2015): Foam formation in full-scale biogas plants processing biogenic waste. In: Energy, Sustainability and Society 2015, 5-1. doi:10.1186/s13705-014-0031-7.
- Lv Z, Hu M, Harms H, Richnow HH, Liebetrau J, Nikolausz M (2014a) Stable isotope composition of biogas allows early warning of complete process failure as a result of ammonia inhibition in anaerobic digesters. *Bioresource Technol* 167:251
- Lv Z, Leite AF, Harms H, Richnow HH, Liebetrau J, Nikolausz M (2014b) Influences of the substrate feeding regime on methanogenic activity in biogas reactors approached by molecular and stable isotope methods. *Anaerobe* 29:91
- Nikolausz M, Walter RF, Sträuber H, Liebetrau J, Schmidt T, Kleinsteuber S, Bratfisch F, Günther U, Richnow HH (2013) Evaluation of stable isotope fingerprinting techniques for the assessment of the predominant methanogenic pathways in anaerobic digesters. *Appl Microbiol Biotechnol* 97:2251
- Koch C, Brumme B, Müller S (2013a) Mikrobielle Gemeinschaften im Visier der Durchflusszytometrie : Aktivitätsanalyse per Barcode. *Biospektrum* 19:396
- Koch C, Fetzter I, Harms H, Müller S (2013b) CHIC—an automated approach for the detection of dynamic variations in complex microbial communities. *Cytom Part A* 83:561
- Koch C, Fetzter I, Schmidt T, Harms H, Müller S (2013c) Monitoring functions in managed microbial systems by cytometric bar coding. *Environ Sci Technol* 47:1753
- Koch, C., Harnisch, F., Schröder, U., Müller, S. (2014a) Cytometric fingerprints: evaluation of new tools for analyzing microbial community dynamics. *Frontiers in Microbiology* 5, art. 273
- Koch, C., Müller, S., Harms, H., Harnisch, F. (2014b) Microbiomes in bioenergy production: From analysis to management. *Curr. Opin. Biotechnol.* 27:65
- Schumann, J., Koch, C., Günther, S., Fetzter, I., Müller, S. (2014a) flowCHIC: Analyze flow cytometric data using histogram information. R package version 1.0.0  
<http://www.bioconductor.org/packages/release/bioc/html/flowCHIC.html>
- Schumann, J., Koch, C., Günther, S., Fetzter, I., Müller, S. (2014b) flowCyBar: Analyze flow cytometric data using gate information. R package version 1.2.0. <http://www.bioconductor.org/packages/release/bioc/html/flowCyBar.html>

### Further information:

Helmholtz Centre for Environmental Research – UFZ  
Permoserstraße 15 | 04318 Leipzig | Germany  
tel +49 341 235-0 | Fax +49 341 235-2791  
[info@ufz.de](mailto:info@ufz.de) | [www.ufz.de](http://www.ufz.de)



## BINDER GmbH

### COMBIMASS® Biogas Flow Metering and Analysis

Binder Group supplies for many years world-wide innovative gas flow metering and control systems for different industrial applications. The COMBIMASS® thermal dispersion biogas flow meter measures the biogas flow unaffected by pressure and temperature fluctuations. They even have an integrated humidity correction and determine the dry biogas flow at standard conditions according to DIN1343. Also in CDM-projects with special requirements on accuracy and recalibration, our high-quality and very precise biogas flow meter can be used. COMBIMASS® portable and stationary biogas analyzer systems fulfill all market requirements.

Only by linking both gas flow and quality parameters brings great advantages in accuracy and process control. Binder offers both under one roof.

#### Further information:

Isabella Dürr  
Buchbrunnenweg 18, 89081 Ulm  
tel. +49 731 18998-14  
E-Mail: [isabella.duerr@bindergroup.info](mailto:isabella.duerr@bindergroup.info)

[www.bindergroup.info](http://www.bindergroup.info)



## Umwelt- und Ingenieurtechnik GmbH Dresden

Umwelt- und Ingenieurtechnik GmbH Dresden (UIT) is a German manufacturer and supplier of Biogas Test Equipment. With the focus to provide high quality and reliable testing equipment we are helping to optimize processes for biogas plant operations and provide useful tools for biogas analysis, research and development with the goal to optimize digester operation and maximize methane production.

As a classic Engineering Company with own production facilities we are in position to provide standard equipment, but also custom-made biogas testing systems that is specific to clients technical requirements - from laboratory scale to mobile container based solutions.

Typically our plants come with:

- Bioreactor vessels
- Stirring units and tools
- Measurement equipment for gas quantity, gas quality, pH and temperature as well as additional parameters
- Data acquisition systems and software solutions



Figure 2: Reactor module incl. heating controller and stirrer



Figure 3: Test-set for identification of frothing of substrates

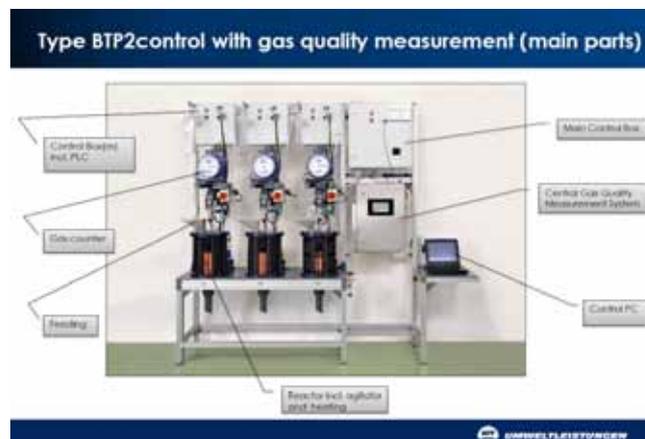


Figure 1: BTP 2 CONTROL incl. central gas analysis module

### Further information:

Umwelt- und Ingenieurtechnik GmbH Dresden  
Zum Windkanal 21 | 01109 Dresden  
Thomas Schneider  
tel +49 (0)3518864682  
t.schneider@uit-gmbh.de | www.uit-gmbh.de

# Notes



# Notes



## **Organiser**

Deutsches Biomasseforschungszentrum gGmbH  
Torgauer Str. 116  
D-04347 Leipzig

Programme support team  
Diana Pfeiffer  
+49 (0) 341-2434-554  
diana.pfeiffer@dbfz.de

## **Scientific coordination**

Dr. Jan Liebetrau  
+49 (0) 341 2434 716  
jan.liebetrau@dbfz.de

**[www.energetische-biomassenutzung.de](http://www.energetische-biomassenutzung.de)**