



des Förderprogramms für energetische Biomassenutzung

# III. Conference on Monitoring & Process Control of Anaerobic Digestion Plants



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# **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 3<sup>rd</sup> International Conference on "Monitoring & Process Control of Anaerobic Digestion Plants" focuses on the requirements of measurement tools, best practice and practically implemented applications of monitoring and control devices.

Furthermore as special events we are proud to announce the Technical Workshop on Biomethane Production in Small to Medium Scale Units (28.03.2017) and the AquaMak conference on Aquatic macrophytes - Ecologically and economically optimized use (30./31.03.2017).

The first workshop will present and discuss the most promising technology solutions along the biomethane supply chain in the sectors substrate pretreatment, digestion and gas upgrading for the use of biomethane as a fuel or for feed-in to the natural gas grid. The second event is held in german and will deal with favorable utilization strategies for aquatic plants.

The 3<sup>rd</sup> International Conference on "Monitoring & Process Control of Anaerobic Digestion Plants" is organized by the program "Biomass energy use" (funded by the Federal Ministry of Economic Affairs), DBFZ Deutsches Biomasseforschungszentrum gGmbH (German Biomass Research Centre), the Helmholtz Centre for Environmental Research (UFZ) and the Hessian State Laboratory (LHL).

# Topics of the conference at a glance

- » Process simulation & control
- » Monitoring and control of plant efficiency
- » Practical experience of process monitoring and control
- » Laboratory measurements: Reliability & validity
- » Monitoring for safety and emission reduction purposes
- » Microbiological analysis: Potential for process characterization and optimization

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

The organizers

32 PRESENTATIONS
& 21 POSTER
FROM 14 COUNTRIES
1 POSTER-AWARD
1 COMPANY
EXHIBITION

# **PROGRAM**

# 1ST DAY

2017-03-29

# 10:00

# Conference opening & Welcome Room 1A

Michael Nelles (DBFZ/Universität Rostock) & Jan Liebetrau (DBFZ)

# Opening session

Chair: Jan Liebetrau (DBFZ)

# 10:10 -10:40 | 10' discussion

Instrumentation and control of anaerobic digestion processes: A review and some research challenges

Jean-Philippe Steyer (LBE - INRA Narbonne Laboratory of Environmental Biotechnology)

# 10:50 -11:15 | 10' discussion

Measuring concept for the biogas measuring programme (BMP-III) Hans Oechsner (University Hohenheim)

# 11:25 -11:45 | 10' discussion

Evaluation of process data from operation of real scale biogas plants – lessons learnt and approaches for practical plant optimisation based on continuous process monitoring Frank Scholwin (Institute for Biogas, Waste Management & Energy)

**11:55 – 13:00** | Lunch break Foyer

# **Session A**

# Laboratory measurements: Reliability & validity

### Room 1A

Chair: Fabian Jacobi (Landesanstalt hessisches Landeslabor, LHL)

# 13:00 - 13:20

The role of inoculum on biomethane potential tests: influence on biogas yield and kinetic parameters

Nils Engler (University of Rostock)

# 13:30 - 13:50 | 10' discussion

The influences of total solids on volatile fatty acids measurement with four titrations in anaerobic digestion process treating pig manure

Shubiao Wu (College of Engineering, China Agricultural University, Beijing, China)

# 14:00 - 14:20 | 10' discussion

Prerequisites for reliable VFA quantification from anaerobic digestion systems Andreas Wagner (Institute of Microbiology, University of Innsbruck)

# 14:30 - 14:50 | 10' discussion

Reliability of VFAs measurements: Evaluation of GC versus HPLC techniques and external standard versus internal standard methodologies

Francisco Raposo Bejines (Departamento de Biotecnología Instituto de la Grasa, CSIC)

# **15:00 – 15:30** | Afternoon break Foyer

# **Session B**

# Monitoring & efficiency of plant operation

### Room 1A

Chair: Frank Scholwin (Institute for Biogas, Waste Management & Energy)

# 15:30 - 15:50 | 10' discussion

Long-term evaluation and modelling the energy efficiency of commercial scale biogas plants

René Casaretto (Flensburg University of Applied Sciences)

# 16:00 - 16:20 | 10' discussion

Detection of short-circuits in full-scale anaerobic digesters using a tracer and CFD simulations in order to optimize the mixing system

Anne Kleyböcker (GFZ German Research Center for Geosciences)

# 16:30 - 16:50 | 10' discussion

Monitoring of operational methane emissions from pressure relief valves of agricultural biogas plants

Torsten Reinelt (DBFZ)

# **Poster Session**

# 5' presentations

Chair: Fabian Jacobi (Landesanstalt hessisches Landeslabor, LHL)

# 17:00 - 18:15

# Room 1A

5' – Modelling anaerobic digestion: From ADM1 to NGS-based metabolic network approaches

Florian Centler (UFZ, Department of Environmental Microbiology)

5' – Anaerobic digestion and near infra-red spectroscopy. Application to process monitoring through the development of five AD parameters models Ronan Tréguer (SEDE) 5' – Development of universal early warning indicators to prevent process failures in single-stage biogas plants fed with maize silage and cattle manure

Patrick Schröder (GFZ German Research Center for Geosciences)

- 5' Genetic analyses of microbial communities in anaerobic digesters reveal enhanced methanogenesis after calcium nitrate dosage Marina Ettl (Yara Industrial GmbH),Thorsten Stoeck (Technical University Kaiserslautern)
- 5' Studies on the influence of moulded feedstocks on the biogas process and the mycotoxicological status of digestates Mathias Hartel (Bavarian State Research Center for Agriculture)
- 5' Towards demand-driven biogas plants: Real-time determination of biogas quality Stefan Palzer (Laboratory for Gas Sensors, IMTEK University of Freiburg)
- 5' On-line monitoring of volatile fatty acids and hydrogen during anaerobic digestion Jacob Joseph Lamb (Norwegian University of Science and Technology Trondheim)
- 5' Operational and seasonal methane emission of open digestate storage tanks Tina Clauß (DBFZ)

# 19:30 | Networking dinner





# Bayrischer Bahnhof Gasthaus & Gosebrauerei

# Bayrischer Platz 1, 04103 Leipzig

The Bayerischer Bahnhof is situated in the south-east of Leipzig (near the big university hospital). You can reach the Bayerischer Bahnhof by tram #9 or #16 (station Bayrischer Bahnhof) or by S-Bahn #S4 (direction Wurzen/Oschatz) from the main station.



09:00
Introduction
Room 1A
Jan Liebetrau (DBFZ)

# **Session A**

Microbiological analysis: Potential for process monitoring and control

# Room 1A

Chair: Sabine Kleinsteuber (UFZ)

# 09:15 - 09:40 | 10' discussion

Compound-specific stable isotope analysis (CSIA) for the assessment of methanogenesis during anaerobic digestion, Marcell Nikolausz (UFZ)

# 09:50 - 10:10 | 10' discussion

Influence of substrate concentration and composition of the microbial community on the biogas production from different carbohydrates

Ellen Euchner (University of Applied Sciences Hamm-Lippstadt HSH)

# 10:20 - 10:40 | 10' discussion

The community sensor – Online flow cytometry concepts for low latency process control Johannes Lambrecht (UFZ)

**10:50 – 11:20** | Morning break Foyer

# **Session B**

# Sensor concepts & applications

# Room 1A

Chair: Michael Mertig (KSI Meinsberg, TU Dresden)

# 11:20 - 11:40 | 10' discussion

Sensor technology for improved characterization of the liquid phase in anaerobic digestion

Stefan Junne (Technische Universität Berlin)

# 11:50 - 12:10 | 10' discussion

Land ahoy or just a mirage – The cabability of a microbial electrochemical sensor to monitor the AD process

Jörg Kretzschmar (DBFZ)

# 12:20 - 14:40 | 10' discussion

Application of extended Kalman filter as soft sensor for anaerobic digestion plants
Daniel Labisch (Siemens AG, Process Industries and Drives Divisions, Technology and Innovations) & Sören Weinrich (DBFZ)

# **12:50 – 13:50** | Lunch break Foyer



# **Session C**

# Practical experience of process assessment

# Room 1A

Chair: Alastair Ward (University of Aarhus, Department of Engineering)

# 13:50 - 14:10 | 5' discussion

Return of experience of SMART control system in a full-scale Biobed® EGSB reactor Santiago Pacheco-Ruiz (Biothane - Veolia)

# 14:15 - 14:35 | 5' discussion

Process monitoring and control for an anaerobic covered lagoon treating abattoir wastewater

Thomas Schmidt (National Centre for Engineering in Agriculture, University of Southern Oueensland)

# 14:40 - 15:00 | 5' discussion

Condition monitoring and lifecycle management of parts and components in the biogas market

Roman Bobik (Boom Software AG)

# 15:05 - 15:25 | 5 discussion

Gasmanagement of anaerobic digestion plantst

Jürgen Kube (Future Biogas)

15:30 | Afternoon break Foyer

# **Session D**

# Simulation, modelling & control of ADs

Room 1A

Chair: Jean-Philippe Steyer (LBE - INRA Narbonne Laboratory of Environmental Biotechnology)

# 16:00 - 16:20 | 10' discussion

Automatic process control of two-phase dry anaerobic digestion of biowaste for hydrolysis

optimization and biogas enhancement Federico Micolucci (University of Verona, Department of Biotechnology)

# 16:30 - 16:50 | 10' discussion

Parameter estimation in anaerobic digestion

- Critical evaluation of different experimental setups and model structures

Sören Weinrich (DBFZ)

# 17:00 - 17:20 | 10' discussion

Modelling of biogas production from single or serial continuous stirred tank reactors using batch assay data

Alastair Ward (University of Aarhus)

17:30 | Guided tour to the AD research plant (optional)

DBFZ | Torgauer Straße 116 |

04347 Leipzig

**Meeting point: Foyer** 

# **ABSTRACTS**

Jean-Philippe Steyer

# Instrumentation and control of anaerobic digestion processes: A review and some research challenges

# instrumentation, modeling, control, spectroscopy, anaerobic digestion, bioaccessibility

To enhance energy production from methane or resource recovery from digestate, anaerobic digestion processes require advanced instrumentation and control tools. Over the years, research on these topics has evolved and followed the main fields of application of anaerobic digestion processes: from municipal sewage sludge to liquid – mainly industrial – then municipal organic fraction of solid waste and agricultural residues. Time constants of the processes have also changed with respect to the treated waste from minutes or hours to weeks or months. Since fast closed loop control is needed for short time constant processes, human operator is now included in the loop when taking decisions to optimize anaerobic digestion plants dealing with complex solid waste over a long retention time. Control objectives have also moved from the regulation of key variables – measured on-line – to the prediction of overall process performance – based on global off-line measurements – to optimize the feeding of the processes. Additionally, the need for more accurate prediction of methane production and organic matter biodegradation has impacted the complexity of instrumentation and should include a more detailed characterization of the waste (e.g., biochemical fractions like proteins, lipids and carbohydrates) and their bioaccessibility and biodegradability characteristics.

However, even if in the literature several methodologies have been developed to determine biodegradability based on organic matter characterization, only a few papers deal with bioaccessibility assessment. In this review, we emphasize the high potential of some promising techniques, such as spectral analysis, and we discuss issues that could appear in the near future concerning control of AD processes.



Jean-Philippe Steyer

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Contact +33 (0) 468.425.151 (direct: 178) jean-philippe.steyer@supagro.inra.fr Frank Scholwin, Johann Grope

# Evaluation of process data from operation of real scale biogas plants – lessons learnt and approaches for practical plant optimisation based on continuous process monitoring

# biogas plants, biogas process monitoring, recirculation

# Background/motivation

Based on scientific work with innovative biogas technologies (e.g. in EU-project), mentoring of PhD thesis on process optimisation and practical optimisation of existing biogas plants a huge data basis of process data is available. As far as a detailed process data evaluation is possible discovery of optimisation potentials happened which could be used in practice. The knowledge about much scientific work for application of e.g. process models in biogas plants, development of innovative sensors and others shows a very low degree of practical implementation of innovative approaches and ideas.

# Aim of the work

The target of the presentation is to highlight the "behaviour" of practical plant operators in the context of optimisation approaches with regard to their plant performance. We want to show practical successful pathways and methods of data evaluation and challenges with visibility of effects of optimisation measures in practical biogas plants. This includes insight into real scale process data as well as an overview of barriers for practical process optimisation.

# **Key research topics**

- » Biological biogas production process optimisation
- » Operation of real scale biogas plants
- » Process visualisation and modelling
- » Behavioural and risk analysis of / for biogas plant operators

# **Methods**

- » Data evaluation and analysis strategies
- » Challenges of data compression and information losses during data compression
- » Application of process modelling for controlling material throughput through the biogas plant units
- » Economic evaluation of process optimisation approaches

# Results

The presentation will highlight the described methods at practical examples of process optimisation regarding economic optimised recirculation of digestate, recirculation of trace elements through digestate separation, identification and prognosis of process disturbances bases on online data analysis.



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scholwin@biogasundenergie.de www.biogasundenergie.de Nils Engler<sup>1</sup>, Andrea Schüch<sup>1</sup>, Thorsten Jakobi<sup>1</sup>, Michael Nelles<sup>1,2</sup>

# The role of inoculum on biomethane potential tests: influence on biogas yield and kinetic parameters

# Biomethane potential Test (BMP)

# **Background/motivation**

Biomethane potential (BMP) tests are still method of choice for characterizing new feedstock materials for anaerobic digestion (AD). Furthermore, BMP-tests are used to compare different kinds of feedstock e.g. in order to evaluate the effect of different pre-treatment methods. In lab practice however there is no standard equipment and hardly a standard protocol for such tests. Parameters like digester volume, measurement of gas volume, measurement of gas composition, recording interval, mixing conditions etc. can vary in wide ranges from lab to lab. Several efforts have been made in the past in order to standardise test routines. One result is e.g. the German directive VDI 4630.

## Aim of the work

All BMP tests require the use of an inoculum in order to start up the microbial degradation chain. Thus, the background biogas and methane yield, caused by the inoculum, has to be measured separately in blank tests. Type, origin and possible pre-treatment of the inoculum have influence on the BMP-test results, which has been shown in several investigations before. Nevertheless there is need for further understanding in order to make BMP-batch-tests more reliable and meaningful.

One key assumption of BMP-tests is that the background biogas production by the inoculum and the gas production from the substrate do not interfere with each other. Experimental works however suggested that the inoculum is generating more biogas in the presence of a substrate than measured from the blank tests. This so called priming-effect requires more attention, as it affects BMP-test results directly.

Aim of the experimental work presented here is to elucidate the role of inoculum in BMP-tests. Special attention is given to the influence on kinetic parameters and to the occurrence of a possible priming effect.

# Methods

Batch-tests in medium scale digesters with a working volume of 60 L respectively 30 L were carried out in order to study the influence of different inocula on the BMP-test results. Tree different substrates (cellulose, maize silage, wheat straw) were tested with two different inocula (sewage sludge from anaerobic waste water treatment and digester content from an agricultural biogas digester). Each variation was tested in n=6. All tests were conducted in the same experimental run to obtain most homogenous test results.

The same batch-test setup was used for the detection of a possible priming effect. A sequenced fed-batch was conducted starting with 30 digesters of which 6 were used as blank test and 24 were fed with glucose. After 28 days, 18 of the 24 digesters were fed with the same amount of glucose again and 6 digesters were considered as blank test for the second sequence. This pattern continued in the third sequence. A possible priming effect should decrease with each sequence.

Small-scale (500 mL) batch-tests with high temporal resolution recording of biogas volume were performed to assess the kinetics of biogas formation. The tests were carried out using the ANKOM® Gas Production System with a recording interval of 30 minutes.

Digester contents from 20 different biogas digesters were used as inocula and a synthetic standard substrate, imitating maize silage, was used as substrate. All tests including the blanks were carried out in triplicates. Kinetics of biogas formation was evaluated by determining the reaction rate k assuming a first order reaction for the biogas production.

# **Results and Conclusions**

All results gained so far indicate that there is only little influence of origin of the inoculum for long reaction times. Differences in specific biogas yields gained with different inocula were small. The typical relative standard deviation of the experimental setup was approx. 4%, differences caused by different inocula were not statistically significant. This leads to the conclusion that BMP-test results from different labs using different inocula can be comparable when the same experimental protocol is used.

No evidence was found that the inoculum is generating more biogas in the presence of a substrate than measured from the blank tests. This finding supports the assumption that blank tests are suitable to consider the background biogas production from the inoculum. Type and origin of inoculum however influence the kinetic parameters of biogas formation. Big differences in cumulative biogas curves were observed when the same substrate was tested with different inocula. Variation of the reaction rate k was statistically significant. Test results from different labs using different inocula seem therefore not to be comparable in terms of kinetic parameters.

Experiments are still ongoing, further results are expected in the next months.

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Verein Deutscher Ingenieure (2006). VDI Richtlinie 4630: Vergärung organischer Stoffe Substratcharakterisierung, Probenahme, Stoffdatenerhebung, Gärversuche. Berlin, Beuth Verlag.

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# The influences of total solids on volatile fatty acids measurement with four titrations in anaerobic digestion process treating pig manure

# Anaerobic digestion (AD), Titration, Volatile fatty acid (VFA), Total solids (TS) Continuobyus stirred tank reactor (CSTR)

# Introduction

The volatile fatty acids (VFAs) concentration has been considered as one of the most sensitive process performance indicators used in various monitoring and control strategies of anaerobic digestion (AD) process. For routine monitoring and control, the titrimetric technique is generally accepted to be superior in simplicity, speed, robustness, user-friendliness, and cost-effectiveness compared with other methods. Various titration methods have been developed and validated for measuring VFAs concentrations in AD process to a certain degree of accuracy in recent decades. However, the VFA measurement difference by titration is still relatively large compared with lab-instrumental methods. Moreover, most of the titration procedures studied require sample filtration or centrifugation prior to titration, which increases the equipment costs and complexity of VFA monitoring of AD processes with titration. Thus, improving measurement accuracy and simplifying sample preparation are two significant aspects of the VFA-monitoring titrimetric technique that need further breakthroughs to achieve on-site practical application.

# Purpose of the work

The aim of this study was to access the relationship between the different TS content and the VFAs measurement differences by titrations between the centrifuged and non-centrifuged samples. In this study, the VFA measurement differences between the centrifuged and non-centrifuged samples, from four CSTR reactors treating pig manure, by four titrations was analyzed firstly. And, the influence of solid interfering subsystems in titrated samples on VFA measurement by four titration methods was evaluated. These results might assist in improving measurement accuracy and simplifying procedure of VFAs monitoring with titration.

# **Methods**

The two pH-end points method, (Nordmann 1977, N2), and four pH-end points method, (Kapp 1984, K4), classified as linear regression and the two pH-end points method, (Anderson & YANG 1992, A2), and the five pH-end points method, (Moosbrugger et al. 1993, M5), classified as solution of linear algebraic equations, generally used with a high representative in the whole world, were used for VFAs monitoring during the experiment. Titration procedure was conducted with an auto-titrator (China, Shanghai, ZDJ-5) with the titrant adding speed of 0.6 mL/min. The electrical conductivity (EC) and temperature of samples were measured with a handheld conductivity meter (Mettler-Toledo) prior to starting the titration procedure. The consumed titrant (0.05 M H<sub>2</sub>SO<sub>4</sub>) volume and the pH was recorded continuously in the same time. Samples were collected from four laboratory-scale continuously stirred tank reactors (CSTR) treating pig manure and tested from the start-up period to the end of the whole experiment with the frequency of every one to three days. The titration was conducted with 10 mL centrifuged and non-centrifuged samples, and diluted by 40 mL deionized water. Reactors were operated for about 300 days with different organic loading rates (OLR). And, three reactors maintained at  $(37 \pm 1)$  °C and one maintained at  $(55 \pm 1)$  °C. Total solids (TS), pH, ammonium-nitrogen (NH<sub>4</sub>-N), phosphate (PO<sub>4</sub>-P), gas production and composition were regularly monitored.

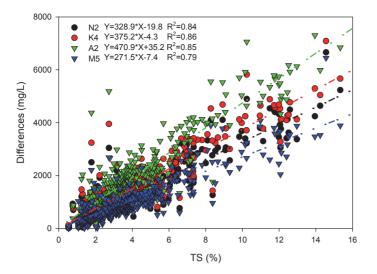


Figure 1 The VFA measurements differences between the centrifuged and noncentrifuged samples by four titrations for four CSTR reactors treating pig manure

### Results and conclusions

In this study, the influence of solid interfering subsystems in titrated samples on VFA measuretment by four titration methods was evaluated based on the analysis of samples from lab-scald pig manure digesters in a long-term continuously routine monitoring with varying OLR condition. The VFA measurement results by titrations from the non-centrifuged samples were higher than the results from centrifuged samples. This mainly attribute to the presence of refractory materials (cellulose and hemicellulose, etc.) and precipitated solids (calcium and struvite, etc.) in the non-centrifuged samples, which can adsorb or react with the titrant during the titration process. Obviously, the over-consumption of titrant cause an over-estimation of the measurement results. The amount of the refractory materials and the precipitated solids in anaerobic digester effluents can be characterized by TS. To quantitatively describe the relationship between TS content in digested samples and the difference in VFA concentrations measured with the centrifuged and non-centrifuged samples, about 450 datas with different TS concentrations (varied from 0.5 % to 15 %) were analyzed. As shown in Figure 1, four promising linear correlation, with coefficients of R2=0.84, R2=0.86, R2=0.85, R2=0.79 (respectively for N2, K4, A2, M5), were established. The liner correlation of the different TS content and the VFA measurement differences by these four titrations might be used for the simplified titration methods (samples were thoroughly mixed rather than centrifuged or filtrated) development and to obtain more accurate results.

For these four titration methods, the measurement differences between the centrifuged and non-centrifuged samples with the same TS were different. The order of the measurement differences from these methods is A2 > K4 > N2 > M5. The differences in mathematicallys and selection of pH end-points of each titration method were the main reason. Furthermore, the comparion of the titration curves shows the over-consumtion of titrant in non-centrifuged samples mainly occurs between pH 5.5 and 7. As shown in Figure 1, the slope of the linear regression curve of N2 (328.9) and K4 (375.2) were quite close, as these two method were in the same category and adopt the closer pH end-points. However, although A2 and M5 method has been classied in the same category, the difference in slope of the linear regression curve was relatively large. This can be attributed to only two pH end-points (5.1 and 3.5) and two subsystems (carbonate and VFAs) were considered in application of A2 method, while five end-points (Initial pH, 6.7, 5.9, 5.2, 4.3) and more subsystems (carbonate, ammonium, phosphate, VFAs and sulfide) were considered in application of M5 method.



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# Prerequisites for reliable VFA quantification from anaerobic digestion systems

# microbiology, ecophysiology, anaerobic digestion, Archaea, VFA extraction, preservation, storage

The anaerobic decomposition process represents a complex interaction of various microorganisms from two domains, the Bacteria and the Archaea. Within this degradation process different phases can be distinguished named hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Gerardi, 2003; Schink, 2002), during which volatile fatty acids (VFAs) appear as intermediate products from the degradation of complex substances on the one hand and the end products of the microbial decomposition process  $\rm H_2$ ,  $\rm CO_2$ , and  $\rm CH_4$  on the other hand.

VFAs are defined as short-chain fatty acids consisting of six or fewer carbon atoms which can be distilled at atmospheric pressure (APHO, 1992). In anaerobic digestion (AD) processes these acids, in particular formic, acetic, propionic, and butyric acid are of central importance for maintaining stable reactor performance and biogas production.

Therefore, they are important process monitoring parameters often used to indicate arising problems or reactor imbalance (Wagner et al., 2014a). However, their role has been discussed controversially but a reliable quantification is crucial for reactor performance assessment (PIND et al., 2003; Wagner et al., 2014b). In the presented study sludge derived form a full-scale anaerobic digester of a wastewater treatment plant (Zirl, Tyrol, Austria) was spiked with formate, acetate, propionate, and butyrate in order to evaluate various commonly used techniques for VFA extraction, preservation, and storage applying HPLC analysis (Wagner et al., 2016). Depending on the respective experiment samples were spiked with  $3.0-5.0~{\rm mmol~kg^{-1}}$  fresh weight each of C1 – C4 VFAs (formate, acetate, propionate, and butyrate, sodium from each) and  $1.8-3.0~{\rm mmol~kg^{-1}}$  fresh weight of phenoxyacetic acid sodium salt (PA) as an internal marker.

Among various tested extraction/separation procedures like filtration, dialysis, and centrifugation it could be shown that VFA extraction after centrifugation (15,000 x g) warranted the highest recovery rates for spiked VFAs. Filtration methods resulted in stable extraction efficiency; however, dialysis dramatically underestimated the spiked VFA concentrations. As a crucial point for reliable VFA quantification from anaerobic digestion systems an immediate sample cooling was figured out. While loss of formate could be limited to 0.37 mmol  $L^{-1*}h^{-1}$  and was < 0.15 mmol  $L^{-1*}h^{-1}$  for other VFAs when a fast sample handling and immediate cooling was secured, uncooled samples completely lost detectable formate and

 $0.62~\mathrm{mmol~L^{-1}*h^{-1}}$  butyrate. For chemical sample preservation prior VFA extraction different Cu and Zn containing chemicals were used. Within a narrow time-frame CuCl $_2$  allowed recovery of 95 % of spiked VFA concentrations, moreover, deep freezing emerged as an alternative to instant VFA extraction. Short time storage of extracted VFA samples at +4 °C was figured out as an option for up to seven days; however, keeping samples longer at this temperature resulted in poor recovery rates. This is particularly important since practically samples have to be stored prior VFA analysis, e.g. samples typically await HPLC analysis using an autosampler at 4 °C. Therefore, for longer periods storage of already extracted samples should secure a temperature of -20 °C in order to allow accurate VFA quantification, where spiked concentrations were found to remain constant for a period of more than one month.

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# Francisco Raposo

# Reliability of VFAs measurements-Evaluation of GC versus HPLC techniques and ESTD versus ISTD standard methodologies

# Analytical performance, analytical method validation, chromatography, interlaboratory study, volatile fatty acids

### Introduction

Measurements of VFAs have a high significance in the anaerobic digestion (AD) research field. By this way, monitoring the concentration of VFAs in anaerobic reactors can be considered as one of the most useful analytical parameters. GC and HPLC are the most common analytical techniques for measuring VFAs profile because both chromatographic techniques are capable of separating the individual components and provide their quantitative determination. Comparable results among laboratories are necessary for the AD research community. Unfortunately, a previous interlaboratory study carried out recently showed a bad overall analytical performance. Taking into account that method validation is a key procedure for testing the suitability of methods as well as the capacity of the analysts and laboratories. Therefore, the way to achieve the comparability of results is through using validated analytical methods.

# **Objectives**

To try to increase the knowledge about VFAs measurements, an international interlaboratory study was carried out among different laboratories, all of them providing services in the AD research field. In this case, the multi-laboratory study was planned including the full analytical validation of different methodologies reported by the participants. By this way, instrument precision, calibration and accuraccy results were reported togheter LOD/LOQ assessment.

# **Materials and methods**

The participating laboratories received all the materials necessary to carry out the analytical validation. By this way, each participant received:

- » Instruction guidelines
- » Standard solutions. Specifically, 18 glass vials containing different aqueous solutions..
- » Fungible materials: volumetric flasks, vials for injection and vials to store some solutions to be prepared in the laboratory.

Instrumental devices and experimental conditions were selected totally free by the partici-

The timetable was set to complete the interlaboratory study within three months after the reception of the materials.

# **Results**

The results reported by 13 participating laboratories (9 using GC-FID and 4 using HPLC) were considered as suitable to further evaluation. Unfortunately, the results of other participants were not considered due to deviations from the established procedure or malfunction to obtain experimental results to reach the correct analytical suitability of the chromatographic systems.

# **Conclusions**

The most important findings from this study were:

- » GC and HPLC can be considered as analytical techniques similarly accurate to carry out VFAs measurements.
- » Both analytical methodologies studied external standard-ESTD and internal standard-ISTD were able to report the different performance parameters studied (instrument precision, accuracy and LOD/LOQ) with no statistical significance. However, ISTD methodology should be considered as valuable due to the normalization of slopes for calibration functions and the incorporation of the quality control concept.



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# Long-term Evaluation and Modelling the Energy Efficiency of Commercial Scale Biogas Plants

# Energy Efficiency in commercial scale, calorimetry, time-series analysis, techno-econmoic assessment

The determination of the efficiency of commercial scale biogas plants is mainly a question of profitability, but also a question of the quality of the digested sludge as a fertilizer, and the environmental impact of the whole supply chain. Particularly, the biogas plants in Germany running on energy crops are highly interested in cheap crops and highly efficient digestion processes. Insofar, an objective and reliable evidence of effect of the measures is essential for the assessment of the operational control and to identify further need for action.

Previous methods for determining the efficiency are based on databases (KTBL, 2013) which are no longer the state of science and technology, like FoTS (Weißbach, 2008) are highly restricted to empiricism derivate from general historic data, and the bio methane potential test needs at least 60 days (Lehner et al., 2009). An analytical procedure only based on the comparison of the physicochemical properties of substrates and products should allow the respective biogas plant allows an objective evaluation of the current efficiency of the system would be possible.

In the presentation a series of common analytical procedures and the evaluation of data collected will be demonstrated (Born et al., 2012). It is based on representative sampling of the residue storage tanks and additionally of the substrates fed, subsequently analyzed for its dry matter and organic dry matter contents and finally its calorific value. The determination of the actual efficiency of the system results from the determination of the specific energy content of the fermentation residual and its comparison with the specific energy content of anaerobically inert but in virtually all substrates present lignin. The determination of the quality and thus the economic value of the substrate is obtained from the measurement of the energy content of the substrate. The comparison of the energy content of the substrate with that of the fermentation residual and the amount of energy generated in each of the CHP design allows the energy balance of the plant and therefore the assessment of losses and slippage. Based on this methodology the efficiencies of more than 140 commercial scale based plants in the Germany were evaluated. The degree of efficiency ranges from 57 % to 86 %.

Even though a single determination of the efficiency of a biogas plant offers already a momentary value and some hints of operational state, a time series analysis will offer the opportunity to get a more reliable insight into the languorous digestion process and the impact of substrate and feeding changes. In a current study 5 different biogas reactor lines of commercial scale biogas plants were examined weekly for 52 weeks. The recorded values were assessed by time series analysis. The therefrom determined energetic efficiencies of the five plants were about 67.0% to 77.4%. During the course of the year the efficiencies fluctuated up to  $\pm 11\%$  per plant. From the fact arises the necessity to measure the efficiency repeatedly or even regularly if reliable economic decisions should be made. The fluctuations in efficiency mainly come from the fluctuating energy input by the substrates. Predigested materials like excrements from animals vary much more in their higher heating value than the energy crops used. Also, changing substrate combinations and higher feeding rates affect the energetic efficiency perceptibly. The long term examination of the efficiency of biogas plants with the use of time series analysis allows it to trace back the changes in energetic efficiency to reasons in the past.

Based on the findings above a model was developed which allows to estimate mass and energy balances for commercial scale biogas plant even though here is a lack of data due to external practical reasons in the running farm management. This model was verified on singular data sets of more than 100 Biogas plants and on five biogas reactor lines in a time series evaluation. The results which will be demonstrated, show that this models offers the opportunity to determine the efficiency not only within days but also over a long period of time.

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# Detection of short-circuits in full-scale anaerobic digesters using a tracer and CFD simulations in order to optimize the mixing system

# Biogas production, Short-circuits, Tracer, Computational Fluid Dynamics, Hydraulic Retention Time

Continuous stirred tank reactors (CSTR) are widespread used as one stage biogas digesters. Hereby, the performance of the biogas process is influenced by the hydraulic retention time (HRT) and the mixing characteristics. The HRT in a CSTR should be equal or greater than one doubling time of the slowest growing microorganism in order to avoid their wash out and thus, to allow for biomass growth and the degradation of organic matter to biogas. Furthermore, mixing is required to avoid dead zones, stagnation zones, and short-circuits and at the best to overcome floating layers and foam formation. Apart from that, the energy demand for the mixing system has to be minimized due to economic and ecological reasons.

At three full-scale anaerobic waste digesters, the methane yield was between 18% and 32% lower than expected. Low concentrations of the volatile fatty acids as well as sufficient methane concentrations in the biogas indicated good biological process performances in the three digesters. Therefore, the residence time distributions of the digesters were investigated using the fluorescent dye uranine as a tracer. The tracer was introduced as an impact load (Dirac Response). In the outflow and at the hydraulic mixing system, the uranine concentration was measured during one to six HRTs. The HRTs ranged between nine and 19 days. The volumes of two digesters were 2300 m³ and for the third one 8000 m³.

The uranine concentration in the digestate dependent on the time resulted in the so called residence time distribution curve. Evaluating this curve revealed short-circuits due to inadequate regulations of the mixing systems as well as the reactor feed and discharge flows. However, the results of the tracer study did not provide detailed information on the characteristics of the flow inside the reactor. Therefore, computational fluid dynamics (CFD) were used for fluid dynamic analyses. CFD simulations showed that small changes in the operation of the mixing systems might severely enhance mixing characteristics and thus, increase the performance of the reactors.



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### Torsten Reinelt

# Monitoring of operational methane emissions from pressure relief valves of agricultural biogas plants

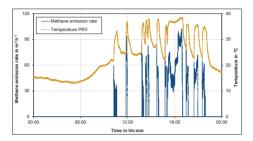
# Operational methane emissions, Pressure relief valves, biogas plants

Biogas plants produce and combust methane to supply electrical and heat energy. The continuous combustion requires the intermediate storage of the produced biogas. In Germany the biogas is mostly stored in low pressure foil storage tanks (lower than 10 mbar overpressure) which are often integrated in the foil roofs of the digesters. Each biogas storage tank has to be equipped with at least one pressure relief valve (PRV) avoiding unacceptable pressure conditions, which could cause damages of the roofs. However, each release event of a PRV simultaneously results in the emission of raw biogas. Since methane is a potent greenhouse gas having a global warming potential of 28 (MYHRE et al., 2013), operational methane emissions from PRV have to be avoided as far as possible. In Reinelt et al. (2016) a method for the long term monitoring of PRVs was developed and validated on two different biogas plants. The method is based on the measurement of the flow velocity and/or the temperature in the exhaust pipe of a PRV. The monitoring of the flow velocity in the exhaust pipe allows the determination of the emitted methane volume flow and the duration of single triggering events. A temperature sensor only registers the triggering of a PRV and the duration of the single triggering events, because the increasing (beginning of an event) and decreasing temperature (ending of an event) is measured. However, advantages of temperature sensors are the much lower investment costs compared to flow velocity sensors. In a current research project ("Operational emissions from biogas plants - BetEmBGA", Funded by the Federal Ministry of Food and Agriculture represented by the Agency for Renewable Resources - FNR, support code: 22020313) the developed method is presently used on four agricultural biogas plants to investigate the relevance of PRVs as a methane emission source from biogas plants. The chosen biogas plants are briefly described in Table 1.

Table 1: Description of investigated biogas plants

	Biogas plant 1	Biogas plant 2	Biogas plant 3	Biogas plant 4			
General	Wet fermentation; Mesophilic temperature level; Manure and energy crops as main substrates; The PRVs are based on a hydraulic weighted system (water seal) with an activation pressure of 3.0 hPa						
CHP (kW <sub>el</sub> )	350 and 180	252 and 190	250	526			
Secondary gas utilisation	Stationary flare, manually operated		Gas boiler; automatically operated (starts only after breakdown of the CHP)				
Digesters (Number of installed PRVs)	Main digester (1) Post digester 1 (1) Post digester 2 (1) Gastight covered dig. storage (1)	Main digester (1) Post digester (2)	Main digester (1) Post digester (1)	Main digester (1) Post digester 1 (1) Post digester 2 (1) Gastight covered dig. storage (2)			
Measurement setup in the upgraded PRVs	Main digester à flow velocity sensor Post digester à temperature sensor	Main digester à flow velocity sensor; temperature sensor Post digester à 2x temperature sensor	Main digester à flow velocity sensor Post digester à temperature sensor	Main digester à flow velocity sensor Post digester à tem- perature sensor			

The emission measurements started in April and May 2016 after the check of the measurement setup by a technical expert according to § 29a of the German Imission Protection law and will go on for a full year. In a next step, further four agricultural biogas plants will be investigated. The measurement results obtained so far show that PRVs are a potential methane emission source of biogas plants. Figure 1 shows the triggering of PRVs in the course of the day.



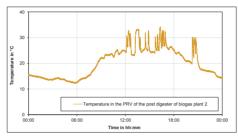


Figure 1 Triggering of PRVs in the course of the day (left: PRV of the main digester of biogas plant 2 equipped with a flow velocity and a temperature sensor; right: PRV of the post digester of biogas plant 3 equipped with a temperature sensor)

The left diagram shows the monitoring of the PRV of the main digester of biogas plant 3, which is equipped with a flow velocity and temperature sensor for the validation of the method. Whenever a flow signal is registered, the temperature also increases, due to the higher temperature of the biogas. When the flow signal stops, the temperature also decreases. This validation shows that for determining the number and duration of triggering events a temperature sensor can be used. The right diagram shows the monitoring of the PRV of the post digester of biogas plant 2, which is only equipped with a temperature sensor. In this figure, the temperature flanks of the single triggering events between 12 and 24 o'clock are also clearly indicated. The evaluation of the emitted methane emission rates as well as the number and duration of the single triggering events will be the next step of the project. The operational methane emissions have to be evaluated together with the operational state of the biogas plants and the atmospheric conditions during the investigation period. For example, a frequent operational cause of triggering of PRVs is the breakdown of the CHP and consequently the missing biogas combustion. Simultaneously, plant operators often work with high filling levels of the biogas storage tanks so that in case of missing biogas combustion, no buffer storage capacity is available.

The first measurement results show that PRVs can be a dominant methane emission source from biogas plants. The permanent monitoring of PRVs is a good option to avoid unwanted methane emissions and to optimize the biogas storage management of biogas plants. The necessary measuring methods are available and have already shown its suitability for this matter (Reinelt et al., 2016).

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# Compound-specific stable isotope analysis (CSIA) for the assessment of methanogenesis during anaerobic digestion

# Compound-specific stable isotope analysis, methanogenesis, ammonia, inhibition, microbial ecology

Biological methane production is associated with an isotope fractionation effect. Stable isotope composition of the produced methane and carbon dioxide is informative about the methanogenesis due to the differences in the fractionation factors of the various methanogenic pathways (Nikolausz et al., 2013). Process inhibition is often associated with shift of the relative contribution of the major methanogenic pathways. Therefore, stable isotope fingerprinting of the produced biogas is a promising tool for monitoring and process control of biogas plants. Although the method is frequently used for environmental samples, it has to be validated for the application in engineered systems.

Anaerobic digestion (AD) will play an important role in the future in the treatment of agricultural wastes including livestock manure. Especially poultry manure is a high potential organic substrate, but it is generally problematic for use in AD due to its high nitrogen content. Ammonia is accumulating during the manure digestion and it is toxic for both bacteria and methanogenic archaea. It has also been reported that acetotrophic methanogens are more sensitive to elevated ammonia concentrations than hydrogenotrophic methanogens. In a previous study, the effect of chicken manure codigestion with maize silage was investigated, and compound-specific stable isotope analysis (CSIA) proved to be an effective early warning tool for complete process failure (Lv et al., 2014).

In the current study, maize silage was used as mono-substrate to avoid the uncertainty introduced by two substrates with different stable isotope compositions in the previous study. Ammonia was added either as ammonium salt or as urea. The inhibitory effect was investigated in batch systems and laboratory-scale semi-continuously operated bioreactors with a reaction volume of 101 under mesophilic temperatures. The compositions of the methanogenic communities were assessed by terminal restriction fragment length polymorphism (T-RFLP) analyses of the mcrA gene according to Bühligen et al. (2016). Potential shifts in the contribution of major methanogenic pathways were further assessed by the stable carbon isotope ( $^{13}$ C/ $^{12}$ C) analysis of methane and carbon dioxide as well as stable hydrogen isotope ( $^{2}$ H/ $^{1}$ H) analysis of methane.

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An important observation of the batch experiment was that a much stronger inhibition was observed in case of urea compared to ammonium chloride treatments; despite the same concentration of total ammonia nitrogen was introduced. This difference was also reflected in the stable isotope composition of the produced biogas. In the experiments with semicontinuous bioreactors, the NH<sub>4</sub>-N level was set to 4g/L with urea in two reactors, while another reactor reached 6g/L final concentration. The specific gas and methane production was lower in the ammonia impacted reactors compared to the control (~ 2 g/L NH<sub>4</sub>-N), but strong process inhibition was only observed in the reactor with highest ammonia concentration (6 g/L). In this reactor, the stable isotope composition of the biogas revealed a shift from a contribution of both methanogenic pathways towards the predominance of hydrogenotrophic pathway.

In the two other ammonia impacted reactors at lower ammonia concentration  $(4\,g/L)$ , the stable isotope composition of the biogas was similar to the ones from the control  $(2\,g/L)$ . This indicates that the relative contribution of the major methanogenic pathways was similar in both reactors with different NH<sub>4</sub>-N level. TRFLP analysis indicated the predominance of the genus of *Methanosarcina*. Members of this genus can utilize both major methanogenic pathways. At highest ammonia concentration  $(6\,g/L)$ , a shift towards the predominance of hydrogenotrophic genus *Methanoculleus* was observed.

CSIA of biogas has a great potential as early-warning tool for indicating process failures caused by ammonia inhibition according to our study, making it a promising technology for process control in biogas plants.

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# Influence of substrate concentration and composition of the microbial community on the biogas production from different carbohydrates

# Biodegradation, process imbalance, model substrates, glucose, starch, cellulose, VFA pattern, microbial community, methane

The project "MOST" (Model Based Process Control of Biogas Plants), funded by the German Ministry for Education and Research aims at optimizing process control of biogas production plants by providing a mathematical model of the anaerobic digestion process calculated from identified key parameters. The consortium consists of ten partners from science and industry producing. In one work package anaerobic biodegradation of model substrates – here cellulose, starch, and glucose – is investigated extensively as a base for mathematically modelling the biodegradation processes in biogas plants. These investigations include a literature review and experimental data.

This paper shows results of bench scale experiments dependent of the concentrations of the biomass and the substrates cellulose, glucose, or starch and associated changes in composition of the microbial community. The fed-batch experiments were performed in 2-L bioreactors equipped with gas meters and sensors for methane and carbon dioxide. Digestate from a biogas plant treating fruit and vegetable wastes was used for inoculation. Total solids and total volatile solids, VOA-TAC (volatile organic acid -to- total anorganic carbon), and pH were measured. VFAs (volatile fatty acids) were analysed with ion exclusion chromatography equipped with a conductivity detector. The microbial community composition was identified using Illumina Amplicon sequencing.

An amount of 209 – 293 mL  $_{methane}/g_{glucose}$ , 275 – 373 mL  $_{methane}/g_{starch}$  and 265 – 356 mL  $_{methane}/g_{cellulose}$  was obtained. During fermentations, the biomass was gradually reduced by 3 to 10 % at an initial substrate concentration of 3 g/L at each substrate feed. The biomass reduction during the experiments with glucose resulted in an increase of the propionic acid concentration (1.8 mmol/L; up to 9.7 mmol/L) and in an increased abundance of Actinobacteria as measured by 16S reads. In the course of the fed-batch experiment with starch, higher concentrations of propionic acid (1.4 – 3.3 mmol/L) and butyric acid (0.8 – 4.4 mmol/L) were detected as well as some additional fatty acids in low concentrations. In the starch reactors the microbial community composition changed mainly in favour of the phylum Bacteroidetes. Representatives of the phylum Fibrobacteres dominated the cellulose-fed reactors. These did not show any elevated concentrations of VFAs. After new inoculation the substrate concentrations were gradually increased by 3 to 5 g/L.

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A concentration of 5 g  $_{\rm glucose}$ /L resulted in a breakdown of the biodegradation process and in accumulation of propionic acid (12.4 mmol/L). Bacteriodetes and Firmicutes decreased in favour of representatives of the phyla Actinobacteria and Cloacimonetes. During the experiment with starch, rising butyric acid production was observed and an increased abundance of Firmicutes and Chloroflexi were measured. The changes in the cellulose concentration caused no significant difference in the VFAs pattern. As before, the microbial community was dominated of the phylum Fibrobacteres.

The biogas and methane yields and the stability of the anaerobic biodegradation process depended not only on the type, but also on the initial concentration of substrate and on the microbial biomass concentration. Reduction of microbial biomass or an increase of the substrate concentration was associated with a more or less impaired degradation process. This effect varied by substrate. The effect was mostly shown during the fed-batch experiments with glucose followed by the experiments with starch. Within repeated feedings, the biomass adapted to all of the substrates tested, but only the adaptation to cellulose occurred without showing an impairment effect.

The experiments are currently continued with glycerol, palmitic acid, oleic acid, and proteins as additional model substrates and will be continued with mixtures of these substrates and complex substrates.



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# The Community Sensor – Online Flow Cytometry concepts for low latency process control

# Microbial community, Flow Cytometry, cofactor  $F_{420}$ , online process control, flexibilisation

After more than a decade of highly dynamic growth fuelled by state subsidies, the German biogas sector will have to face profound changes to keep its position as an integral and profitable part of the energy mix of the future. Utilizing the unique "on demand" capabilities of the biomass – biogas – electricity conversion chain, a focus on flexible bioenergy provision might be a way to go.

Flexible biogas production is an essential cornerstone of this strategy. It most commonly refers to temporally flexible methane provision, but can also encompass other approaches, such as the combined production of chemicals and energy in a two stage system. In contrast to the steady state process doctrine deployed today, comprehensive coverage with low latency process control would be indispensable for a truly flexible and still functionally stable process.

By sensing changes of the microbial community rather than the abiotic parameters which microbial activity is eventually influencing, much faster response times might be achieved. Flow cytometry's cost effective, high throughput single cells analysis, make it a promising candidate for the sensor module of the envisioned process control.

The concepts and methods currently developed along the way to an online / on site flow cytometer will be presented. Current approaches involve flow cytometric community finger-printing and  $F_{420}$  fluorescence based quantification of methanogenic archaea in a dynamically operated industrial scale plug flow digester.

More information on the working group Flow Cytometry at the UFZ and the available data processing algorithms:

https://www.ufz.de/index.php?de=38422 http://bioconductor.org/packages/release/bioc/html/flowCyBar.html http://bioconductor.org/packages/release/bioc/html/flowCHIC.html



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# Sensor technology for improved characterization of the liquid phase in anaerobic digestion

# multiparameter measurement, dynamic feedstock load, electrooptical measurement, cell polarizability, vitality

### Motivation and aim of the work

Monitoring tools for the liquid phase of anaerobic digestion are often restricted to a very few parameters. This might be sufficient for an operation at steady-state conditions, but not suitable at dynamic changes of feedstock and loading rate or when – in general – a poor yield is achieved and process optimization and plant retrofitting is required. Some examples of improved monitoring in the liquid phase in industrial plants, combined with suitable monitoring methods to transfer conditions of the lab into the large scale are presented.

### **Methods and Results**

Two novel techniques are introduced that allow the optimization of biogas production on site. The first method describes an automated vitality measurement of cells based on the polarizability in an electric field. The cells' polarizability is dependent on the membrane integrity and transport capacities of ions inside the cell. When different process conditions are compared, the polarizability provides a good indicator of the ability of cells to convert carbohydrate sugars.

The second technique describes the monitoring of the cell size distribution directly in culture broth based on laserlight back-reflection. This method can be used to evaluate the efficiency of pre-treatment methods. A narrow distribution of small particle sizes indicates a suitable pre-treatment. Both, the monitoring of the cellular polarizability and the particle size distribution, provide further insight about the suitability of operation points and give advise to process optimization, e.g. an adopted feedstock loading profile.

# **Conclusions**

The examples serve as possible integrated process analytical tools for a better understanding of the conditions inside the liquid phase. The concepts might reduce operational risks, especially at unstable conditions like a changed loading rate or when the feed composition changes. Hence, the concept can increase the flexibility in the operation of a biogas plant, supporting a sustainable integration into local and seasonal fluctuating material cycles.

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# Characterization of a microbial electrochemical VFA sensor for anaerobic digestion process control

biosensor, bioelectrochemical systems

Online monitoring and steering of the anaerobic digestion (AD) process becomes increasingly important, especially in the light of efficient demand driven energy production from biogas (e.g. Boe et al. 2010). Microbial electrochemical sensors for monitoring volatile fatty acids (VFA) are one emerging sensor platform on the pathway to online process control of AD (Tront et al. 2008; Liu et al. 2011; Kaur et al. 2013; Kretzschmar et al. 2016; Jin et al. 2016). Today, the measurement of VFA depends on laboratory analysis, e.g., gas- or liquid chromatography, that is expensive, time consuming, mostly offline and usually not available at AD plants. Based on a living and self-regenerating recognition element (receptor) microbial electrochemical sensors are cost effective and long-time stable. The receptor is based on an electroactive biofilm that is permanently connected to an electrode, i.e. anode. The bacteria oxidise acetate and other VFA and use the anode as terminal electron acceptor for their metabolism, i.e. electrode respiration (Bond and Lovley 2003). The metabolic electron flux is measured as current signal that correlates to the amount of oxidized VFA respectively their concentrations (Kretzschmar et al. 2016). The aim of this work was to characterize the dynamic response behaviour and the stability of such a sensor with respect to potential technical failures and AD relevant inhibitors, e.g. NH,-N and salinity. The results show amongst others biphasic response behaviour of the sensor that seems to be related to the metabolism of the electroactive bacteria (Kretzschmar et al. under review). The sensor can be shut down up to 10 consecutive days without losing its functionality. Paving the way towards application, future research will focus on the long time stability of the sensor under AD conditions as well as engineering and testing of a sensor device that can be implemented into existing AD reactors.

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# Application of Extended Kalman Filter as Soft Sensor for Anaerobic Digestion Plants

# Extended Kalman filter, soft sensor, monitoring, parameter estimation, simplified kinetic model

To evaluate the current state of a process, measurements of physical, chemical or biological values are necessary. In some cases the measuring of specific values is difficult or only possible using offline laboratory analysis. A few values might not be measureable at all due to high costs. Examples are the reaction heat of an exothermic process, the number of bacteria in bio reactors, or the concentration of nutrient in a fermenter. Using a soft sensor is an obvious solution. Based on existing measurement values a soft sensor computes further not measureable values. In this contribution an extended Kalman filter (KF) as model-based soft sensor is considered (Brown & Hwang 1997). A KF uses a simulation of a rigorous dynamic model in parallel to the process, whereby the model contains the unknown values and the existing measurement.

The algorithm of the KF consists of two main steps. First, the model is simulated starting at the current process state for one time step into the future to predict the future state. As measurements are available in the next time step, they are compared to the values computed by the prediction. The predicted state is then corrected such that the error of the measurement is minimized in stochastic average. This corrected state contains also the estimation of the not measurable values and can be used for displaying purposes or further computations.

Due to limited amount of computing power, memory capacity or suitable model structures KF applications are rarely used in control system for monitoring of industrial processes. Especially, anaerobic digestion plants often lack reliable automated monitoring systems. The anaerobic digestion process depends on many influencing factors; many of them (such as detailed concentrations of volatile organic acids, VOA) are only available as laboratory measurements and are analysed seldomly. Available methods for process evaluation or simulation are rarely used in digestion plants. Due to the large number of state variables and unknown model parameters, complex models – such as the established Anaerobic Digestion Model No. 1 (ADM1) (BATSTONE et al. 2002) – still cannot be applied for automatic monitoring and robust simulation of varying substrates and process conditions. Thus, the development of simplified model structures as well as their implementation in soft sensors is of great importance for a standardized application of model-based process monitoring and control systems for anaerobic digestion plants.

This contribution presents KF application regarding practical process monitoring of anaerobic digestion plants using a simplified kinetic model. During 2017 Siemens will offer an extended KF fully integrated into the distributed control system (DCS) SIMATIC PCS 7 for the first time. The model for the KF can be easily provided using the Kalman Configurator. The values estimated by the KF can directly be visualized in the DCS using the corresponding faceplate or can be connected to further computations. Another benefit of this implementation of the KF is the consideration of sporadic laboratory analysis (LOHNER et al. 2012).

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The utilized model is based on the stoichiometric structure of the ADM1 (Batstone et al. 2002). Since hydrolysis is the rate-limiting process phase during anaerobic digestion of fibrerich and particulate agricultural substrates, the gas production rate for uninhibited process conditions can be entirely described by first-order kinetics (Weinrich & Nelles 2015). Therefore, the complex model structure of the mass-based ADM1 was simplified to simulate the anaerobic digestion of carbohydrates, proteins, and lipids to biogas based on the superposition of four first-order reactions (including first-order biomass decay).

The model is converted in nonlinear state-space form to perform control engineering analysis. A KF can only be applied if the model is observable, i.e. the trajectories of the states can be completely reconstructed only using the input and measurement information. The derived model is proven to be locally observable (Numeuer & van der Schaft 1990) in the neighbourhood of a working point and subsequently it is implemented in the configurator. Together with measurement data of the biogas plant unkown model parameters can be estimated and the weighting matrices of the KF are tuned using simulation in the configurator.

Full scale experiments were conducted at the research biogas plant of DBFZ (Deutsches Biomasseforschungszentrum) in Leipzig. The semi-continuous digestion of maize silage was carried in a 208 m³ ( $V_{liq}$  = 165 m³) continuously stirred tank reactor at mesophilic temperature (40 ± 1 °C). Detailed gas production rates as well as gas compositions were logged online. Moreover, standard process analytics (such as pH, total VOA or ammonia nitrogen concentrations) were measured weekly to ensure stable and uninhibited process conditions.

The results prove the successful application of KF for process monitoring of anaerobic digestion plants using a simplified version of ADM1. The soft sensor can continuously estimate different state variables such as degradable nutrient concentrations as well as the unknown kinetic parameters. Thus, the observed concentrations of remaining degradable nutrients in the digestate can be used for detailed efficiency evaluation. The adjusted model can be utilized to predict biogas production for demand-oriented biogas utilization and for application in model-based control strategies (MAUKY et al.2016).

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### Return of experience of SMART control system in a full-scale Biobed® EGSB reactor

# EGSB, online performance monitoring, continuous process control, automatic load control

#### Background/motivation

Anaerobic treatment is a proven and well established technology of a wide range of industrial wastewaters. Process stability is a crucial factor for the application of anaerobic treatment plants. This however, is highly sensitive to dynamic alterations in organic loadings and/ or wastewater composition (Punal et. al, 2002, von Sachs et. al, 2003). The complexity of the anaerobic processes makes its monitoring and control very challenging and demanding. So far, the control applied to most of the anaerobic processes has been restricted to environmental variables such as pH and temperature, etc.. However, control strategies based on the monitoring of a number of process indicators could give complementary information which could enable an optimum operation; resulting in better designs (Punal et. al. 2002). Therefore, there is growing need to develop systems for anaerobic process management, advanced monitoring and control that combines available real-time performance online data, off-line measurements and detailed knowledge of the process in order to achieve a reliable and optimum operational performance (Punal et. al, 2002; von Sachs et. al, 2003). Biothane has developed, in cooperation with Kruger, a sludge management and reactor control techniques (SMART) system for UASB or EGSB reactors that offers several features such as: automatic plant load control, automatic buffer and emergency tank management, online system capacity test, indication of pant performance and advice on OPEX reduction. This online control system represents a significant advantage for the real-time control of the COD loading rate which can significantly improve the performance of anaerobic systems, potentially improving the effluent quality, increasing the biogas production and reducing the chemical dosing. The SMART system is designed to increase the easiness, reliability and effectiveness of operation of the reactor by regulating the operating loading rate closer to the real-time maximum capacity of the reactor, resulting in a smaller volume of buffer tank required to deal with fluctuations and consequently a smaller reactor design.

#### **Aim**

This case study aimed to evaluate and quantify the impact of the SMART system on the performance of a full scale Biobed® EGSB system as well as the reliability and effectiveness of operation before and after its installation.

#### **Methods**

The SMART system was installed in a Biobed® EGSB reactor of a soft drink production facility in Germany with troublesome operation due to strong fluctuations in COD and toxic components in wastewater. The SMART system comprises the following modules:

- » System capacity: in this module the maximum capacity of the anaerobic reactor in terms of COD load (kg/d) is defined by applying a short stepwise increase in load and a check of the maximum biogas production rate. The maximum treatment capacity is dependent on several factors such as the amount of biomass inside the reactor, the activity of this biomass, the type of wastewater applied, the temperature, pH, etc. Since all parameters can (quickly) change over time, this test gives valuable real time information that makes it easier to define loading rates to the anaerobic plant.
- » System performance: in this module several checks are done to judge the performance of the reactor, making use of online measurements (such as COD in effluent by online TOC/COD measurement, pH in effluent, CH<sub>4</sub> % in biogas).
- » Emergency tank management: Diversion of off-spec streams to an emergency tank and automatic return when the buffer tank has the capacity to receive it.

- » Automatic and fixed load control: This module controls the organic load into the anaerobic reactor, targeting for a fixed COD load for optimum biological performance. The module optimises the operational protocol based on buffer tank level, system performance and maximum treatment capacity. Applying a constant COD load results in general in a more stable (and better) performance a more constant biogas production rate. It protects the system against sudden peaks in COD load and the negative effects of this such as organic overloads, peaks in chemical consumption, a drop in pH and reduced reactor performance.
- » OPEX optimisation: this module is aiming for a reduction in operational expenses of the anaerobic treatment plant. Advice is given to the user on reduction of nutrient dosing rates and optimization of setpoints such as pH and temperature.

#### **Results**

As shown in Table 1, the performance of plant increased significantly in first four months of continuous operation with SMART system when compared to the previous 14 months without it. The EGSB plant with the SMART module was capable to increase VLR by 13 % while increasing the COD load by 12 %, but yet increasing the COD removal rate by 4 % and specific gas production by 31 % and reducing the specific caustic consumption by 19 %. Table 1. operational parameter comparison before and after the installation of SMART system

Parameter	Unit	Without SMART	With SMART	Difference
Flow (WA)	$[m^3/d]$	354	351	-1 %
Influent COD	[mg/L]	4732	5534	14 %
VLR	[kgCOD/m <sup>3</sup> .d]	9.73	11.21	13 %
COD load	[kg/d]	1694	1931	12 %
COD removal	[%]	86.12	89.42	4 %
Specific biogas prod.	[N-L/gCOD influent]	0.29	0.42	31 %
Biogas production (WA)	[N-m³/d]	427	740	42 %
Specific caustic consumption	[L NaOH/kgCOD in influent]	1.68	1.41	-19 %
Average effluent COD	[mg/L]	620	551	-13 %
Average effluent pH	[-]	6.8	7.06	4 %

**Table 1** operational parameter comparison before and after the installation of SMART system

#### **Conclusions**

The presented case study shows the impact of the SMART control installed in a Biobed® EGSB system of a soft drink production facility in Germany which enabled to operate the full-scale EGSB reactor closer to the design values and lower operational costs due to the increase in biogas production and reduction in caustic consumption.

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# Process monitoring and control for an anaerobic covered lagoon treating abattoir wastewater

# AD technology, process control, abattoir wastewater

This presentation will show results from the scientific monitoring of an anaerobic covered lagoon treating abattoir wastewater and accompanying lab scale experiments such as feedstock analyses, biomethane potential tests, sludge activity tests, mass balancing and characterisation of different wastewater streams, and continuous digestion tests in Continuous Stirred Tank Reactors (CSTR). Covered anaerobic lagoons are the preferred anaerobic treatment option for the meat processing sector in Australia and the lagoon monitored in this study is the first of its kind due to implementation of some process innovations, such as two stage design and biomass sludge recovery. At the abattoir, a part of the daily wastewater is treated in a two-stage process with open (aerobic) hydrolyses and anaerobic stage in an unheated covered lagoon.

After more than one year of operation, the evaluation of process parameters has shown a relatively low biogas production and accumulation of volatile organic acids. With reduction of the organic loading rate pH and volatile organic acid level have recovered and sludge activity has increased. Continuous digestion experiment have shown, that high levels (>1000mg/L) of FOG (fat, oil, and grease) did not inhibit the anaerobic process and that use of a stream with high COD and FOG levels (green stream) is improving the NH4 concentration, alkalinity, and buffer capacity.

The presentation will show data describing the lagoon performance and problems as well as results from a wastewater mapping study, which assessed the individual flows and characteristics of different streams (kill floor, boning room, paunch wash, cattle wash,...) and will present strategies to identify potential solutions to overcome these. A further focus is on lab scale continuous digestion experiments and the comparison of results to the data obtained from the lagoon.



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### Condition Monitoring and Lifecycle Management of Parts and Components in the Biogas Market

# Condition Monitoring, Maintenance 4.0, Predictive Maintenance, Lifecycle Management (LCM), Smart Components

A biogas plant structure is similar to any other chemical engineering plant. Vessels (fermenters), pumps, valves, pipes, control units, electrical components (i.e. motors and hydraulic parts), and agitators are common. Biogas plants typically have one or more combined heat and power units (CHP) and/or a gas processing station to feed gas into the grid. Most of those components have a need for maintenance. This maintenance is typically still based on hours of operation or time intervals. Inspection and maintenance is performed based on rules issued by the component or subsystem vendor, but often doesn't consider the actual abrasion. Failures are typically resolved only when they occur. All of these tasks are generally documented in a (paper) file by the owner operator (OA). While almost mandatory among the OA and engineering contractors in the process industry, the biogas OAs and vendors are typically far behind other industries in employing software lifecycle management (LCM) solutions to document these maintenance and troubleshooting actions.

In the future, tighter safety regulations will enforce the use of software-based LCM solutions in the biogas industry. In this context the availability of "smart" parts and components, which enable a more predictive and proactive maintenance regimen, will be an interesting step forward. Maintenance and malfunction information will calculated by an embedded controller using various sensors in various parts and components. Similar to "Industry 4.0", there will be "Maintenance 4.0." Maintenance will be based on actual abrasion data, and malfunctions will be predicted ahead of time.

A key challenge is the algorithm to combine all sensor information so that maintenance actions can be predicted reliably and in real time. One method could be to generate an n-dimensional vector with all sensor information. If the head of the vector is within the predefined (limited) n-dimensional vector space, abrasion is within a certain limit. If the vector head is outside this "space", maintenance or reconditioning of the part or component is required. Since this information could be securely transferred over the Internet, it would ideally feed into the LCM/Service Management (SM) Systems. In combination with mobile computers (tablets) for the field service organizations for scheduling and distributing the tasks to the work force, including completion confirmation and checklists fed back into the system, this could be an ideal "21st Century LCM solution."

Conclusion: Several vendors of components (particularly "pump manufacturers") are working on advanced "smart pumps" with condition monitoring capabilities. The internet of things (IOT) will be a key driving factor to push for "smart components and parts" as the controllers and sensors required become cheaper and more integrated when produced in larger quantities. So the future is us!



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### Gasmanagement of anaerobic digestion plants

# Gas management, flexible power production, gas domes

Biogas plants can fulfil a vital role in the transition of the energy system towards a renewable supply. They can fill the gaps in the energy supply provided by cheaper sources like wind and solar power by storing biogas on site in gas domes and convert it into electricity in times of high power demand. To make this system work, gas domes must be managed actively – especially if more than one dome is used on a biogas plant.

This presentation describes the relationship between gas dome level and gas dome pressure for different kind of gas domes. Further, it describes what happens if two or more gas domes are coupled and gas can flow freely between them.

Greater detail is provided for the design of blower and pressure regulation valves for air-inflated double membrane gas storage tanks, as these gas domes are now the most prominent.

Examples are given for unsuccessful gas management systems on large scale biogas plants and how problems were remedied during operation.

The presentation then describes Agraferm's patented controller (EP 2 535 402 B1) which ensures that all gas domes on the biogas plant have the same level and that 100 % of the gas volume can be used for storage purposes.

The presentation is wrapped up by showing a best-practice example of a 2.76 MW biogas plant with three coupled gas domes operating in flexible mode and using the whole gas storage volume.



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# Automatic process control of two-phase dry anaerobic digestion of biowaste for hydrolysis optimization and biogas enhancement

# 2-stage anaerobic digestion, hydrolysis, biowaste, pH control, percolation

#### Introduction

This study was part of an R&D project on energy yield optimization of a 2-stage anaerobic digestion (AD) technology for the organic fraction of municipal solid waste (OFMSW) as implemented in full-scale at the AIKAN® plant in Holbæk, Denmark. The aim of the study was to increase hydrolysis of the waste by pH control of the  $1^{st}$  stage and thereby enhance the subsequent biogas production in the  $2^{nd}$  stage. Hydrolysis is the rate-limiting step for AD of solid organic matter like OFMSW (Ghosh & Klass 1978) and separation of the AD process into 2 phases may take advantage of adjusting the pH in each stage to the respective optimum, i.e. pH 5 – 6 for hydrolysis to produce volatile fatty acids (VFAs) and pH 7 – 8 for methanogenesis to convert VFAs into biogas (AMANI et al., 2011). pH adjustment by recirculation of the digestate from the methanogenic stage has recently been successfully applied in a 2-stage process for production of hydrogen and methane from OFMSW (Micolucci et al. 2014).

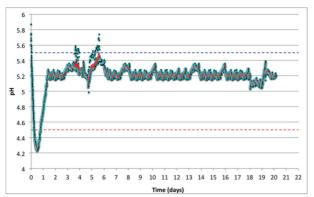
#### **Methods**

Two lab-scale reactors (4.5L total volume), were used to mimic the 2-stage AD process. In the 1st (hydrolytic) stage reactor (F), 148.5 g OFMSW was loaded on a sieve (mesh size 1 mm) and 2 L of water was sprinkled on top of the OFMSW. The  $2^{nd}$  (methanogenic) stage reactor (R) was filled with 2.6 L of inoculum from the full-scale 2nd stage reactor of the AIKAN® plant and both reactors were operated at mesophilic conditions (37°  $\pm$  0.3 °C). The pH of the leachate from the  $1^{st}$  stage reactor was measured by a pH electrode-probe (KnickTM with a Stratos Evo 4-wire analyser) immersed in a leachate reservoir (0.6 L) after the 1st stage reactor. 2 pumps and 3 valves were connected to the two reactors to be able (1) to pump the leachate from the  $1^{st}$  to the  $2^{nd}$  stage reactor or (2) to recirculate the leachate directly to the  $1^{st}$  stage or (3) to recirculate digestate from the 2nd stage to the  $1^{st}$  stage. A hardware device, cRIOTM (National InstrumentsTM), was programmed by a virtual instrument (VI) interface to control valves and pumps (1) to keep the pH in the  $1^{st}$  stage in the range of pH 4.5 – 5.5 by either direct leachate recirculation (to decrease the pH) or by digestate recirculation (to increase the pH) and (2) to secure hydraulic retention time (HRT) of 24 days in the  $2^{nd}$  stage reactor.

Biogas production of the combined 2-stage system was recorded on-line, methane content of the biogas was analysed by GC while TS, VS, total Kjeldahl nitrogen, ammonium nitrogen, total and soluble chemical oxygen demand (COD) were analysed daily and measured according to standard methods (APHA 2005).

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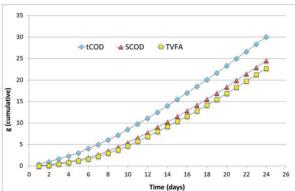


Figure 1 pH (left) and cumulative COD and VFA release (right) in the leachate of the 1ST stage reactor

#### **Results**

The pH of the 1st phase was maintained at pH 5.20  $\pm$  0.15 by the pH control and a steadily increasing release of VFAs, total and soluble COD in the leachate from the 1st stage (fig. 1) was observed for the 24 days operation period. The total COD release of 30.1 g was equivalent to a COD conversion of the input COD of the waste of 71 %. The cumulative methane yield from the 2nd stage was 227 mL-CH<sub>A</sub>/g-VS.

#### Conclusion

pH control of the 1<sup>st</sup> stage of a 2-stage AD process of OFMSW through direct or indirect leachate recirculation from the 1<sup>st</sup> stage keeping the pH to pH 5.2 lead to an efficiency of the hydrolysis of 70 % and an overall methane yield of 227 L-CH<sub>4</sub>/kg-VS of the waste input.

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Sören Weinrich, Jürgen Pröter

# Parameter estimation in anaerobic digestion - Critical evaluation of different experimental setups and model structures

# Process simulation, kinetic parameters, biogas potential, operating conditions, scale-up

The efficient operation of a biogas plant primarily depends on the individual degradation characteristics of the utilized substrates and the respective process conditions. Commonly, substrate characteristics – such as the total methane or biogas potential as well as the degradation kinetics (e.g. first-order reaction constants) – are determined based on laboratory batch tests. Due to extensive research and comparative ring tests the standard protocols (such as VDI 4630) for the conduction of anaerobic batch tests have been optimized and standardized in the past years (Holliger et al. 2016). However, the results and significance of discontinuous digestion tests are still affected by numerous influencing factors, such as the source or pre-treatment of the utilized inoculum. Furthermore, the validity of batch tests to describe continuously operated anaerobic reactors is rarely investigated or proven (Batstone et al. 2009). Continuous laboratory tests are used to examine the degradation efficiency, inhibitory effects or long term process stability during anaerobic digestion of numerous substrates. However, the direct comparability of continuously operated laboratory, pilot or industrial scale processes (scale-up) is still unkown.

The current contribution presents a critical comparison and detailed evaluation of different experimental setups and model structures for estimation of the total biogas potential and kinetic parameters during anaerobic digestion of maize silage. Thus, parallel digestion tests were conducted in discontinuous (batch) and continuous operation mode under mesophilic process conditions. Batch tests were carried out in different test setups and reactors sizes (0.5 and 10 L) according to VDI 4630. Additionally, two inocula from a waste water treatment plant (sewage sludge) as well as from a running biogas plant (digestate, mono digestion of maize silage) were compared. Continuously operated laboratory reactors (10 L) were run in parallel to a pilot-scale biogas plant (153 m³). All experiments were fed with the same cultivar of maize silage; analytical substrate characteristics (such as DM, oDM or composition of nutrients) were determined to calculate the stoichiometric biogas potential. Detailed gas and/or methane production rates were logged for all experiments. Moreover, standard process analytics (such as pH, total VFA or NH<sub>4</sub>-N concentrations) were measured weekly to ensure stable and uninhibited process conditions.

Different simplified kinetic models – based on the superposition of single- or two-step first-order reaction kinetics – were implemented (Matlab) and applied to simulate gas production in batch or continuous operation mode; an extended simplex algorithm was used for numerical parameter estimation of the experimental biogas potential and kinetic constants. Furthermore, typical objective functions based on the absolute or squared error between the simulated and measured biogas production rates were investigated and compared during parameter optimization.

The results clearly evince differences between the different experimental setups and model calculations. Typically, batch tests and established stoichiometric calculation methods underestimate the effective biogas potential in long term continuous operation mode (Batstone et al. 2009). It also can be proven, that the maximum methane yield as well as the degradation kinetics in batch mode are strongly affected by the source of the utilized inoculum. Due to adaptation processes the inoculum of a running biogas plant (continuously and entirely fed with maize silage) evinces faster degradation kinetics and higher methane potentials. Moreover, the different test setups, measuring systems and reactor sizes (hydrodynamics) also influence batch test results. The logged gas production rate of the pilot-scale biogas plant is affect by numerous operational factors (e.g. pumping processes) and strong measurement errors. In the current investigation the experimental biogas potential and kinetic parameters estimated on the biogas production rate of the continuously operated laboratory reactor – run under optimal process and measuring conditions – define the most reliable parameter estimation and substrate characterisation method during anaerobic mono digestion of maize silage.

The selection of an appropriate model structure (model complexity) as well as the objective function also affect the estimated kinetic parameters and respective simulation results. Even though, complex model approaches can depict the gas progression curves in more detail, the high number of unknown model parameters hamper a deterministic and realistic estimation of the kinetic constants. The current study presents an introduction and first results on critical aspects and fundamental comparability of typical experimental and modelling methods for parameter estimation in anaerobic digestion. Thus, further research is conducted for an extensive evaluation of numerous substrates, process conditions, plant types and sizes to develop a reliable and precise determination method for experimental substrate characterisation.

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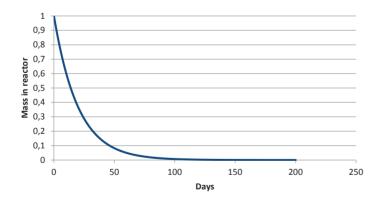
#### Alastair James Ward

# Modelling of biogas production from single or serial continuous stirred tank reactors using batch assay data

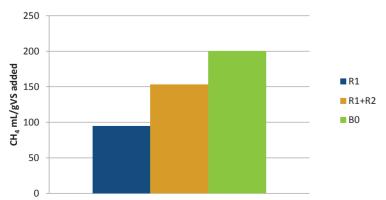
# Biogas, NIR, multivariate analysis, pre-treatment

Biogas research relies on batch testing to find the biochemical methane potential (BMP or B<sub>a</sub>) of substrates. The method provides information regarding the maximum methane yield possible under anaerobic conditions as well as information regarding rate of degradation. However, the biogas industry is dominated by continuous processes, particularly the continuous stirred tank reactor (CSTR) design. In the batch assay an inoculum from a functioning biogas reactor is placed in a container; the substrate of interest is added and the mixture is incubated at the required temperature for a period of up to several months. In a continuous process a little digested material is frequently removed from the reactor and a corresponding mass of fresh substrate is added. The volume of the reactor and substrate added define the hydraulic retention time (HRT). Thus, a continuous process is usually very stable in terms of volatile fatty acid (VFA) concentration and biogas production rate. Conversely, a batch assay is very dynamic, starting with a sudden large organic load which is subsequently hydrolysed, fermented and converted to biogas at rates dependent on many variables. Thus the biogas flow rate is far from constant, often starting very slowly then increasing to a maximum before falling to zero, at which point B<sub>o</sub> has been reached and the assay is finished. The concentration of substrate fed to a single CSTR reactor will undergo an exponential decay over time as the substrate is flushed from the reactor (Fig. 1).

It is considered good practice to configure multiple CSTRs in series to give a better retention in the system and thus an improved product yield (Fig. 2). This has been demonstrated empirically in studies where, for example, 13 - 17.8 % more biogas was produced in serial CSTR configurations divided 70/30 % and 50/50 % (respectively) when compared to a single CSTR (KAPARAJU et al. 2009). Models such as the Tanks in Series or Dispersion models



**Figure 1** Mass flow in a single CSTR reactor with a 20 day HRT



**Figure 2** Modelled cumulative methane yields of a serial CSTR process (20 day + 20 day HRTs) using a B0 value of 200mL CH<sub>4</sub>/gVS and a reaction rate of 10 mL CH<sub>4</sub>/gVS/day

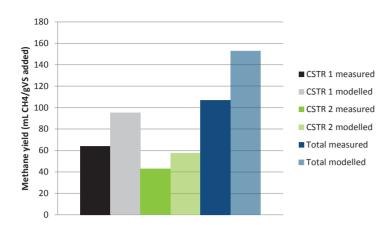


Figure 3 Measured and modelled performance of a serial (10 + 10 day HRT) reactor configuration digesting cattle manure with grass. Models use the rate calculated from the modified Gompertz

can model the flow of material through serial CSTRs (Levenspiel 1999) although the models are generally restricted to equal sized reactors, a situation that is not always the case in full scale biogas plants.

The work presented here is in two parts: first there is the mass flow model of a system of unequally sized CSTRs, and secondly the application of batch assay data to the mass flow model to gain an estimate of CSTR yield. The batch data can be modelled (Kafle & Chen 2016) and applied to the mass flow model. An initial validation of the model using batch assay modified Gompertz modelled results to estimate performance of single and serial CSTRs demonstrated that the model gave a considerable overestimate of the CSTRs performance of up to 55 %, although the relative proportions of gas production modelled in the serial CSTR system were promising (Fig. 3). The inaccuracy of the results was not surprising due to the dynamic nature of the batch assay; the reaction rate of a given substrate is highly dependent on inoculum to substrate ratio, inoculum activity and acclimation to the substrate (Moset et al. 2015). The model will therefore be subjected to validation using empirical data to find the relationship between the I:S ratio of the batch assay data and the loading rate of the CSTR.

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# POSTER PRESENTATION

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### Modelling Anaerobic Digestion: From ADM1 to NGS-based Metabolic Network Approaches

# Anaerobic digestion, modelling, ADM1, flux-balance-analysis, community modelling

Anaerobic digestion is a microbial community-driven process that transforms organic material to methane in a multi-step process under anaerobic conditions. Current modelling approaches consider these consecutive process steps, for example in the ordinary equationbased model ADM1, but fall short on reflecting the high diversity of involved communities. Current high-throughput methods including next-generation sequencing (NGS) based metagenome and metatranscriptome analysis can unravel both the composition and function of such communities. Such data, however, are not compatible with the aforementioned modelling approaches. To improve our understanding of microbial community dynamics in terms of composition and function, and their link to reactor performance, the aim of this BMBF-funded project - "McBiogas - metabolism centered predictive modelling of anaerobic digestion for biogas production" - is to harness high-throughput data to inform detailed models of metabolic activity within the community. For this purpose, lab-scale biogas reactors are operated under well-controlled conditions. Bioinformatics pipelines are developed that allow for the construction of organism-specific metabolic network models from metagenome and metatranscriptomic data obtained from reactor biomass. Constraint-based modelling techniques including Flux-Balance-Analysis are then used to predict metabolic turnover and growth of single organisms. Multiple species models can be coupled to a dynamic community model by defining a common pool of metabolites which can be exchanged between species. Integrating this approach with ADM1 results in a high-resolution dynamic model based on NGS data. This improved modelling framework will fundamentally enhance process understanding and be used as a platform to identify indicators of looming reactor breakdowns, to test promising intervention strategies, and to develop novel online monitoring and control strategies. These are essential for making biogas production more flexible towards an on-demand energy production scheme.

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### Anaerobic digestion and near infra-red spectroscopy. Application to process monitoring through the development of 5 AD parameters models

# Near infra-red spectroscopy, methane potential, volatile fatty acids, process monitoring

Reliable and real-time information is imperative for the monitoring and control of anaerobic digestion (AD) plants to ensure good performance and stability of the process. Some parameters are already being followed instantly and continuously, giving valuable information for plant control and operation. Other important parameters, such as volatile fatty acids (VFA) concentration, alkalinity, total and volatile solids content, etc., still require sampling and off-line laboratory analysis implying a delay in the response depending on the technology used. Near InfraRed Spectroscopy (NIRS) is a proven technology used to measure in-line organic matter properties in fields such as agro-food and pharmaceutical industries where it is becoming an important tool for process control (Rosas et al. 2012). Its application on the characterization of organic substrates and follow-up and control of anaerobic digestion of waste is growing rapidly, with several studies reporting the performance of this technology to determine the methane potential of different substrates (Doublet et al. 2013) or for process control (Stocklet et al. 2013).

#### Aim of the work and methods:

The current study presents the development of NIRS-based mathematical models in regards to parameters of interest for AD process monitoring: dry solids (DS), volatile solids (VS), volatile fatty acids (VFA), ammonium (NH $_4$ ), and alkalinity (Alk) concentrations. This development has been performed by relying on an existing database gathering analysis from multiple studies, waste types and process steps. The NIRS spectra were acquired using a Thermo Antaris II analyzer and samples collected were analyzed (by classical methods used in anaerobic digestion related laboratories) for the above-mentioned parameters of interest.

#### Results and conclusion:

Three main types of models have been used to achieve this development: the PLS (Partial Least Squares), the local-PLS (which allows more precise predictions but implies a more complex integration), and the PLS-DA2 (with the same principle as the PLS, but resulting in classification purposes rather than quantification).

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### Development of universal early warning indicators to prevent process failures in single-stage biogas plants fed with maize silage and cattle manure

# Optimization of biogas production, renewable raw material, early warning indicators

Process failures like over-acidification or floating layers are common problems for the production of biogas. The aim of the project "ProGas" is to develop recommendations for the operation of single-stage biogas plants fed with renewable raw materials and to optimize the process for a demand-driven energy supply. In case of a high energy demand, a rather quick increase of the biogas production is required but for a stable process rather low changes in the substrate supply are necessary. Early warning indicators (EWI) are required to predict the risk of a process failure and to start countermeasures in time.

To develop EWIs three lab-scale over-acidification experiments were conducted with a fermentation volume of 20 L each and continuous stirring digesting 95 % maize silage and 5 % cattle manure based on the volatile solids (VS). The organic loading rate (OLR) was increased every 8 days by 1 kg VS  $\rm m^{-3}$  d<sup>-1</sup>.

Raising the OLR led to an increase in the gas production rate (GPR) while the pH and the concentration of organic acids stayed constant until a critical OLR was reached and process failures occurred. The critical OLRs for the processes were between 4.5 and 6.5 kg VS  $\,\mathrm{m}^{-3}\,\mathrm{d}^{-1}$ . The concentration of organic acids increased from 800 to 6000 mg  $\,\mathrm{L}^{-1}$  and the GPR decreased by 90 %.

The classical process parameters such as pH, GPR and organic acids warned 3 to 4 days before the complete over-acidification occurred. In one of the experiments the EWI FOS/ TAC-value warned first with a warning time of 8 days while for the other two experiments the warning time was only 4 days. In former studies at the GFZ the EWIs oS/Ca, P/Ca and P were developed for biogas plants fed with sewage sludge and high-fat co-substrates (KLEY-BÖCKER et al. 2012). In the present experiments those EWIs warned 14 to 3 days prior the over-acidification, thus being suitable for the digestion of maize silage and cattle manure.

Besides the already mentioned EWIs a new, online-measurable EWI was developed that warned in two experiments even two days earlier. However, the robustness and operation of this new EWI has to be validated in further experiments.

Additionally, the microbial communities dominating in the experiments will be characterized by molecular biological methods such as genetic fingerprinting, qPCR and next generation sequencing approaches to extent the chemical EWIs by microbial indicators.

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### Genetic analyses of microbial communities in anaerobic digesters reveal enhanced methanogenesis after calcium nitrate dosage

# Anaerobic digestion, biogas production, microbial communities, calcium nitrate, methanogenesis

Plants operating anaerobic digesters aim at a maximum yield of methane ( $CH_4$ ) while minimizing the production of other gases such as hydrogen sulphide ( $H_2S$ ). In the past, we found that the dosage of calcium nitrate to anaerobic fermenters resulted in several improvements of the fermentation process. Monitoring data of several biogas plants showed indeed a higher quality of biogas (more  $CH_4$  and less  $H_2S$ ), substrate savings were achieved, and operators reported a more robust plant operation (FRANKE et al. 2016; ETTL and FRANKE 2016). Thus far, these measurements and observations were empirical. Therefore, the aim of this study was to reveal the mode of action of calcium nitrate dosage in anaerobic digesters on microbial communities and their activities. Considering the complexity of microbial communities in anaerobic digesters and the processes mediated by these communities, we hypothesised that calcium nitrate interacts in the competitive processes of denitrifiers, sulphate reducing bacteria (SRB) and methanogens in favour of methane production.

To test this hypothesis, we have analysed the microbial community structures in a calcium nitrate-dosed fermenter of an operating plant as well as in a control fermenter of the same plant. Therefore, we extracted nucleic acids from samples of a time series collected in both fermenters and used high-throughput sequencing of taxonomic marker genes followed by taxonomic assignment of the obtained DNA barcodes. Furthermore, we have analysed the activities of methanogens and SRBs using quantitative real time PCR of transcribed RNA molecules extracted from all samples.

Indeed, we found that despite negligible changes in microbial community structures in the calcium nitrate-treated fermenter in comparison to the control fermenter methanogenesis significantly increased after calcium nitrate dosage, while sulphate reduction ( $\rm H_2S$  production) decreased significantly. Based on these results, we have developed a mechanistic model on the mode of action of calcium nitrate dosage in anaerobic digesters, which results in a higher quality of the produced biogas.

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# Studies on the influence of moulded feedstocks on the biogas process and the mycotoxicological status of digestates

# Activity tests, mycotoxins, moulds, biogasprocess, digestate, carry over effect, hygienization

There are indications that silage spoiled by mycotoxin producing fungi can affect the biogas process. In addition to mass loss by microbial degradation, the secondary metabolites formed by moulds may affect the activity of the microorganisms in biogas plants. In order to test this, activity tests were conducted with selected mycotoxins and continuous process fed with differently moulded silages were analysed. Analyses comprised of Biogas production, effects on the methanogenic biocenosis at the molecular level and the fate of mycotoxins in digestate.

High-quality substrates are the basis of optimal biomass utilization in biogas plants and contribute significantly to the efficiency of the overall system. Silage spoiling may not only cause mass loss by microbial respiration but also can result in the formation of secondary metabolites by the growth of moulds. These substances can be toxic and show antibiotic effects. Thus, mycotoxins may affect the metabolic activity of microorganisms in biogas plants and decrease the efficiency of the biogas plant. In addition to metabolic products such as aflatoxin B1, which may be contained in purchased feed, many fungal toxins of stock moulds can be toxic for higher organisms. However, the metabolism of the mycotoxins in the complex biogas process and their fate in the digestate are largely unknown.

The results of the batch tests showed that effects of the toxin addition depend on the state of the digester contents and the nature and concentration of the added substance. Of the investigated mycotoxins in pure form, only mycophenolic acid in vivo relevant concentrations caused a weak reduction of the biogas productivity. The impact on the biogas productivity was seen with both, the stable and unstable biocenosis. Results of the molecular biology analysis provided no explanation for the reduced gas production. With regard to the recovery of the toxins and the influence on the biogas rate, no correlation between reduced biogas productivity and the compounds detected in the digester was seen. In the experiments with continuous addition of moulded silages the digester with Monascus ruber, the methane productivity collapsed after 70 days and the variant. Penicillium roqueforti after 90 days. These effects were accompanied by an increased acetic acid concentration from 700 mg/kg (FM) nearly up to 10000 mg/kg (FM) and 2000 mg/kg (FM) nearly up to 6000 mg/kg (FM) for propionic acid. The results for the recovery of toxins in the digestate could only verified for the toxins Monacolin and Roquefortin, Mycophenolic acid was not detectable.

Spoiled silage by mycotoxin producing fungi can be affecting the biogas process. It remains to be investigated which key processes were inhibited at the molecular level. It seems regarding the removal mycotoxins the recovery rate corresponds to degrading metabolism.

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# Towards demand-driven Biogas plants: Real-time determination of biogas quality

# Photoacoustic NDIR, low-cost sensors, real-time measurements

Biogas is the only renewable energy source with the possibility for on-demand production (GBA 2015) and consequently it is set to become an even more important pillar in a future, sustainable energy landscape. Currently available technologies for flexible usage of biogas plant outputs mostly rely on cost-intensive use of gas storage tanks. Due to their limited volume, they are unable to cope with gas surpluses for more than a couple of hours. Furthermore, gas storage poses a serious safety hazard.

One appealing possibility to enable on-demand supply of biogas is to control the output itself via flexible feeding. To do this, the online monitoring of the gas composition of biogas plants is a prerequisite. However, currently employed sensing technologies for the determination of the most important biogas components carbon dioxide (CO<sub>2</sub>) and methane (CH<sub>4</sub>) are costly and rely on extracting gas samples from the fermenter. Moreover, the acquisition time for a data point is typically on the order of several tens of minutes, which is too slow for an online process control. Here we present an approach that employs a so-called non-dispersive infrared absorption spectroscopy (NDIR) setup, but makes use of an alternative detector scheme based on the photoacoustic effect (Scholz et al. 2016). With this novel sensor technology, we are able to considerably increase the sensitivity towards CO2 and CH4 while at the same scale down the system size. This enables the measurement of these gases with high temporal resolution of a few seconds. Because we use low-power consuming light emitting diodes (LEDs) instead of the usually employed thermal emitter as a light source, it is possible to operate the sensors intrinsically safe, which in turn allows for low-cost packaging as compared to explosion proof enclosures. Hence, it becomes possible to monitor the process online and at low overall cost, which consequently may lead to a widespread use of the technology.

The gas concentrations are determined without cross-sensitivities towards humidity by probing the main absorption bands of CH $_4$  and CO $_2$  at 3.4 and 4.2  $\mu m$  using appropriate MID-IR LEDs, respectively. To generate a photoacoustic signal the respective LEDs are intensity modulated at around 250 Hz and a new gas concentration reading is obtained every 1.6 s. The sensors are able to reliably determine the gas concentration in the range from 40 % – 100 % for methane and 0 % – 60 % for carbon dioxide.

In conclusion, we present a novel NDIR-setup based on a non-resonant photoacoustic detector technology, which shows significantly higher response to the probe gases than solid-state detectors but without the typically occurring cross-sensitivities towards humidity. Because of this, it is possible to build low-cost sensor systems for monitoring anaerobic digestion plants with small footprint, high sensitivity and high temporal resolution. Based on this information, the processes inside biogas plants can be monitored in real-time and thus enable a number of new possibilities for control of the processes.

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# On-line monitoring of volatile fatty acids and hydrogen during anaerobic digestion

# Biogas, hydrogen, volatile fatty acids, optical fiber, chemical sensors

Current methods for on-line calculation of dissolved  $\rm H_2$  concentration in anaerobic biogas production involve detection of the fraction of  $\rm H_2$  in the gas phase of the digester. Although this measurement is straight forward, the limited  $\rm H_2$  mass-transfer coefficient suggests a significant delay in  $\rm H_2$  concentration equalization between the liquid and gas phases of the digester. This is a severe limitation to maintaining optimal control of the biogas production process (Spanjers & Lier 2006). Furthermore, off-line dissolved  $\rm H_2$  monitoring requires extraction methods that are both time consuming and disadvantageous to continuous process control. Although many electrochemical sensors that can measure dissolved  $\rm H_2$  have been developed, some require the use

of H<sub>2</sub> permeable membranes that are susceptible to fouling resulting in a short sensor lifetime, low selectivity, low signal-to-noise, and are therefore are not in use by the industry (Huck et al. 2013). Titrimetry and various gas chromatography techniques are commonly used methods for assessing VFA's, but are susceptible to biofouling and are time-consuming measurements. Alternative methods include using NIR- and IR-spectrometry techniques, but the required automation and sample processing need advanced systems to maintain sufficiently rapid (< 2 h) measurement (FALK et al. 2015). The use of chemical sensors (including electrochemical, electronic, and optical technologies) appears to be ideal for monitoring of the bioprocess, but their limited selectivity, robustness, consistency, and stability may prove to be challenging for use in bioreactors (PERIS & ESCUDER-GILABERT 2013; BIECHELE et al. 2015). Therefore, the application of artificial noses and tongues (array of multiple chemical sensors where each sensor is only partially selective) may be a solution (Peris & Escuder-Gilabert 2013; Askim et al., 2013). Although there has been attempts to apply such artificial tongues to biogas production (Rudnitskaya & Legin 2008; Buczkowska 2010), the complexity and low reproducibility of the process media composition can lead to sensor contamination (Rudnitskaya & Legin 2008).

This research intends to address the lack of on-line monitoring of the two imperative process parameters; concentration of VFA's, and dissolved  $\rm H_2$  concentration. A new optical fiber based sensor system allowing the live monitoring of both  $\rm H_2$  and VFA's during the biogas production process will be pursued. This may be in the form of some-what indiscriminate optical sensor arrays, various chemometric sensors (using the artificial tongue concept), or a combination of both. If successful, the on-line measurement of  $\rm H_2$  and VFA's will allow continuous manipulation of externally produced  $\rm H_2$ , increasing the productivity of the biogas production process.

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# Operational and seasonal methane emissions from open digestate storage tanks

# Methane emission, open digestate storage tanks, biogas plants, open path tunable diode absorption spectrometry

Open (not gas tight) digestate storage tanks are a main methane emission source on agricultural biogas plants (LIEBETRAU et al., 2013). As methane is a greenhouse gas with a 28 times higher warming potential than carbon dioxide (MYHRE et al. 2013), it is of great importance to determine the methane emissions during the biogas production process. The emissions of the open digestate storage tanks depend strongly on the filling level and the process parameters of the biogas plant and, hence, on the particular season.

Until now, only a few studies concerning the methane emissions from open digestate storage tanks exist, e.g., Rodhe et al. 2015; Baldé et al. 2016. To estimate the total annual methane emissions from the open digestate storage tanks and also the contribution to the methane emissions from the whole biogas plant, more investigation concerning the dependency of the emissions from the operational parameters and the season are necessary.

During the current project BetEmBGA (Operational emissions from biogas plants – Funded by the Federal Ministry of Food and Agriculture represented by the Agency for Renewable Resources), the methane emissions from the open digestate storage tanks from four different biogas plants in Germany were determined over a time period of one year (from Sep. 2015 until Sep. 2016). As the emissions are dependent on the particular season, the measurements were performed in 20 measurement time slots (5 measurement campaigns for each of the four biogas plants). The methane emission rate was determined with open path tunable diode absorption spectrometers (GasFinder 2.0 and GasFinder 3 from Boreal Laser Inc., Canada) combined with a weather station including an ultrasonic anemometer and a subsequent calculation with a Lagrangian backward model (using the program Windtrax).

The results of the open path measurements are compared to in-situ measurements performed directly on the surface of the digestate using a closed or an aerated chamber (Liebetrau et al. 2013). With the open path measurement, the emission of the whole storage tank is determined, but the measurement is extremely dependent on the weather conditions and the topography of the area around the biogas plant, whereas the in-situ method is used on a limited area of the digestate surface only, but it determines the emission rate directly and is not sensitive to the extern conditions. The comparison of both methods provides a basis for a future method harmonization on the determination of methane emissions, e.g., for open digestate storage tanks, and demonstrates the potentials and shortcomings of each method.

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# POSTER EXHIBITION

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### Measuring biogas composition with a compact MEMS based spectrometer

# MEMS, spectrometer, methane, machine learning

This project evaluates a newly developed MEMS (Micro-Electrical-Mechanical Systems) infrared sensor for use in measurement of biogas. The performance of the sensor was tested for the measurement of Methane and Carbon Dioxide. Currently, the commercially available possibilities for measuring the composition of biogas in an industrial environment are either using a catalytic sensor or an IR sensor using either one or two wavelengths. Catalytic sensors have a drawback that high concentrations of Hydrogen Sulphide can damage the sensor.

When compared to a conventional IR sensor, there is also a benefit as the MEMS sensor records a spectrum at multiple wavelengths, allowing a profile to be recorded. This profile is used for calculation of methane concentrations, and provides an improved robustness to the problem of cross sensitivity when compared to one or two measurement points. These conventional measurement systems need investment costs in the area of thousands of Euros for systems designed to withstand the hydrogen sulphide concentrations and moisture content, and furthermore some systems require regular maintenance.

The overall aim of the work was to evaluate the suitability of the newly developed MEMS based spectroscopic sensor for the biogas application. Machine learning methods are tested and evaluated in order to predict the concentration of methane and carbon dioxide from the sensor data.

The sensor that was used measures mid infrared light from 3000 nm – 3700 nm, using a MEMS tuneable Fabry-Perot Interferometer. This interferometer functions as a tuneable optical filter, and combined with a wideband light source and wideband sensor, and this allows the system to function as a compact spectrometer. A gas measurement cell with a 10 cm path length was used to hold the gas that was being measured.

Testing was performed in three locations, firstly in a laboratory, secondly, on a 1000 L pilot scale biogas digester at the :metabolon research facility, and thirdly at a 1.2 MW landfill gas collection site, also at the :metabolon research facility.

For the laboratory testing, the response to methane was measured at different concentrations, mixed with methane. Initially, nitrogen, which is inactive in this infrared region, was used for mixing in order to measure the response only due to methane. Then mixtures of methane and carbon dioxide were tested. Finally, testing was performed with gas mixtures bubbled through water in order to increase the humidity of the gas and evaluate the output due to changing humidity.

For the testing on the pilot scale digester, the sensor was installed for 109 days. The digester was also connected to a commercial gas analysis unit which measured carbon dioxide and methane every hour. Machine learning methods were developed using these values as reference values, to estimate the gas concentrations from the measured spectra. The machine learning methods that were investigated include both linear and non-linear regression such as PLS-R, PCR, SVM, and Random Forest. From the 109 days of data, the first 76 days data used for training and the remaining 33 days for testing.

Averaging of the data was also performed to reduce the signal to noise ratio, which provided an increase in accuracy. The current scan settings required 35 seconds per scan, and 10 scans were averaged to give the output. Further work is underway in order to reduce the number of wavelengths measured in order to produce faster measurements. Presently the entire range of the sensor is used at regular intervals, but further analysis can indicate which measurement points can be removed, increasing the measurement rate. Analysis software was written in Python, in order to run the program on a Raspberry Pi, which allows measurements and data analysis to be performed on the device.

For the data from the pilot scale reactor, methane prediction is performed with an RMSEP (Root Mean Square Error of Prediction) of 0.95 % and carbon dioxide prediction with an RMSEP of 3.4 %. The gas concentrations ranged from 20 % to 55 %, with rapid changes due to maintenance allowing ambient air into the reactor. These fast transients were also well tracked by the sensor. The optimum machine learning technique for predicting methane concentration was PCR, and for predicting carbon dioxide was Random Forest. With the current performance, a measurement value is generated approximately every 6 minutes, however it is anticipated that this can be substantially reduced, and is expected that the final time will be under one minute.

The MEMS sensor, when produced in an industrial scale, will have a relatively low cost, with the possibility to reduce the cost of a gas sensor system compared to today's prices. Due to the sensor construction and use of a glass measurement cell for the gas, the electronics are completely separated from the gas, and as a result will have no exposure to the corrosive Hydrogen Sulphide present in biogas. Consequently the sensor will not have any issues with damage or degradation from the gas. Furthermore, for the entire duration of testing, no degradation in the amplitude from the light source was observed which also indicates the long life of the system.

The capabilities of the sensor combined with a Raspberry Pi has enabled a powerful system to be built which is compact and portable whilst offering prediction for multiple gases. In the biogas application, the system can perform online measurement of both carbon dioxide and methane simultaneously, allowing it to fulfil the role of two different sensors with good performance. MEMS based sensors offer a new alternative technology for monitoring of biogas composition which can be used for maintaining stable plant operation.



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# Monitoring the composition of biogas through impedance spectroscopy

# Impedance, spectroscopy, ammonia, biogas

The biological metabolic processes in biogas plants are very complex; therefore operators usually view them as a "black box". There are few reliable and easy to measure indicators for early detection of emerging disorders resulting from anaerobic biodegradation processes. Within the project "Model-based process control of biogas plants, MOST", monitoring the biogas composition was identified as a possible. For the main components methane and carbon dioxide affordable long-term stable sensors are present. The determination of the ammonia content in the raw gas, however, is much more difficult.

This paper describes a method for efficiently determining the ammonia content in the raw gas. The measurement method is based on the following constraints:

- » The raw gas is saturated with water vapor
- » Ammonia is the sole water-soluble gas, which contributes significantly benefiting the electrical conductivity of the condensed water vapor.
- » The ammonia is dissolved in the condensed water vapor
- » The conductivity of the condensate depends on the ammonia content
- » The contribution of carbon dioxide in the conductance is low and can be compensated for if necessary, since this is also measured.

The measurement system consists of an interdigital electrode; which is cooled below the dew point of the biogas by means of a Peltier element. On the measuring surface water vapor condenses and forms the measuring surface filling drops. For determining the electrical properties, impedance spectroscopy is performed on this condensate. In Figure 1 the magnitude and the phase of the impedance are shown.

It can be seen that the sensor behaves like an ideal capacity in the dry state. In other cases, a distinct plateau formation in the range of about 5 to 100 KHz can be observed, the level of which decreases with increasing ammonia content. After calibration, this system can be used for monitoring of the ammonia content.

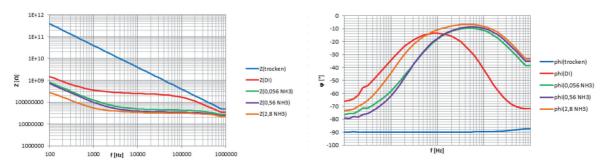


Figure 1 Magnitude and phase of the impedance

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### Selectivity and long term stability of metal-oxide sensors for volatile fatty acids in biogas

# Metal-oxide sensors, sensitivity, reproducibility, selectivity, long term stability, volatile fatty acids (VFA)

The kinetics of the biogas process is characterized by volatile fatty acids (VFA) fingerprints measured with multi-sensor arrays of differently doped metal-oxide sensors. The sensors are subject to reversible chemical reaction with the VFA in the biogas. Dependent on the doping of the semiconductors the acidic carboxyl and the hydrocarbon groups of the VFA reduce the semiconductor. Filtered and dried air reoxidizes the semiconductors. The transient behaviour due to reduction and reoxidation is superimposed by a long term irreversible reaction of the semiconductors, e.g. by reduction due to sulphur-containing compounds in the biogas. The sensors provide a sufficient selectivity to the aliphatic hydro-carbon acids and exhibit also a sufficiently low cross-sensitivity to the residual gas composition of the biogas.

In a 1st step the dynamical sensor signal functions (DSSF) are analyzed with respect to the short term transient functions of reduction and reoxidation. Then the long term drift behaviour due to the irreversible reduction of the semiconductors is evaluated. Time series of diffusion processes with different kinetics of chemisorption, charge carrier diffusion, integration of spatial current density to the integral resistance show that the transient functions can be expressed as n

 $\sum_{k=0}^{\infty} (c_{1,k} + c_{2,k} e^{-c_{3,k} t})$ 

The experienced sensor dynamics exhibits nonequilibrium behaviour with a hierarchical structure composed of different dynamics on different time scales. With a set of  $(c_{1,k},c_{2,k},c_{3,k})$  the deterministic function can be represented by a probabilistic approach from which the first passage time is defined as the time when the measured signal set as a random variable passes for the first time an absorbing boundary. This can be a certain degree of sensor degradation, or (with sensors still inside the function intervals) a certain stage of the process.

The probability density is  $f(t) \propto t^{-\frac{3}{2}} \, e^{-\frac{(b-x_0)^2}{4Dt}}$ 

with t the time, b the boundary value,  $x_o$  the start value, and D a diffusion coefficient. From this hitting probabilities are calculated for the cases

- (i) of the degradation of the sensors, and
- (ii) for reaching a critical limit of the VFA concentration spectrum.

The sensor dynamics can be written as a stochastic differential equation  $d\vec{X} = \vec{K}(\vec{X})dt + \vec{G}\,d\vec{W} \quad \text{where} \quad d\vec{X} \quad \text{is the vector-valued random variable of the sensor function,} \quad \vec{K}(\vec{X}) \quad \text{the transient and drift vector,} \quad \vec{G} \quad \text{the chemical reaction matrix, and} \quad d\vec{W} \quad \text{the Wiener noise vector with} \quad dW_i \quad dW_j = \delta_{ij} \quad dt \quad \text{.} \quad \text{Stochastic analysis of combined chemisorption, diffusion of charge carriers, and of electrical noise yields finally the complete function parameters of sensor dynamics.}$ 

Usually equilibrium is assumed between the liquid phase in anaerobic digestion and the VFA components of the biogas. Buffering agents in the liquid influence the accuracy of the VFA measurement. Furthermore nonequilibrium is possible due to rapid changes in the composition of the liquid phase. As a provisional result it can be concluded that the accuracy of the evaluated experimental data is sufficient for the required sensitivity and reproducibility. In the online monitoring the time delay is <10 s. The specific sensor functions with cross sensitivities, and individual scattering are calibrated in the laboratory with use of pure VFA mixtures in different combinations and concentrations. The sequences of DSSF are separated into the short term and the long term kinetics. The stochastic method for calculating first passage times can be used (1) for prediction of the usage time of the sensors, and (2) for the process drift analysis. The relative sensor drift rates depend on the gas flow rates and range between  $10^{-10}$  and  $10^{-8}$  s<sup>-1</sup>. The sensitivities range down to the ppm range. Practical experience with continuous monitoring of a digester yields average sensor lifetimes >6 months.



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### Biogas production from maize with different inocula in batch reactor

# Maize silage, batch fermentation, digestate, microbial population

Long-term success of biogas production from renewable resources depends i.a. from the biogas yield. Technical parameters as well as the operation mode of the biogas plant affects the yield and duration of biogas production. Beside these parameters the microbial community plays an essential role for the efficiency of the degradation process.

To achieve the full potential yield of biogas in the fermenter the microbial community as well as suitable process parameters are taken into account. Within the project HoLaFlor the biogas production from maize silage should be enhanced to achieve high biogas yields in process. Therefore methane emission in residual digestate would be reduced.

#### **Key research topics**

The objective of this project "HoLaFlor" is to operate a biogas plant in lab scale as a high-load process with maize silage as mono-substrate. A comparatively short hydraulic retention time will characterize the operation mode. The process will be investigated regarding the expected enhanced degradation efficiency and biogas production from the substrate maize silage. At the same time a second biogas plant will be operated in a conventional operation mode as a reference. As the inoculum of the biogas plant plays an important role, a screening for the best inoculum will be executed first. Due to limited knowlegde of the microbial populations in anaerobic sludges microbial communities will be analysed during the project. Both biogas plants will be started using the best suited inoculum aiming to achieve an enhanced biogas yield in process.

#### **Methods**

For screening a suitable inoculum digestates from 10 different biogas plants were analysed comparatively. The composition of the solids as well as the biogas potential were investigated. Standard batch digestion tests were carried out in 1L-batch reactors according to VDI 4630. Each digestate was prepared as inoculum and seven 1L-batch reactors at 37 °C were operated parallel. Maize silage was fed to three batch reactors as mono-substrate to evaluate its conversion to biogas. As a control substrate microcrystalline cellulose was used. After feeding biogas production was measured continuously for each reactor.

#### **Results and conclusion**

Results for the analysis of the digestate used as inocula will be presented. Biogas yield and course from the substrate maize silage achieved with 10 different digestate will be shown. Accordingly, the dependence of biogas production on the different inocula will be presented.

#### Scientific innovation and relevance

The screened inoculum with the most efficient biogas production will be used as inoculum for the high-load digestion with maize silage as mono-substrate.



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# Simulation of biogas plant run based on chosen substrates under laboratory conditions

# Biogas, continuous fermentation, biogas plant run, swine slurry

Global climate changes and the threats related to conventional systems for power generation have affected the rapid development of renewable energy technologies production (Mesarić and Krajcar 2015). One of the alternative energy sources is the biogas produced in methane fermentation process (Budzianowski 2012). Moreover, it is considered that new Renewable Energy Sources support system in Poland, will promote construction of biogas plants. Due to the large energy potential in terms of biofuels production, in the near future the construction of new biogas plants is expected in Poland (Piwowar et al. 2016). However, before any investment in biogas market, it is necessary to perform research tests of the efficiency of methane production from the used substrates and to proceed simulation of initial start-up on a laboratory scale. This action helps to avoid financial losses in real scale resulting from the process interference.

The aim of the study was to simulate the start-up of an agricultural biogas plant in laboratory scale using pig manure and maize silage. Therefore, there is a scientific problem that claims: it is possible to start-up a biogas plant with pig manure supplemented with pulp fermentation without the use of cattle manure as a source of microbial methane fermentation. The research tests on methane efficiency of the substrates in continuous fermentation (in glasses fermentation reactor BTP-2 Umwelt- und Ingenieurtechnik GmbH) were carried out in the Laboratory of Ecotechnology at the Institute of Biosystems Engineering at the Poznań University of Life Sciences pursuant to adapted standards VDI 4630, commonly used in Europe. The analyses of basic physicochemical parameters of the substrates and inoculum were conducted according to Polish standards: dry matter (PN-75 C-04616/01), dry organic matter and ash (PN-Z-15011-3). In continuous process, pH analysis has been carried out every single day.

The substrates used in fermentation reactor were swine slurry and maize silage. These materials were characterized by dry matter content (TS) at the level of  $5.47\,\%$  and  $41.97\,\%$  respectively. While organic dry matter content (VS) was more than  $75\,\%$  T.S. During fermentation process the volume of produced biogas amounted  $2069.72\,\text{dm}^3$  with an average content of methane approx.  $56\,\%$ .

The results obtained allowed to conclude that the start-up of an agricultural biogas plant using pig slurry without supplementation leads to a cease of the fermentation process and intensive production of ammonia and hydrogen sulfide. Reduction of fermentation process efficiency resulted from low content of methanogenic bacteria in swine slurry, used in simulation of fermentation process. It led to accumulation of hydrolysis and acidogenic bacteria (Deublein and Steinhauser 2008, Lindmark et al. 2014).

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The use of digestate as supplement allows for a stable production of biogas and methane with a load volume of 4.5 kg·m<sup>-3</sup>·d<sup>-1</sup>. This result is corresponding for organic load rate in one-step fermentation reactor (BISCHOFSBERGER et al. 2005, FNR 2005, AHMAD et al. 2011, OWAMAH AND IZINYON 2015).

On the basis of obtained research results, the following conclusions have been formed:

- 1. It is possible to run the start-up of an agricultural biogas plant at using swine slurry supplemented with digestate without the use of cattle manure as a source of methane fermentation microflora
- 2. Organic load rate in fermentation reactor at the level 4,5 kg VS·m³·d¹¹ allow stable production of biogas and methane.
- 3. In order to define maximal organic load rate limit in fermentation reactor, at use swine slurry and maize silage, it is necessary is to perform further research.
- 4. Used swine slurry as mono-substrate in the start-up of an agricultural biogas plant lead to breakdown of methane fermentation process and intensive production of ammonia and hydrogen sulfide.

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### Effect of sulphate addition on the microbial community during the anaerobic degradation of cellulose

# Process efficiency, methanogens, sulphate reducing bacteria, microbial competition

The microbial community in a biogas plant is complex, as a range of different microbial groups is involved in a complete degradation of organic substance and a suitable and balanced composition of the community is essential for a quantitatively and qualitatively high gas yield. Some substrates, e.g. wastewaters from slaughterhouses, food processing or paper production can contain high loads of sulphate (Colleran et al., 1995). Unfortunately the presence of sulphate during anaerobic digestion (AD) enables sulphate reducing bacteria (SRB) to compete with methanogenic archaea for hydrogen and may lead to severe process disturbances. The resulting product of the sulphate reduction, hydrogen sulphide, can further have an inhibitory effect on various members of the microbial community and endangers technical equipment, due to its corrosive impact.

Although there is a range of studies concerning the effect of sulphate on the gas production during AD, little information about the accompanied dynamics of the microbial community is available (Paulo et al., 2015). Thus the present work investigates the effect of the addition of sulphate to an AD process with special focus on the shifts in the bacterial and archaeal communities during sulphate caused inhibition of methane production.

The examined anaerobic community originated from a biogas plant in Roppen, Tirol. This thermophilic plug flow fermenter operates according to the Kompogas® dry fermentation procedure. as described in Illmer & Gstraunthaler (2009). A medium containing different amounts of carbon sources, mainly cellulose, was degraded over a period of 4 weeks. During the incubation, several chemical parameters, such as gas quantity and quality, concentrations of volatile fatty acids, and pH were determined along with a DNA based characterization of the involved microorganisms. The performed analysis gave an interesting insight into the processes, which accompany the adaption of a functioning anaerobic community to an abrupt rise of the sulphate concentration. Irrespective of carbon concentrations the samples with and without sulphate addition were distinctly different concerning the composition and the amount of the produced gas. While control communities without sulphate shock constantly produced methane, those exposed to an initial sulphate load of 3 g/L ceased methane production after 7 days of incubation. The results clearly demonstrated the competitive effect of SRB, which established quickly after the addition of sulphate. The presence of sulphate had also a significant influence on the dynamics of the volatile fatty acids, resulting e.g. in an accumulation of acetate in the sulphate containing samples. Depending on sulphate presence denaturating gradient gel electrophoresis (DGGE) showed distinct differences regarding the dominating bacterial and archaeal species at the end of the incubation. However these community shifts did not happen simultaneously with the changes in the gas production, which as an indicator of microbial activity, occurred more immediately after the sulphate addition.

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### The influence of hydrothermal and enzymatic hydrolysis on biogas efficiency of sewage sludge

# Sewage sludge, enzymatic hydrolyse, hydrothermal hydrolyse, economic calculations

Ensuring low-cost and highly productive substrates and efficient technologies is a key issue to secure the profitability of the biogas plants. Besides the silage, huge potential has a sewage sludge, produced in the world in the amount of tens of billions tons per year.

The aim of this study was to investigate the influence of technical and technological factors on the process of decomposition of substrates under methane fermentation with a separated step of hydrolysis process. Moreover, the research aim focused on the use of hydrolysis as initial stage of fermentation process in order to increase the amount of produced methane and profitability of biogas investment in Polish biogas market.

The study was conducted in Laboratory of Ecotechnologies – the biggest biogas laboratory in Poland, laboratory working within the Institute of Biosystems Engineering (Poznan University of Life Sciences). The research based on modified German standard DIN 38 414/S8, while chemical and physical analytical methods based on Polish Standard System. The concentration measurements of methane, carbon dioxide, hydrogen sulphide, ammonia and oxygen in the produced biogas have been carried out with the use of certified Geotech gas analyser GA5000. The substrate – sewage sludge, has been investigated under fermentation without any preprocessing (as a control) and subjected to two types of hydrolysis – hydrothermal and enzymatic. On the basis of obtained results related to biogas efficiency, also the energy and cost-effective balance of developed solutions use in the hypothetical biogas plant with a capacity of 1 MWe has been calculated basing on two kinds of governmental subsidies – certificates of origin and auction system.

The highest increase of methane efficiency of the sewage sludge was observed after enzymatic hydrolysis (29.4 %), however hydrothermal technology was very similar and reached 29 %.

Economic calculations based on actual market price of electric power and financing of renewable energy sources (RES) in the form of certificates or auctions proved that the use of sewage sludge for biogas purposes in any of the tested hydrolysis is not capable to increase the biogas efficiency enough to cover the investment costs. Economic factor NPV (Net Present Value) calculated for both kinds of hydrolysis had negative values, which means it is unprofitable and should not be implemented in industrial scale.

The key-point of economic success of biogas plant investment under Polish conditions (low subsidies, more than twice lower in comparison with Germany) is to find cheap technology because increasing biogas efficiency even for 30 % not always goes with better profitability of investment.



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### Eco-physiological Adaption of Methanogenic Communities: Potential for Biotechnological Applications

# Methane, methanogenic communities, natural habitats, adaptation, process optimization

Methane ( $\mathrm{CH_4}$ ) is of profound global interest both as potent greenhouse gas that is emitted from anthropogenic and natural sources as well as the primary component (60-70%) of biogas. Methane formation in engineered digesters is performed by providing and maintaining favorable conditions (e.g., constant temperatures and neutral pH-values) for methanogenic communities to retain process stability. In contrast, methanogenic organisms in natural habitats are exposed to a large climatic and seasonal variability as well as changing pH-values. It is yet unexplored if the indigenous organisms in natural habitats have evolved the ability to tolerate frequently changing conditions and whether this ability could be used to improve the overall process efficiency and stability of biogas plants. The ecologically- and/or community-based strategies providing tolerance toward changing environmental conditions are scarcely understood thus far and studies of the niche adaptation of methanogenic organisms in natural habitats are still in their infancy. Hence, the aims of the present study are (i) to identify and enrich methanogenic organisms with a broad eco-physiological potential and high resilience out of soil samples and (ii) to subsequently implement the enriched cultures to lab scale standard digestions and estimate the effect on fermenter performance.

Four different study sites have been chosen including two swamps (M1 and M2) and two springs (S1 and S2). The collected soil samples have been characterized via profound analyses regarding soil- and microbiological properties and in situ measurements of the CH, flux were performed employing a static chamber technique. Subsequently we enriched the engaged organisms out of the soils and established stable mixed cultures. With the purpose of evaluating the effect of changing environmental conditions in lab scale experiments, single-parameter variation studies including temperature and pH were performed to assess their impact on both the establishment and diversity of anaerobic communities as well as methane production potentials of these mixed cultures. Samples were regularly withdrawn to analyze the gases in the headspace and intermediate volatile fatty acids via GC and HPLC, respectively. Common molecular-biological techniques (DGGE) were employed to provide an insight into microbial diversity in order to detect possible adaptive capabilities of the engaged organisms. The *in situ* results revealed a highly positive methane balance at all four study sites. Furthermore, site-specific differences were observed, with methane flux rates determined at the swamp sites exceeding those assessed at the spring sites. These site-specific differences were also apparent during lab scale culture experiments regarding methane production potentials. The engaged organisms of both swamps showed their optimum for methane production at 37 °C and pH 6.5, whereas the ones inhabiting the springs showed different optima (S1: 25 °C and pH 6.5; S2: 25 °C and pH 7.5). Surprisingly, these optima of the mixed cultures remain unchanged for weeks. Fingerprinting-techniques (PCR-DGGE) revealed a distinct impact of the prevalent environmental conditions on the diversity of archaeal species. Interestingly, DGGE band numbers and patterns showed a contrary picture depending on varied temperatures or pH-values, suggesting that both temperature and pH have a regulatory but contradictory effect on the methanogenic community structure and composition in complex ecosystems.

The results are therefore not only of ecological relevance but could also contribute to a stable process and prevent (upcoming) instability and thus calls for new approaches to investigate the potential and characteristics of indigenous microbial communities out of natural habitats as a function of varying environmental conditions.



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# Integration of microalgae cultivation for enhanced biogas production at the municipal wastewater treatment plant

# Process simulation, microalgae AD, WWTP

Sustainable wastewater treatment is possible by applying different processes and technologies such as chemical enhanced primary treatment, deammonification and anaerobic digestion (AD), where energy consumption reduction on one side and energy recovery from wastewater on the other side can be achieved. AD is commonly used for energy generation from biosolids (i.e. primary and secondary sludge). On the other hand, bio-solids processing causes additional nitrogen and phosphorous load to the mainstream bioreactors in form of rich in ammonia and orthophosphates reject waters. In this research, microalgae were investigated as an sustainable option for biogas intensification in AD but also as a post treatment method for reject waters after the Anammox process. Study found that microalgae biomass boost biogas production but also side steam microalgae process reduce nitrogen and phosphorus concertation in reject waters. The key objectives of the project were

- (1) to investigate the nutrient removal performance and
- (2) to determine the biogas productivity for native algae (single-strain and multi-strain) grown in pretreated effluent from full scale anaerobic digester, and additionally
- (3) to evaluate the energy cost of biomass processing (cultivation, harvesting, pretreatment, biogas production) against the energy production and nutrients removal throughput flow sheet process modeling.

The implementation of side stream microalgae process for reject water treatment has potential for nutrients removal and could be applied as an additional sustainable biomass source for biogas production. Application of additional biomass could cause a exploitation of larger working volume of digesters, thereby the reduction of HRT and boost the methane production in WWTP. Technoeconomic analysis revealed the advantages and disadvantages on integrating microalgae processing into municipal wastewater treatment plant.

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# Automated H<sub>2</sub> and H<sub>2</sub>S measuring device with intelligent overload protection

# Biogas sensor, H<sub>2</sub> sensor, H<sub>2</sub>S sensor, overload protection

As part of the public founded (BMBF) program "BioProFi - BioEnergy - Process-oriented Research and Innovation" the aim of the project "MOST - model based process control in biogas plants" was to develop a compact measuring device with multiple parallel working sensors for the biogas analysis. For a better understanding of the bioprocess itself, the participating microorganisms and their interactions during the process a reliable stable mathematical model had to be designed. Based on this new model a sensor based control of the process should be established. As part of this project a new modular sensor for H<sub>2</sub> and H<sub>2</sub>S in the biogas of even small laboratory fermenters was developed. Beginning with the examination of different electrochemical detectors for H<sub>2</sub> and for H<sub>2</sub>S the key problem for using these types of detectors in biogas is, that the amount of oxygen which is necessary for the electrochemical reactions inside the detectors cannot be provided by the anaerobe digestion the biogas process itself. Another challenge is the wide range of possible gas concentrations that can be found in the biogas. The concentrations of interest for the process control are in the range of a few 10 ppm up to some 100 ppm. Sometimes much higher amounts of several 10.000 ppm are possible. The new sensor uses a unique method of precise automatically adapted dilution of the biogas. With this new method both problems are solved. Prototypes of the sensors are currently used and evaluated by the project partners and will be optimized afterwards regarding the requirements of the model based process control strategy.



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# Near-infrared spectroscopy (NIRS) for a real-time monitoring of the biogas process

# NIRS, process monitoring, volatile fatty acids, flexibility

A more flexible energy production from renewable energy sources like photovoltaic, wind and biogas is a key point in German energy policy. As biogas is only temporarily storable due to its large volume, a flexible and demand-oriented production gains more and more importance to cover diurnal peaks in power demand. One possible approach to adapt the biogas production is the adjustable feeding of the digester.

However, this is at odds with the fact that possibilities to gear into the complex biological system of anaerobic fermentation are still limited. Disturbances occurring during the biological fermentation process are still monitored by time-consuming wet chemical analyses. This often prevents timely intervention to avoid operational failures. To monitor the anaerobic digestion an exact knowledge of relevant physico-chemical process parameters, such as the acetic, propionic and the total acid, the organic dry matter (VS), ammonium content (NH $_4$ ) and the carbonate buffer (TIC), is crucial. Therefore, real-time process control will play a decisive role in enabling the assessment of the current digester state.

The application of near infrared spectroscopy (NIRS) allows new approaches of analysis and process control within the production of biogas.

In our experiments we investigated, how quickly an impact loading of substrate was going to be microbially converted and whether this increase in organic loading rate stressed the fermentation process.

A laboratory scaled biogas digester was continuously fed every two hours by an automatic feeding system to achieve an organic loading rate of 2.5 kg organic dry matter per cubic meter digester volume and day. Impact loading varying in its frequency was performed with shredded wheat up to an organic loading rate of 8 kg organic dry matter per cubic meter digester volume and day.

In this context, NIRS measurements were carried out to document digester reactions to seasonal fluctuations in feed composition or to singular impact loadings, and to determine how quickly biocoenosis can recover following a change in loading. The frequency in which changes in substrate or impact loadings can be done without leading to process instabilities was to be observed.

The good results of the other process parameters indicate the excellent suitability of NIRS for monitoring the fermentation process in biogas plants. Nevertheless regular servicing and maintenance of the equipment is essential. Furthermore an application in practice requires the adjustment of the calibration models to the substrate matrices of every new biogas plant by the equipment manufacturer.



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#### Pocket Power! -

### Increasing the potential of anaerobic digestion at farm-scale

# Small-scale anaerobic digestion, Greenhouse gas emissions, Full-scale measuring campaign, Sector scan

To date the region of Flanders counts already more than 100 small-scale digesters. Such installations convert proprietary biomass to provide energy for the company itself. However, almost all installations use cattle slurry as input. Other agricultural sectors can still not benefit from the (partial) fulfillment of the energy requirements this technology can offer while rising energy prices becoming a more and more determining cost. The project Pocket Power, funded by Flanders Innovation & Entrepreneurship, wants to meet these shortcomings by exploring the possible extension of the positive experiences with small-scale anaerobic digestion of cattle slurry to other agricultural streams (e.g. pig manure, crop residues). In addition, Pocket Power will investigate to what extent pocket digestion could serve as a climate measure by quantifying the reduction in greenhouse gas emissions accomplished by implementing a farm-scale digester and in that way avoiding the uncontrolled anaerobic digestion that will take place during long-term biomass storage.

A physical-based model (based on mass balances) was set up considering hydrolysis as the rate-limiting step and including its temperature dependency. Simulation studies were performed for an average Flemish dairy farm taking into account constraints for manure storage as imposed by the Flemish legislation. Scenario-analysis was carried out through simulation of a default dairy farm with a manure pit under the stable (reference) in comparison to an integrated digestion farm with a manure slide, short-term external manure storage, small-scale anaerobic digester and digestate storage. The first simulations proved that the implementation of a farm-scale digester could have significant potential in reducing methane emissions originating from the reference taking into account assumptions for methane emissions due to digester leakages, fossil fuel production and manure spreading. Storing manure externally instead of in a manure pit under the stable could even without a digester diminish methane emission losses. Besides, minimizing the age of biomass fed to the digester demonstrated to improve the process performance. An intensive full-scale measuring campaign will be performed to collect field data on biogas concentrations in the pocket digester, manure pit and digestate storage. These data will be combined with model refinements and additional simulation studies to get a more detailed view on greenhouse gas emissions associated to pocket digestion. Moreover, emissions by potential leakages, methane slip or an active overpressure safety device will be monitored. Additional measuring campaigns may be set up to explore seasonal variability of the emission rate. Furthermore, a sector scan will be performed for Flanders to define the potential of a transfer to other agro sectors. Based on a multi-criteria analysis the two subsectors with the highest potential will be selected. Since new feedstocks could cause new technical problems, hands-on solutions will be sought to enable the design of an adjusted pocket digestion concept. This design will be evaluated economically and ecologically. Finally, Pocket Power will assist in the implementation of a pilot installation within one selected subsector.

The overall aim of Pocket Power is to respond to the demand of other sectors than the dairy sector for a profitable valorization of their agricultural residues as well as to provide a framework for the development of operational strategies to further reduce greenhouse gas emissions. Moreover, the the economic and ecological impact studies – if positive – may offer constructers a real help in expanding their business and may convince stakeholders of the benefits of this technology.

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# Enhancement of biogas production from residual agricultural feedstock in small-scale biogas plant by cavitation-based pretreatment strategies

# Cattle manure, ultrasound, hydrodynamic cavitation, lignocellulosic feedstock, anaerobic digestion

Development of a sustainable bioenergy market is now based on bio-based resources to meet climate protection and resource efficiency. However bio-based resources, that can substitute fossil resources, have to minimize the competition between food supply and biomass production. Consequently, there is a need for searching the non-food biomass to rebalance the support of biofuels production versus the food feedstock use.

Animal manure is a massively under-exploited biomass resource which have to be treated in environmentally-friendly way to minimize GHG emissions (FAO 2013). Worldwide GHG emissions from livestock supply chains are estimated to produce 7.1 gigatonnes of  $\mathrm{CO}_2$  per annum, which represents 14.5 % of all human-induced emissions. Anaerobic digestion (AD) of organic biomass into biogas is now evaluated as one of the most energy-efficient technology for bioenergy production (Montingelli et al. 2015). Thus, AD of animal manures may be an effective way to reduce a fugitive methane emissions from slurry storage, and can also displaces the fossil fuels consumption. In fact, animal manures are depleted of much of its energy in the animal gut, and co-digestion strategy may improve its relatively small amount of energy recovered.

This work was aimed at carrying out a small-scale ABP for biogas production from dairy cattle manures mixed with residual wheat straw. Wheat straw is now recognized as one of the most abundant biomass produced in the world, making it highly interesting as a substrate for biogas production (Sun et al. 2013). However, due to the complex lignocellulosic structure, its degradability and gas yield are low. To enhance the feedstock digestibility, cavitation-based pretreatment strategies: ultrasonic and hydrodynamic cavitation were tested. The use of pretreatment methods is justified only if profits exceed the expenditure of energy.

#### Aim of work

The study analyzed the cost-effectiveness of energy use ultrasonic or hydrodynamic cavitation in relation to the system without disintegration.

#### Methods - Pretreatment equipment and procedure

Ultrasonic pretreatment (UP) was performed in 5 connected rectangular square hollow tubular sections (0.96m x 0.96m x 0.85 m) made of stainless steel with a total working volume of 40 L (8 L per section). Sections were placed in an ultrasonic equipment made of 60 transducers, each with a power of 10 kW and a frequency of 24 kHz. The hydrosonic pump used for hydrodynamic cavitation pretreatment (HCP) was constructed of cylindrical rotor fixedly attached to the shaft and placed in 0.25 L tank (Patent No PL 214335 B1). The rotor was turned by electric motor (4 kW, 2800 rpm). A feedstock inlet port was placed at the bottom of the tank, while the outlet port was on the top. As the feedstock injected in the tank travelled across the rotor and was mixed by centrifugal force, areas of vacuum were generated within the liquid from its own turbulence, expansion and compression resulted into cavitation.

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Depending on the pretreatment approach, ultrasonic disintegrator or hydrodynamic disintegrator were installed behind the rotary lobe pump. Starting the pump involved the filling of pretreatment device and was associated with the inflow of pretreated feedstock to MFT and the outflow of fermented feedstock from MFT to PFT. Both pretreatment devices worked in a batch mode. In UP, the number of cycles per day was 10 (1 min feedstock filling/15 min working/1 min feedstock removing), while in HCP it was 16 (1 min feedstock filling/10 min working/1 min feedstock removing). It was established on the results of laboratory experiments performed for optimization of the cavitation conditions on pretreatment of the mixture of cattle manures and wheat straw. Feedstock was pretreated with Es ranged from 0 to 8064 kJ/kg generated by ultrasonic equipment and hydrosonic pump. Energy inputs provided the highest biogas production from pretreated feedstock was 4034 kJ/kg TS for both UP and HCP (data not published).

#### Results - Energy balance of biogas plant

The energy input and output for the biogas process and the pretreatments were calculated as kWh/d. The lowest energy input was noted for ABP operation, but the gross energy output was as low as 76 kWh/d. Ultrasonic equipment used in ABP-UP doubled energy needs. However, the efficiency improvement of the biogas production ensured the highest gross energy output on the level of 97 kWh/d. In case of the ABP-HCP operation, the gross profit of energy was 91.6 kWh/d.

Energy consumption to produce 1m3 of biogas was the lowest in ABP. Comparing the two pretreatment methods, lower energy needs for hydrosonic pump operation resulted in lower energy requirement per unit of biogas produced in ABP-HCP. The final net energy output was of 61 kWh/d was calculated for ABP-HCP, which turned out the most energy-effective variant for residual agricultural feedstock management. High energy requirement for ultrasonic equipment operation contributed to lowest net energy gain of 52 kWh/d, despite the highest gross energy output. During ABP operation, where no feedstock pretreatment was done, the net energy output was 56 kWh/d.

In conclusion, the biogas plants based on hydrodynamic cavitation feedstock pretreatment are the most profitable, however in the current industrial practice of wastewater treatment, hydrodynamic cavitation is not used. Our study showed that low energy requirement for hydrodynamic cavitation makes the potential to become energy efficient technique for lignocellulosic biomass pretreatment in a large scale. The electricity consumption associated with the motor was not greater than profits derived from extra biogas yield.

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# SPONSORS



# Awite Bioenergie GmbH

Gas analysis systems, automation solutions, microbiological desulfurisation and flow measurement from a single source: Awite is specialised on gas analysis, automation and measuring technology in the field of biotechnological plants.

#### **Portfolio**

- » Gas analysis systems (incl. service) for more than 2000 biogas plants
- » Automation system for more than 100 biogas plants
- » Desulfurisation systems
- » Flow measurement
- » Sensors & Electronis



# **Bioprocess Control AB**

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 20 years of industry leading industry 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.

#### **Portfolio**

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



## Institute for Biogas, Waste Management & Energy

Based on many years of experience in research and consulting Prof. Dr.-Ing. Frank Scholwin founded in 2012 the Institute for Biogas, Waste Management & Energy in Weimar. In close conjunction with a network of national and international experts independent scientific consulting services are provided.

The main targets are to implement biogas technology in order to contribute to the sustainable transition of the energy system from fossil fuels to renewables and to create sustainable material life-cycles.

A large number of research and consultancy projects incorporating the linked and sometimes conflicted fields of biogas technology, energy management, agriculture and waste management was carried out or coordinated. This has already resulted in large numbers of projects providing consulting services to plant operators, power supply utilities, financial institutions, professional bodies and government agencies, enabling Professor Scholwin to acquire an expert and up-to-date understanding of the organisational, technical and economic needs and concerns of that client base.

One of the key elements of the expertise during the last years was the evaluation of the biogas market and the biogas potentials for future development. A large number of publications on biogas topics has been written during the last years. Prof. Scholwin is member of a number of scientific bodies and scientific boards/committees.

#### **Portfolio**

- » Consulting for industrial and political decision-makers
- » Independent expert evaluations
- » Development and evaluation of in-depth technical and economic concepts
- » Cooperative analysis for identifying and resolving weak points in plant operations
- » Concept and execution of seminars, conferences and expert presentations
- » Coordination and execution of scientific work in national & international research projects

#### **Further information**

www.biogasundenergie.de/publications.html

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### BlueSens gas sensor GmbH

#### BlueSens - understanding bioprocesses

BlueSens gas sensor GmbH develops and manufactures sensors and software for bioprocess applications. The company is located in Herten, Germany and has been founded 15 years ago by the founders and managing directors Dr. Udo Schmale and Dr. Holger Müller, who are still in charge today. BlueSens has an own research and development department and is constantly evolving its reliable products. In addition to its own research and development, BlueSens is also very active in the field of research projects funded e.g. by BMBF. The main ambition of BlueSens is that their customers are able to gain a comprehensive understanding of their bioprocesses so that they can get the best out of their processes. Depending on the individual need that could be a perfect understanding and documentations of the metabolic processes for a scientific workgroup, the maximum yield in an anaerobic digestion plant or the total quality for a PAT process in the pharmaceutical industry. To archive that, BlueSens is offering a variety of gas sensors to analyze the key parameters of the bioprocess. Over the last years, software such as BlueVis is becoming more and more important in BlueSens' portfolio. The software can collect the data, it calculates, visualizes and logs additional information via soft sensors, and it controls actuators such as pumps, stirrers, thermostats etc.. BlueVis has got a high grade of connectivity to sensors and probes of other brands and is even able to send and receive data from process lead systems. In general, the BlueSens gas sensors are installed directly in the process and measure in real-time vital gases (such as  $CH_4$ ,  $O_2$  /  $CO_2$ ,  $CO_3$ ,  $CO_4$ ,  $CO_4$ , and Ethanol). Through the measurement data, the conditions in the fermenter can be consistently observed and documented and the process can be better analyzed and controlled. The sensors are easy to integrate in the bioprocess and they have no direct contact to the substrate and are applicable for all process scales. With BlueSens it is easy for you to gain a deeper understanding of your bioprocess and to run a controlled process.

#### BlueSens, the "hidden champion"

A lot of branches know the so called "hidden champions". Hidden champions are smaller companies that are quite unknown but still the market leader in their specific branch. In the field of gas analysis for bioprocesses BlueSens is such a hidden champion. More than 15,000 sensors for  $\rm CO_2$ ,  $\rm O_2$  and  $\rm CH_4$  were sold over the last 15 years to the pharmaceutical industry, biofuels companies and a lot of universities. Many of these sensors are sold directly together with a complete fermentation system by major players in that field, for instance Sartorius Stedim, Infors HT, Applikon or Eppendorf. This means that quite often the customer doesn't even recognize that he already owns BlueSens' sensors. If you own a fermentation system of one of these big players with an integrated gas analysis, you will also have components of the hidden champion BlueSens included. BlueSens is more than just a component supplier for the major companies. The gas analysis technique and new developments of BlueSens are influencing the complete approach of bioprocessing. The old fashioned mass-



specs are becoming obsolete for a lot of applications and the less expensive gas analyzer facilitate an in-situ analysis of the complete process in real time, especially for parallel processes. Knowing the key parameters in real-time is the prerequisite for gaining a deeper understanding of the process and to control the process efficiently. More and more people in universities, laboratories and the industry become aware of the unique measurement solutions of the hidden champion and it is just a question of time until BlueSens is no longer a hidden champion but a well-known company for everyone involved in fermentation processes, bioprocessing and the production of biofuels.

#### Monitoring the key parameters of the bioprocess with BlueVis

Monitoring the main process parameters in the bioprocess in real-time is the key to be able to optimize and to control the process. For a lot of processes the OUR, CER, RQ, and the growth rate are main indicators of the metabolic state. BlueSens' bioprocessing software BlueVis is capable of calculating these important parameters via the integrated soft sensors. BlueVis will utilize the measurement data from the connected sensors and probes to compute the key parameters in real-time. For other processes different measurement values (e.g. pH value,  $CH_4$  concentration etc.) could be the main process parameters. If the parameters are not in the expected or desired range, it might be necessary to adjust the process actuators via BlueVis. For example a substrate feed via pumps could be adjusted, the speed of the stirrer, the temperature via the cryostat or the gas flow at the inlet via mass flow controller. BlueVis also executes pre-programmed controls. For instance, if the CER of your process drops under a desired value BlueVis could activate a pump to feed the process with glucose. In this way the new BlueVis fits perfectly to the BlueSens philosophy and will assist you in understanding bioprocesses.

#### **Portfolio**

- » Gas Sensors for various biofuels (CH<sub>4</sub>, H<sub>2</sub>, Ethanol & Methanol) and for other gas components (CO<sub>2</sub>/O<sub>2</sub>/CO)
- » Software for the monitoring, logging, visualisation and management of fermentation processes

#### Web

# **SIEMENS**

#### Siemens AG

The Siemens Process Industries and Drives division is the trusted partner for continuous improvements in biofuels, especially biogas.

Siemens AG is a global technology powerhouse that has stood for engineering excellence, innovation, quality, reliability and internationality for more than 165 years. The company is active in more than 200 countries, focusing on the areas of electrification, automation and digitalization. One of the world's largest producers of energy-efficient, resource-saving technologies, Siemens is a leading supplier of efficient power generation and power transmission solutions and a pioneer in infrastructure solutions as well as automation, drive and software solutions for industry.

Over many years, Siemens has built up a strong experience with worldwide proven customized solutions for biofuels, especially biogas: Our capabilities range from the smallest privately held farmer's biogas plant up to the worldwide-scale production of bioethanol or biodiesel by "blue chips" companies. Customers have chosen Siemens to maximize their profitability and performance in over 500 plants targeting performance and cost optimization, both over the processing workflow as the entire lifecycle. Our comprehensive portfolio of solutions, systems, products and services is specifically geared to meet the needs of producers, planners (EPCs), equipment manufacturers (OEMs), and system integrators. With our innovation capabilities, we provide state-of-the-art solutions and constantly optimize our portfolio to effectively address your production challenges:

- » Shorter time to production thanks to a high level of integration, fast engineering and commissioning
- » Low initial investment combined with maximum sustainability for solutions according to your requirements, global and locally
- » Increased profitability with high plant availability, optimum product quality, efficiency and flexibility
- » High plant safety and IT security for staff, assets and environment

Products, systems, solutions, software and services from the process control system SIMATIC PCS 7 for continuous and batch process, through intelligent motor control centers and drives, gas analysis, weighing, to field instrumentation (sensors), Siemens offers a broad portfolio for biogas. The offer also extends to process design and engineering, project management and nowadays digital solutions on the way to Industry 4.0.



#### **Portfolio**

http://w3.siemens.com/mcms/sensor-systems/en/Solutions-for-industries/Renewable-Energy/Pages/Renewable\_Energy.aspx#w2g-/mcms/sensor-systems/en/Solutions-for-industries/Base4Layer/Pages/Casestudies\_Erneuerbare\_Energie.aspx

#### **Further information**

Web link: www.siemens.com/biogas www.siemens.com/sensors/renewable

Speech given at  $3^{rd}$  International Conference on Monitoring and Process Control of Anaerobic Digestion Plants, 30.03.2017, 12:20:

"Application of Extended Kalman Filter as Soft Sensor for Anaerobic Digestion Plants", Daniel Labisch (Siemens AG)

#### Address

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#### Web

www.siemens.com/biogas





# 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.

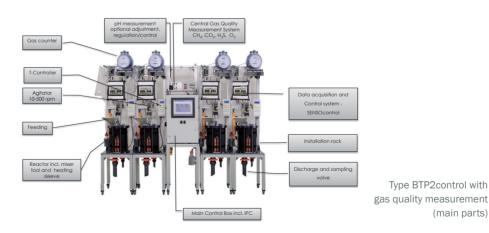
As a classic Engineering Company with own production facilities we are in position to provide standard equipment, but also custom-made biogas testing systems that is specific to clients technical requirements - from laboratory scale to mobile container based solutions.

Typically our plants come with:

- » Bioreactors
- » 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 SENSOcontrol

#### **Portfolio**

Co-operations together with several national and international research and development institutions, as well as universities



Web

www.uit-gmbh.de

Type BTP2control with

(main parts)

#### **Address** Zum Windkanal 21 01109 Dresden

Germany

#### Contact

**Thomas Schneider** +49 (351) 8864682 t.schneider@uit-gmbh.de

#### Green conference

The 3<sup>rd</sup>conference "Monitoring & Process Control of Anaerobic Digestion Plants" was planned mostly as a resource-saving and thus environmentally sustainable conference. Following aspects were taken into account:

#### **Venue**

The Leipziger KUBUS is run by the Helmholtz Centre for Environmental Research – UFZ, which is certified according to EMAS III, a rigid eco-audit for public institutions. By building the Leipziger KUBUS in 2004 ecological aspects were taken into consideration, e. g. the foyer is heated by geothermal energy during winter, coated glass slats in front of the glass façade reduce the heat load in summer and the rooms` heat recovery is provided by exhaust air. For all events hosted in the Leipziger KUBUS is waste separation obligatory.

#### Food

Keeping the climate footprint of our conference in sight, we decided to offer only vegan/vegetarian food and no meat. Many of the dishes are made from regional and seasonal products.

#### Preventing leftover food

In order to avoid leftovers the catering company "ENK Leipzig GmbH" calculates the food as exactly as possible. In case there are leftovers – as registered participants do not appear at the conference – doggy bags can be handed over to the guests.

#### Recycling paper

Printed products like the conference reader are made from recycled paper and comply with the standards of the eco-label "Blauer Engel".

#### Preventing paper waste

If required, participants can get conference material at the registration desk. Additional information materials and publications are also available in the registration area.

#### **Mobility**

The conference takes place in the excellent located Leipziger KUBUS. Participants reach the KUBUS from the main station within 15 min by public transport (Tram 3/3E, heading for "Taucha" or "Sommerfeld", get off at stop "Torgauer / Permoserstraße") . Participants travelling by car reach the KUBUS from the motorway A14 within just a few minutes (exit "Leipzig-Ost", heading "Stadtzentrum" and turning left into "Permoserstraße") . Nevertheless, the organizers recommend to travel by public transportation.

#### Compensation of CO, emissions:

As organizers we tried our best to keep  ${
m CO}_2$  emissions connected to the conference as low as possible and chose an environmentally friendly venue, catering and promoted travelling by public transport.

Participants can also contribute to a better climate footprint by e. g. compensating CO<sub>2</sub> emissions of their own journey. Special registered companies are for instance:

Atmosfair >> www.atmosfair.de or My Climate >> de.myclimate.org

# Organizing instituions



## 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.



#### 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.



# LHL - 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.

# **Organiser**

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