

MIR spectroscopy for monitoring of AD processes – Prospects & Challenges

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- 1 Middle-infrared spectroscopy - background**
- 2 Middle-infrared measurement system design**
- 3 Lab- and full-scale testing of an MIR probe**
- 4 Results and challenges**
- 5 Prospects – MEMS-based MIR sensors**



Middle-infrared spectroscopy - background



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760nm 2μm 10μm 1mm

near-infrared	middle-infrared	long-infrared
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- The mid-infrared region is interesting as
 - Biochemical process variables show stronger absorption.
 - Spectral data is clearer due to less overlap.
- In 2006 first MIR experiments were conducted by Spanjers et al. at wastewater treatment plants [1]:
 - Problems with water content, length of optical fibre, fibre material and spectrometer costs

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Agenda



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Requirements for a MIR online measurement systems

Measurement of
multiple process
variables

Robust probe and
probe fitting design

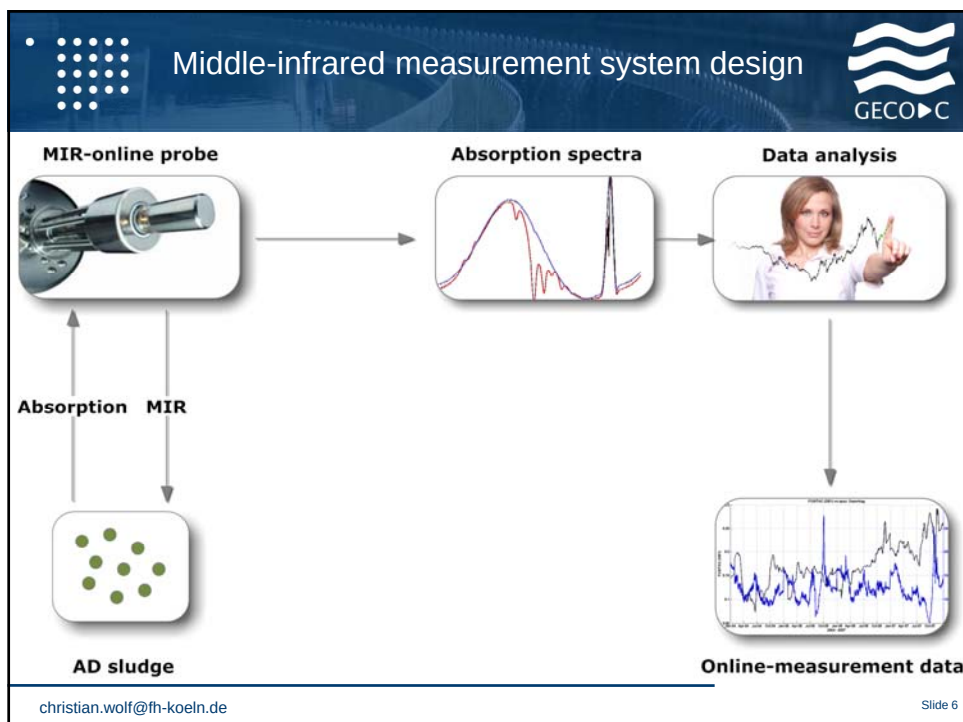
Fully automated
cleaning and
recalibration system

Data Analysis and
intelligent software

Reasonable pricing 10,000-15,000€

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Middle-infrared measurement system design



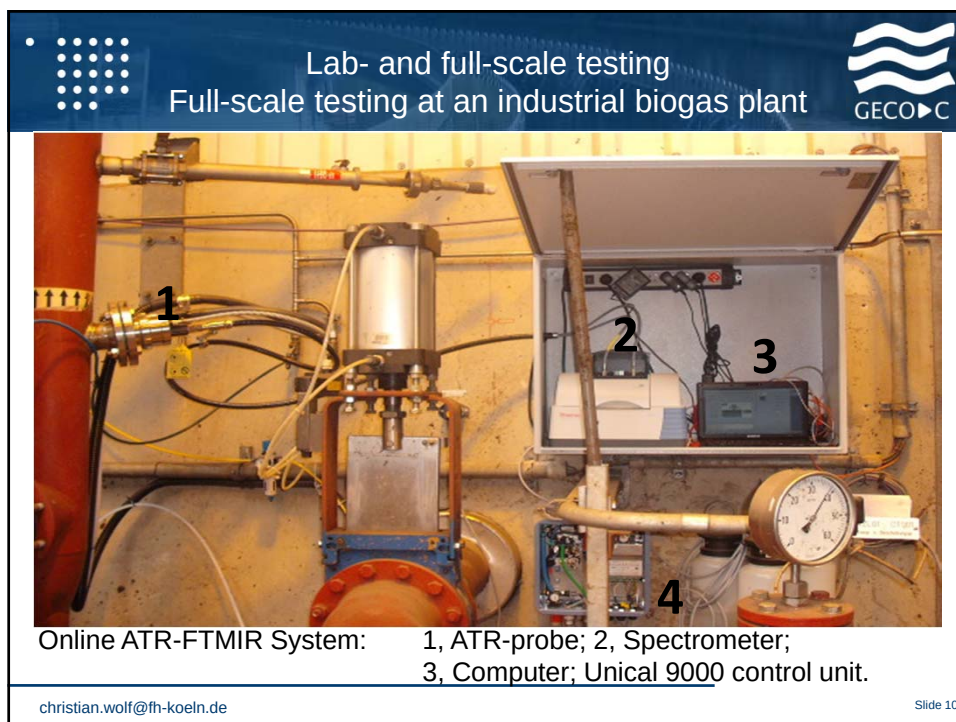
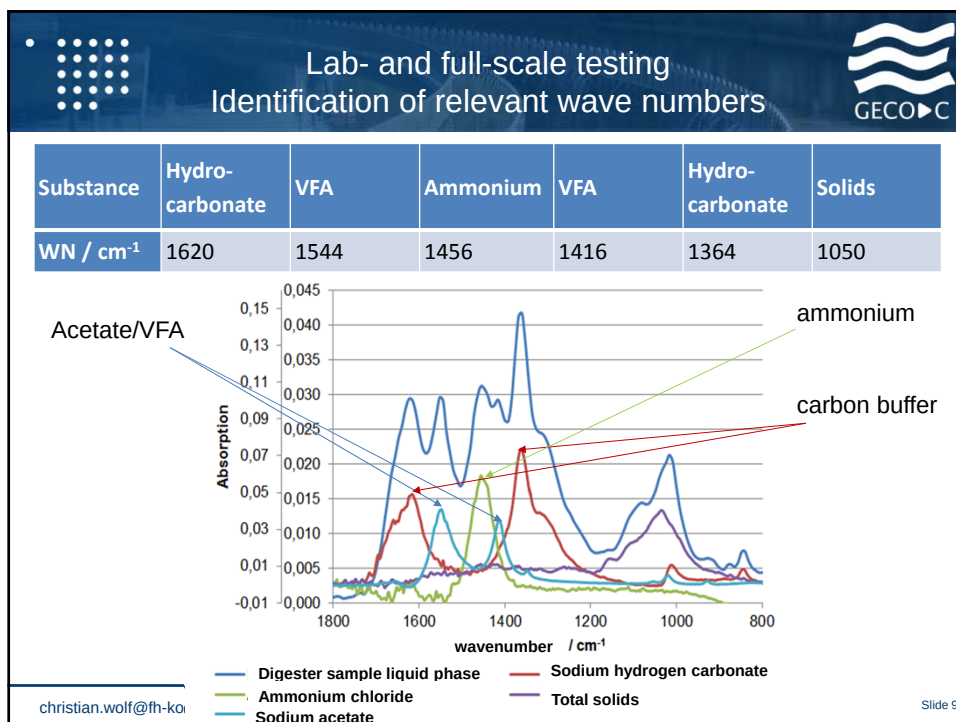
- A diamond-tipped ATR probe from the company art photonics was used ($4000 - 400 \text{ cm}^{-1}$).
- The MIR-light is transmitted through a Polycrystalline fibre (PIR) made from Ag/Cl and Ag/Br.
- The MIR-spectrometer is from Thermo-Fisher (iS5).
- The fully automated process interface comes from the company Knick.



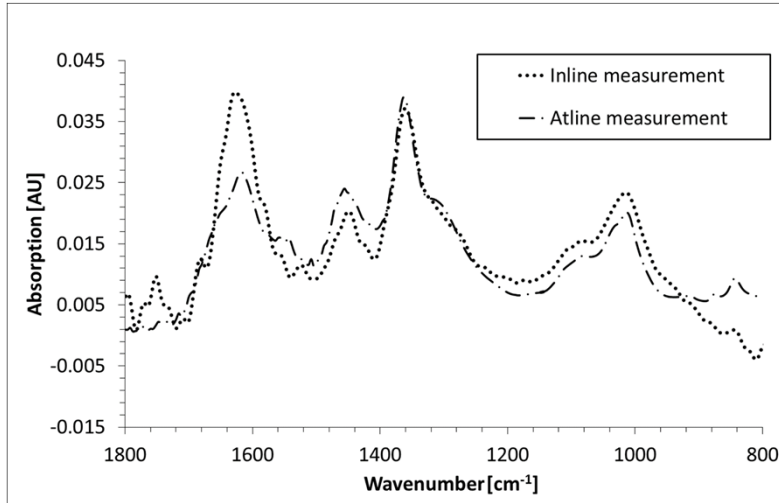
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Lab- and full-scale testing Comparison inline and lab spectrums



Agenda



- 1 Online monitoring of biogas plants – state of the art
- 2 Middle-infrared measurement system design
- 3 Lab- and full-scale testing
- 4 **Results and challenges**
- 5 Further development based on MEMS-technology

Results and challenges

Calibration results using Machine Learning

Process variable	Method	b_1	R^2	RMSECV / g/l
VFA	PLS1	0.950	0.971	0.372
VFA	v-SVR	0.939	0.937	0.386
Bicarbonat	PLS1	0.992	0.996	0.336
Bicarbonat	v-SVR	0.990	0.995	0.259
NH ₄ -N	PLS1	0.985	0.992	0.171
NH ₄ -N	v-SVR	0.990	0.993	0.110

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Results and challenges

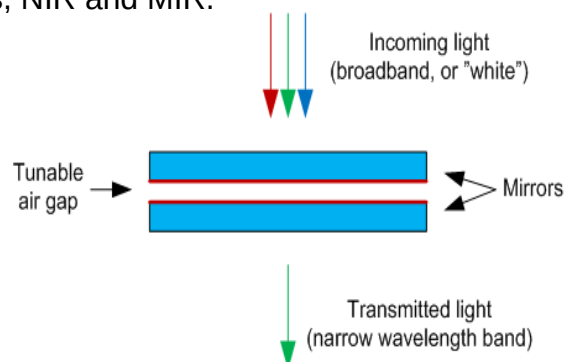
- It is possible to detect VFA, TA and ammonium in AD processes using MIR spectroscopy.
- Sensitivity and accuracy of the MIR system is poor! Only high concentrations can be detected. Reasons are:
 - long optical fibre 3m.
 - vibrations (full-scale application)
 - signal loss due to fibre coupler
- The full-scale test showed:
 - The automated process interface works.
 - Automatic cleaning and recalibration reduced maintenance to a minimum.
- Overall, costs for the system are too high. (40,000€ +)

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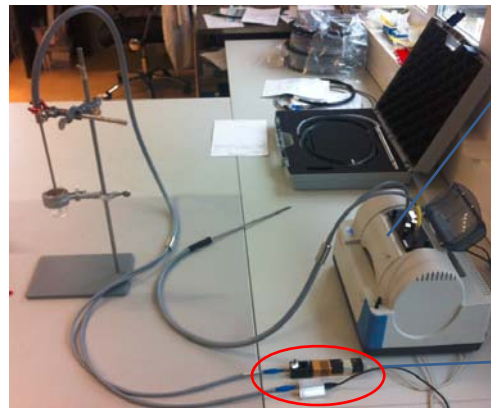
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Prospects MEMS-based MIR sensor

- MEMS-based Fabry-Perot-Interferometers are small (10 x 5cm) and cheap (~70-100€) and are available for UV/vis, NIR and MIR.



Prospects MEMS-based MIR sensor



FT-IR spectrometer iS5
before



MEMS-FPI
after

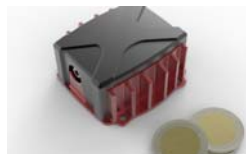
Totally integrated MIR sensor design



Diamond tipped ATR
probe



MEMS-FPI spectrometer
from 6-8 μ m and 8-10 μ m



Raspberry PI for probe
control and data analysis





Summary and outlook



- MIR spectroscopy is a promising technology that requires less calibration than NIR spectroscopy.
- Main issues are S/N ratio and equipment costs.
- MEMS-based FPI sensors allow reduction of fibre length which improves S/N ratio.
- MEMS-FPI sensors will bring down prices for NIR and MIR sensors if produced in large numbers.



Thank you!



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- [1] Spanjers, H., Bouvier, J., Steenweg, P., Bisschops, I., van Gils, W. and Versprille, B., 2006. Implementation of in-line infrared monitor in full-scale anaerobic digestion process. *Water Science and Technology*, 53 (4-5), 55–61.