



Spectroscopic Measurements for the Anaerobic Digestion Process

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Agenda

- Why Spectroscopic systems?
- Liquid Phase Measurement.
- Gas Phase Measurement.
- Conclusions.

WHY SPECTROSCOPIC SYSTEMS?

Spectroscopic Measurements

- Advantages
 - Can predict multiple parameters using the same data.
 - Doesn't need sample preparation (e.g. centrifuging, diluting, filtering).
 - Inline measurements gives faster results.
- Disadvantages
 - Not a true measurement, only a “prediction”.
 - Data analysis necessary to convert from spectra to concentration.
 - Better measurements with cleaning.
- Types of spectroscopic system
 - MEMS sensors less sensitivity to vibration.
 - FTIR systems more sensitive to vibration due to moving mirror.

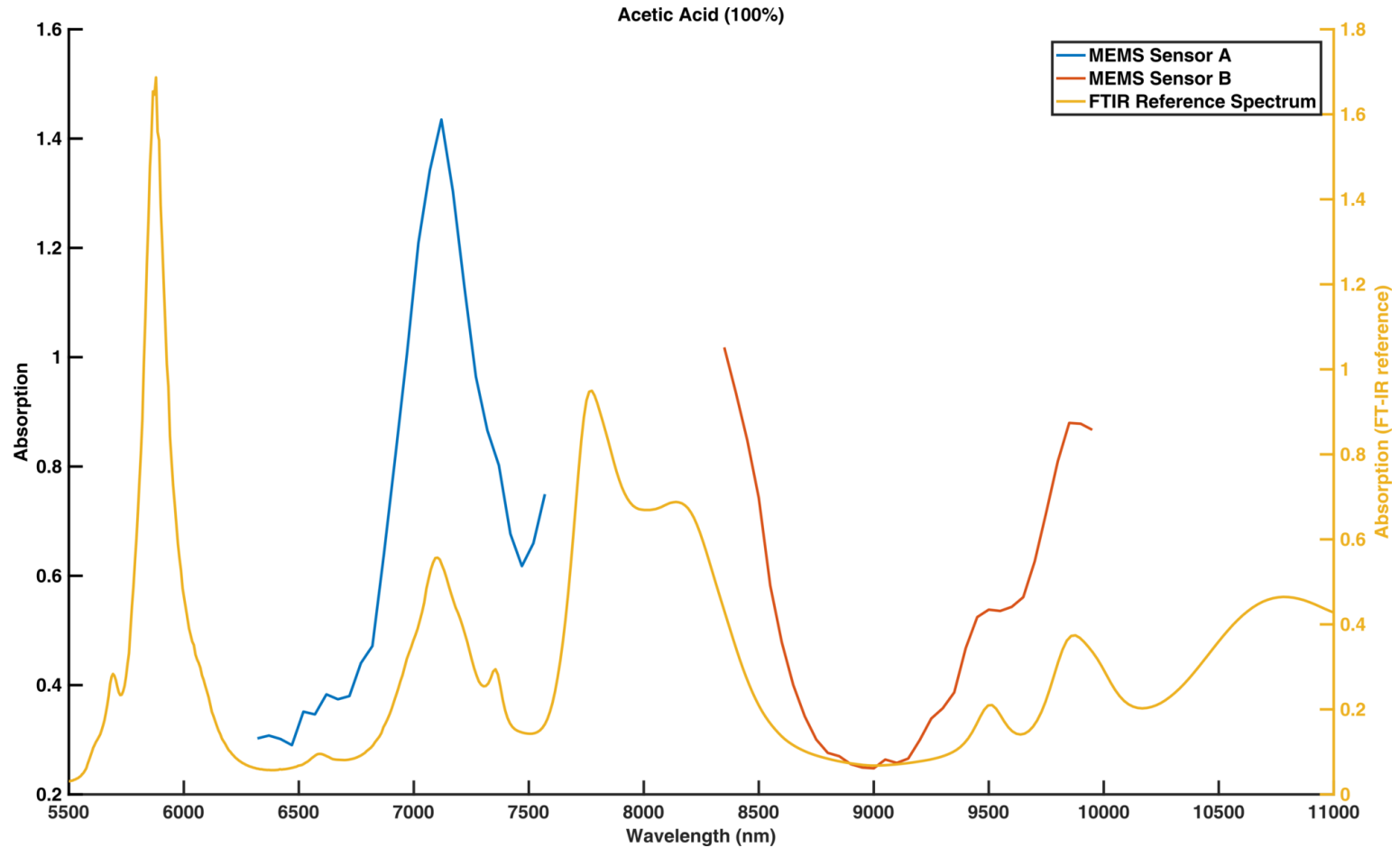
LIQUID PHASE MEASUREMENTS

MEMS VFA Sensor Details

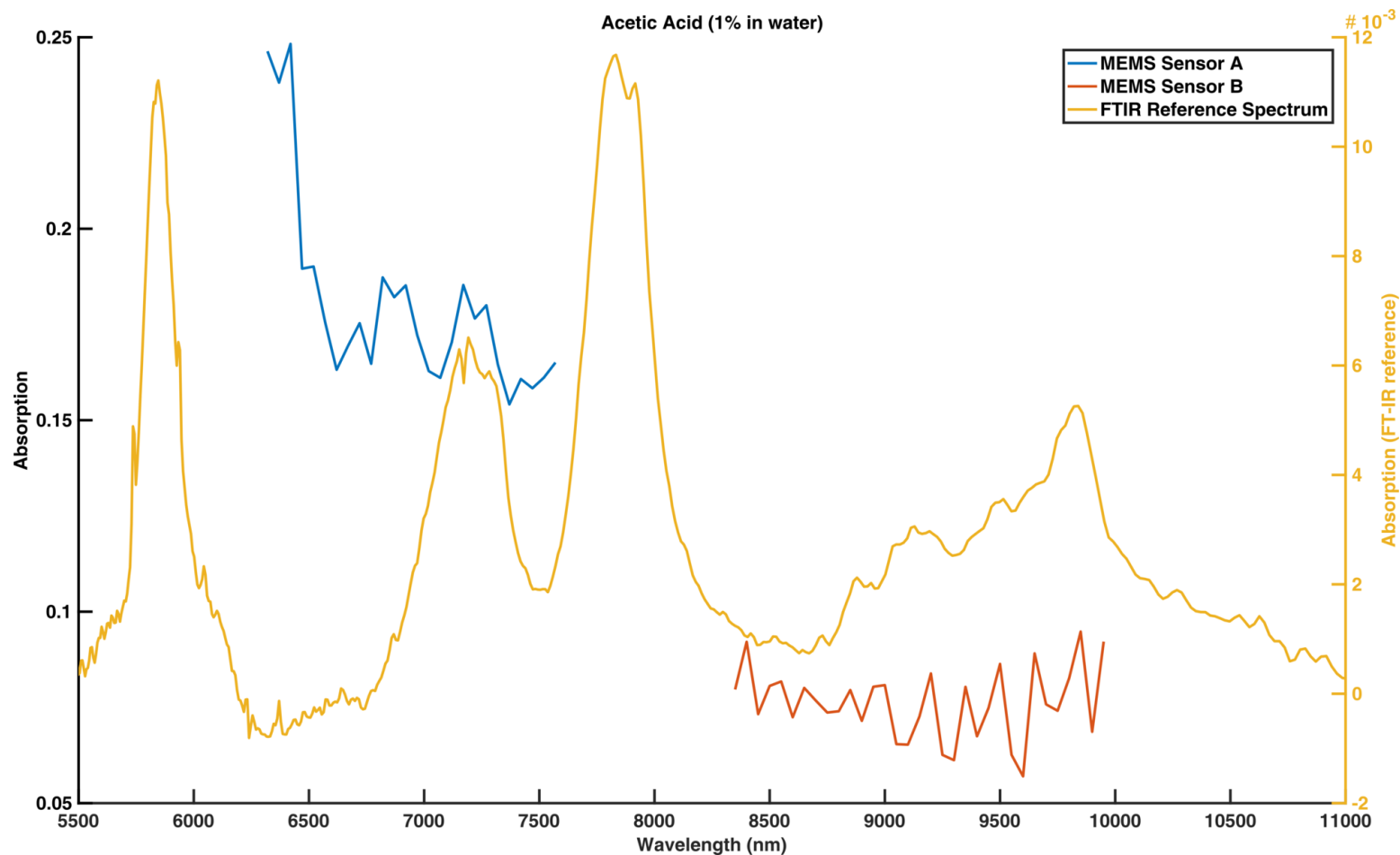
- Two sensors:
 - $6.320 - 7.570\mu\text{m}$
 - $8.350 - 9.970\mu\text{m}$
- MEMS Fabry-Perot interferometer.
- Approximately 11 minutes per scan per sensor.
- Total measurement time around 45 minutes with BG scans.
- Coupled with Diamond ATR probe from art photonics.
- ATR single measurement surface can be cleaned more easily.



Acetic acid (100%)



Acetic Acid (1% in water)



MEMS VFA Measurement Results

- Issues with SNR.
- Doubling scan time increases SNR by factor of 1.4.
- 10x SNR improvement needs 100x longer measurement.
- Challenge is low concentrations of VFA in AD process.
- Acetic acid typically highest concentration VFA.
- 0.2% acetic acid can cause inhibition [1].
- Sensor could be used in other applications with higher concentrations or other process parameters.

[1] Fachagentur Nachwachsende Rohstoffe e. V. (2012). Guide to Biogas - From Production to use. url: <http://mediathek.fnr.de/broschuren/fremdsprachige-publikationen/english-books/guide-to-biogas-from-production-to-use.html>.

BIOGAS COMPOSITION WITH MIR



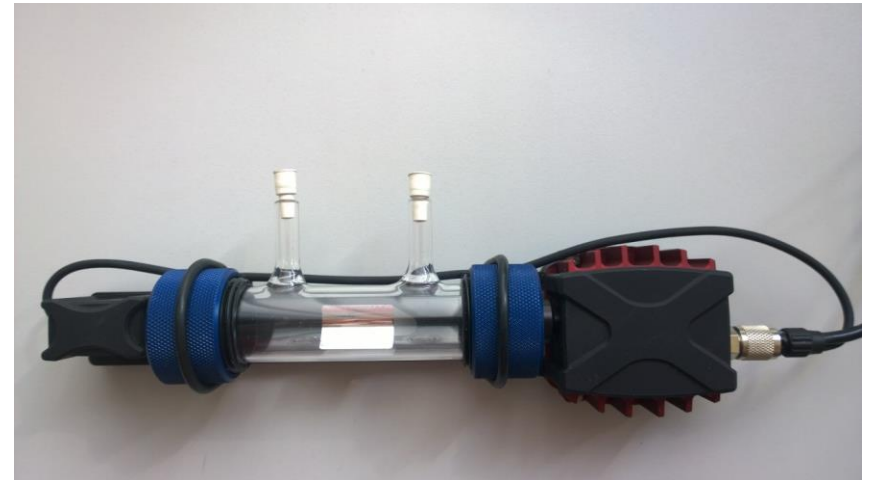
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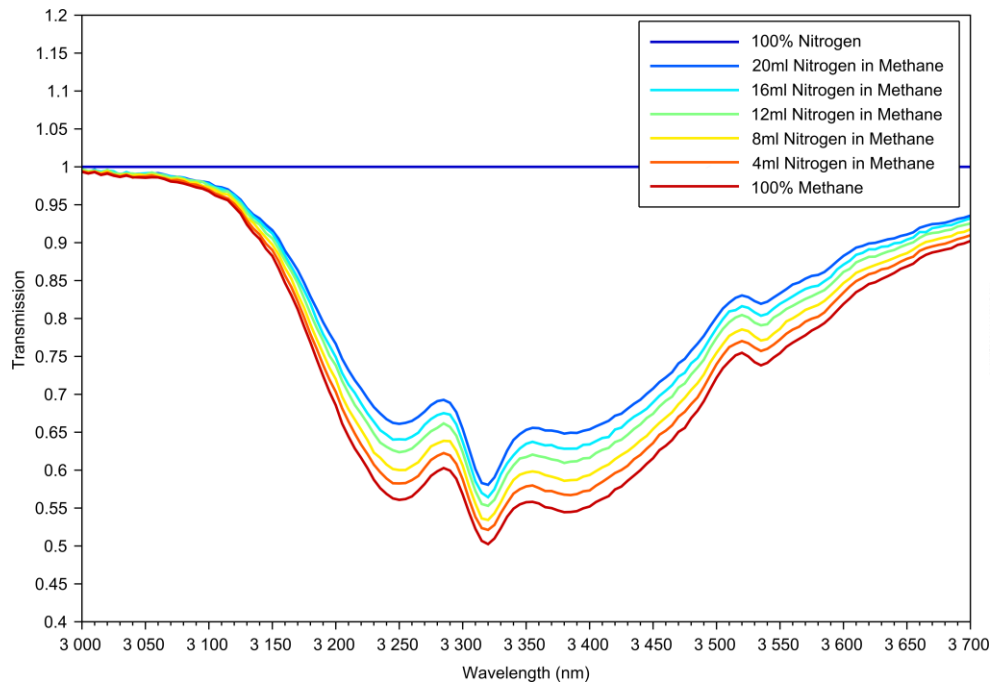
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Sensor Details

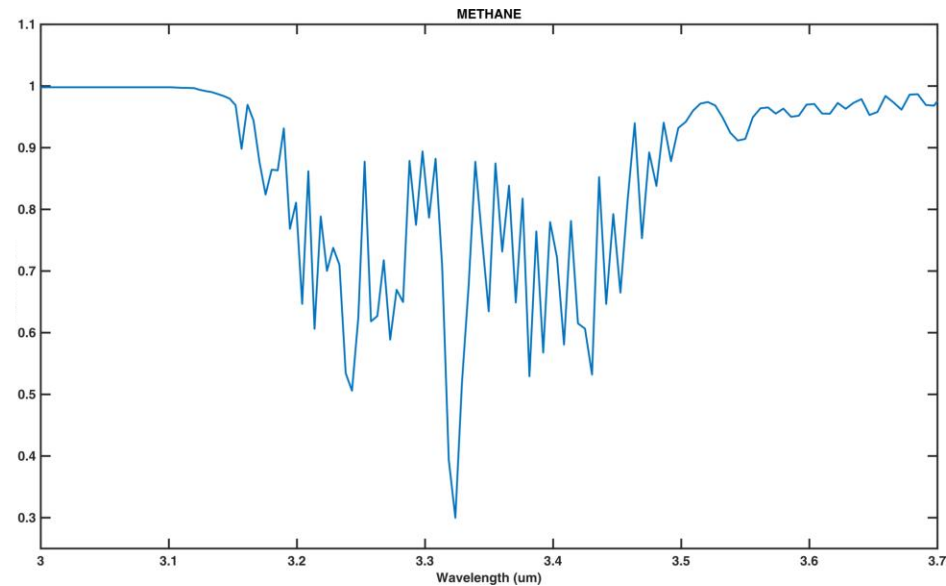
- System measures from 3.0 μ m – 3.7 μ m.
- 10cm gas measurement cell.
- Measurement of 141 wavelengths takes ~45 seconds.
- Averaged results of 10 measurements, for time of ~ 7 minutes.



Spectral Response



MEMS Sensor Results



NIST Reference Spectrum [2]

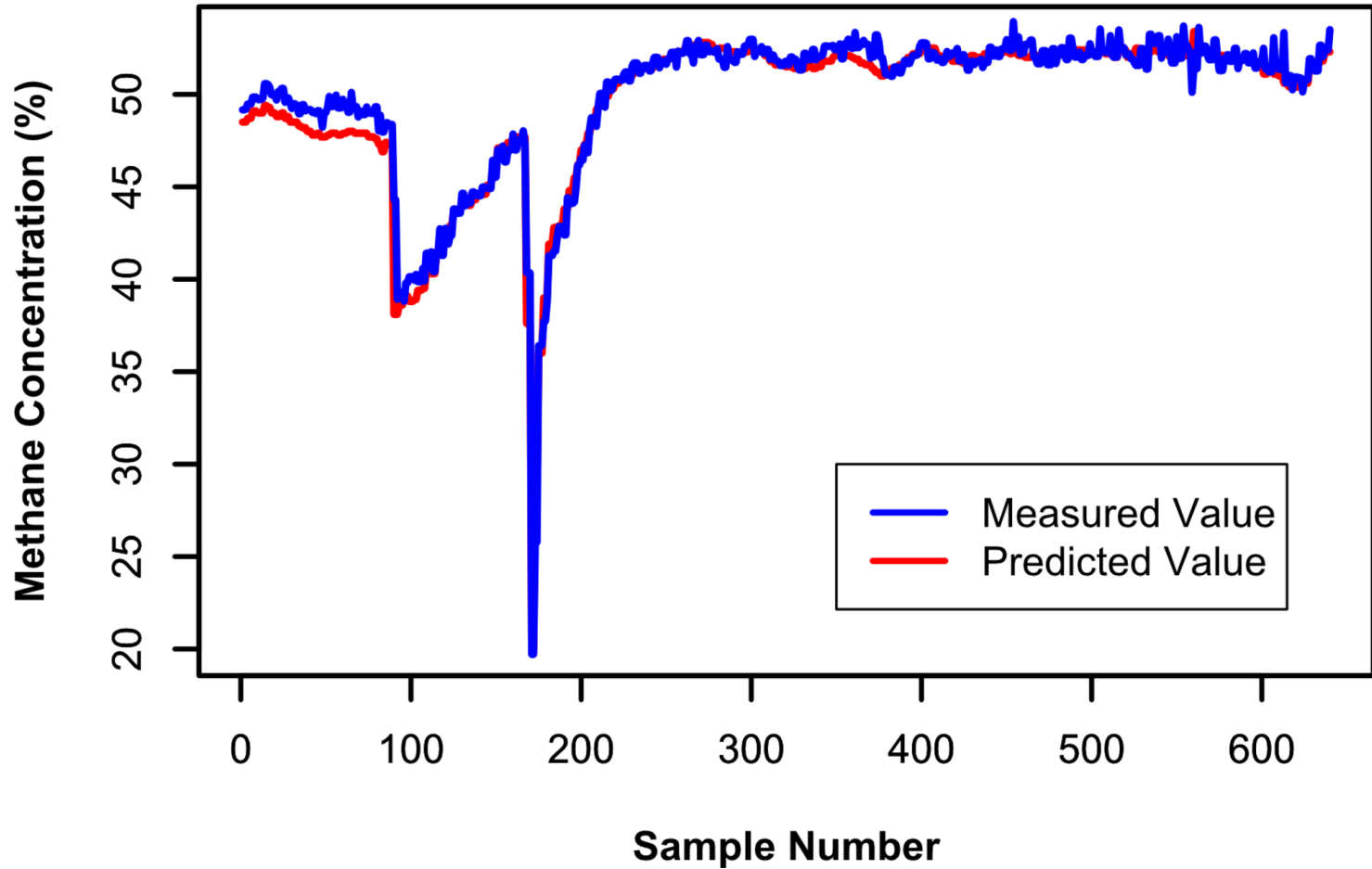
[2] NIST Mass Spec Data Center and D. S.E. Stein, "Infrared Spectra," in NIST Chemistry WebBook, NIST Standard Reference Database Number 69, P. J. Linstrom and W. G. Mallard, Eds. National Institute of Standards and Technology, Gaithersburg MD, 20899, 2016.

Pilot Scale Plant Test



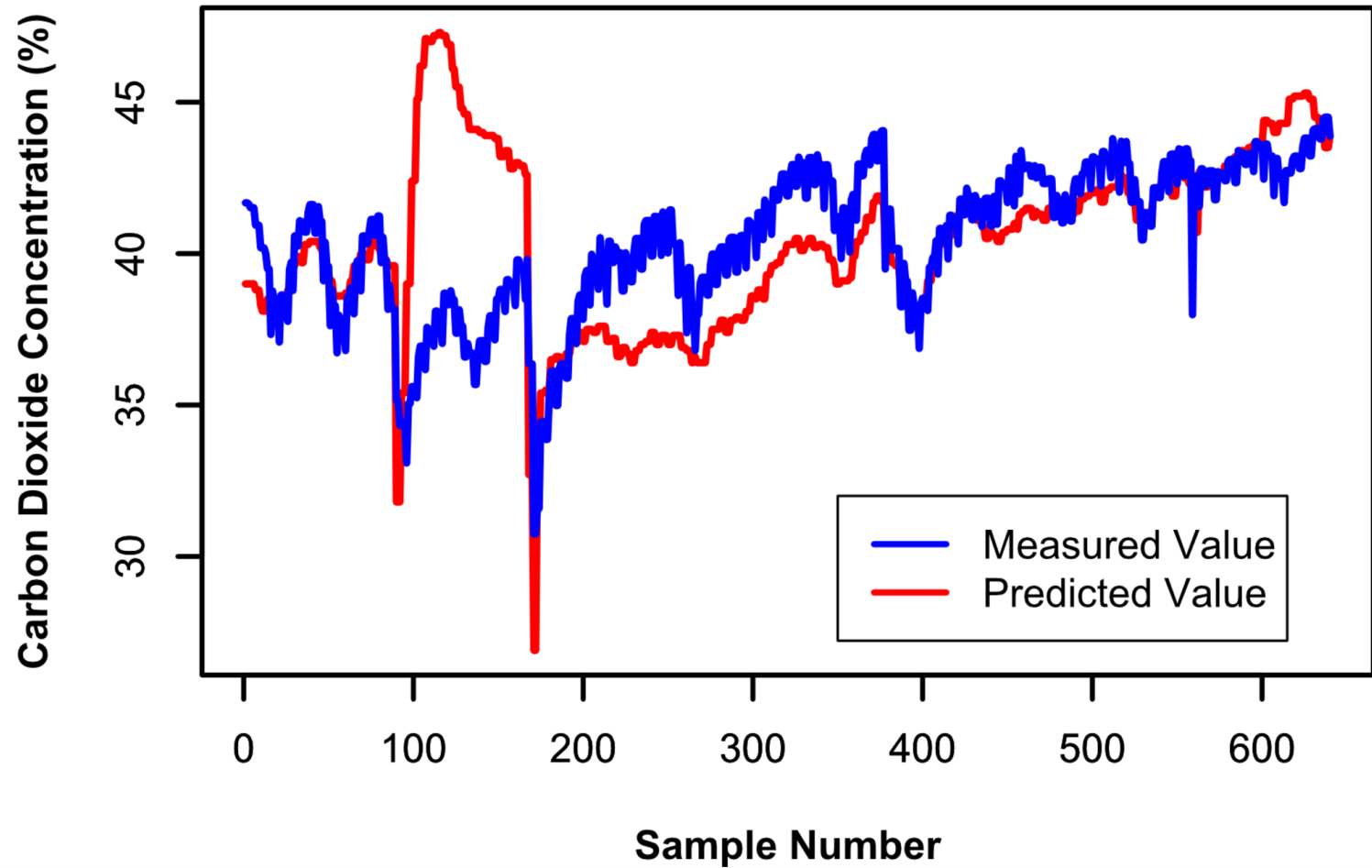
Methane

Plot of Measured VS Predicted Values



Carbon Dioxide

Plot of Measured VS Predicted Values



CONCLUSIONS

Conclusions

- Spectroscopy offers advantages over laboratory analysis.
- Liquid Phase System
 - Works well with diamond ATR.
 - Sensitivity not high enough for measuring VFAs in AD process.
 - Alternative applications would be interesting.
- Gas measurement system
 - Better results, more suited for AD process.
 - Measured methane with 0.824% RMSEP.
 - Carbon dioxide measurements with 2.681% RMSEP.



Thanks for listening



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