DOE BIOENERGY TECHNOLOGIES OFFICE (BETO) 2023 PROJECT PEER REVIEW

"AN ADVANCED PRETREATMENT/ANAEROBIC DIGESTION (APAD) TECHNOLOGY FOR INCREASED CONVERSION OF SEWAGE SLUDGE TO BIO-NATURAL GAS IN SMALL-SCALE WASTEWATER PLANTS OF LESS THAN FIVE DRY TON SEWAGE SLUDGE PER DAY"

4/6 2023
ORGANIC WASTE

Principal Investigator: Birgitte K. Ahring, *Ph.D.*

Washington State University

<u>Partners:</u>

- Washington State University (lead) *(Birgitte K. Ahring)*
- Pacific Northwest National Lab *(Peter Valdez)*
- CleanVantage LLC *(Richard Garrison)*
- City of Walla Walla *(Frank Nicholson)*
- Jacobs Engineering *(Willy Breshear)*

PROJECT OVERVIEW

Project in Budget Period 3

***Problem*:**

Energy production from sewage sludge especially at small-scale using Anaerobic Digestion (AD) typically do not produce significant value with the current available AD technology due to low Carbon Conversion Efficiency (CCE) (generally less than 50%).

We propose a novel concept, which will overcome this problem and produce significant more energy in the form of Renewable Natural gas (RNG)

Project goals:

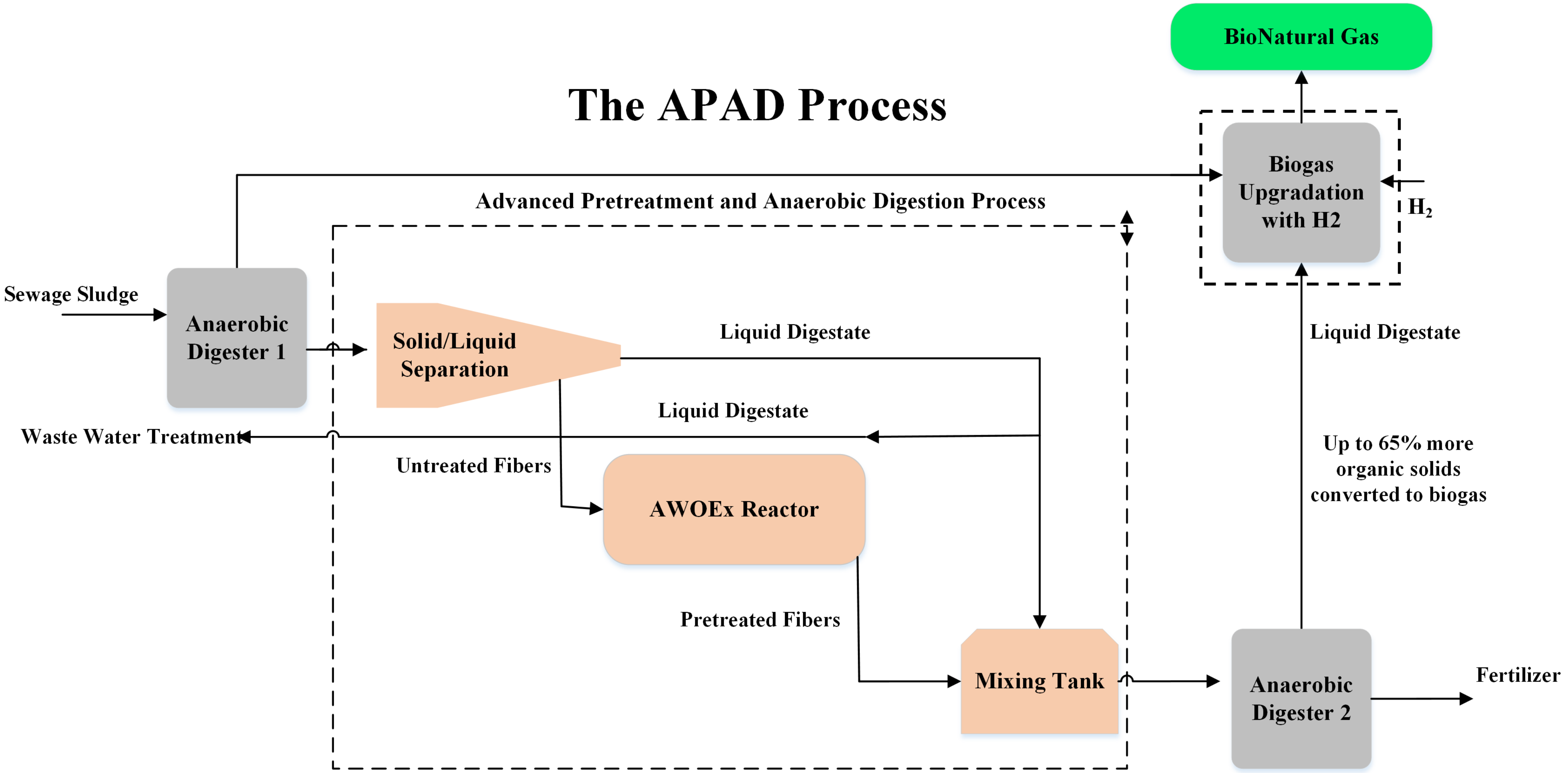
1. *Significantly increase the CCE of AD with 40% end of BP2 and 50% increase end of BP3*
2. *Upgrade biogas (60% CH_4/40% CO_2) to RNG. Converting 80% of CO_2 with H_2 to more CH_4 end of BP2 and 95% of CO_2 end of BP3*

Project goals fully aligned with DOE EERE's goals of FOA 2019 under Area of Interest (AOI) 9 - Rethinking AD:

- Improve carbon conversion efficiency by at least 50%; **and/or**
- Reduce disposal costs of the wet-waste feedstock streams in question by 25% or more
- *The project further includes the specific interest area of conversion of CO_2 with hydrogen from electrolysis of water*

We met all our targets in Budget Period (BP) 2

APPROACH



The dashed line boxes are the innovative steps of the APAD Process

APPROACH

Advanced Wet Oxidation & Steam Explosion Pretreatment (AWOEx)

AWOEx was originally developed for pretreating lignocellulosic biomass materials for producing biofuels

AWOEx scaled up with manure/straw: AD-Booster (3 dry ton/hour), Ribe Biogas Denmark (40% increased production CH_4 production)

This is the first test of AWOEx on sewage sludge

Oxygen

Biomass

Dewatering

Wet Oxidation

Process Parameters:
Temp: *170 – 210 °C*
Time: *20-30 min*
Oxygen *< 10 bar*

Flash valve

Flash Tank

Pretreated Biomass

APPROACH

Advantages of pretreating remaining solids after first anaerobic digestion (AD1)

- Reduces the volume of biomass to be pretreated, which lower cost of pretreatment
- Performing the pretreatment only on the recalcitrant portion of the sludge not converted in AD1
- The second anaerobic digestion (AD2) reactor can operate at low retention time at high solids loading due to changed rheology of the pretreated sludge
- Economically sound and efficient (uses maximum 18% of extra energy produced)

An energy efficient setup for improved carbon conversion efficiency of Anaerobic Digestion

APPROACH

Process Workflow

AD 1 of raw sewage sludge → Concentration of digested sewage sludge **Task 2** → AWOex pretreatment **Task 3** → AD 2 of pretreated digested sludge **Task 4** → Upgrading of biogas to pure CH4 **Task 5** → Pilot Testing **Task 6** (BP 3)

TEA & LCA **Task 7**

Budget Period 2 (BP 2):

- Examining and optimizing each process stepwise separately in the laboratory starting with concentration of sludge after AD 1. Development of a mass-, and energy-balance for the optimized process

Budget Period 3 (BP 3):

- Testing and optimization of the integrated process in small pilot scale corresponding to 11 Gallons AD 1 sludge/day
- Verification of the final mass-, and energy-balance

Optimizing each step of the APAD individually in BP2 and all integrated in small pilot in BP 3

APPROACH

Optimizing AWOEx of Sewage sludge

- We used **JMP Pro statistical software** for optimization of AWOEx pretreatment

- The effect of the pretreatment conditions (20) were evaluated by Anaerobic digestion of the material in duplicate small bioreactors

- **Parameters tested**:
  – Temperature range 165-200 °C
  – Oxygen dose (based on organic input (VS)) between 1-10 %
  – Residence times between 15 and 45 minutes

Optimal pretreatment conditions determined for both mesophilic and thermophilic anaerobic digestion

APPROACH

Collaborations and Diversity activities

- **Collaborations:**
  - APAD Partners meets every 2 month for discussing progress and solving problems
  - Quarterly meetings have been held between our BETO team members and the project team
  - Yearly whole day meeting with all APAD partners
  - Collaboration with NREL and APAD team lead by City of Walla Walla through DOE's Waste-To-Energy Technical Asistance for modernization of the biosolids operations

- **Diversity, equity, and inclusion activities:**
  - APAD team collaborates with the local TRIO Program* by providing educational support for students from disadvantaged backgrounds through laboratory sessions and workshops to encourage undergraduate underrepresented students (over 40 students) to pursue careers in STEM
  - As part of the APAD project, we have further employed 3 BSc students from the TRIO program as laboratory assistants for APAD research staff

* The Federal TRIO Programs (TRIO) are Federal outreach and student services programs designed to identify and provide services for individuals from disadvantaged backgrounds

Project Risks and Proposed Abatements

Project Risk	Project Abatement
Concentration of AD digested sewage sludge to 25% or more is found to be difficult	We will solve this by using other flocculants and concentration methods
AWOEx pretreatment does not produce the CCE's as anticipated	We will use other oxidants than oxygen to enhance the effect
The CO_2 conversion with H_2 to CH_4 do not produce the expected reductions in CO_2	We will operated the methanogenic fermentation in two steps after each other to increase the overall efficiency

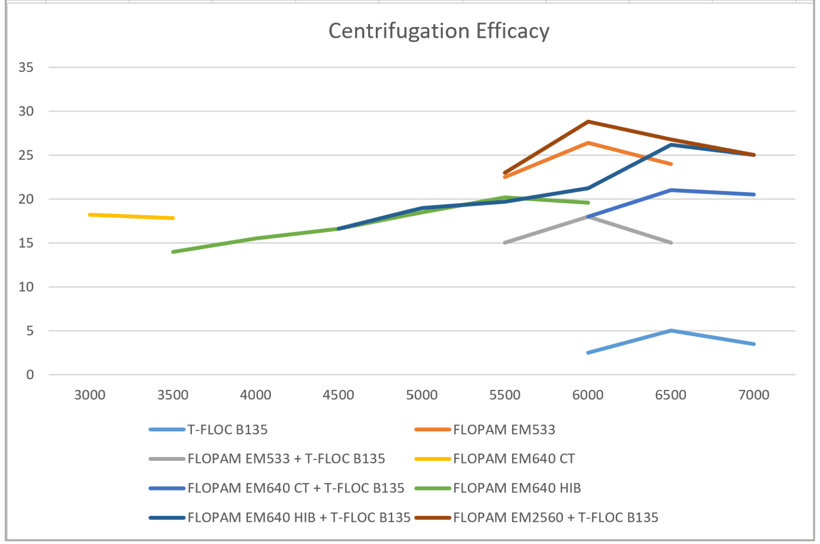
Until now there has been no need for abatements

Results from concentration of digested sludge from AD 1 using decanter-centrifugation (Task 2)

- We collaborated with Thatcher company for identifying optimal flocculants and received a variety of polyacrylamide flocculants (PAM) for testing
- Several of these performed better with 100 ppm of coagulation agent (Tfloc-B135)
- Flopam EM2560 (200 ppm) with Tflock-B135 (100 ppm) showed the best performance

We found a useful flocculant for production of over 25% TS from the 2.2% digested sewage sludge from AD 1.

The positive results of adding a coagulation besides polyacrylamide could improve concentration of sewage sludge.



PROGRESS AND OUTCOMES

Cost-Benefit Analysis of Different Dewatering Equipment

Make	Model	Type	Input %TS	Input Capacity	TS Capacity	Output %TS
Amcon	ES-353 (2ea)	Screw Press (stage 1)	2.20%	130 gpm	250 lbs/h	18.00%
Alfa Laval	Aldec G3-75	Centrifuge (stage 1)	1.79%	100 gpm	186 lbs/h	20.00%
Elode	EODS-2000	EM Osmosis (stage 2)	13.00%	2,400 lbs/h	312 lbs/h	40.00%

Make	Model	$ Capital	$ Installation	$ Maint/yr	kW Power	$ Power/yr	$ Stage 1/yr	$ Stage 2/yr	Total Cost/yr
Amcon	ES-353 (2ea)	$375,000	$70,000	$9,000	30 kW	$7,188	$38,438	$0	$38,438
Alfa Laval	Aldec G3-75	$350,000	$80,000	$10,000	30 kW	$7,188	$38,688	$0	$38,688
Elode	EODS-2000	$1,250,000	$50,000	$75,000	120 kW	$28,754	$38,438	$168,754	$207,192
		Costs are annualized over 20 years							

✓AlfaLaval, AMCON, and ELODE tested AD 1 Sludge from Walla Walla WWTF in their laboratory using their standard flocculent

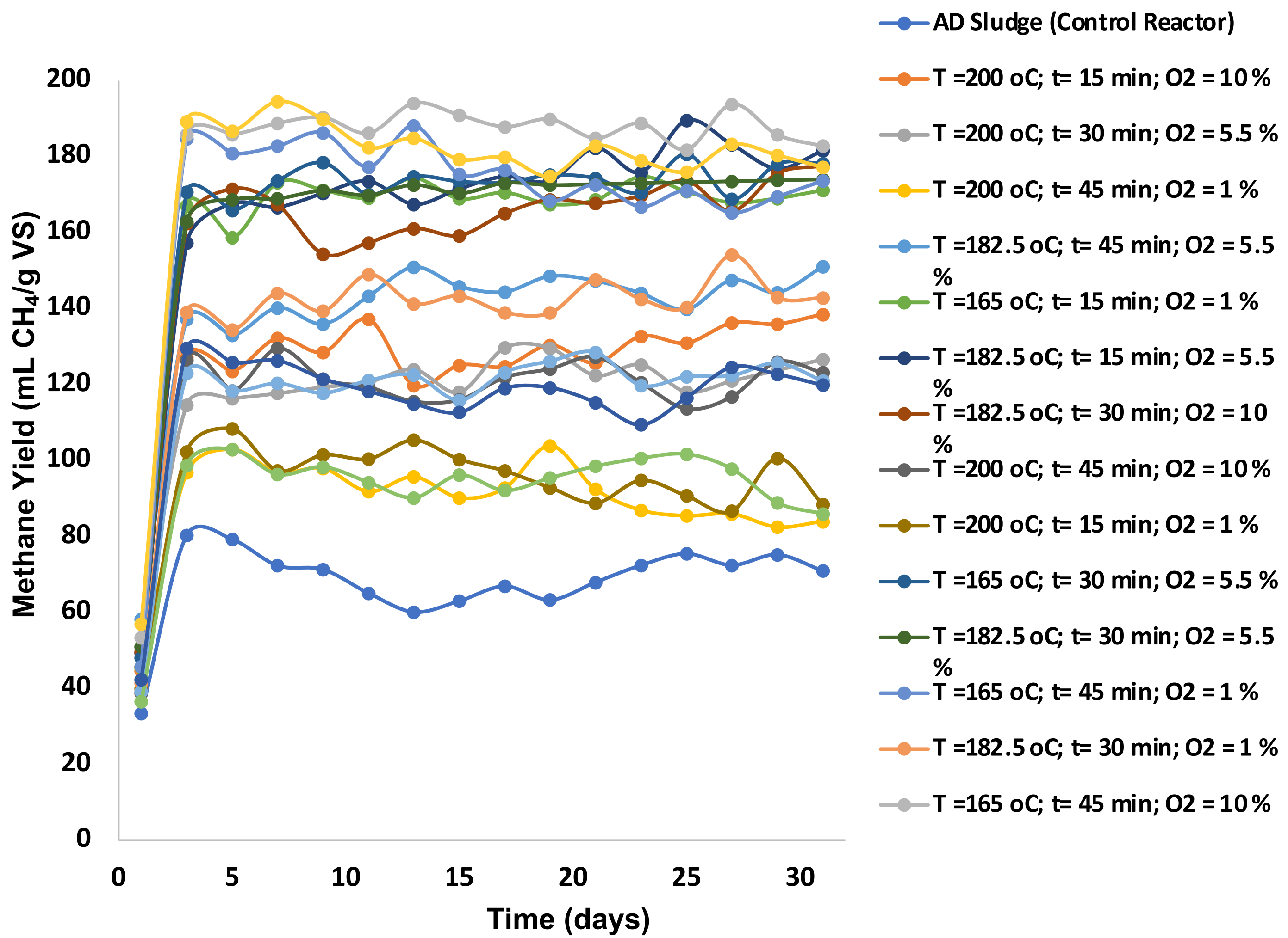
✓Besides providing the data from the testing, each company also provided cost and power consumption data

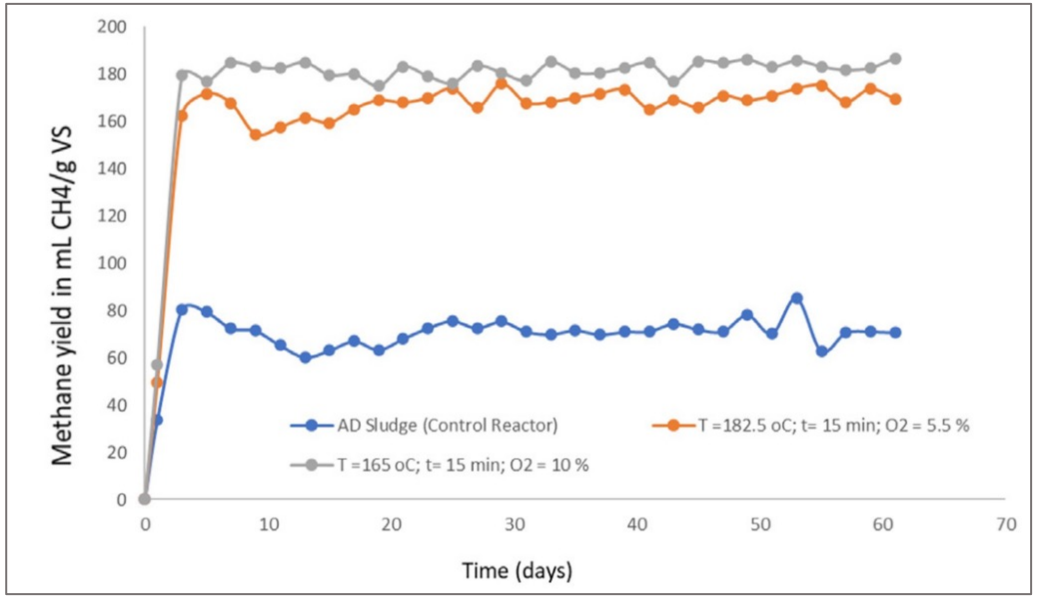
✓Analysis of the data shows that screw press and decanter centrifuge are directly comparable in performance and cost, while Elode using a 2 step approach concentrate to far higher concentration but with a prohibitive 5.4 times higher cost

✓An analysis of the extra heating cost for pretreatment compared to the cost of concentration to lower TS % showed no advantages of concentration to 25% compared to 20% TS

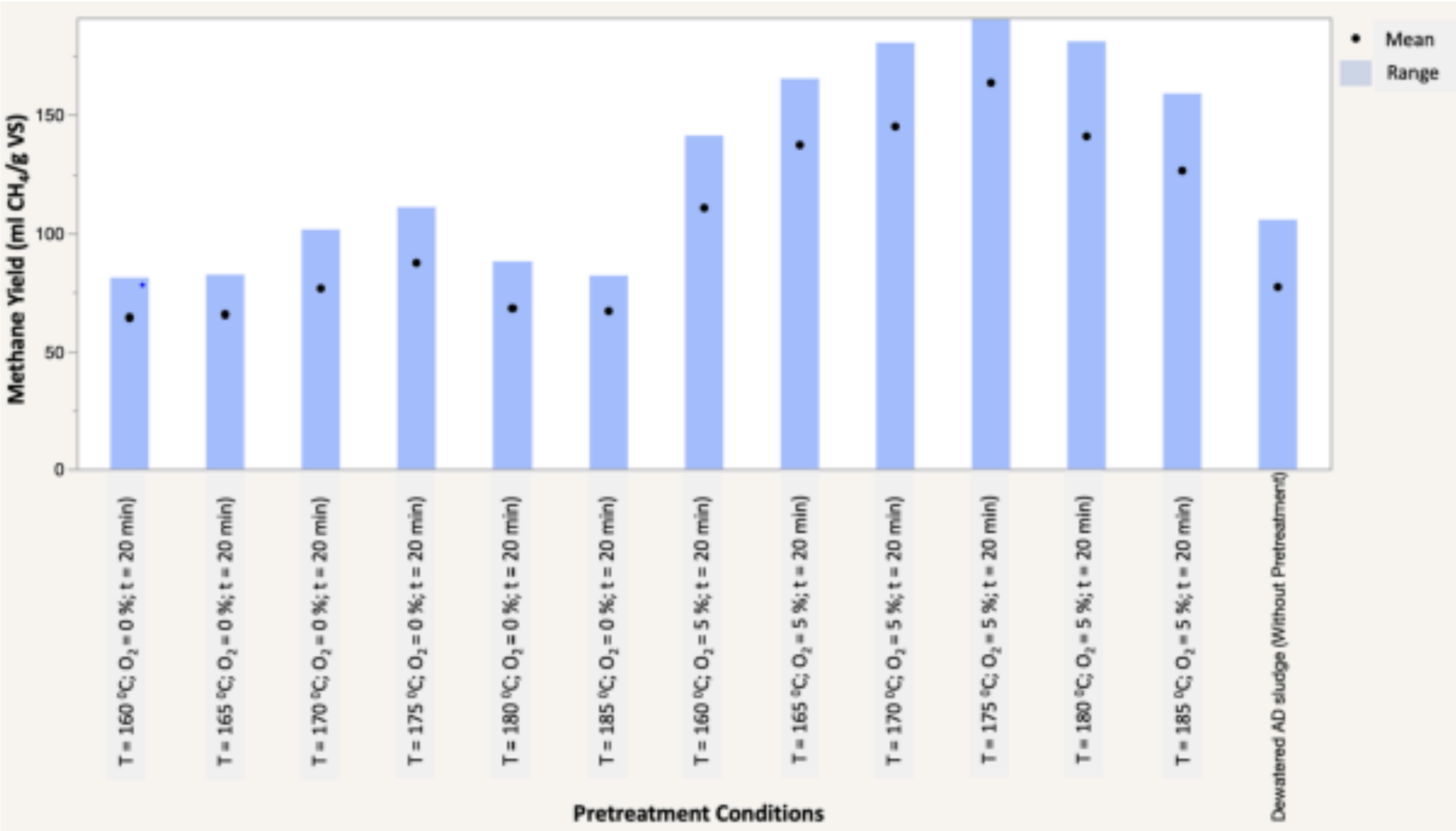
PROGRESS AND OUTCOMES

Methane Yield



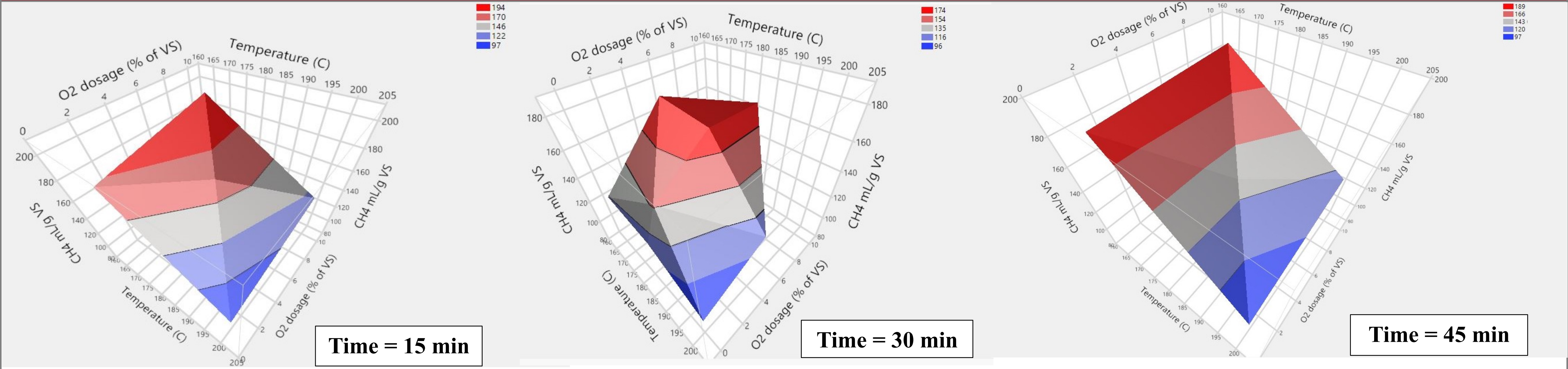


Finding the optimum pretreatment for secondary thermophilic conditions (AD2)



Comparing the effect of the two tested pretreatment variables on methane yield: DSS pretreated with and without oxygen at temperatures ranging from 160 to 185 °C. Range of the methane yield is plotted with mean marked for each pretreatment conditions (bar)

PROGRESS AND OUTCOMES



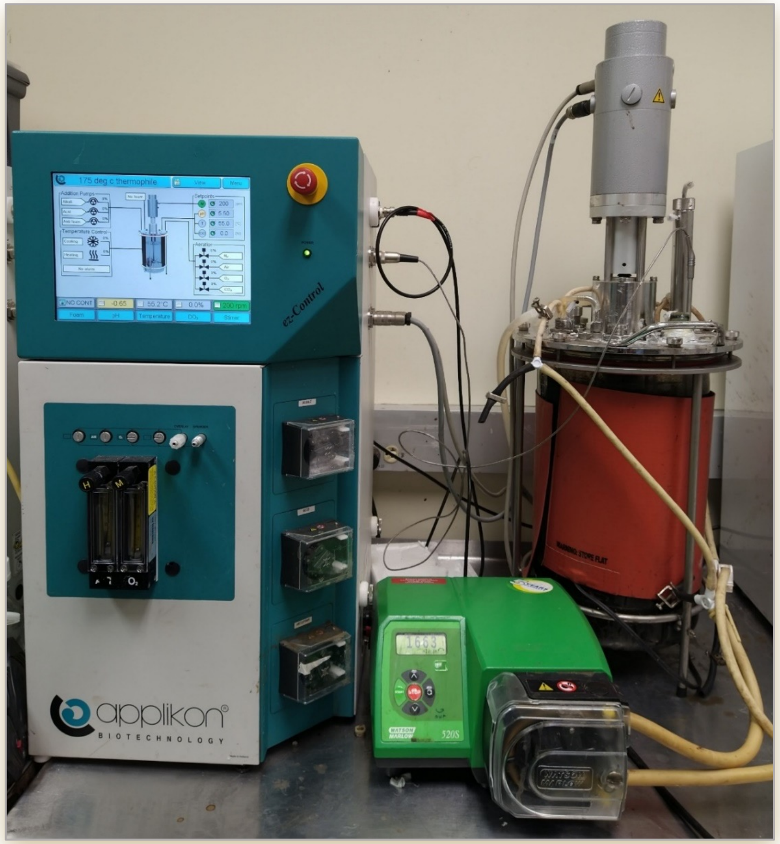
Source	LogWorth	PValue
Temperature	4.804	0.00002
O2 dosage (%)	2.509	0.00310
Residence time	0.103	0.78845

- All process parameters had influence on methane yield.
- **Temperature and O_2 dosage had significant effect on methane yield as indicated by high LogWorth value.**
- The positive effect of higher temperatures on methane yield at low oxygen dose was compensated by higher O_2 dosage at low temperatures.

Anaerobic digestion of the AWOEx pretreated sewage sludge in continuous 15 L bioreactors (Applikon) for making the data for the BP 2 verification data (Task 4)

- We did thermophilic digestion of materials pretreated under the optimal AWOEx conditions for thermophilic digestion determined by statistic design [T= 175°C; t= 20 min; O_2= 5 %]. We determined methane yield, TS and VS conversion, conversion of the major components of the sewage sludge due to the digestion (lipids, proteins, carbohydrates, cellulose, hemicellulose and lignin.
- The HRT was set at 15 days at 55^{o}C
- The main components of the sewage sludge (proteins and lipids) showed high conversion besides the small amount of lignocellulosic polymers

Results show optimal solids input is between 9 and 12 % for maximum methane yield (184 mL/ g VS)



15 L bioreactor (Applikon) in operation

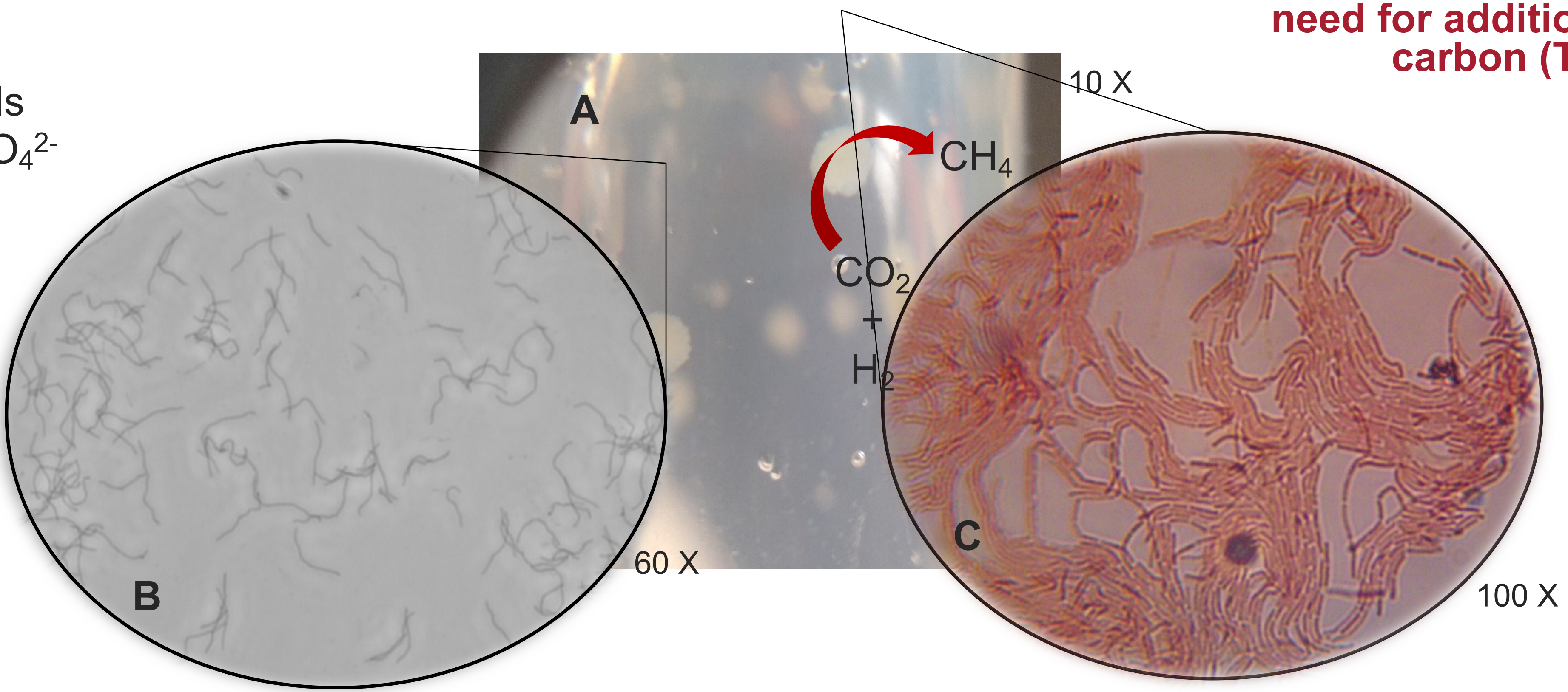
PROGRESS AND OUTCOMES

Methanothermobacter wolfeii strain BSEL

Mineral media:

NH_4Cl
Vitamins
Trace metals
HCO_3^- or HPO_4^{2-}

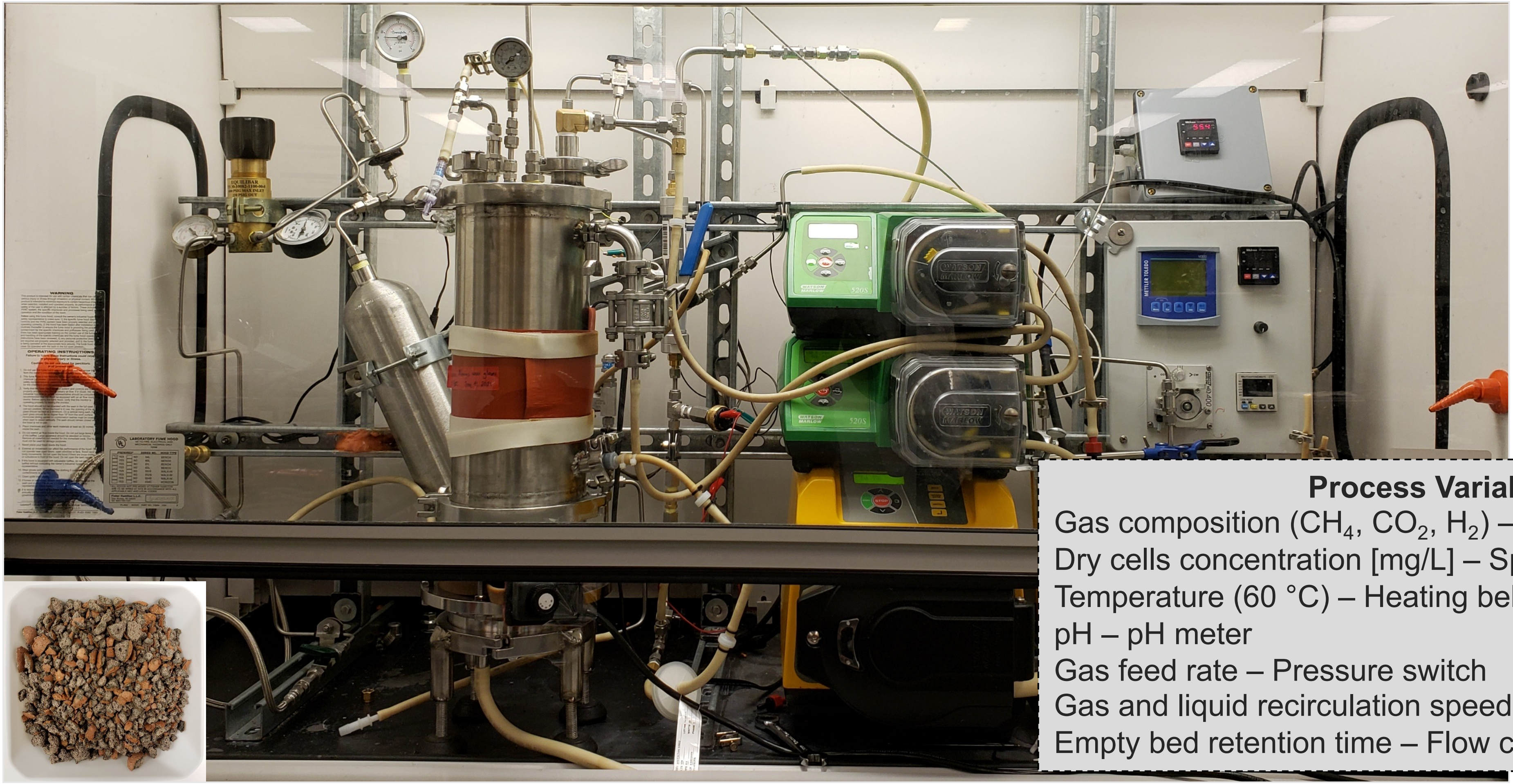
We isolated a thermophilic methanogenic strain without need for addition of organic carbon (Task 5)



Axenic culture of *Methanothermobacter sp.* **A**.- Colonies in roll tubes. **B**.- Phase contrast image. **C**.- Gram stained cells in optical microscope.

PROGRESS AND OUTCOMES

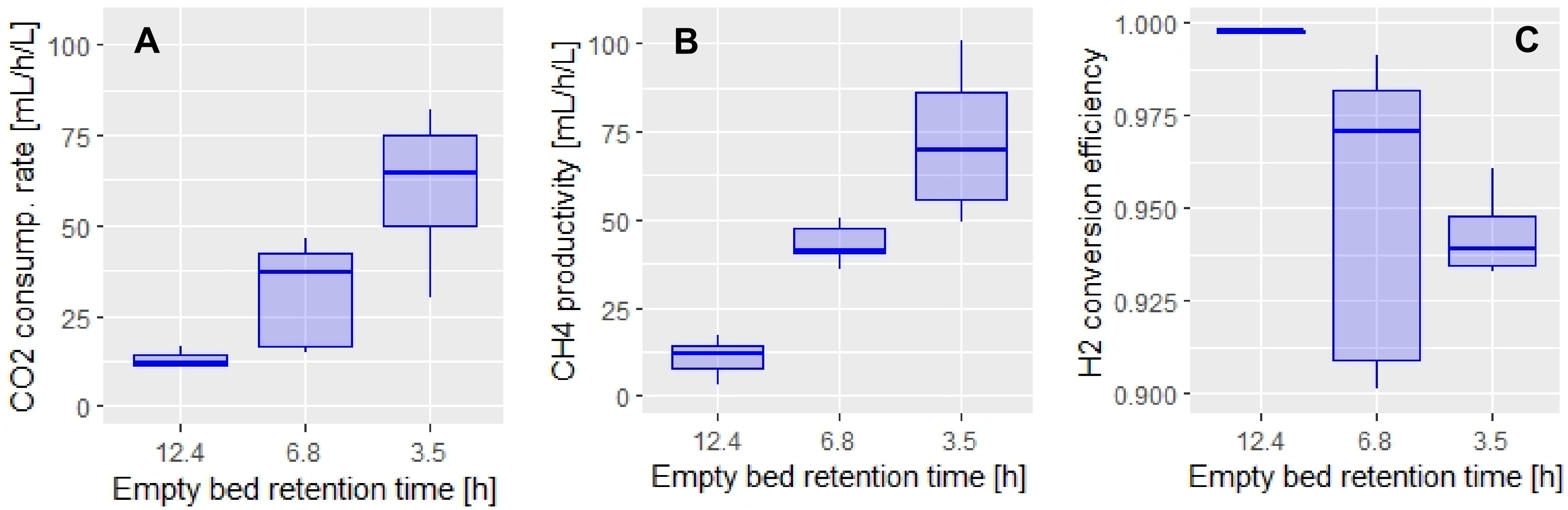
Growing *M. wolfeii* BSEL in our Biogas-to-RNG reactor system



Process Variables:

Gas composition (CH_4, CO_2, H_2) – Mass spectrometer
Dry cells concentration [mg/L] – Spectrophotometer
Temperature (60 °C) – Heating belt and control loop
pH – pH meter
Gas feed rate – Pressure switch
Gas and liquid recirculation speeds – Peristaltic pumps
Empty bed retention time – Flow control valve

Optimal conditions for maximum conversion of CO_2



Effect of EBRT on CO_2 consumption rate (A), CH_4 productivity (B), and H_2 conversion efficiency (C) during continuous operation of the trickle-bed bioreactor. Gas volumes reported at standard conditions (1 atm; 273.15 K)

Biogas upgrading milestones

- ✓ Microbial isolation and characterization
- ✓ Reactor construction (Enhanced mass transport)
- ✓ Determination of optimal flows (Reduction of CO_2 to less than 5 %)

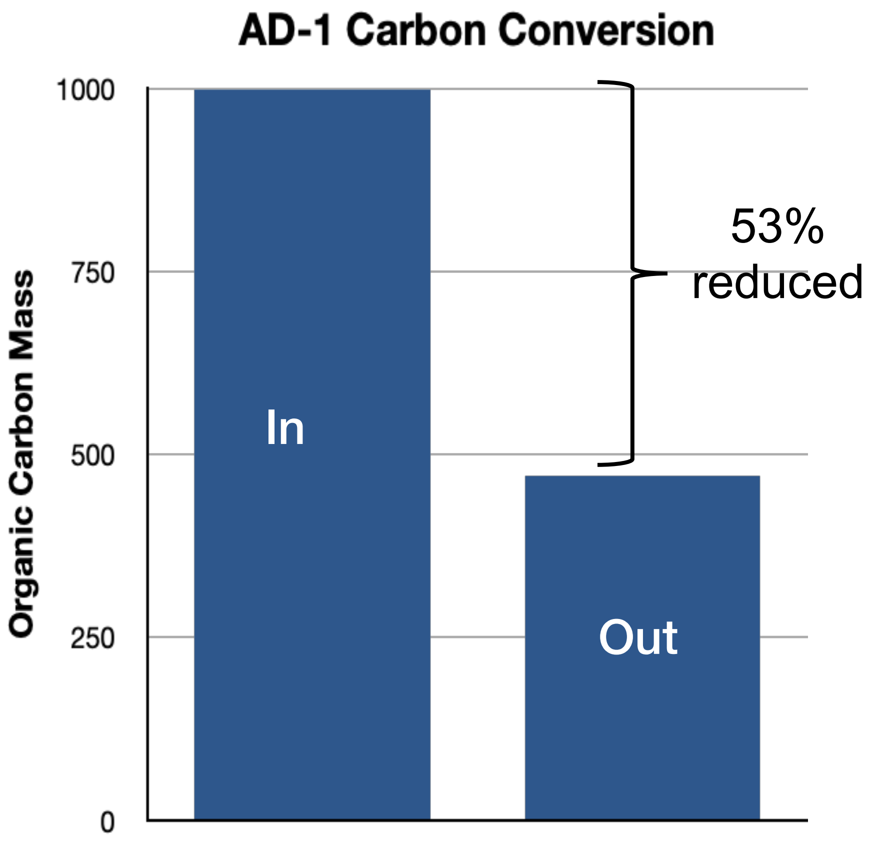
Biogas upgrading milestones

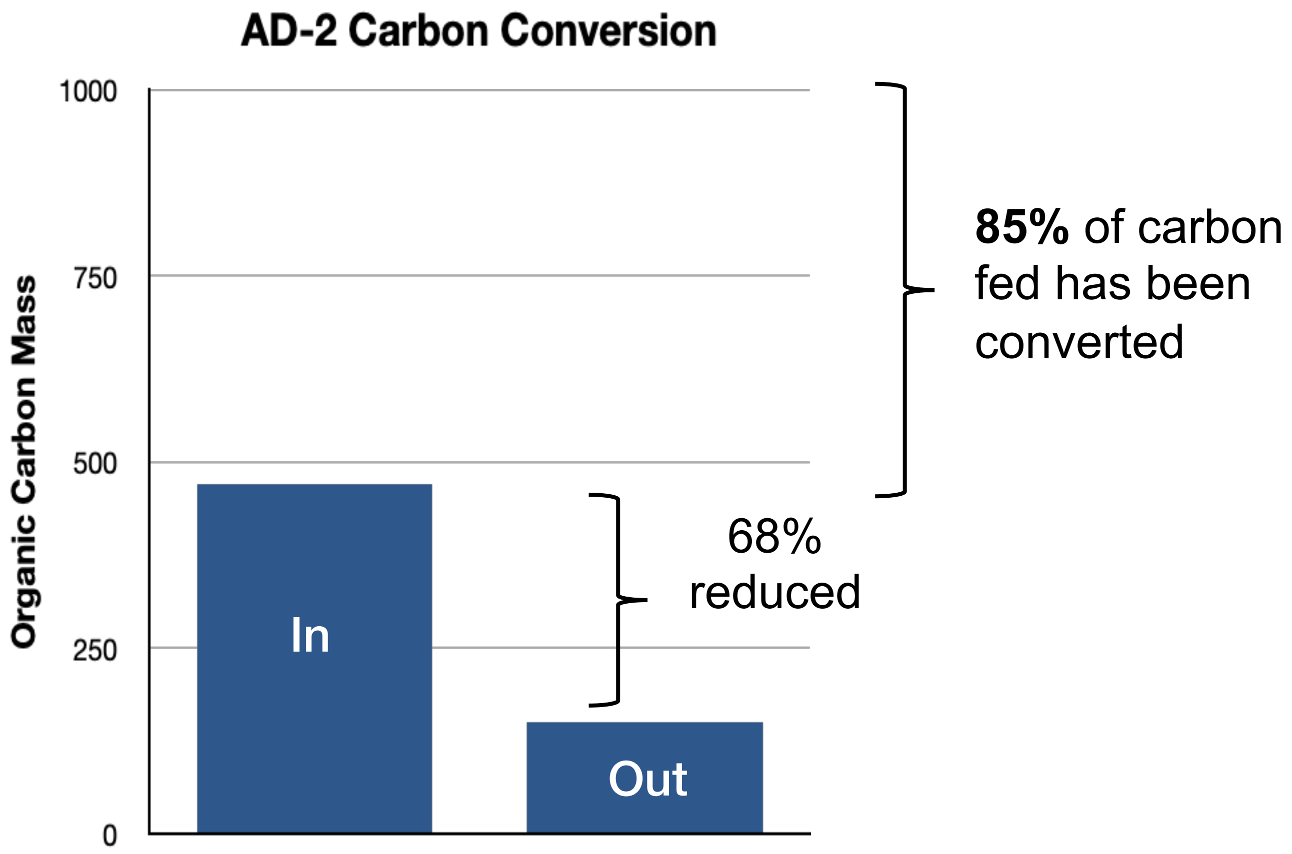
Milestone	Final Goal	Achieved in the Project
CO_2 conversion efficiency	87.5%	**95%**

PROGRESS AND OUTCOMES

Volatile solids (organic carbon) conversion

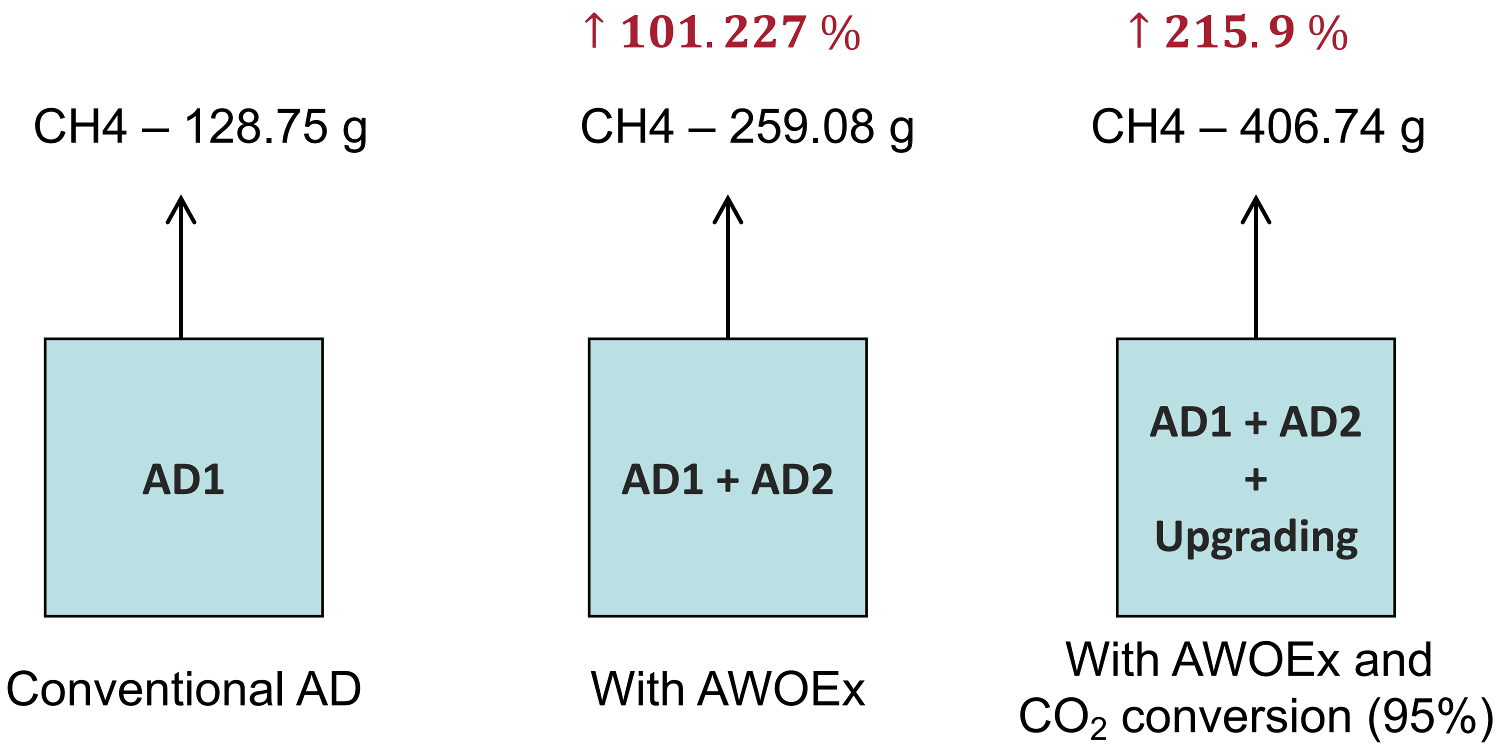
60.4% increase in CCE





PROGRESS AND OUTCOMES

Comparison to Conventional AD



We have shown that APAD can improve the CCE of 60% and the methane yield of over 3 times

Birgitte K. Ahring Ph.D.
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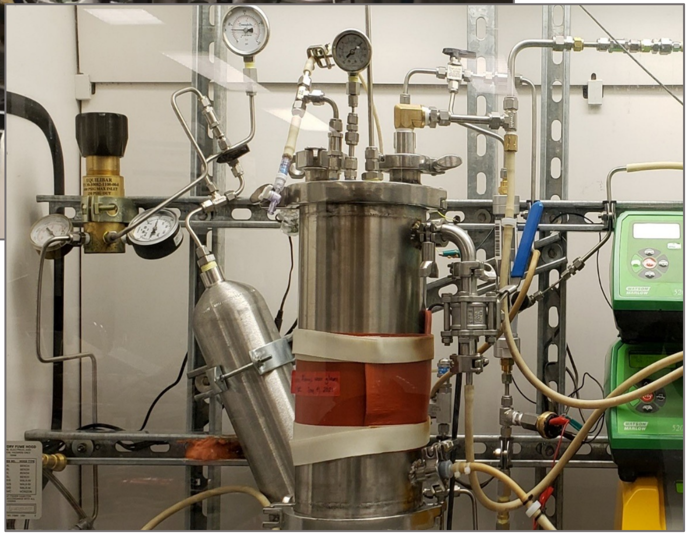
Pilot Plant Testing (BP3)

CleanVantage℠

	Design	Fabrication	Testing
Fermentation Vessels	NA	January	February 2023
Biogas Collection System	January	February	March 2023
Gas Upgrading System	December	February	March 2023

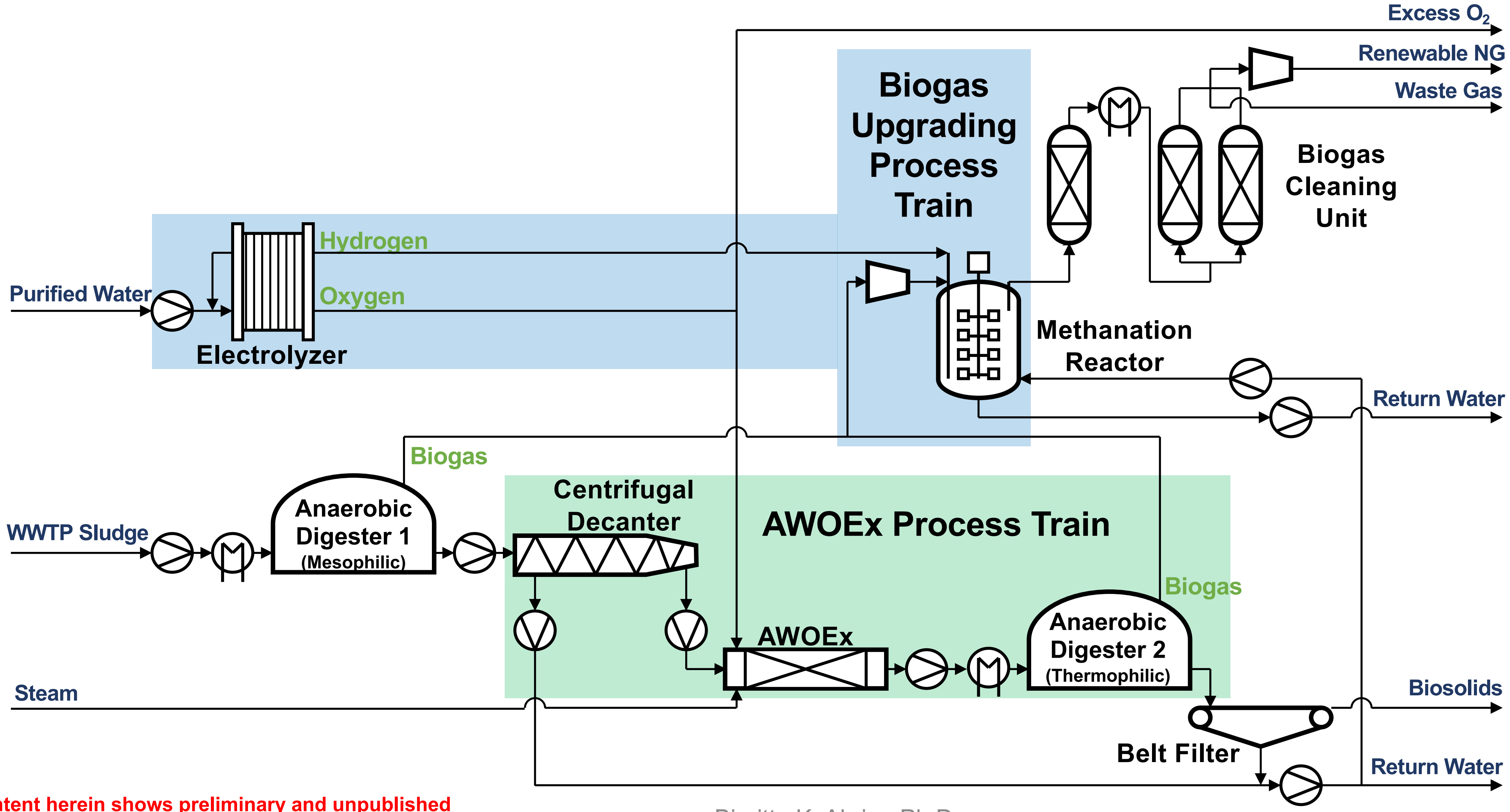
Fermentation vessels



Gas upgrading device

PROGRESS AND OUTCOMES

Process Flow Diagram for TEA (Task 7)



The content herein shows preliminary and unpublished results that are subject to change

PROGRESS AND OUTCOMES

Benefits of Processing Scenarios

Scenario	Anaerobic Digestion 1	Biogas Upgrading	AWOEx + Anaerobic Digestion 2	Biosolids Class	Biosolids Reduction (%)	Biogenic Emissions Offset (ton CO_2/y)	Levelized Cost of Sludge Treatment ($/ton dry sludge)
1	✗	✗	✗	B	-		$530
2	✓	✗	✗	B	26%	21	$600
3	✓	✓	✗	B	26%	31	$490
4	✓	✗	✓	**A**	**59%**	50	**$79**
5	✓	✓	✓	**A**	**59%**	**72**	$260

- AWOEx and Anaerobic Digestion 2 **minimize** net costs of sludge treatment.
- Complete APAD processing **maximizes** emissions offset with renewable natural gas.
- Both scenarios create **class A biosolids** with **reduced volumes** of solids to be managed.

The content herein shows preliminary and unpublished results that are subject to change

IMPACT

- The APAD project has demonstrated for the first time a way to significantly increase the CCE of sewage sludge by AD

- Implementing AWOEx and AD 2 can improve recycling and decarbonization of sewage sludge while reducing the cost of waste disposal

- Producing RNG by upgrading CO_2 from the biogas with H2 from renewable sources will further reduce the greenhouse gas emission of handling of sewage sludge

IMPACT

A Few of the Publications in the World Press

WaterWorld

New sewage treatment step improves biogas conversion

Researchers from Washington State University find that, by adding oxygen to a high temperature and high pressure environment before the anaerobic digestion process, a wastewater treatment plant could raise its biogas production by 98 percent.

Nov. 9, 2022

The Chronicle

Division of CT Publishing

WSU Develops New Biogas Process

Posted Sunday, November 6, 2022 2:36 pm

WSU is working with Richland-area clean-tech startup Clean-Vantage to help further develop and commercialize the technology, which was funded through a grant from the U.S. Department of Energy.

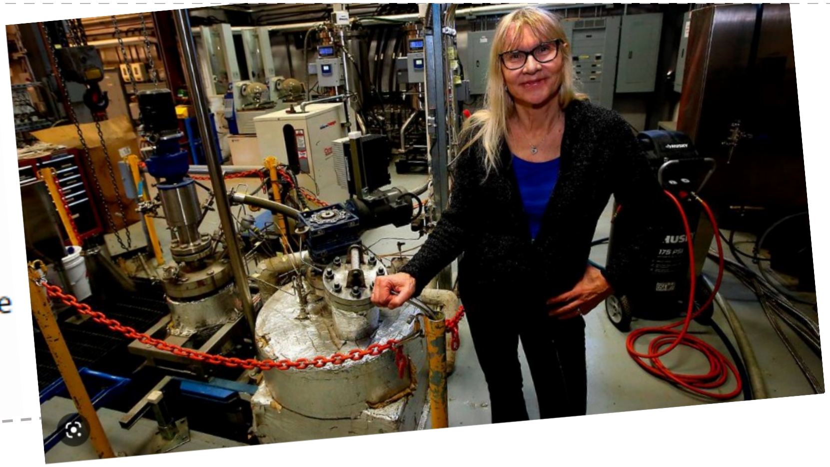
Tri-City Herald

Part of the McClatchy Media Network

WSU Researcher more efficiently turning poop into energy

BY NOVEMBER 25, 2022 AT 10:23 AM

Professor Birgitte Ahring explains how a new waste treatment process being tested at the WSU-Tri-Cities campus in Richland, WA converts more than 85% of biosolids into biogas which can be used to produce electricity or power vehicles.



IMPACT

Dissemination of Results

"We want to implement the data from the DOE project at Walla Walla WWTF in 2023/24"

***Frank Nicholson**, City of Walla Walla to Tri-city Herald November 24, 2022*

Peer-reviewed publications (3) in 2021-2022.

Journal Impact factors: (7.45-16.8)

Improved valorization of sewage sludge in the circular economy by anaerobic digestion: Impact of an innovative pretreatment technology. Nalok Dutta, Anthony T. Giduthuri, Muhammand Usman Khan, Richard Garrison, Birgitte K. Ahring . 2022. Waste Management 154,105-112. Impact factor 8.816

Enhancing methane production of anaerobic digested sewage sludge by Advanced Wet Oxidation & Steam Explosion pretreatment. Nalok Dutta, Richard Garrison, Muhammad Usman, Birgitte K. Ahring. 2022. Environmental Technology & Innovation 28, 102923-102931. Impact factor 7.45

Current status of biogas upgrading for direct biomethane use: A review. Muhammad Usman Khan., …., Birgitte K. Ahring. 2021. Renewable and Sustainable Energy Reviews 149, 111343-111355. Impact factor 16.8

IMPACT

Summary

- **Overview:** The APAD project uses AWOEx to enhance the CCE of digested sewage sludge and a biological upgrading process for converting CO2 from biogas into RNG.
- **Approach:** The major process step were optimized separately and then integrated in the laboratory/pilot to provide input for the Mass and Energy Balance, TEA, LCA etc.
- **Progress & Outcome:** Digested sewage sludge can be concentrated to 25% or more using a combination of polyacrylamid flocculent mixed with a small amount of a coagulant and standard concentration equipment.
- **Progress & Outcome:** Temperature and oxygen are of significant importance for the AWOEx pretreatment, while residence time is of lower importance. The effect of a lower temperature can be compensated by adding more oxygen and the oxygen concentration can be reduced by increasing the pretreatment temperature. The optimal parameters producing the highest CCE during secondary thermophilic digestion is 175^{o}C, 5% O_2 and 20 min retention time.
- **Impact:** AWOEx followed by a secondary AD step results **in 85% conversion of organic carbon** or an **improvement of the CCE of 60.4 %** significant higher than our end goal. It further gives over **100% more methane** than from the primary AD reactor without APAD.
- **Impact:** Conversion of the CO_2 from the primary AD step along with conversion of CO_2 from APAD will result in a final development of **219% more methane**.
- **Impact:** The project has demonstrated that pretreatment and CO_2 upgrading significantly increase the bio-energy production of sewage sludge and reduce cost of disposal of sewage sludge.

QUAD CHART OVERVIEW

Timeline
- *10/01/2019*
- *10/31/2023*

	FY22 Costed	Total Award
DOE Funding	*(10/01/2021 – 9/30/2022)*	*$2,088,146*
Project Cost Share *		*$607,070*

TRL at Project Start: 2
TRL at Project End: 5

*Only fill out if applicable.

Project Goal
The main goal of this project is to develop and demonstrate a concept for significant increased Carbon Conversion Efficiency of sewage sludge compared to conventional Anaerobic Digestion. A second goal is to develop a biological process for cost-effective conversion of CO_2 from biogas into pure methane with hydrogen addition

End of Project Milestone
End case: 50% improved Carbon conversion efficiency with conversion of 95% of the CO_2 in the biogas with hydrogen to results in a total increase of 135% CH_4 compared to conventional AD

Funding Mechanism
FOA FY 19 DE-FOA-0002029

Project Partners*
- Washington State University
- Pacific Northwest National Laboratory
- Clean-Vantage LLC
- City of Walla Walla &Jacobs Engineering

Questions?

WASHINGTON STATE UNIVERSITY

Bioproducts, Sciences, and Engineering Laboratory

Team members in front of an AD reactor at Walla Walla WWTP

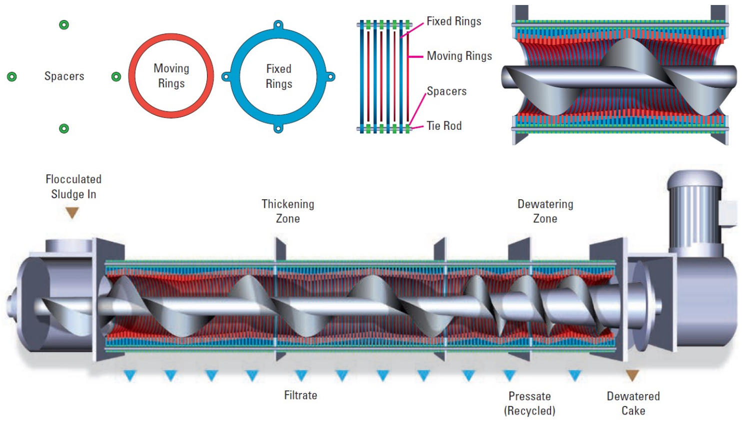
Contact Information: bka@wsu.edu

APPENDIX

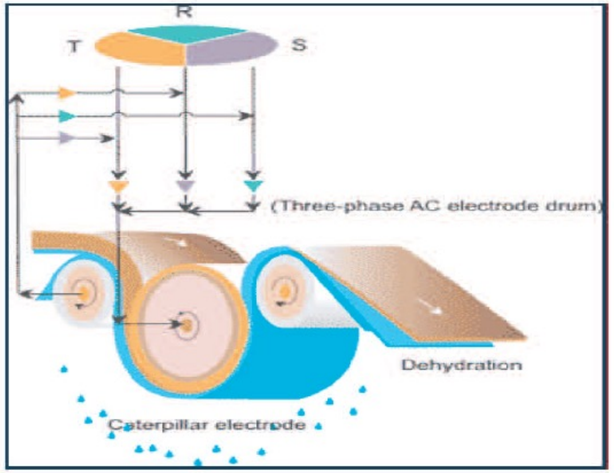
PROGRESS AND OUTCOMES

Testing of different equipment for sludge concentrating

AlfaLaval G3 Centrifuge



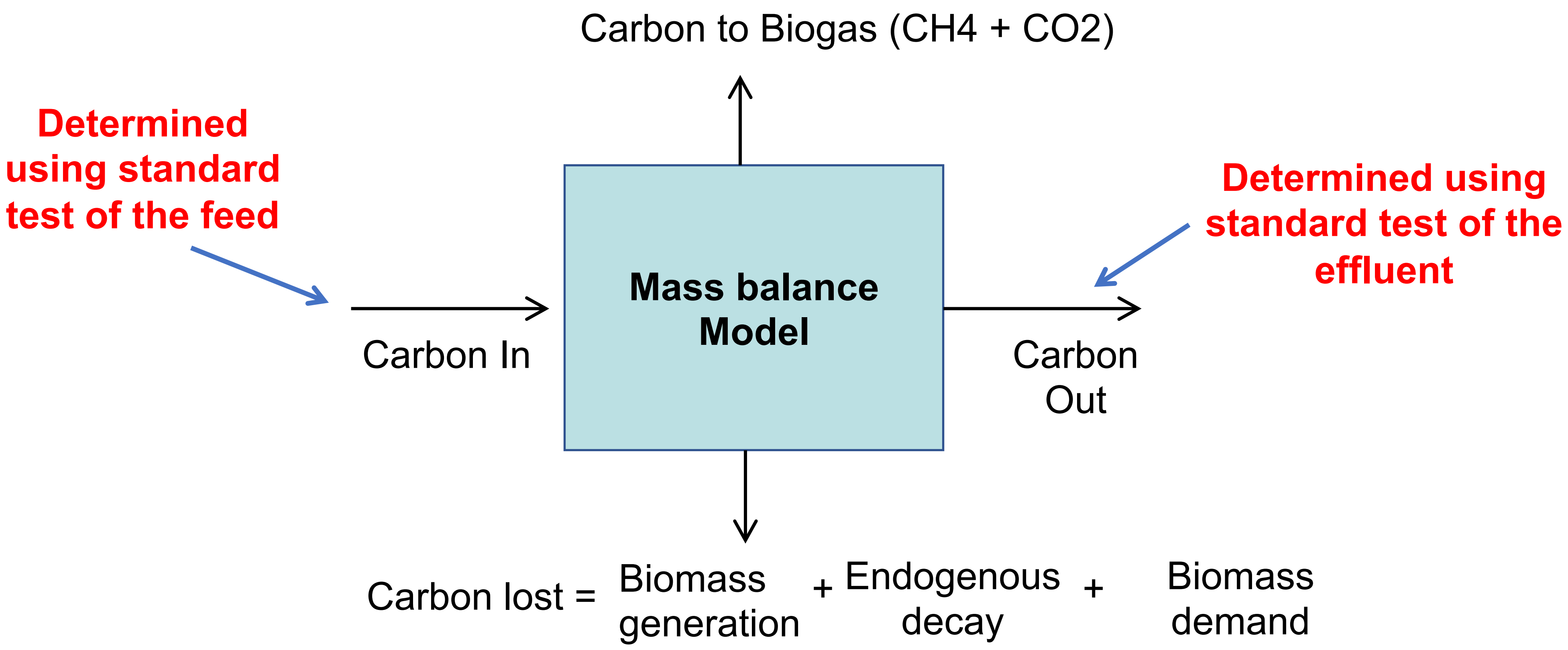
AMCON Screw Press



ELODE Electro-Osmotic Press

AlfaLaval, AMCON, and ELODE are material separations companies. We collaborated with them for a side-by-side dewatering test for understanding the best and most cost-effective equipment to use for dewatering AD 1 sludge

Mass Balance Model



Birgitte K. Ahring Ph.D.
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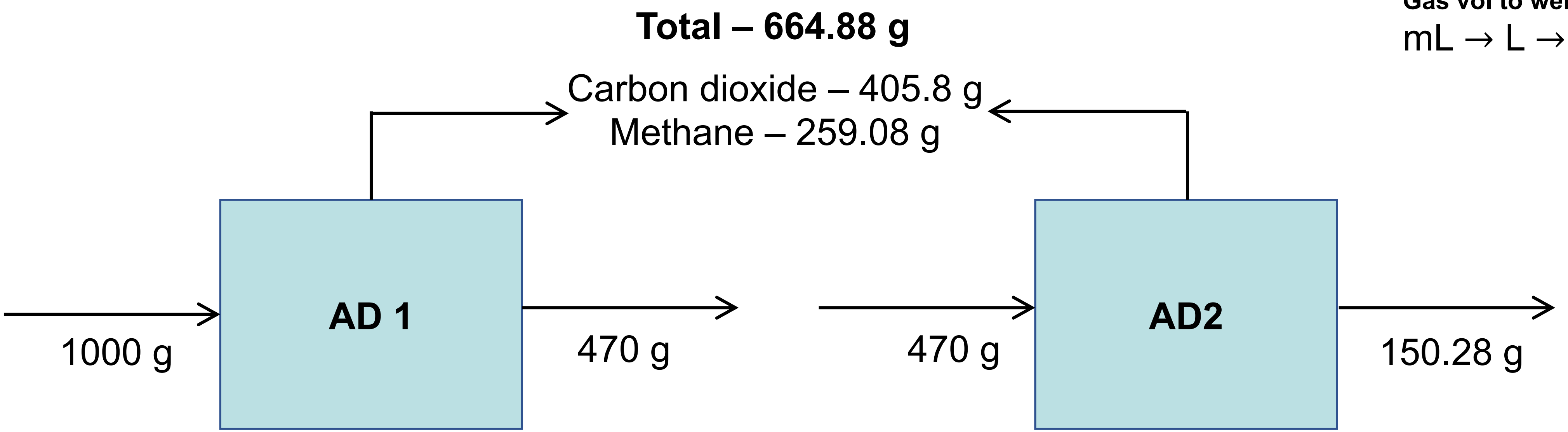
Mass Balance & Efficiency Calculations

Gas vol to weight conversion

mL → L → moles → g



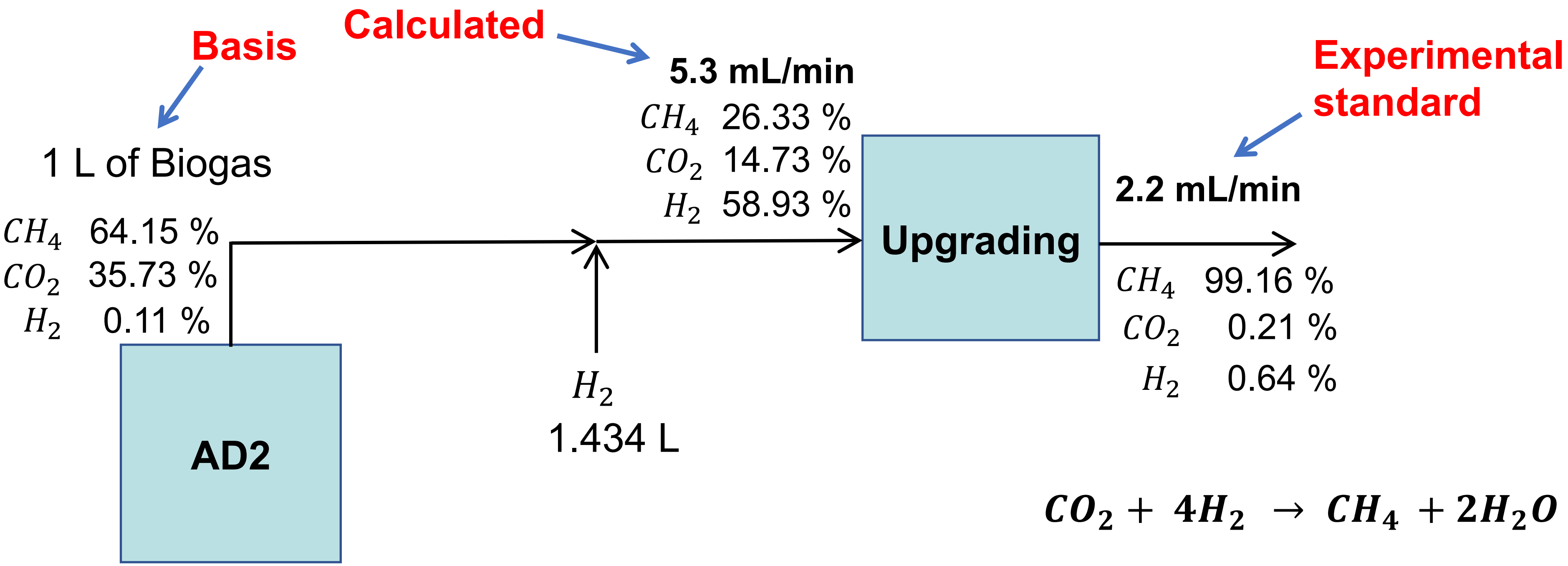
Carbon in - Carbon out = Carbon converted to gas + Carbon demand by biomass + Carbon losses during the unit operations

% carbon converted = 85

% carbon converted to biogas = 78.22

Mass Balance & Efficiency Calculations

Carbon dioxide to methane conversion (Based on upgrading biogas from AD-2):



$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O$$

PROGRESS AND OUTCOMES

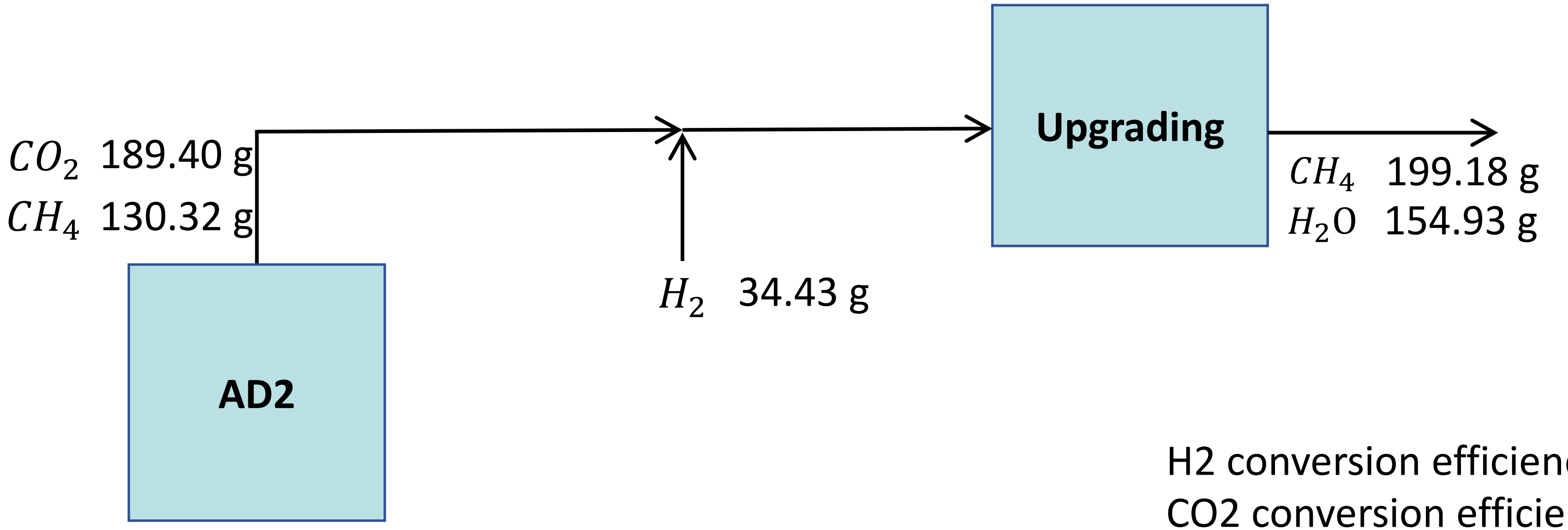
Mass Balance based on stoichiometry & experiments

$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O$$

	CO_2	$+ 4H_2$	$\rightarrow CH_4$	$+ 2H_2O$	
Volume (ml)	46.9449	187.8114517	46.9768		
Moles	0.002094	0.008379203	0.002096	0.004191737	
Mass	0.092156	0.016758406	0.033534	0.075451273	
Mass (left hand side)		0.109069007	Mass diff	-1.56264E-14	
Mass (right hand side)		0.109069007			
Inlet gas volume		318.7026162	Outlet gas volume		132
CO2 in (ml)		46.94489537	CH4 out (ml)		130.8912
CH4 in (ml)		83.91439885	CO2 out (ml)		0.2772
H2 in (ml)		187.8114517	H2 out (ml)		0.8448
H2 removal efficiency		99.55%			
CO2 removal efficiency		99.41%			

Mass Balance & Efficiency Calculations

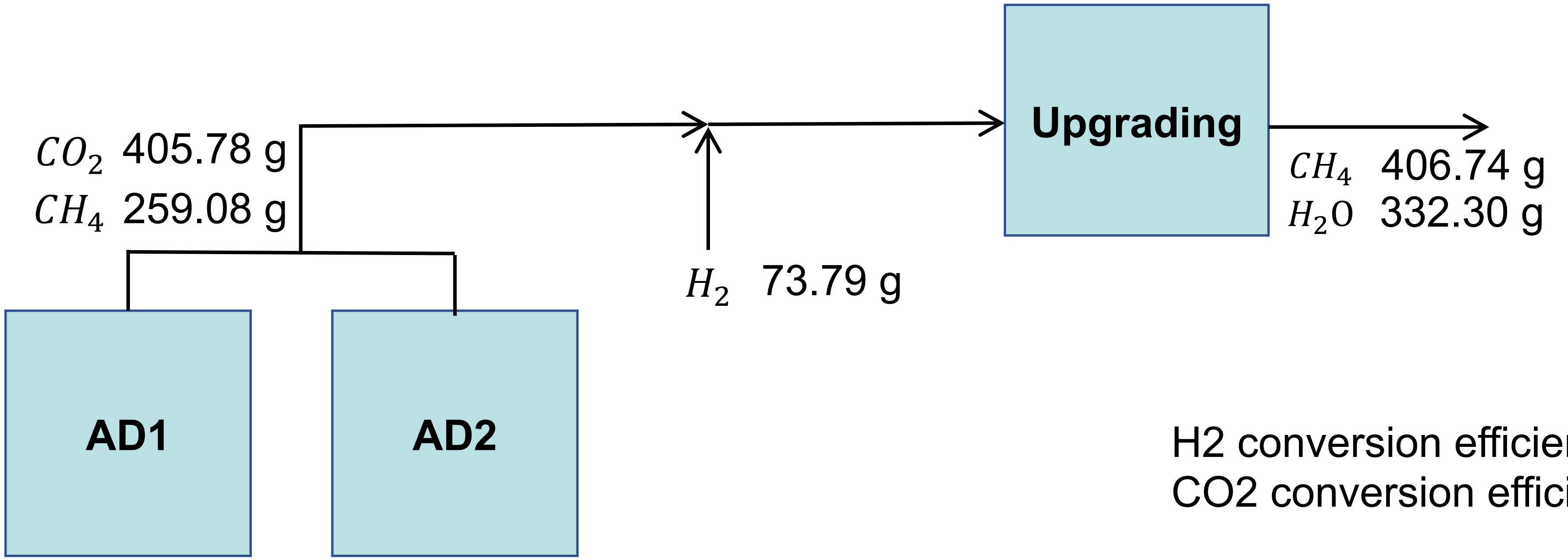
Mass balance for AD2 + Upgrading (based on experiments):



H2 conversion efficiency – 99.55 %
CO2 conversion efficiency – 99.41%

Mass Balance & Efficiency Calculations

Extended mass balance to AD1 + AD2+ Upgrading:



H2 conversion efficiency – 99.55 %
CO2 conversion efficiency – 99.41%

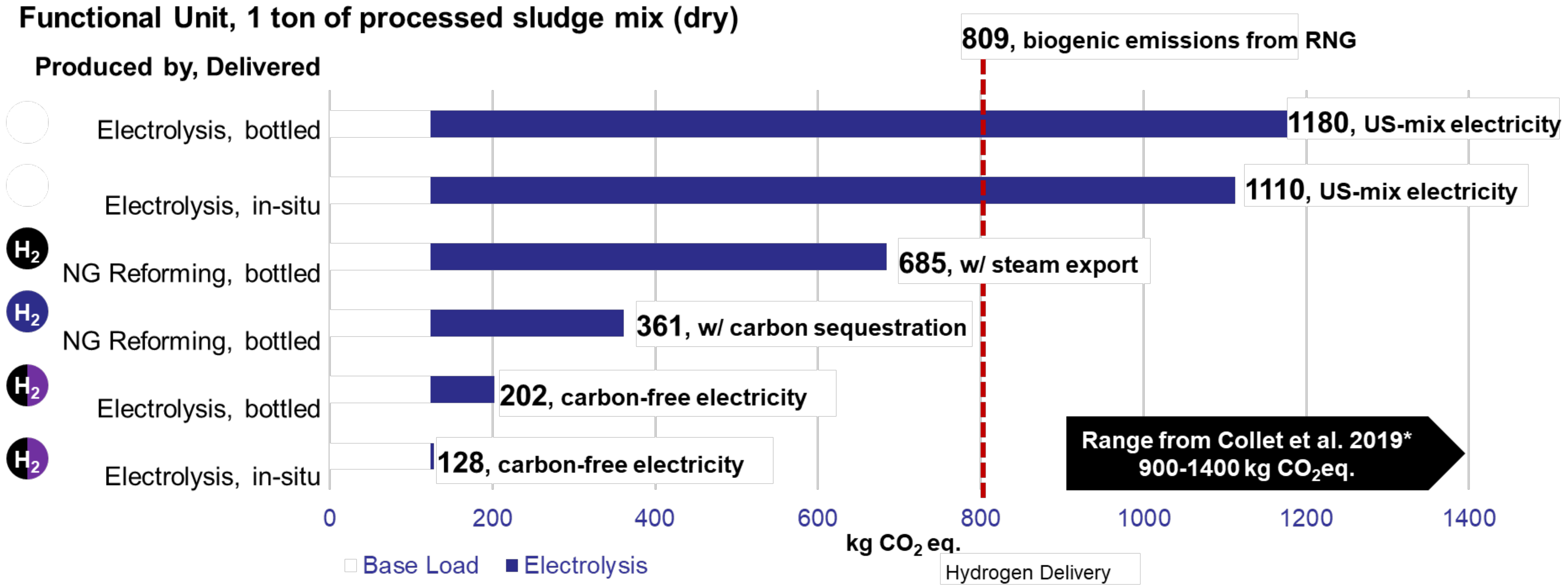
Process Performance Metrics

Parameter	Conversion efficiency
AD1 only	0.243
AD2 only	0.407
AD1 + AD2	0.305
AD1 + AD2 + Upgrading	0.479
g CH4/ g Carbon converted (overall)	0.479
g CH4/ g Carbon fed	0.407

$$\textit{Carbon to } CH_4 \textit{ Conversion efficiency} = \frac{g\ CH_4\ produced}{g\ VS\ (carbon) converted}$$

PROGRESS AND OUTCOMES

Total greenhouse gas emissions for APAD



Hydrogen from steam reformation or electrolysis powered by carbon-free electricity sources are necessary to achieve carbon-neutral processing

The content herein shows preliminary and unpublished results that are subject to change

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*Collet et al. examined AD and biomethanation using H_2 from electrolysis
https://doi.org/10.1016/j.apenergy.2016.08.181

PROGRESS AND OUTCOMES

WSU Pilot facility used for APAD

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