DOE Bioenergy Technologies Office (BETO) 2015 Project Peer Review

Recycling of Nutrients and Water in Algal Biofuels Production

March 25, 2015
DOE Bioenergy Production Technologies Office
Algae R&D Activities Peer Review

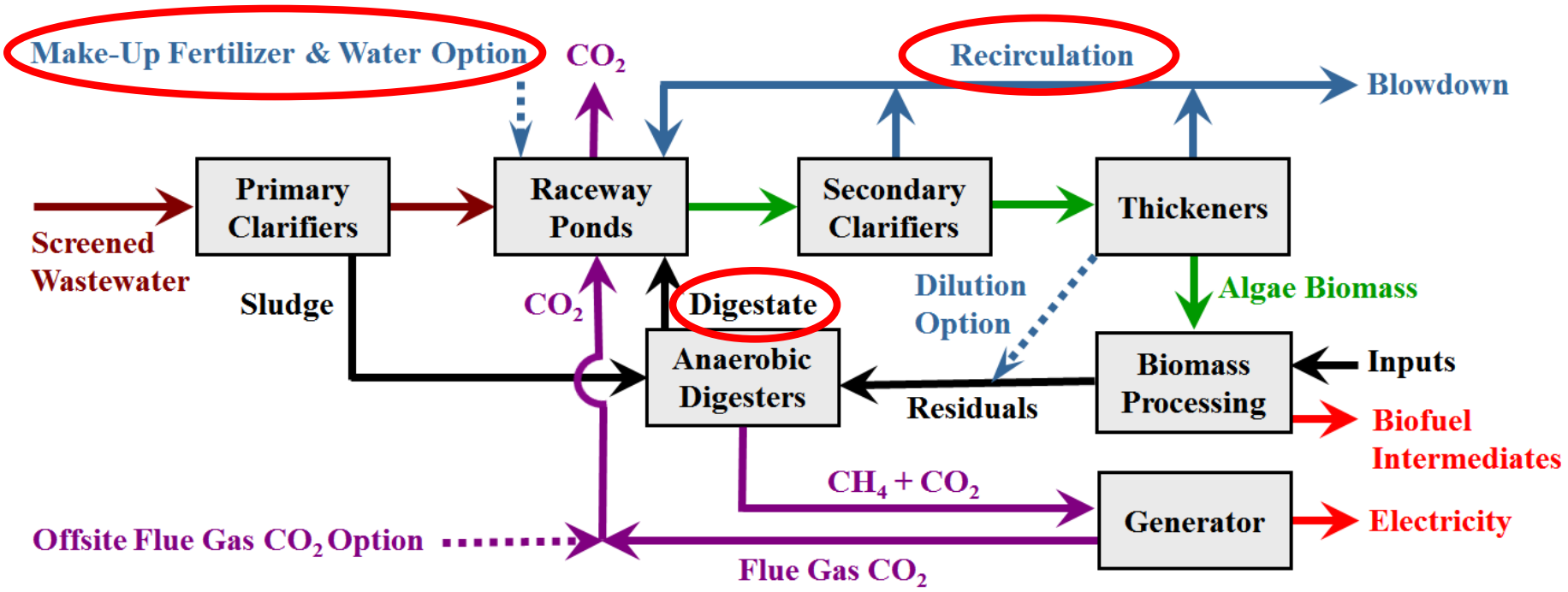
PI: Tryg Lundquist, Ph.D., P.E. (Engineering)
Co-PI: Corinne Lehr, Ph.D. (Chemistry)

Civil and Environmental Engineering California Polytechnic State University San Luis Obispo, California

MicroBio Engineering, Inc. San Luis Obispo, California

Goal Statement

- Improve the sustainability of algae biofuels by developing and demonstrating efficient recycling of water, nutrients, & some carbon.
- Without significant loss in culture stability and productivity, achieve at least 75% recycle efficiency of:
  - The water recovered after harvesting the biomass
  - The nutrients added (N, P, K and minor nutrients)
- Water and nutrient recycle rates of up to 90% will be tested.



Quad Chart Overview

Timeline

- Started February 2013
- Ends February 2016
  - Go/No-Go January 2015
- 85% complete

Barriers

- Ft-N Algal Feedstock Processing
  - Recovery and recycling of nutrients and water

Budget

	Total Costs	FY 13 Costs	FY 14 Costs	Total Planned Funding (FY 15-Project End Date
DOE Funded	$1,306k	0	$294k	$951,982
Project Cost Share (Comp.)*	$372k	0	$252k	$120k

Partners

- Cal Poly (100%)
- MicroBio Engineering, Inc. (cost share)
- City of San Luis Obispo (cost share)

1 - Project Overview

- Cal Poly operates an algae production pilot facility at a municipal wastewater treatment plant. Nine raceways @ 33 m^2 (10 m^3) each.
- Nutrients and carbon will be re-solubilized using anaerobic digestion, with digestate fed to the raceways.
- Recycled water will be monitored for build-up of inhibitory compounds and removal methods tested.
- Model recycling: processes, lifecycle, techno-economics.

Critical Success Factors

- ***We are attempting to demonstrate key technical and sustainability aspects of a common model of algae biofuel production.***
- ***Technical Challenges***
  - ***Achieving at least 75% water and nutrient recycling capability.***
  - ***Achieving rapid and extensive degradation of cell matter in digesters and raceway ponds to release nutrients.***
  - ***Overcoming inhibitors (free fatty acids, turbidity, etc.) with low cost methods.***
  - ***Maintaining low-cost bioflocculation harvesting during recycling.***
  - ***Variability among replicate ponds***

2 – Approach (Technical)

- Lab studies will establish the methods and initial modeling [Select a scalable cell lysing method; Determine biomass degradation parameters; Characterize inhibitory compounds from algae production].
- Pilot experiments will each be operated continuously over several months, in replicate and with controls. Cells will be lysed prior to digestion.
- Algal biomass will be harvested by bioflocculation, with centrifugation as needed.
- ***Go–No Go at end of Year 2: Was pilot plant performance measured with separate nutrient and water recycling, compared to controls? If yes: Proceed with integrated nutrient & water recycling pilot studies.***
- Up to 90% water recycling will be tested.
- Lifecycle and cost assessment studies based on pilot data.

2 – Approach (Management)

- *Critical success factors*
  - *Technical: Achieving 75% water and nutrient recycling*
  - *Market & Business: Achieving at least 25% lower cost than conventional wastewater treatment.*

- *Top challenges*
  - *high annual productivity,*
  - *nutrient losses*
  - *winter nutrient removal*

- *Management approach*
  - *Intensive use of milestones*
  - *Knowledge integration + research economy-of-scale with Cal Poly ABY and ATP^3 projects and MicroBio Engineering, Inc. market knowledge.*

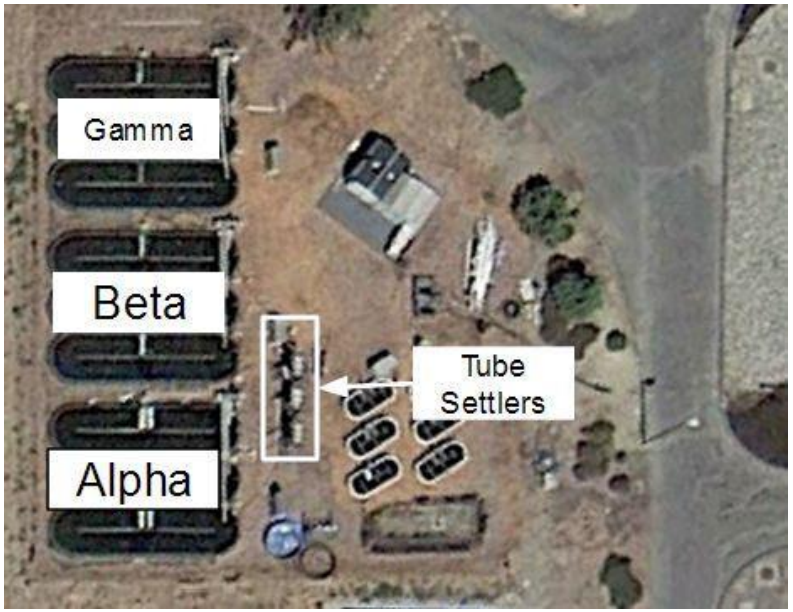
3 – Technical Accomplishments/ Progress/Results

Task 1: Pilot plant setup and shakedown

May, 2013 to August, 2013
Completed August, 2013

Task Description
Ponds
Harvesting Units – Settlers and evaluation of filters
Digesters
Biomass Pretreatment

Conclusion: Granular filtration was not practical due to rapid clogging; instead use coagulant + settling when bioflocculation is insufficient.



Pilot plant process schematic

Grow 2-6 days

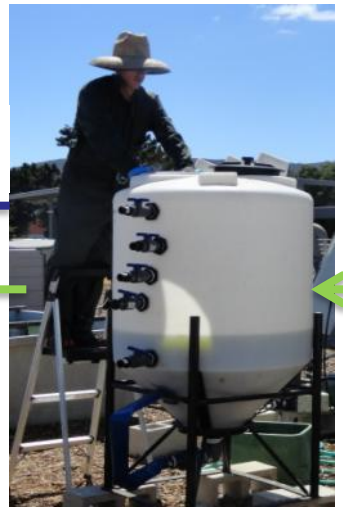
Tube settler
supernatant

Algae

Digest 40 days

Supernatant

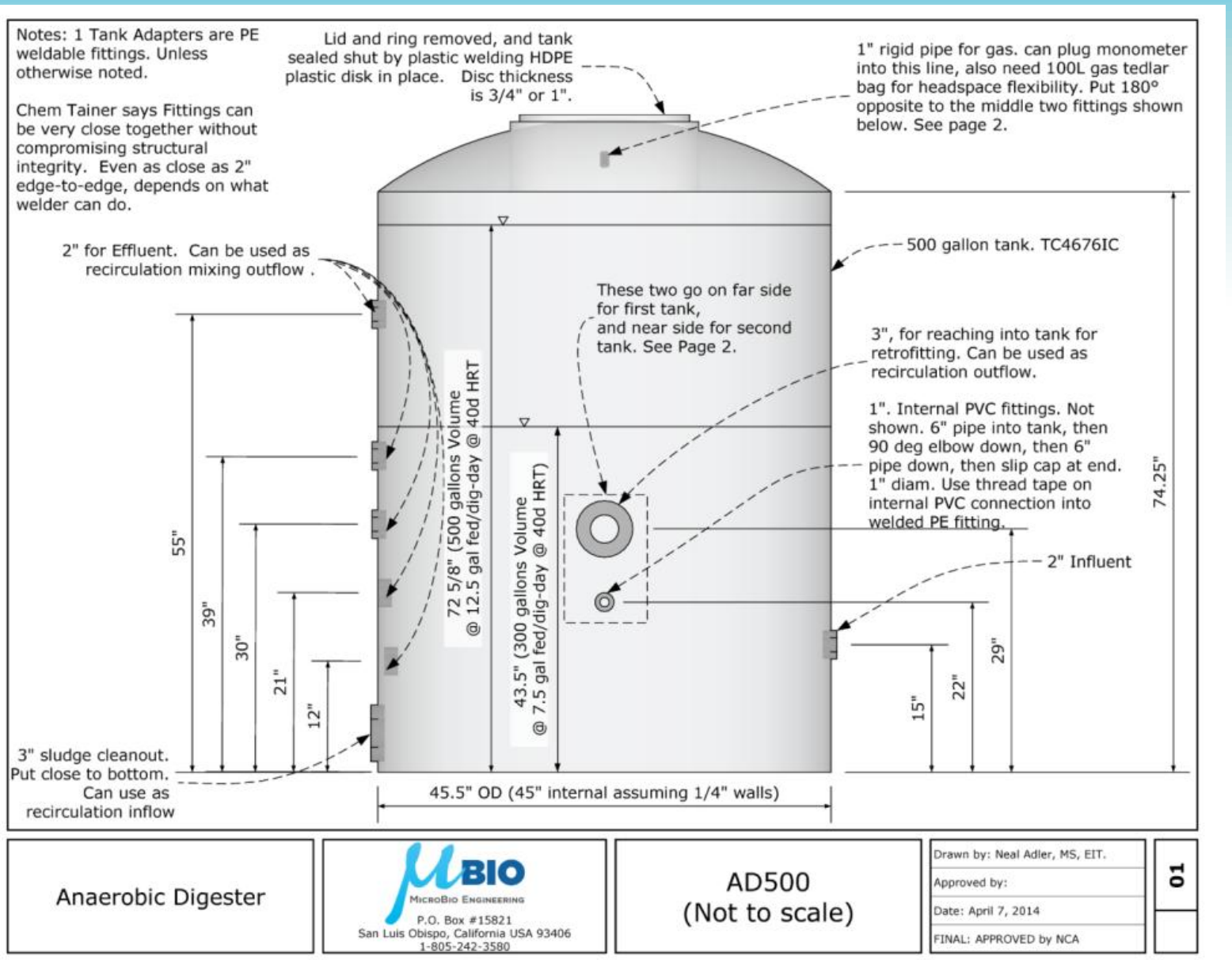
0.5-4%
VS
Algae



Thicken 12-24 hours

Harvest

Custom digester units were designed and manufactured

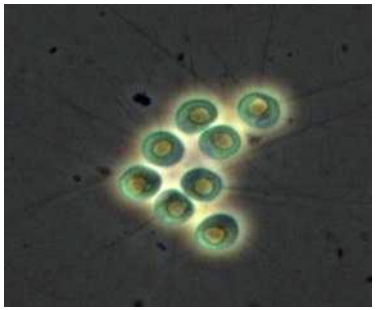


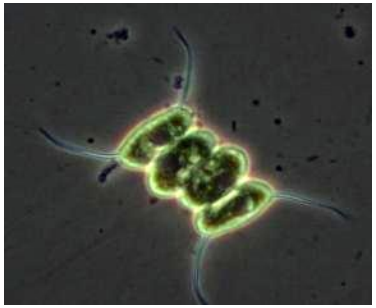
Milestone 2.1: Perform continuous operation and monitoring of the control ponds for an initial ML period of three months

June, 2013 to August, 2013
Completed August, 2013, continued under ML2.3 and 2.4

Work performed

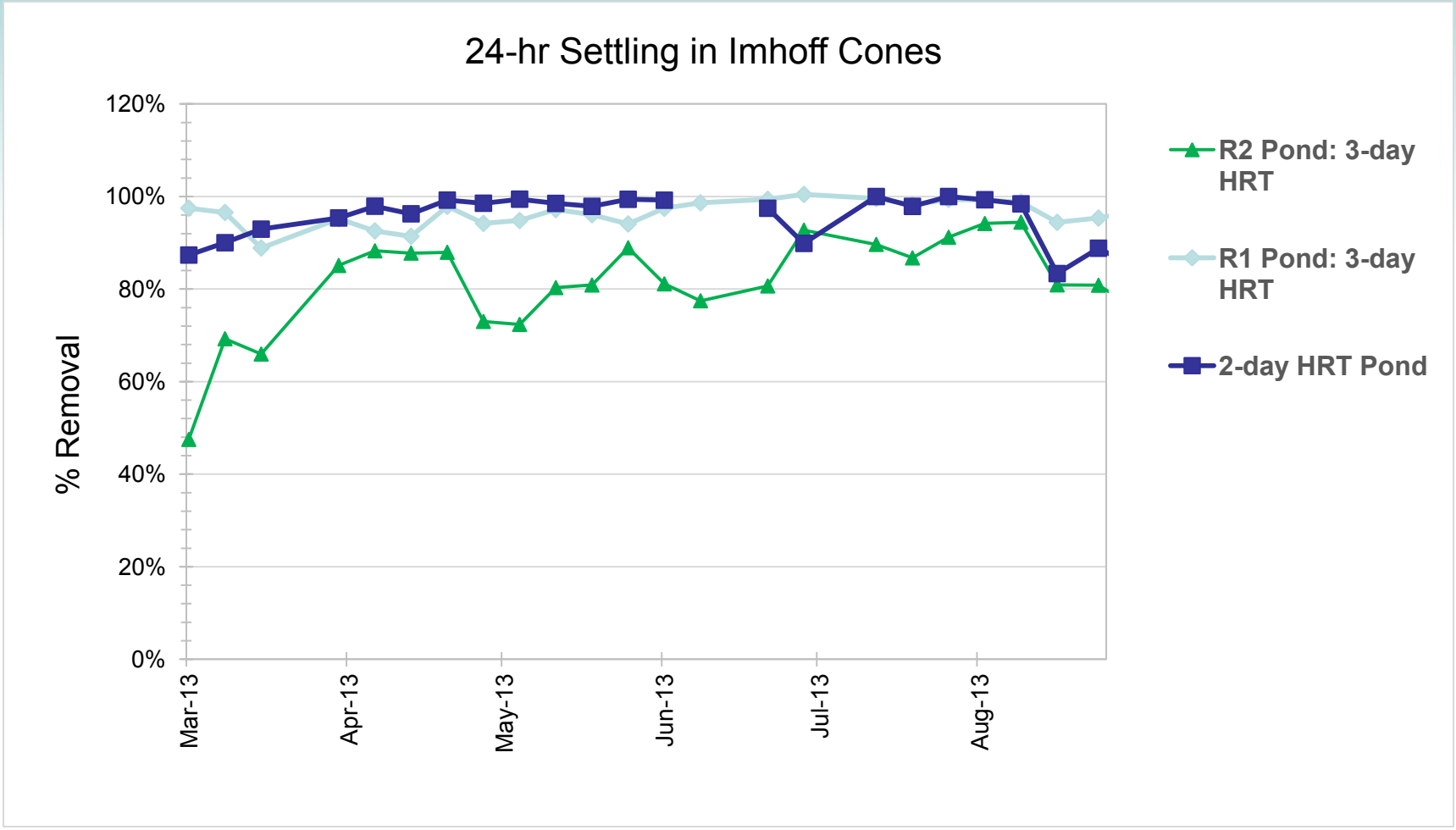
Control ponds monitored at 2 and 3 day HRT. "Round Two" ponds grown on Round 1 effluent was monitored. Continued pond operation activities reported under Subtasks 2.3 and 2.4.





Net Productivity (monthly average) (g VSS/m^2-day)		Round 2 with 3-day HRT	Round 1 with 3-day HRT	Round 1 with 2-day HRT
2013	Jun	14.1	22.2	25.7
	Jul	15.8	28.3	36.5
	Aug	14.1	26.6	29.0
	Sept	11.5	16.7	18.6
	Oct	7.7	16.7	23.9
	Nov	7.7	9.0	12.7
	Dec	2.9	7.9	11.5
2014	Jan	8.4	9.1	12.8
	Feb	10.1	14.3	17.2
	Mar	8.0	14.3	18.6
	Apr	7.5	16.9	26.2
	May	13.2	15.5	39.1
	Jun	20.3	18.1	39.6
	Jun-Jun avg	10.9	16.6	24.0

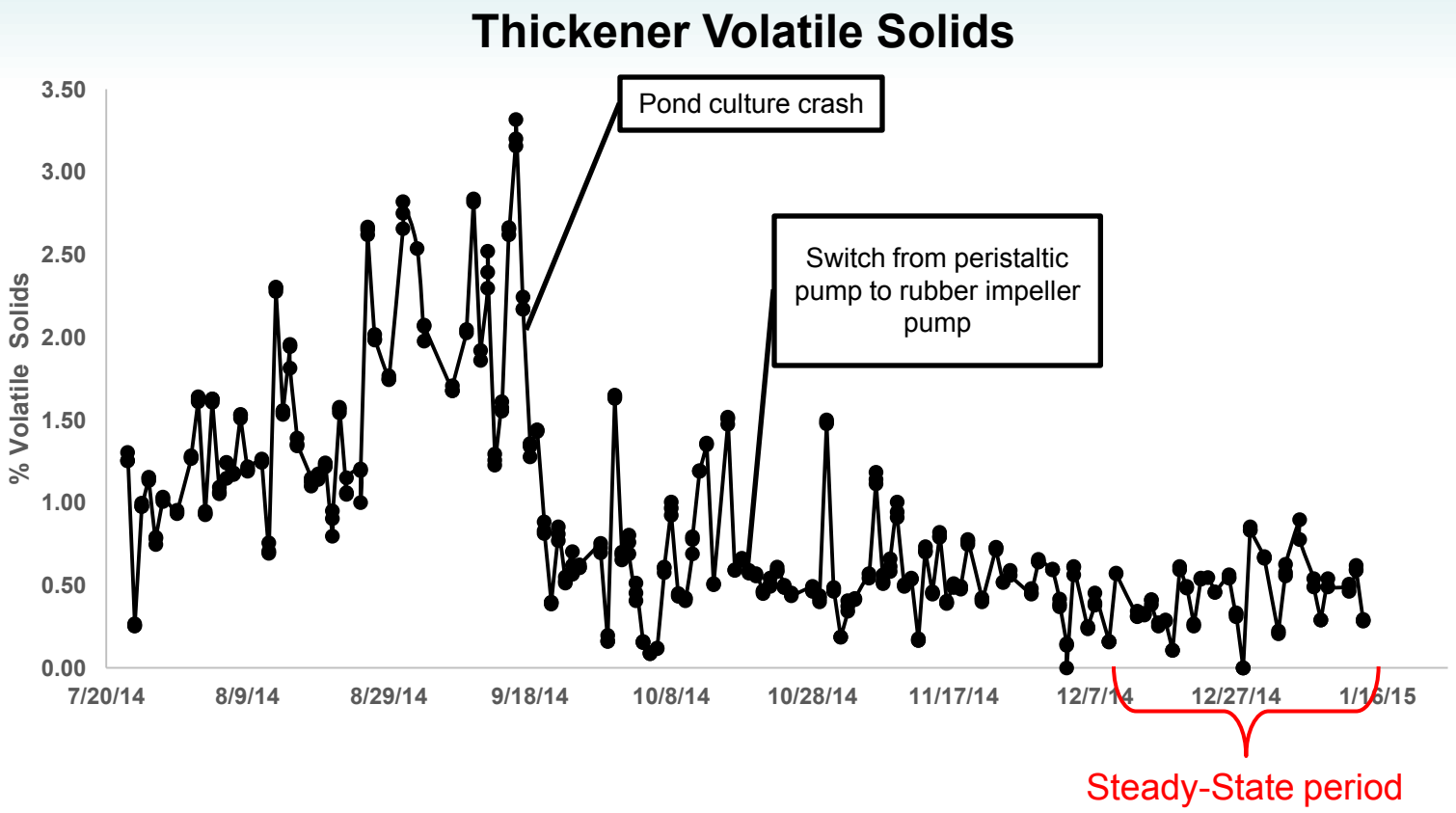
Bioflocculation: excellent summer settling performance for Round 1 ponds



Milestone 1.3: Evaluate the need for secondary harvesting or thickening units

April, 2013 to May, 2014
Completed May, 2014, with monitoring continued under ML2.4

Conclusion: Thickening in tube settlers was sufficient initially, but productivity overloaded the tube settlers, so secondary thickener cone tanks were installed.



Results: *Gravity thickener solids over time, with pond culture crash and change in pumping apparatus*

Milestone 1.4: Evaluate the need for a cell homogenizer

April, 2013 to December, 2013 → August, 2014
Completed May, 2014

Task Description
Evaluate the need for a cell homogenizer. Select, install, and successfully shakedown cell homogenizing unit.

Conclusion: Homogenization did not substantially improve nutrient solubilization after anaerobic and aerobic digestions. Biogas production increased by 15%, but that is insufficient to cover the energy input of homogenization. Homogenization was dropped.

Milestone 2.2a: Anaerobic lab-lysed incubations

Task description: Determine in anaerobic lab incubations the kinetic parameters for resolubilization of organic carbon and nutrients from biomass lysed by lab methods. The anaerobic resolubilization tests will also allow estimation of biogas production potential.

Milestone 2.2b Aerobic lab-lysed incubations

April, 2013 to October, 2013
Completed June, 2014

Determine in aerobic lab incubations the kinetic parameters for resolubilization of organic carbon and nutrients from biomass lysed by lab methods and anaerobically digested.

Milestone 2.2c: Anaerobic pilot-lysed incubations

1-Aug-13 to 31-May-2014

Task description: Determine in anaerobic lab incubations the kinetic parameters for resolubilization of organic carbon and nutrients from biomass lysed by the pilot method. The anaerobic resolubilization tests will also allow estimation of biogas production potential.

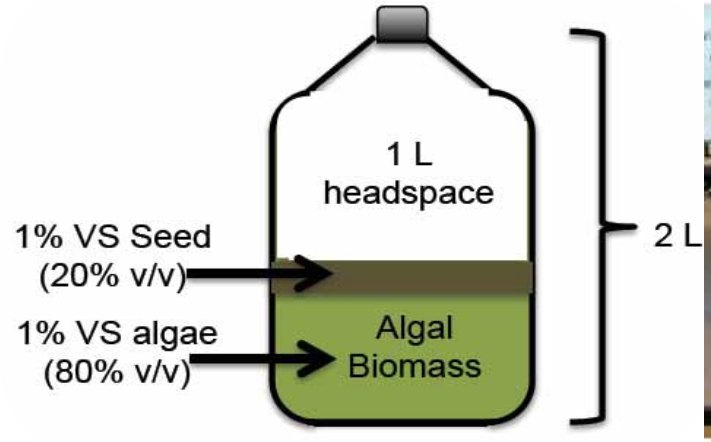
Milestone 2.2d Aerobic pilot-lysed incubations

August, 2013 December, 2013 → October, 2013
Complete September, 2013

Determine in aerobic lab incubations the kinetic parameters for resolubilization of organic carbon and nutrients from biomass lysed by the pilot method.

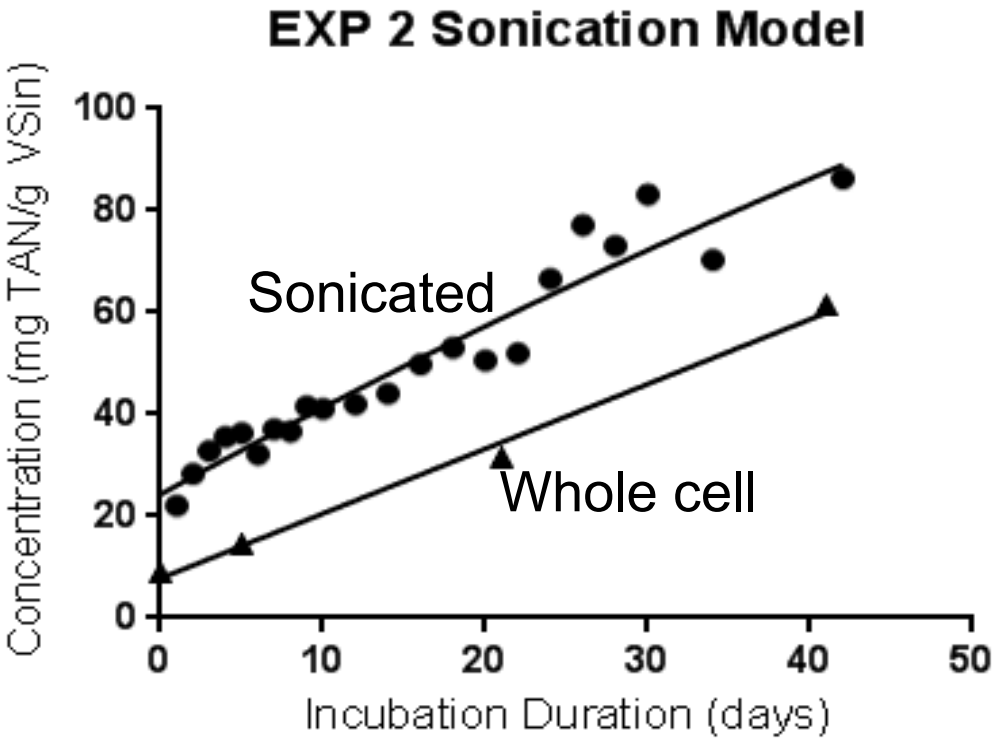
ML 2.2a, 2.2b, 2.2c, 2.2d Experimental Set-up

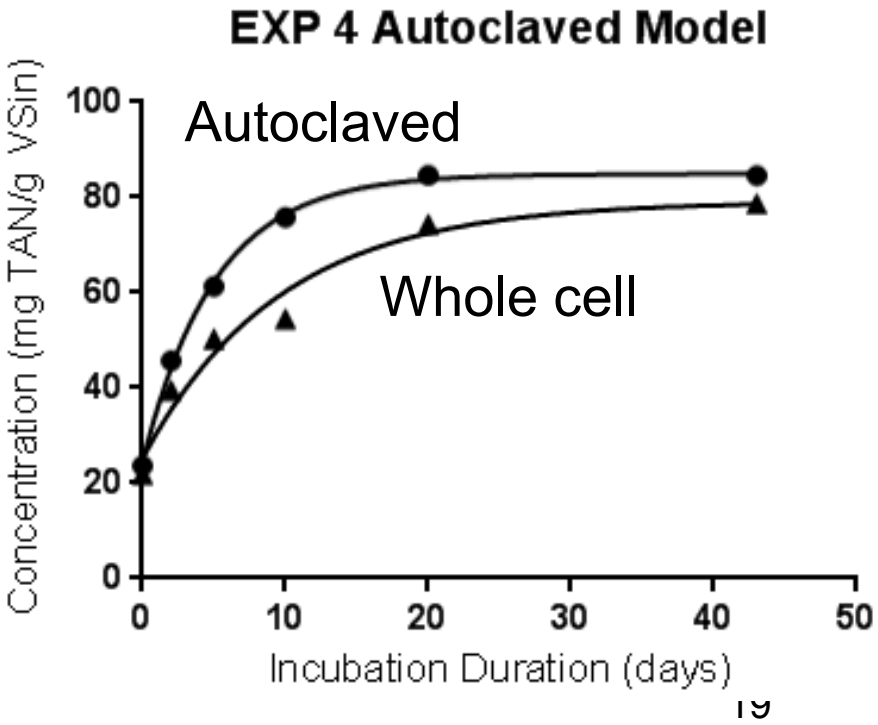
- Cell disruption methods: sonication, high pressure homogenization, boiling and autoclaving
- Batch digesters, 40 day digestion
- 80% by volume algae, 20% seed (digested municipal WW sludge -60 day HRT)
- Anaerobic digestion followed by aerobic digestion for 107 days



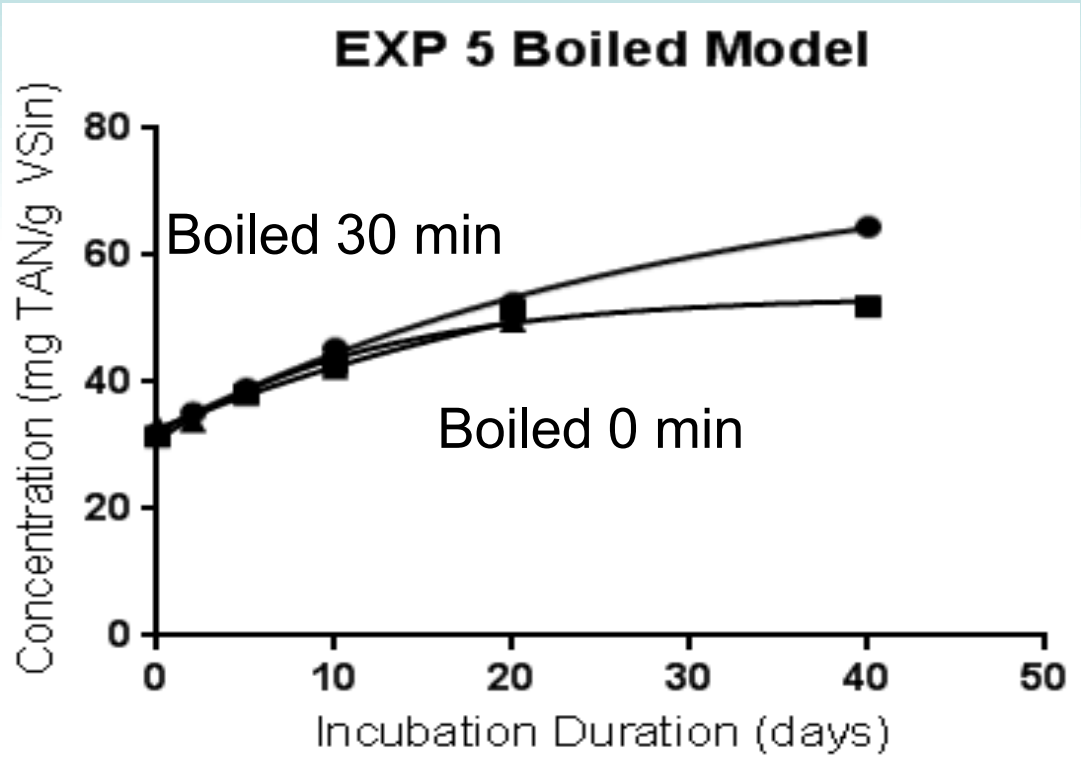
ML2.2A Kinetic parameters for resolublization of nitrogen

Conclusion: Nutrient solublization tended to follow a characteristic saturation curve, with maximum solublization occurring around 40 days of digestion. Both disrupted and whole cell algae tended to follow similar resolublization rates and final resolublization for whole cell and disrupted algae was similar.



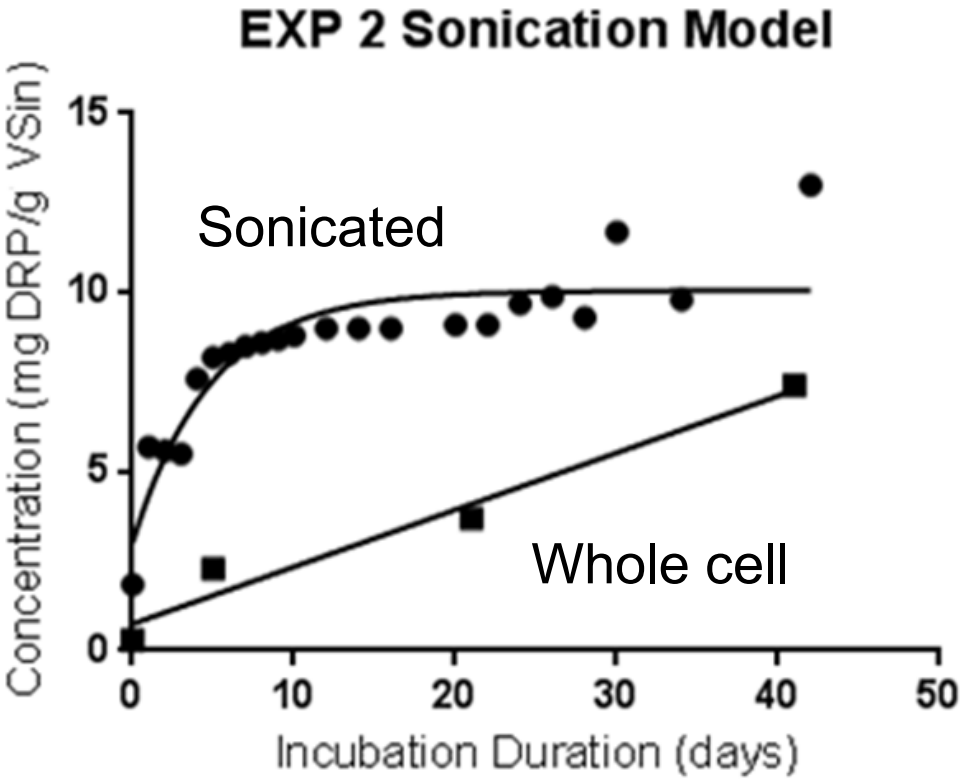


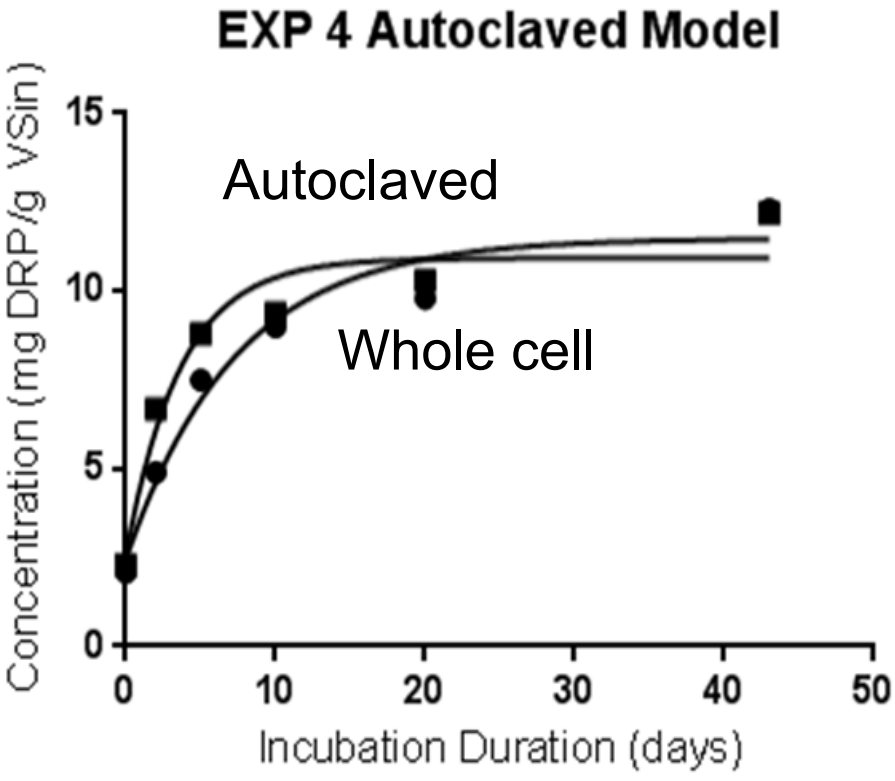
ML2.2A Kinetic parameters for re-solublization of nitrogen continued



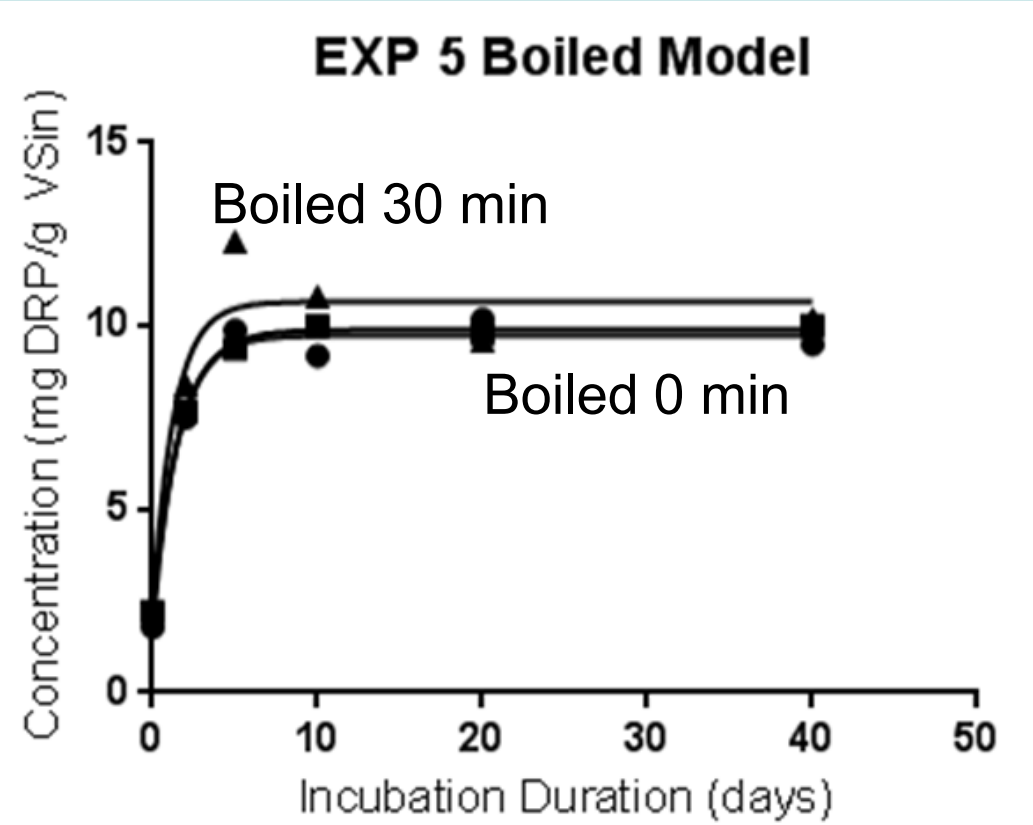
ML2.2A Kinetic parameters for resolublization of phosphorus

Conclusion: Phosphorus solublization tended to follow a characteristic saturation curve, with maximum solublization occurring around 40 days of digestion. Both disrupted and whole cell algae tended to follow similar resolublization rates and final resolublization for whole cell and disrupted algae was similar. Precipitation led to misleading readings.



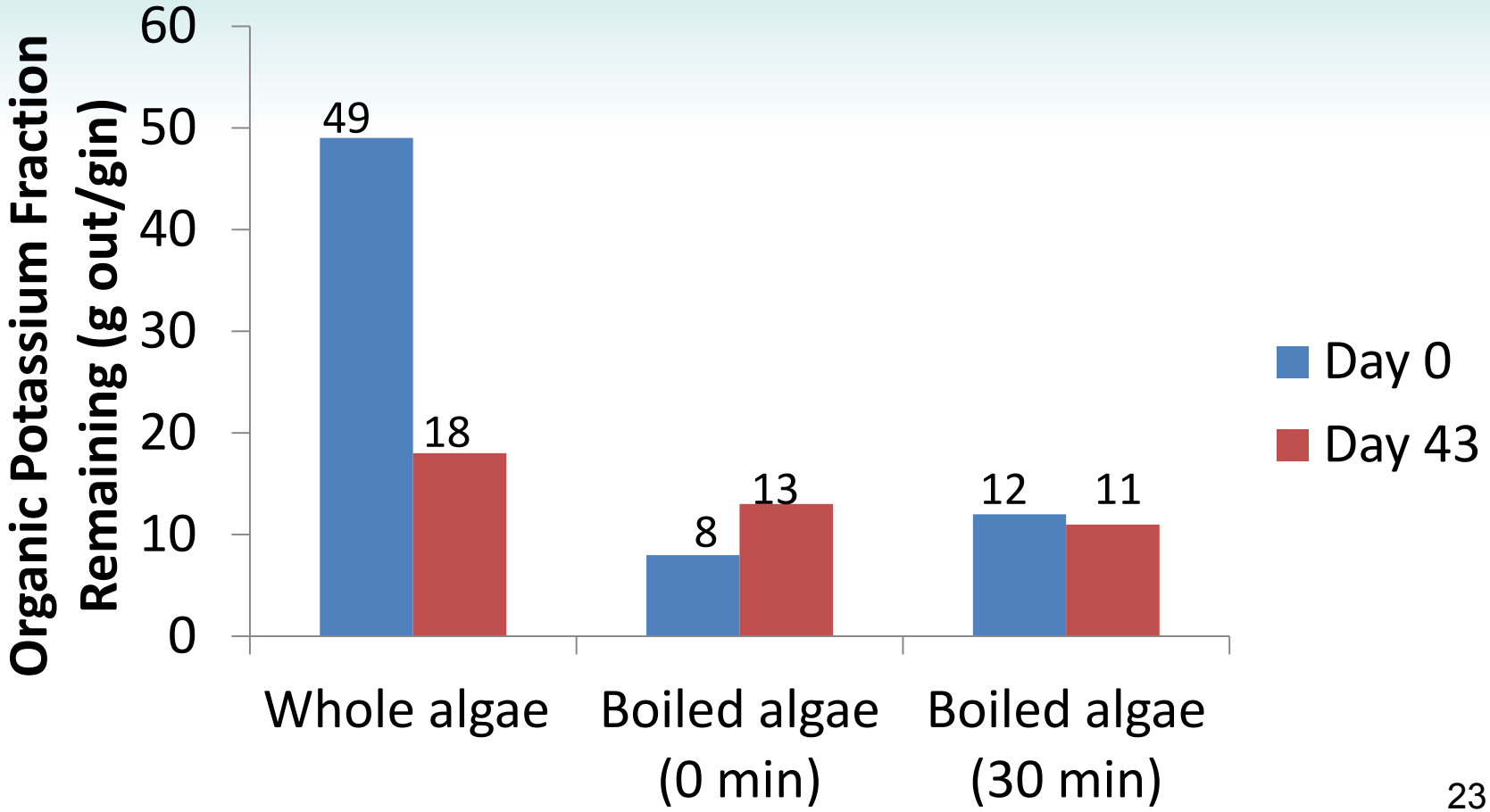


ML2.2A Kinetic parameters for resolublization of phosphorus continued



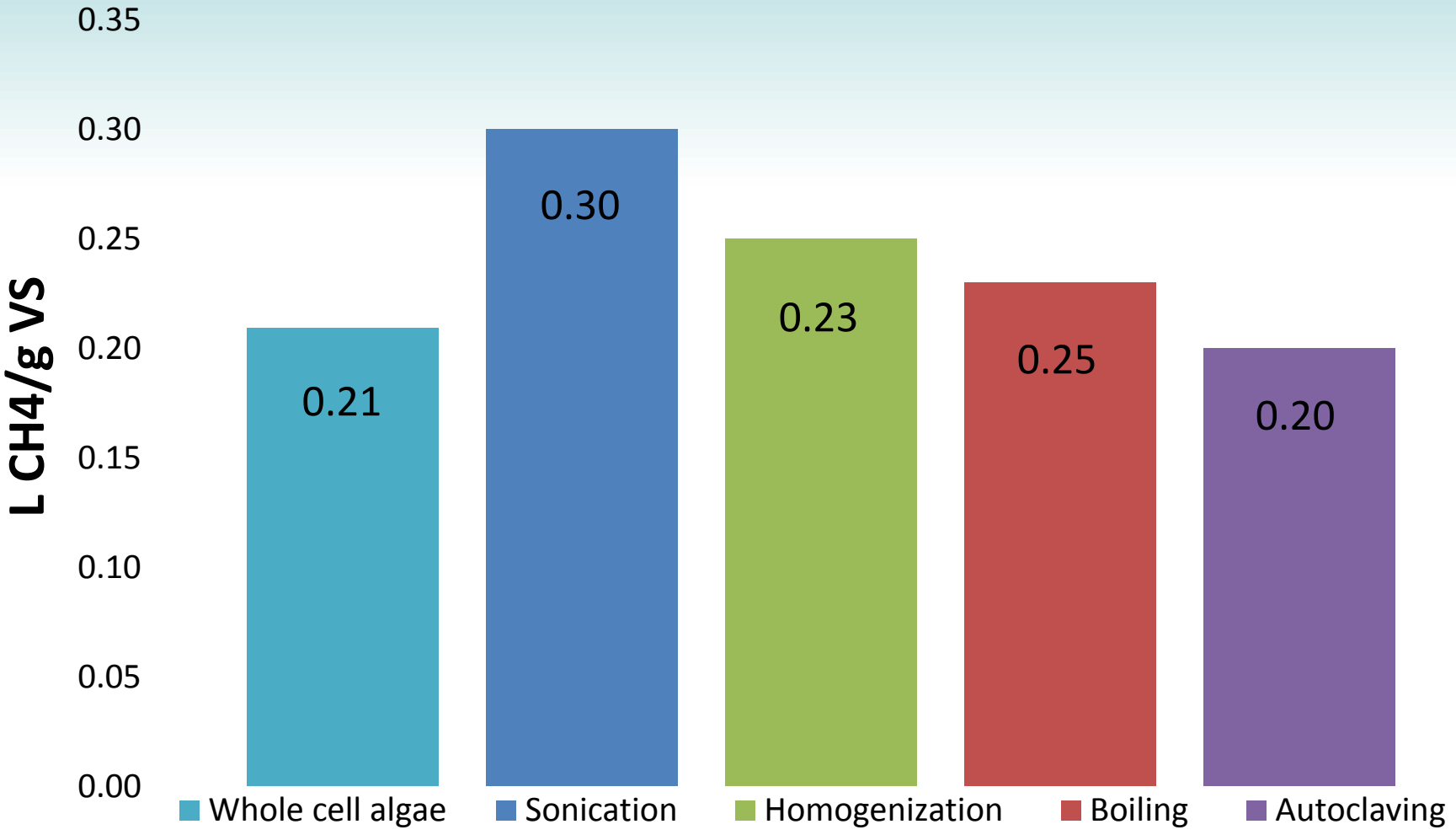
ML2.2A Resolublization of potassium in digesters

Conclusion: Pretreatment alone results on significant potassium solublization, but after 43 days of digestion whole algae have reached similar resolublization levels

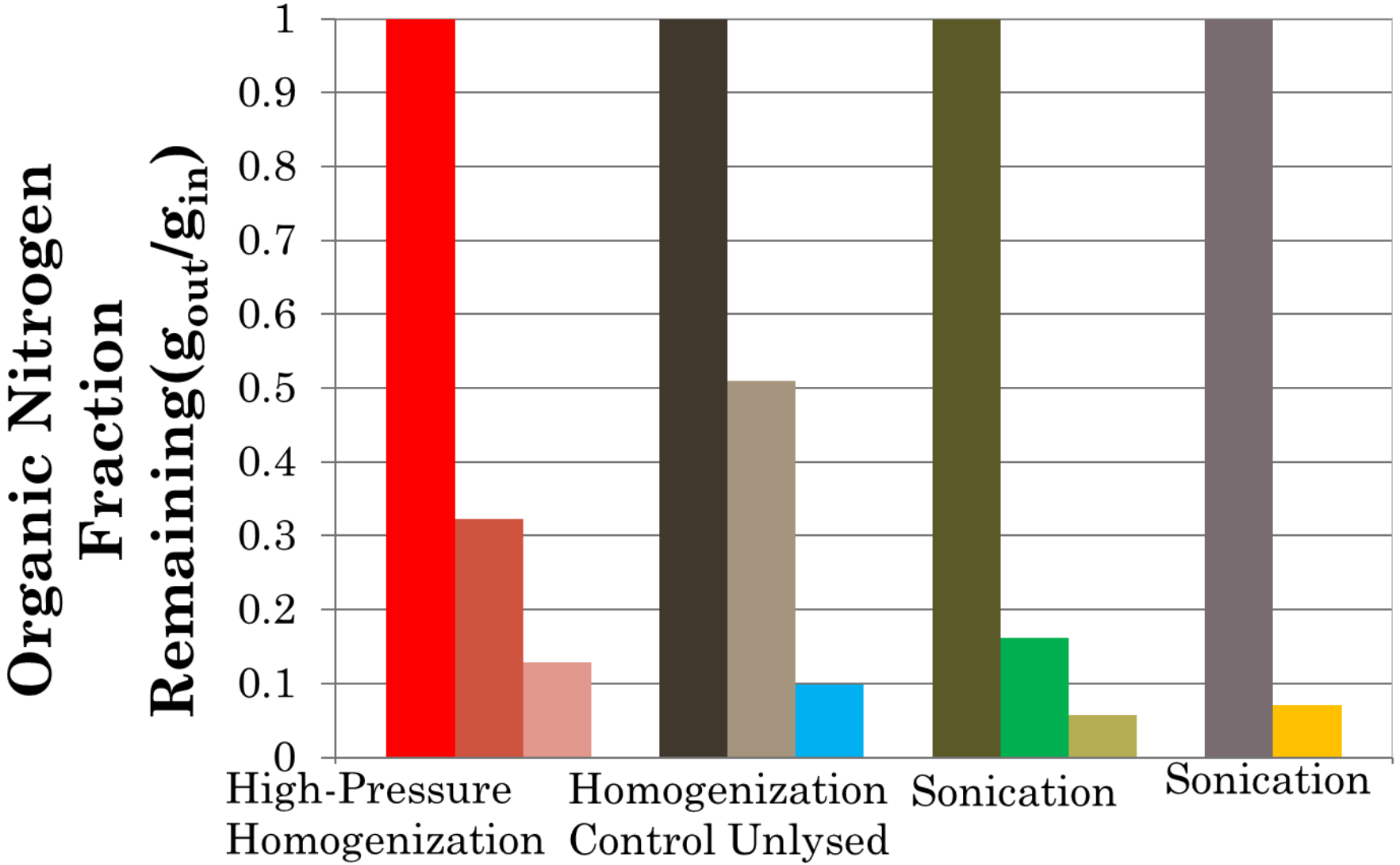


ML2.2a Biogas Production potential for lysed and whole cell algae

Conclusion: Sonication had the highest methane yield but also was the most energy intense disruption method.



ML2.2a, ML 2.2b Nitrogen



ML2.2a, ML 2.2b Nitrogen

Conclusion: Pretreatment did not improve nutrient resolublization

HOMOGINIZATION

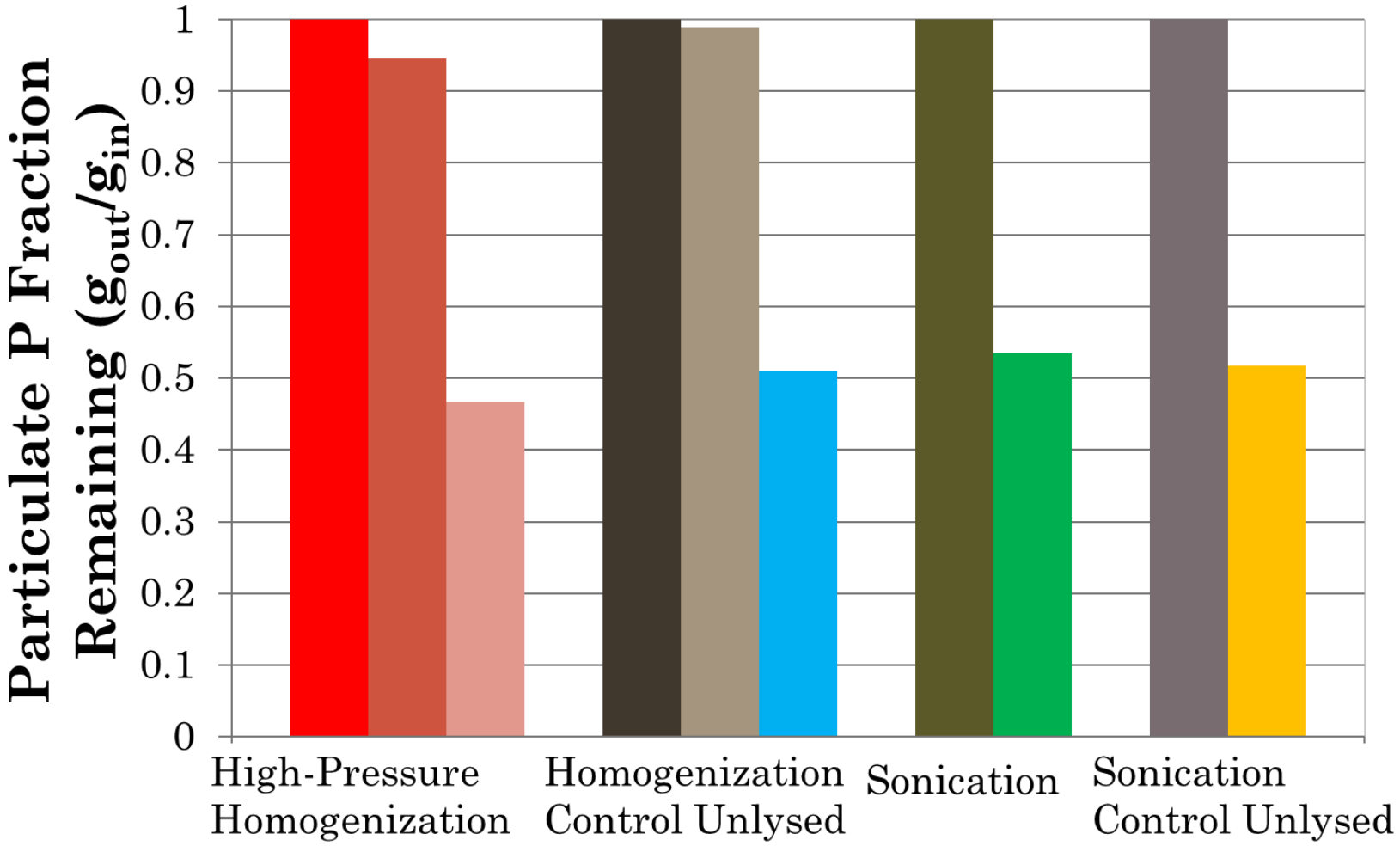
- 68% solublization in digester for disrupted cells, 49% for whole cells
- 19% increase in solublization with aerobic digestion for disrupted cells, 41% for whole cell

SONICATION

- 84% solublization in digester for disrupted cells, 93% for whole cells
- 10% increase in solublization with aerobic digestion for disrupted cells, whole cell not aerobically digested

Sonication showed the highest solublization of all disruption techniques. Solublization for boiled and autoclaved algae was similar to homogenization.

ML2.2a, ML 2.2b Phosphorus



ML2.2a, ML 2.2b Phosphorus

Conclusion: Pretreatment did not improve nutrient resolublization

HOMOGENIZATION

- 5% solublization in digester for disrupted cells, 1% for whole cells*
- 48% increase in solublization with aerobic digestion for disrupted cells, 48% for whole cell*

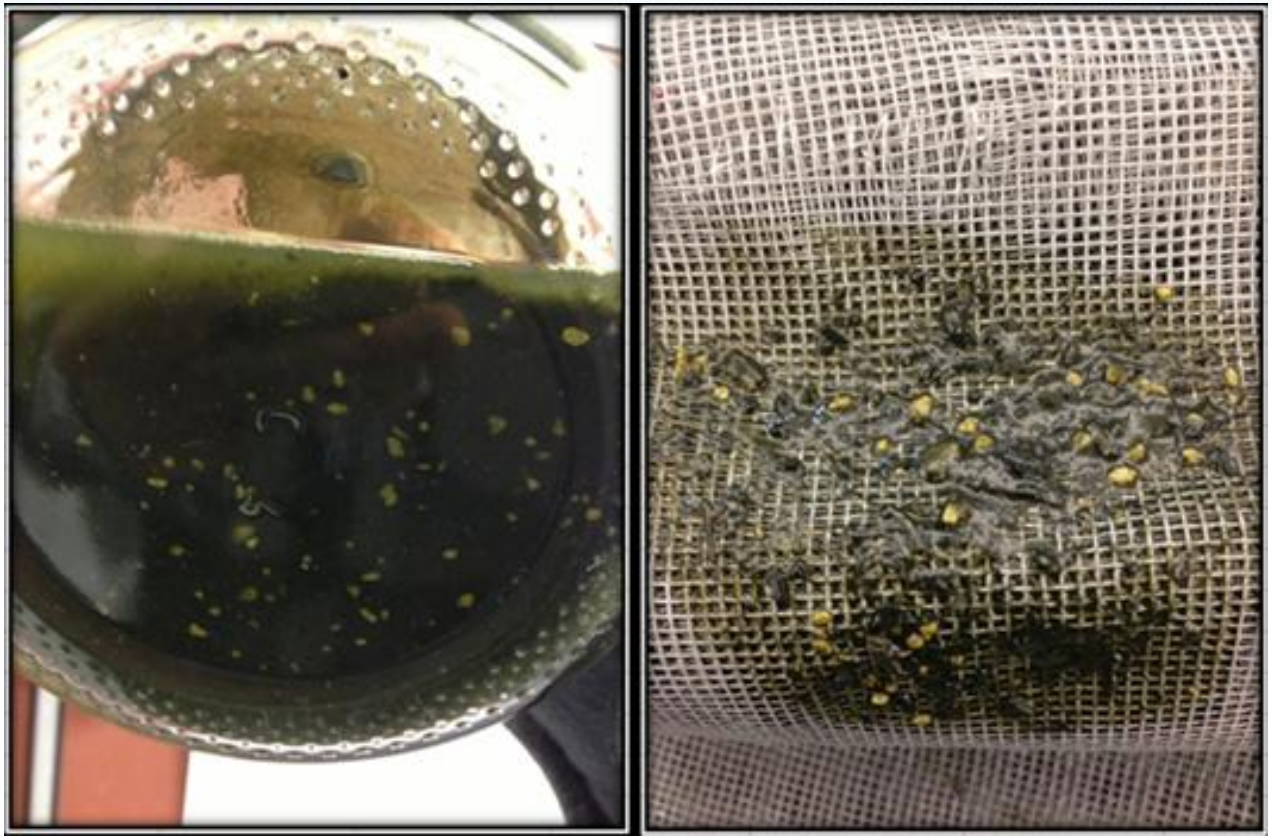
SONICATION

- 48% solublization in digester for disrupted cells, 47% for whole cells

Other cell disruption techniques, including boiling and autoclaving resulted in ~ 40-50% P solublization

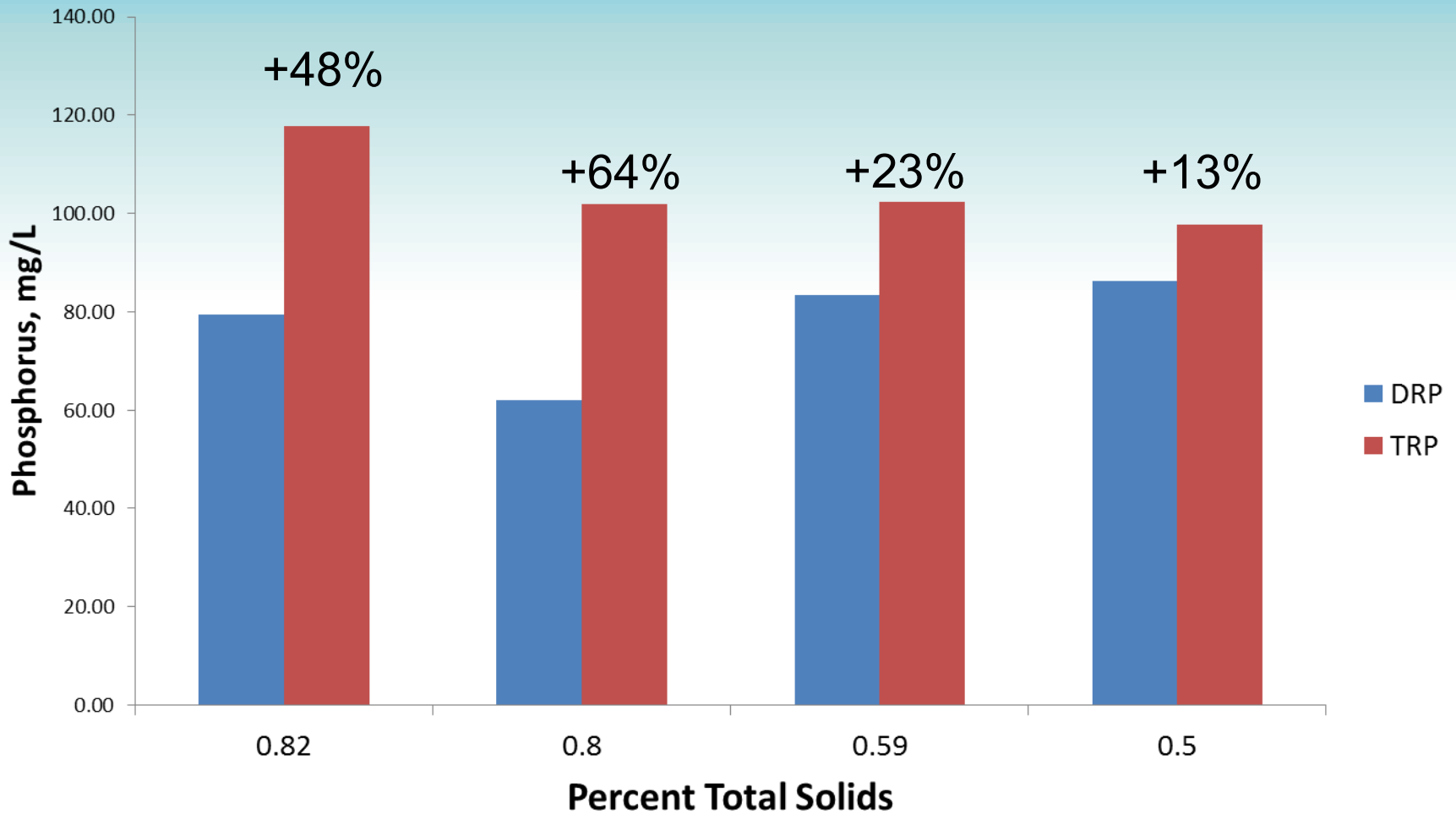
ML2.2a, ML 2.2b Aerobic Resolublization

Conclusion: Aerobic treatment did not significantly increase nutrient resolublization after anaerobic digestion. Most nutrient release in aerobic experiment was likely due to dissolving precipitates of nitrogen and phosphorus that formed in the digester. Better analytical methods are needed to measure nutrient solublization in digesters.



Bioavailable Phosphorus

Results: *Difference between Dissolved Reactive Phosphorus & Total Reactive Phosphorus at different solids concentration*

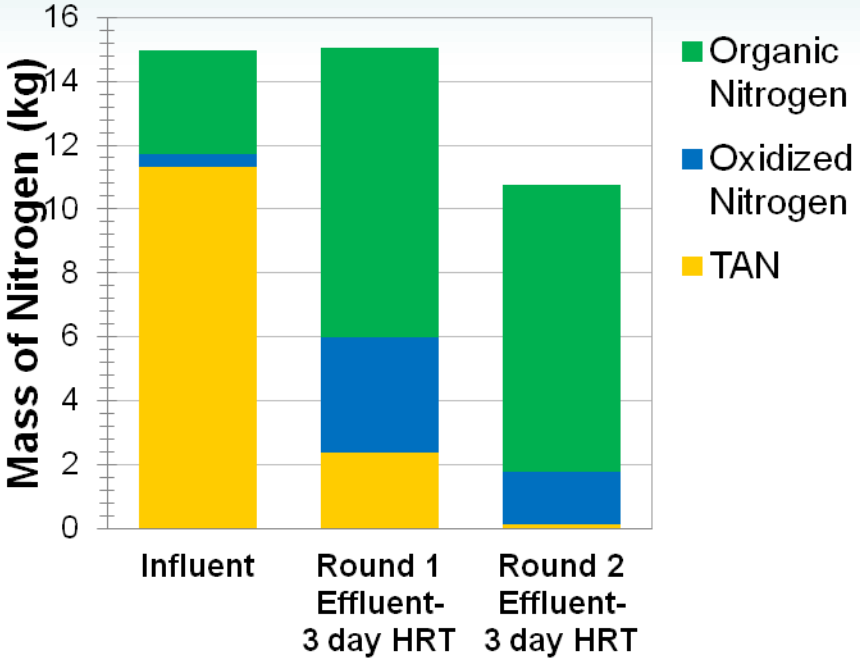


TRP includes phosphorus adsorbed to solids, and precipitates formed in the digester
Difference between TRP and DRP increases with higher solids concentration

Milestone 2.3c continued - Nitrogen Mass Balance: Ponds in Series

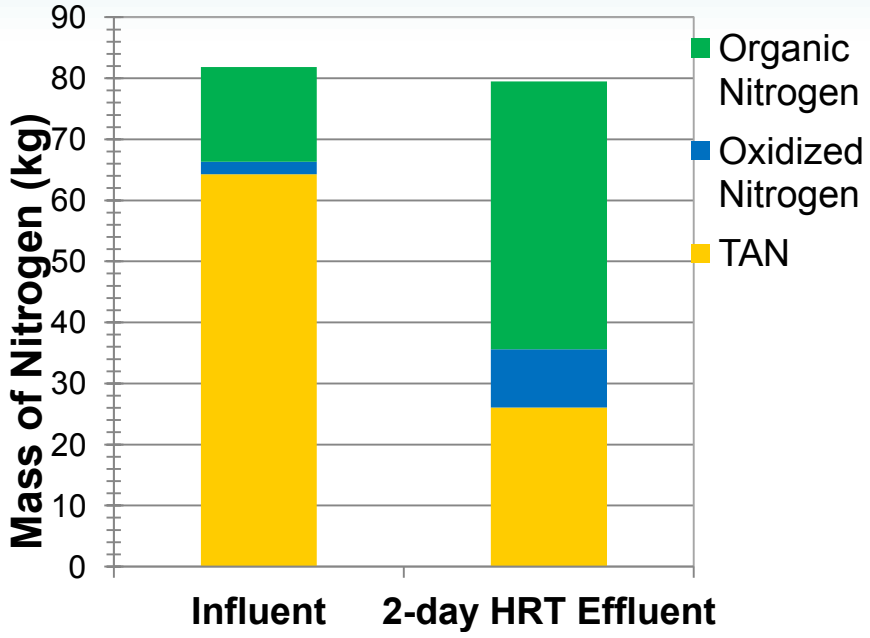
Mass Balance

Time duration = **13** weeks (June 19, 2014 – September 11, 2014). Data shown reflects **12** weeks. **1** week is missing because data for at least one nitrogen component was not run or failed QA/QC.



Mass Balance for entire experiment duration

Time duration = 77 weeks (March 3, 2013 – September 11, 2014). Data shown reflects 59 weeks. 18 weeks are missing because data for at least one nitrogen component was failed not run or failed QA/QC.

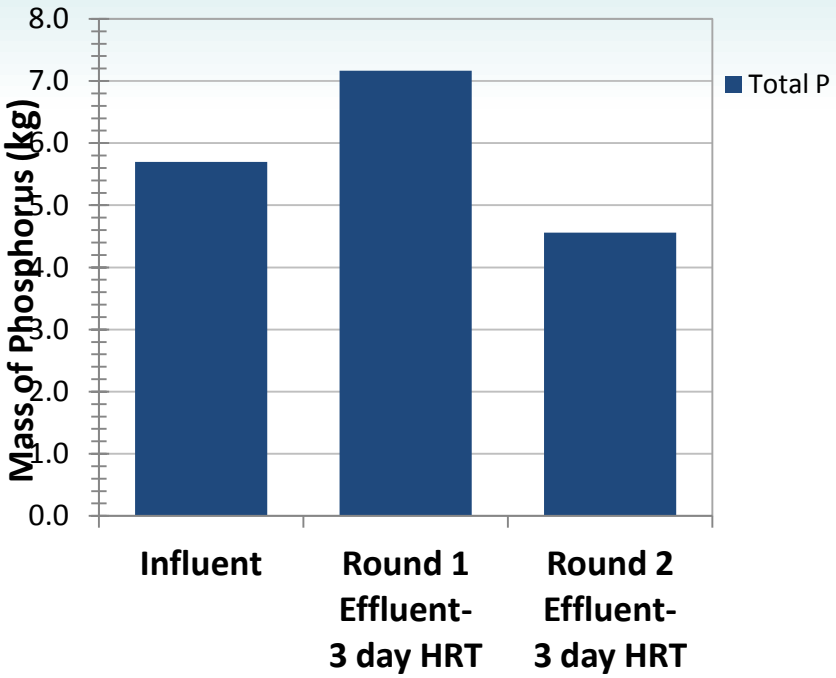


Conclusion: The fate of nitrogen can be accounted for using mass balances when sampling and analytical work is carefully done.

Milestone 2.3c continued – Phosphorus Mass Balance – Ponds in Series

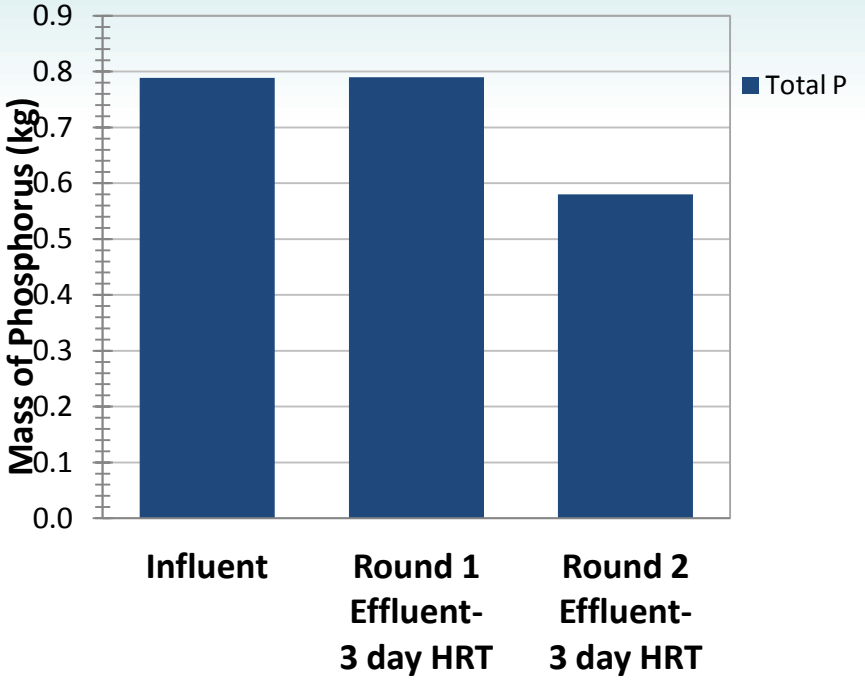
Mass Balance for entire experiment duration

Time duration = 61 weeks (July 3, 2013 – September 24, 2014). Data shown reflects 43 weeks. 18 weeks omitted because data for total phosphorus was not run or failed QA/QC.



Mass Balance with standpipe overflow samples

Time duration = 4 weeks (September 4, 2014 – September 24, 2014).

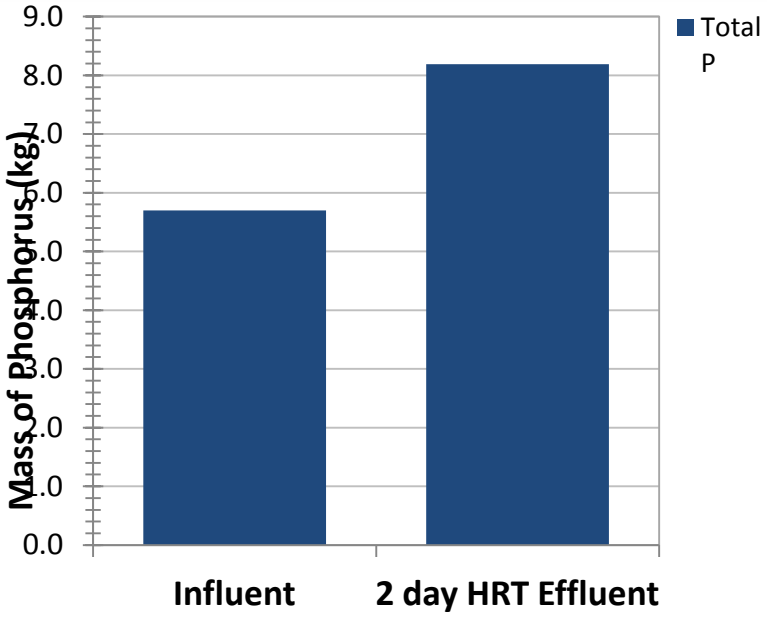


Conclusions: Improved sampling method improved the P mass balance. (Round 2 Effluent is lower because phosphorus was removed by the harvesting system.)

Milestone 2.3c continued – Phosphorus Mass Balance – 2-day HRT effluent

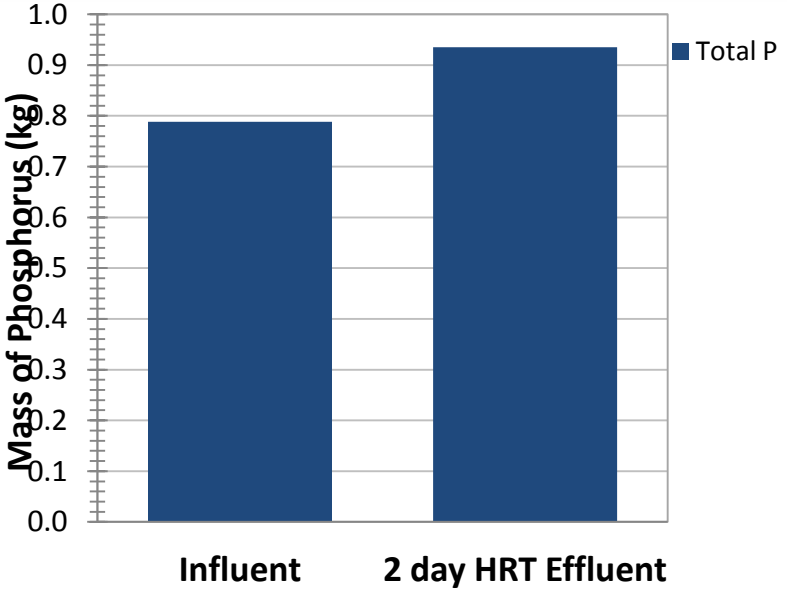
Mass Balance for the entire experiment duration

Time duration = 61 weeks (July 3, 2013 – September 24, 2014). Data shown reflects 43 weeks. 18 weeks are missing because total phosphorus data was either not run or failed QA/QC



Mass Balance with standpipe overflow samples

Time duration = 4 weeks (September 4, 2014 – September 24, 2014).



Milestone 2.4a: Over an initial four months, measure productivity, nutrient recapture efficiency by mass balance, and bioflocculation/settling efficiency

January, 2014 to July, 2014 → April, 2015

Work performed

<u>April 1, 2014 to June 30, 2014</u>

Additional tube settler trials conducted to see if change can improve harvesting without adding harvesting equipment. Cone-bottom tanks setup for use in thickening experiments and to generate feed for the pilot digesters.

<u>July 1, 2014 to Sept 30, 2014</u>

Ponds and harvesting units reconfigured to recycle water continuously. Two cone-bottom tanks added and backup coagulant dosing system planned. Two sets of control ponds in operation. Biomass from control ponds feeding digesters. One set of ponds is being used to test the recycling of water and nutrients. Productivity measurements are shown in the following table:

Productivity (g/m^2-day)															
	3 Day Control					Recycling Water					1.5 Day Control				
Date	Pond 1	Pond 2	Pond 3	Avg	Std. Dev.	Pond 4	Pond 5	Pond 6	Avg	Std. Dev.	Pond 7	Pond 8	Pond 9	Avg	Std. Dev.
10/8/2014	15	30	37	27	9						25	27	33	29	3
10/15/2014	15	14	19	16	2	10	6	11	9	2	14	18	21	17	3
10/22/2014	7	12	15	11	3	14	17	44	25	14	15	18	17	17	1
10/29/2014	2	12	28	14	11	9	15	10	11	2	15	9	11	12	2
11/5/2014	4	15	9	9	4	5	6	3	4	1	11	11	4	9	3
Average				**16**	**6**				**12**	**5**				**17**	**3**

Milestone 2.4a continued

Batch growth experiment

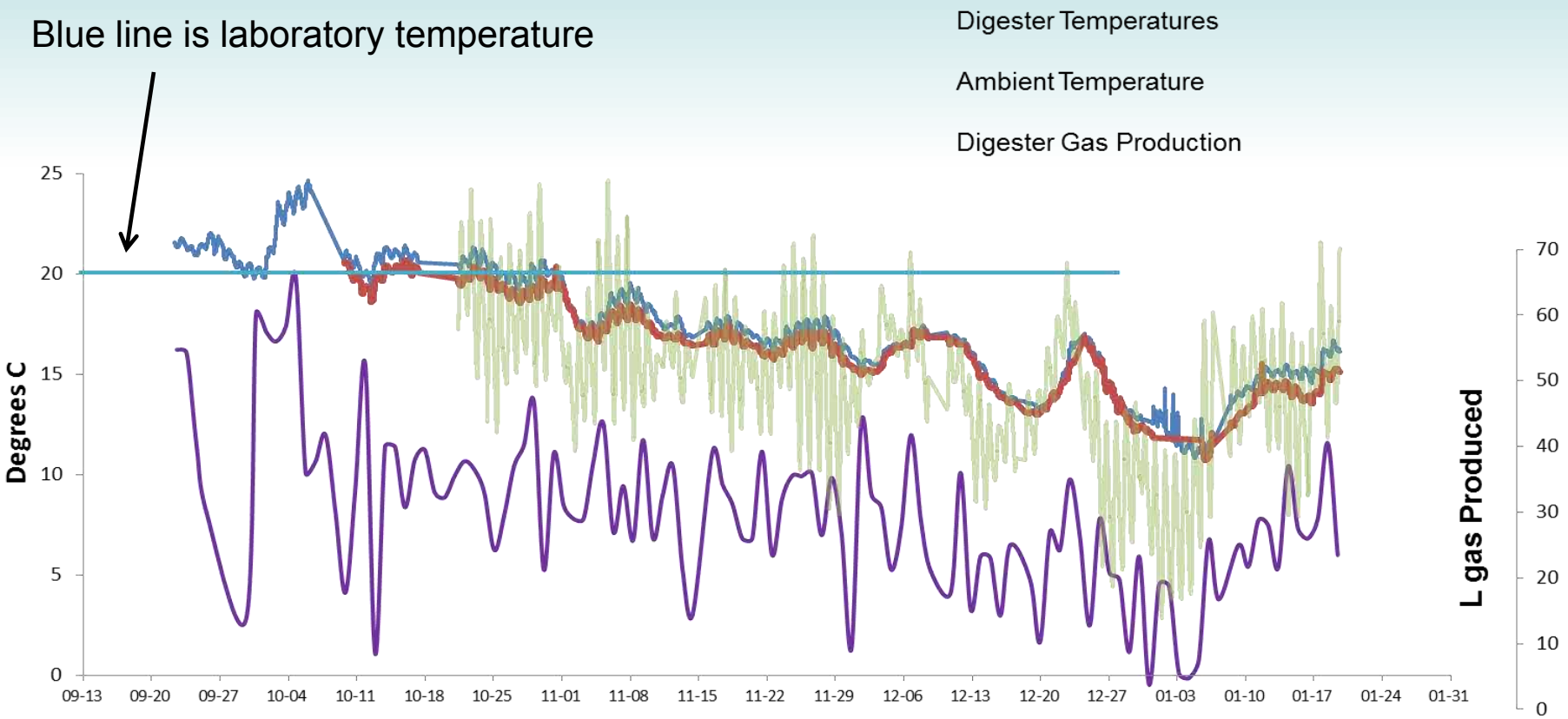
Objective: Prove feasibility and potential for water and nutrient recycling.
Experiment: Compare in a batch test the growth and nitrogen uptake of ponds grown on fresh wastewater, and recycled water with an addition of nutrients from anaerobically digested algae.
Outcomes:

Growth Media	Peak Productivity (g VSS/m2-day)
Fresh Wastewater	19
Recycled Water & Digestate	23
Reclaimed Water Only	9

Conclusion: Addition of digested algae to recycled water provides sufficient nutrients for comparable productivity to ponds fed fresh wastewater.

Milestone 2.5: The purpose of this task is to operate the digesters in a stable, continuous process that allows a high level of water recycling to support S2.2 and 2.4.

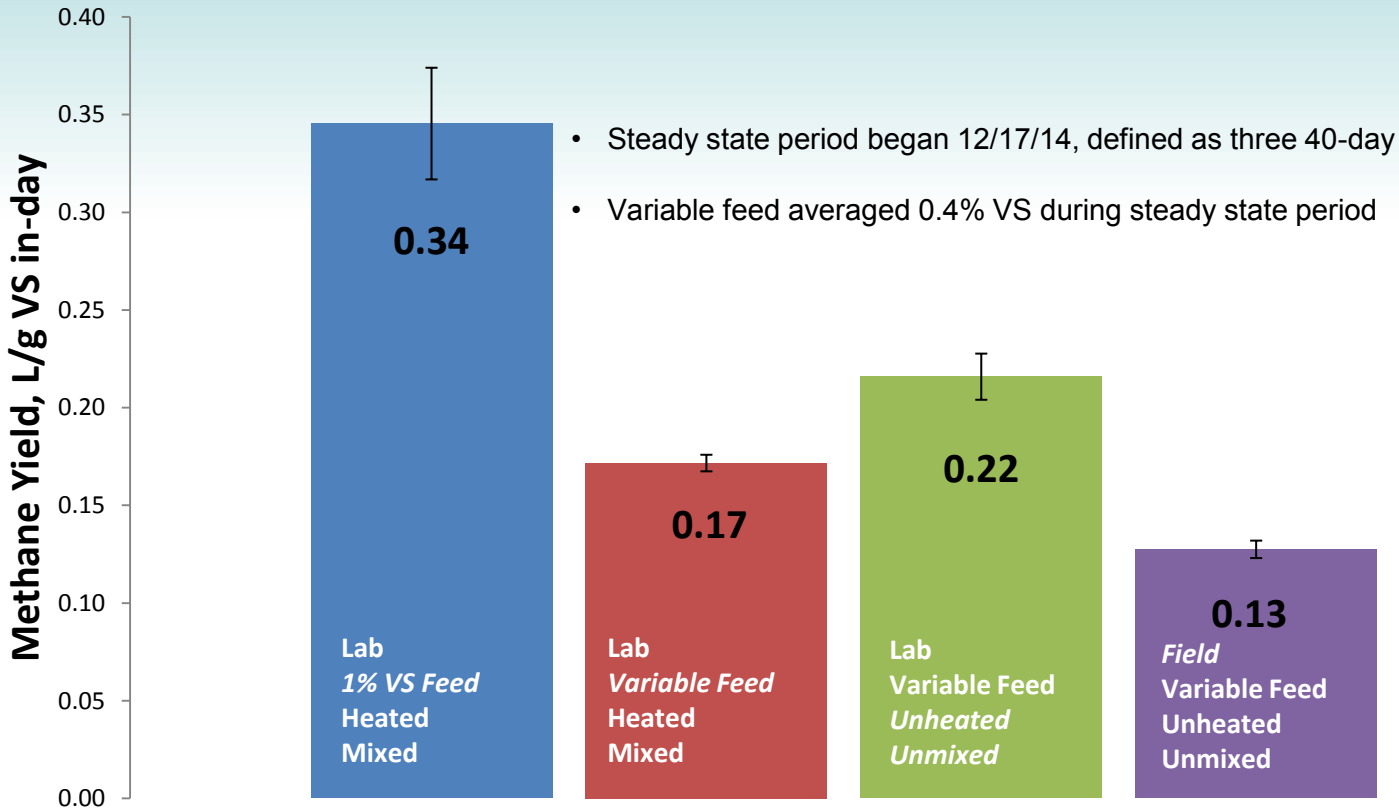
Results: *Impact of outdoor temperature on digester gas production*



Seasonal changes in temperature have large impacts on gas production
Sudden drops in temperature can affect digester health

Milestone 2.5 Continued

Results: ***Influence of organic loading rate, temperature, and mixing on methane yield (L/g VS in – day) during steady state period***

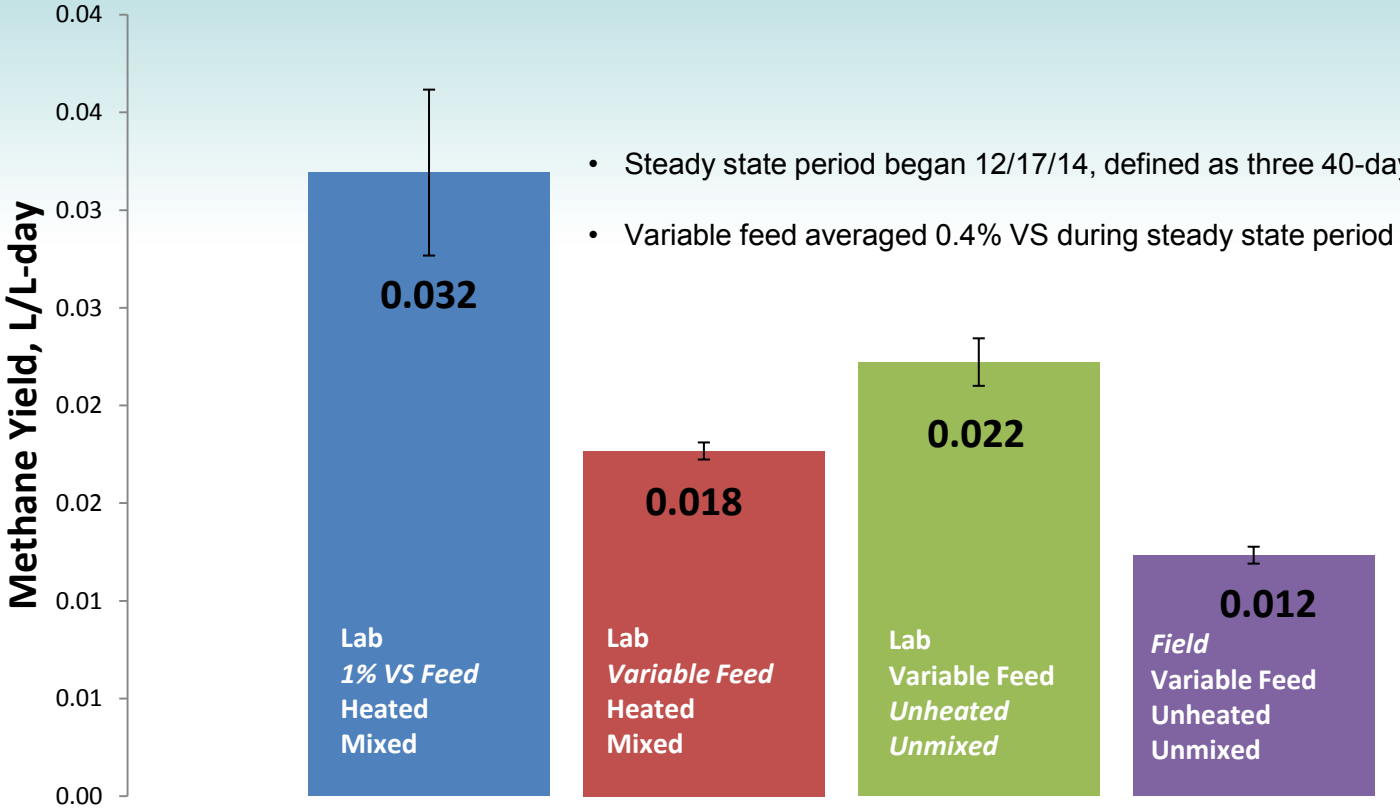


- Steady state period began 12/17/14, defined as three 40-day residence times from startup
- Variable feed averaged 0.4% VS during steady state period

Consistent organic load is beneficial for methane yield
Unmixed digesters perform better than mixed in low organic loading

Milestone 2.5 Continued

Results: ***Influence of organic loading rate, temperature, and mixing on volumetric methane yield (L/L - day) during steady state period***

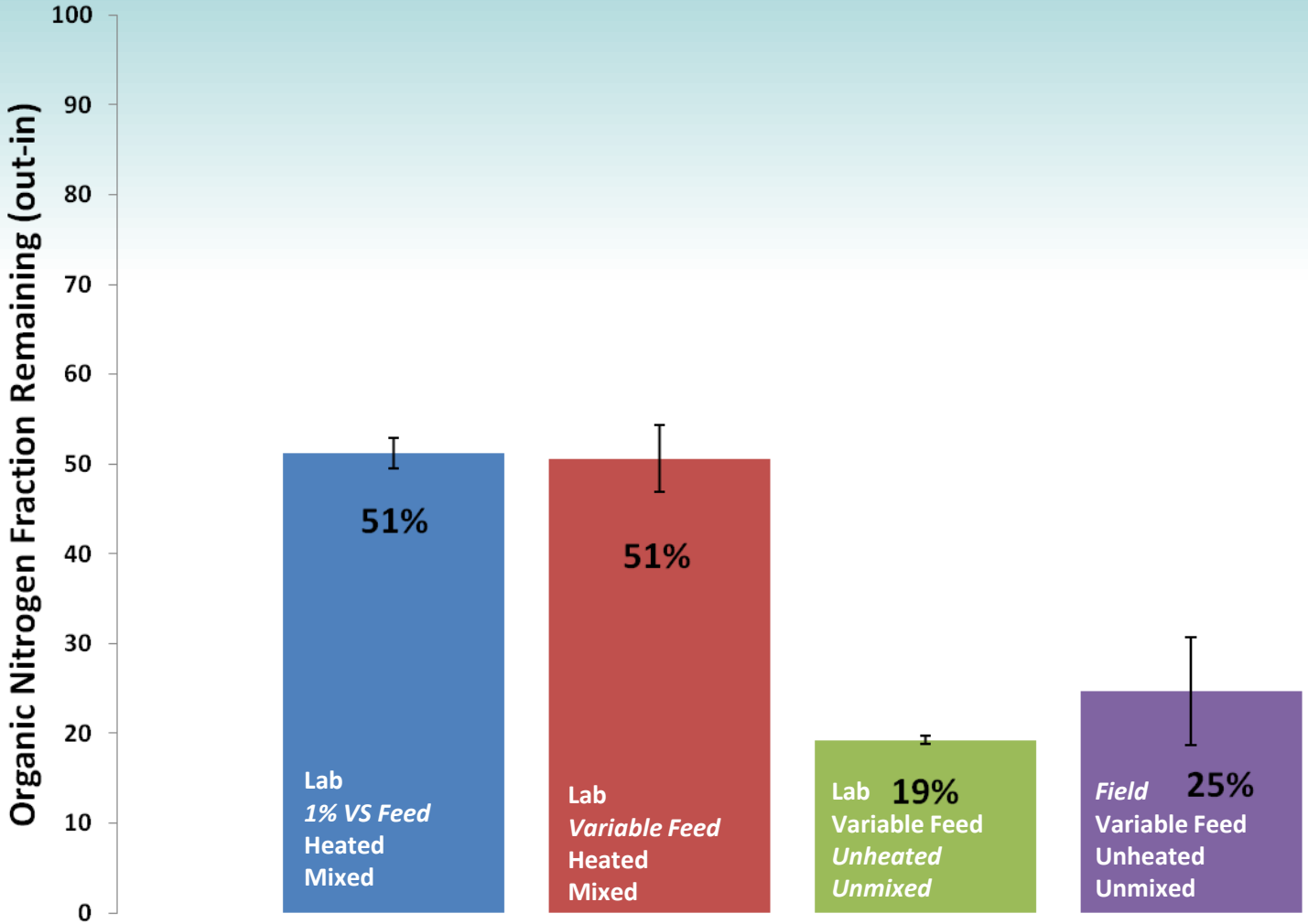


- Steady state period began 12/17/14, defined as three 40-day residence times from startup
- Variable feed averaged 0.4% VS during steady state period

Low organic loading results in poor volumetric yield, but high yield per gram VS in

Milestone 2.5 Continued

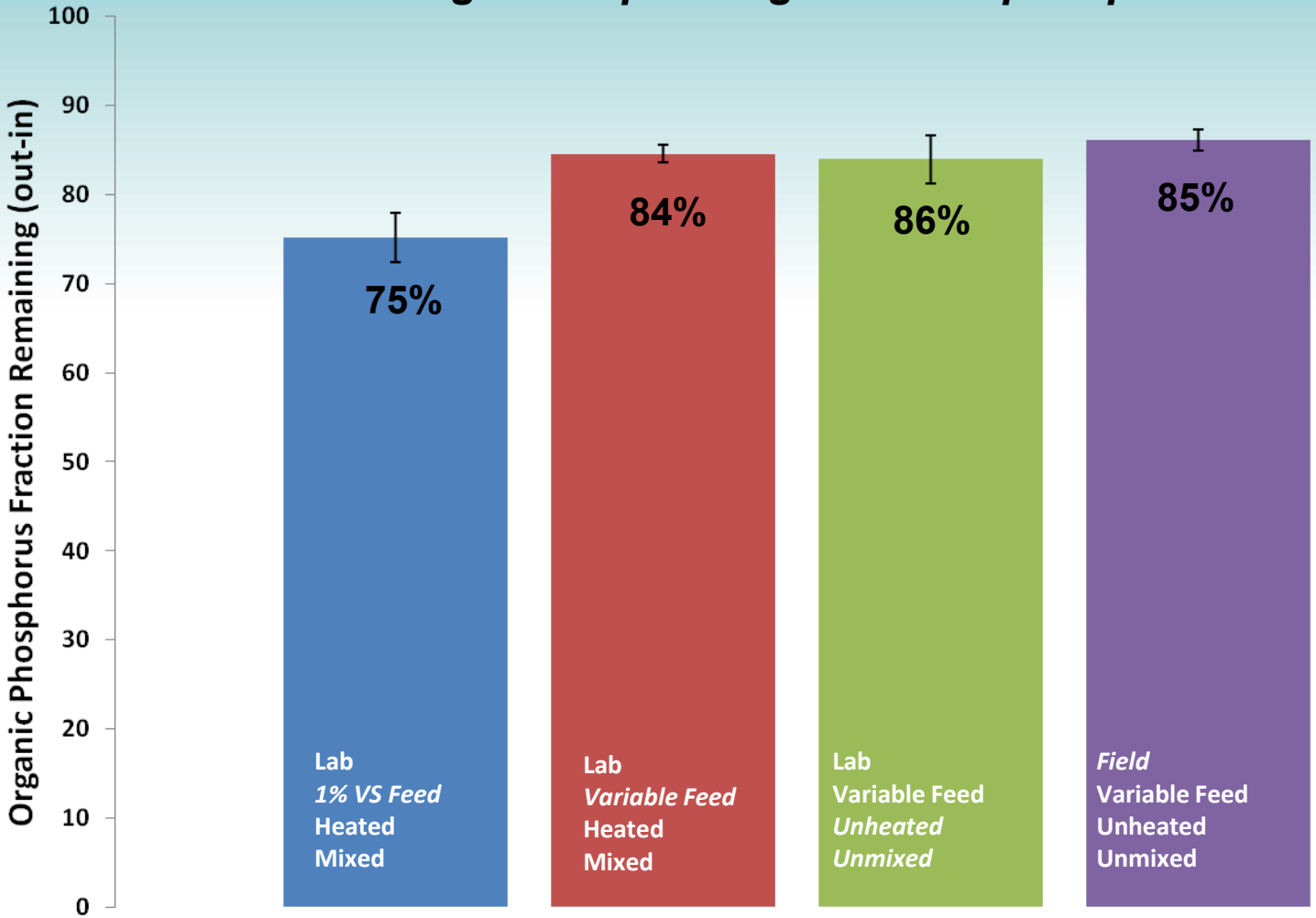
Results: ***Influence of digester operating mode on nitrogen solubilization***



Better nutrient solubilization in unmixed digesters due to longer solids retention time

Milestone 2.5 Continued

Results: ***Influence of digester operating mode on phosphorus solubilization***

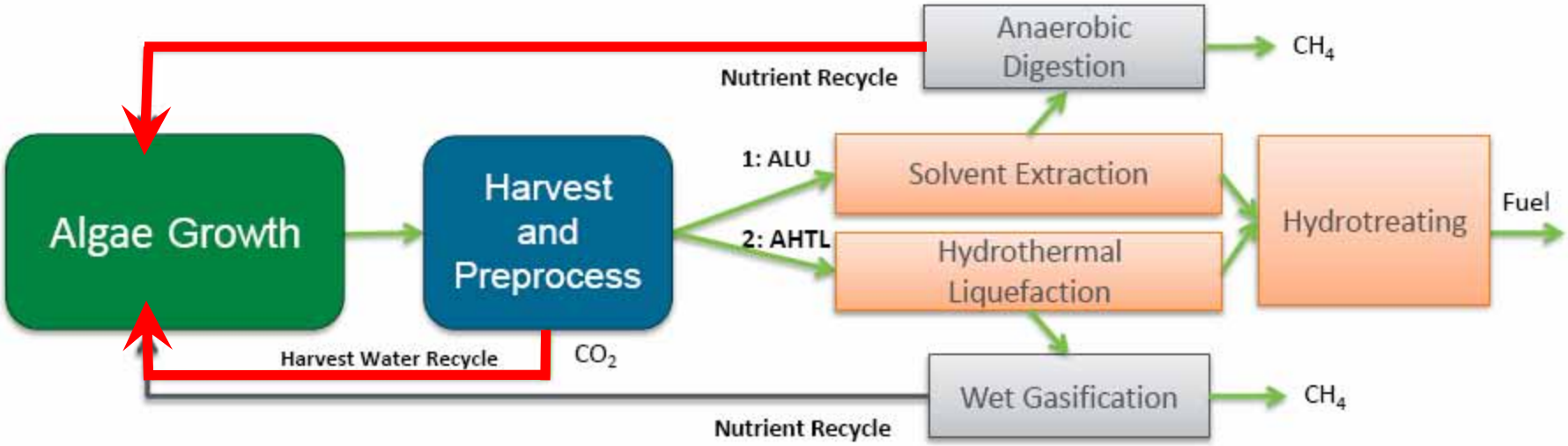


Phosphorus is more difficult to solubilize than nitrogen
Minimal effect of digester operating mode on solubilization

4 – Relevance

BETO Multi-Year Program Plan topics addressed:

- *R.9.2 Sustainability*
- *R.9.2.1 Pathway & Cross-Pathway Analysis*
- *R.9.2.1.1.8 Environmental - Algae*
- *R.9.2.1.3.8 Systemic Sustainability – Algae*
- *R.9.2.2 Sustainability Standards & Adoption*



Fishman, DOE, 2012

5 – Future Work

Task 1: Integrated water & nutrient recycling

Issue: Organic compounds from WW, digestate, from algae (allelopathy).

- **Pilot:** Operate with algae digestate (with WW digestate & fertilizer as needed for max. growth) winter vs. summer with controls. 10-50% imported WW. **Monitor:** productivity; nutrient recovery; buildup of sediment, soluble C & salt; bioflocculation; costs; coagulant use, etc.
- **Lab:** Simulate field operation without the environmental variability=better for inhibition detection. **Determine:** productivity vs. number of water and biomass recycles. **Monitor:** as above

Task 2: Digestion for nutrient recycling & power

Pilot: Operate digesters with algae slurry through 4 seasons. **Monitor:** CH_4 yield; nutrient solubilization; soluble C & salt; costs; etc.

- Long-term digester operation to monitor affect of algae production/thickening on digester performance.

- **Lab:** Accumulation of non-digestible algae and other compounds due to biomass recycling
- Determine polymer effects on digestion
- Same but for a range of organic loads and residence times

Task 3: Harvesting

Issue: Bioflocculation efficiency needs to be higher for biofuels

- Biomass recycling to select for settling biomass
- Bacteria biomass flocs from WW used for adsorption of algae. How many reuses?
- Chemical coagulant backup: What's the cost?

Task 4: Nutrient Recycling via HTL

Issue: Overcoming toxicity with better TEA/LCA results

- HTL aqueous treatment to allow recycling
  - Coordinate with other BETO efforts
  - Aerobic – anaerobic pretreatment
  - Anaerobic – aerobic pretreatment
  - Anaerobic methanogenesis for plant power
  - Compare to CHG (obtain from PNNL)
  - Validate with algae growth/inhibition studies

Task 5: Water Co-Product for Revenue

Wastewater treatment revenue to support biofuel production in near-term

- **Issue:** Removing P with dearth of N
  - **Field** in conjunction with Task 1: Assimilate by adding missing N
  - **Lab:** Precipitate with pH (photosynthetic + chemical)

Task 6: TEA and LCA studies

Summary

- Some key elements of sustainable algae biofuel production are the following:
  - Efficient recycling of water, nutrients, and carbon
  - Low-cost, low-input biofloccuation and sedimentation harvesting
  - Renewable electricity production from biogas to offset other GHG-generating inputs to the overall algae biofuel process.
- We will generate basic information and model parameter values and demonstrate integrated cultivation recycling in lab.
- We will attempt to recreate and confirm lab results in the pilot facility.
- LCA and TEA analyses will be updated based on the results.

Thank you