Biochemical Process Pilot-Scale Integration Project

March 7, 2017
Biochemical Conversion Area

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NREL

Goal

Goal**:**
Produce pilot scale **integrated performance data for techno-economic analysis (TEA)** and **resolve process uncertainties and scale up issues** using a **well-maintained and relevant pilot plant** to support BETO's mission to deploy cost-effective biofuels production technology.

Aerial View of the Pilot Plant's Two High Bays

See additional slides for more information on pilot plant equipment and capabilities.

Outcomes:

- Generate pilot-scale integrated performance data producing a modeled cost meeting BETO's 2022 biofuel cost target of $3.00/GGE.
- Provide a ready-to-use pilot plant for BETO's and industry's use.

Relevance:

- Identify and solve scale-up issues while still manageable before deployment.
- Provides facility for initial scale up and process integration testing to de-risk technology.

Quad Chart

Timeline

- Project start date: FY15
- Project end date: FY17
- Percent complete: ~80%

Barriers

- Ct-J: Process integration
- It-A: End-to-End Process Integration
- Project addresses MYPP goal to verify modeled fuel production cost of $3/GGE by 2022 using pilot-scale performance data (page 2-66).

Budget (MM$)

Total FY12–FY14 Costs	FY15 Costs	FY16 Costs	Total Planned Funding FY17
6.42	1.94	1.89	2.00

Subcontracts

- Aeration studies: Benz Technology, Katzen International, Genomatica
- Pilot plant maintenance

Other collaborations

- Membranes: Membrane, Science, Engineering and Technology Center (see additional slides for information on this center)

Project Overview

We solve scale-up problems to reduce technical risk of successful technology deployment since:

- Many issues that manifest at the pilot scale are usually not seen during early process development work.
- It is absolutely critical to address these issues during scale-up before technology is taken to demonstration or commercial scale.

Project Organization

Pilot Scale Integration (PSI) Project

Pilot Scale Integration R&D

- Perform pilot-scale applied R&D to generate performance data for TEA.
- Produce process-relevant scale-up data to improve process understanding or reduce process uncertainty.

Pilot Plant Support

- Maintain/repair/calibrate process equipment, instrument, and utility systems.
- Evolve in-house equipment/capabilities or acquire new equipment needed to perform the 2022 integrated pilot-scale demonstration runs or work for industry.

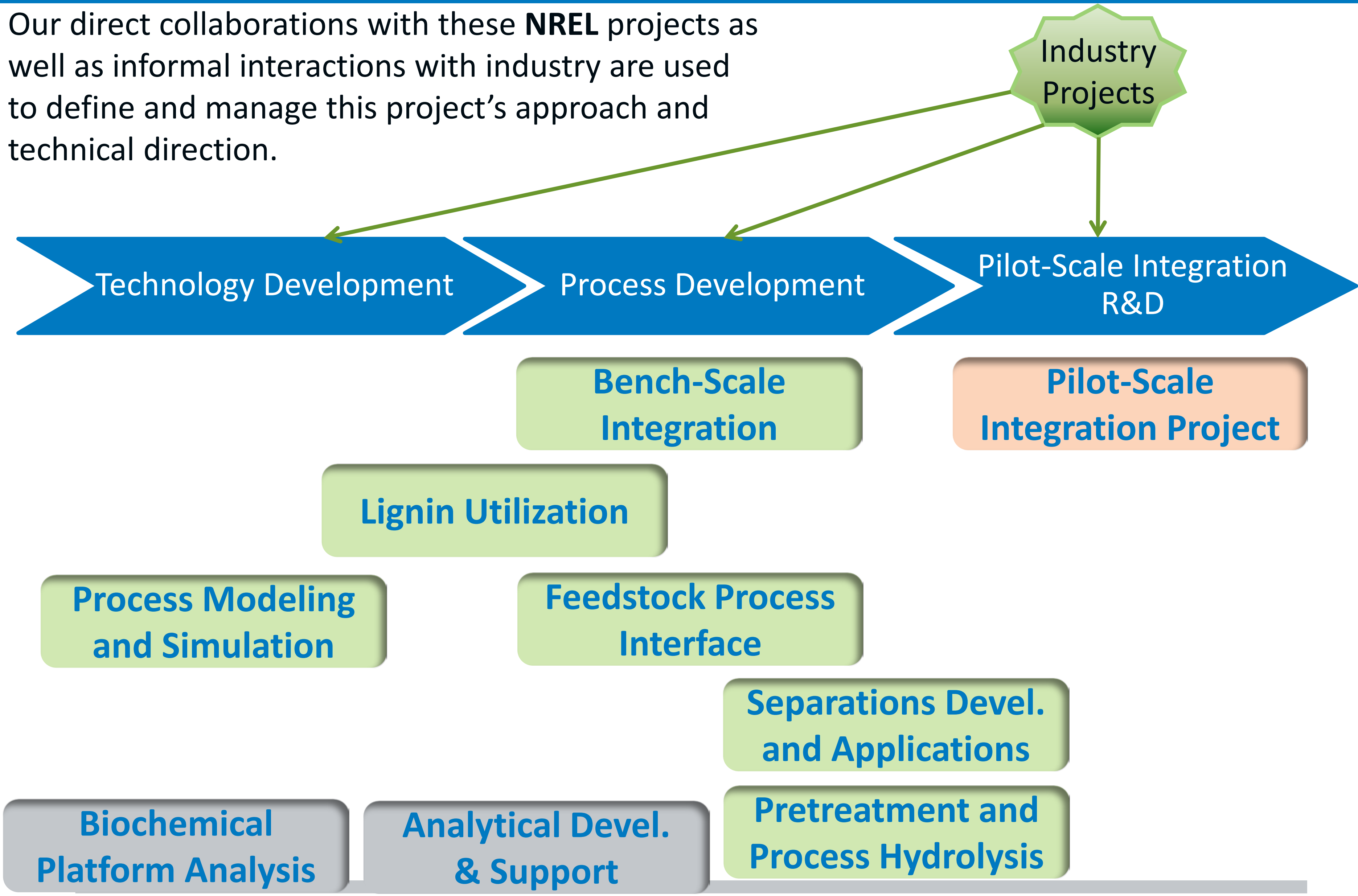
Biochemical Conversion Projects in the NREL Portfolio

Enabling/fundamental technologies	Synthesis and upgrading technology	Process development	Process integration, scale-up, verification

- Biochemical Process Modeling and Simulation
- Targeted Microbial Development
- Feedstock-Process Interface
- Pretreatment and Process Hydrolysis
- Biological Upgrading of Sugars
- Enzyme Engineering and Optimization
- Bench-Scale Process Integration
- Biological Lignin Depolymerization
- Catalytic Upgrading of Biochem. Intermediates
- Separations Development and Application
- Lignin Utilization
- Pilot-Scale Integration
- Analytical Development and Support
- Biochemical Platform Analysis

Management Approach

Our direct collaborations with these **NREL** projects as well as informal interactions with industry are used to define and manage this project's approach and technical direction.



Approach (Technical)—Pilot-Scale R&D

Objective

- Investigate scale-up issues affecting process performance, cost, and reliability and generate pilot-scale data for TEA.

Technical Approach

- Identify process relevant issues/problems in collaboration with other projects.
- Conduct statistically designed experiments/engineering studies that generate performance information.
- Take project management-driven approach using milestones, performance metrics, and heavy reliance on go/no go decision points.

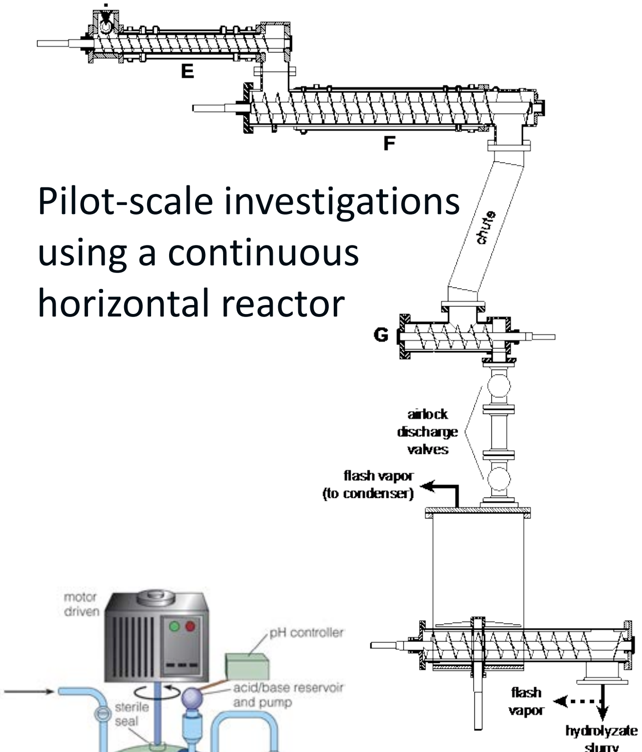
Challenges

- Lack of publicly available information
- Difficult pilot-scale operations

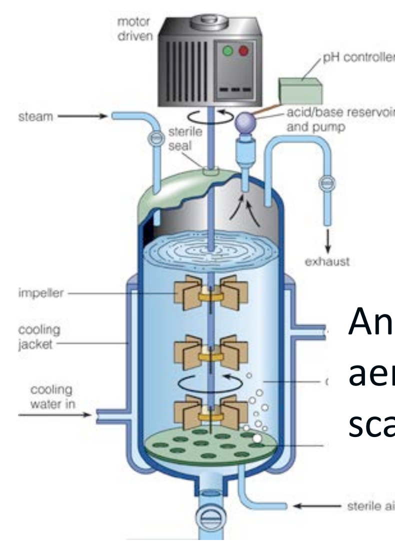
Critical Success Factors

- Near-term: Identify lower cost aeration strategy; Demonstrate successful alkaline pretreatment process
- Longer-term: Produce integrated pilot-scale performance data meeting BETO’s 2022 cost target.

Primary FY15/16 Activities



Pilot-scale investigations using a continuous horizontal reactor



Analyzing cost of aerating large-scale bioreactors

Approach (Technical)—Pilot Plant Support

Objective 1

- Maintain pilot plant functionality.

Technical Approach

- Perform routine maintenance, repair, and calibration activities.
- Maintain and improve plant safety and ergonomics.
- Maintain/upgrade SCADA system.
- Document/track activities.

Challenges

- Unique, one-of-kind equipment
- High repair cost
- Material handling difficulties

Critical Success Factor

- Equipment/systems available when needed for R&D and industrial client work.

Pilot Plant Management

- Utilities/ Equipment
  - Maintenance
  - Repair/upgrades
- Safety
  - Process hazard analysis
  - Readiness reviews
  - Key safety devices
  - Management of change
- SCADA*
  - System/network upkeep
  - Instrument calibration
  - Hard/software upgrades

*Supervisory control and data acquisition

Approach (Technical)—Pilot Plant Support

Objective 2

- Add new pilot-scale capabilities needed for process demonstration work.

Technical Approach

- Identify new needs in collaboration with other BETO projects and industry partners:
  - Yearly brainstorming session to identify needed equipment and capabilities
  - Informal interaction with industry partners
- Define and implement milestones/key decision points to manage acquisition and installation activities.

Challenges

- Sufficient resources to acquire new equipment.
- Long implementation time for equipment design, fabrication and installation.

Critical Success Factors

- Capabilities available to perform 2022 pilot-scale runs.
- Ability to support industrial projects and collaborations.

Primary FY16/17 Activities*

Acquisition, installation, and testing of new separation and concentrating equipment

Disc stack centrifuge

Feed system improvements

Feed hopper and weigh belt

***New focus beginning in FY16 after cancellation of the FY17 biochemical demonstration runs**

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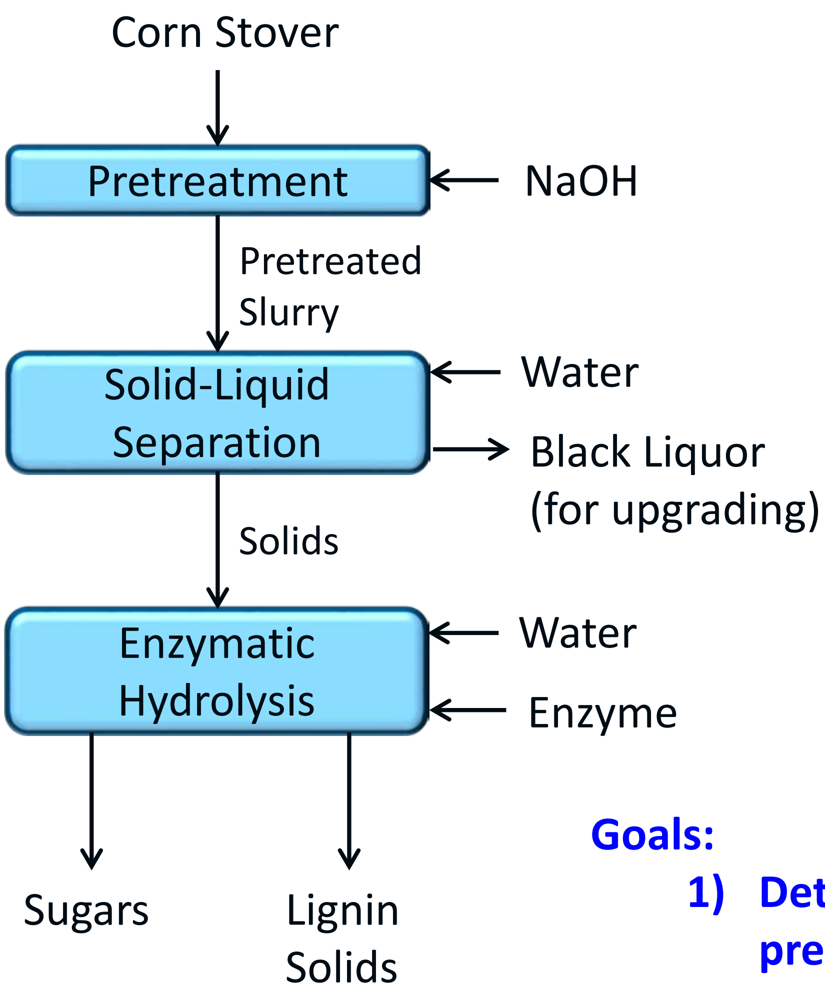
Technical Accomplishments
Pilot-Scale Integration R&D

- Pilot-scale alkaline pretreatment
- Pretreatment reactor scale-up study
- Aeration of large-scale bioreactors

Pilot-Scale Alkaline Pretreatment



Pilot-scale alkaline pretreatment was researched as one method of obtaining soluble lignin for valorization (with Lignin Utilization project).

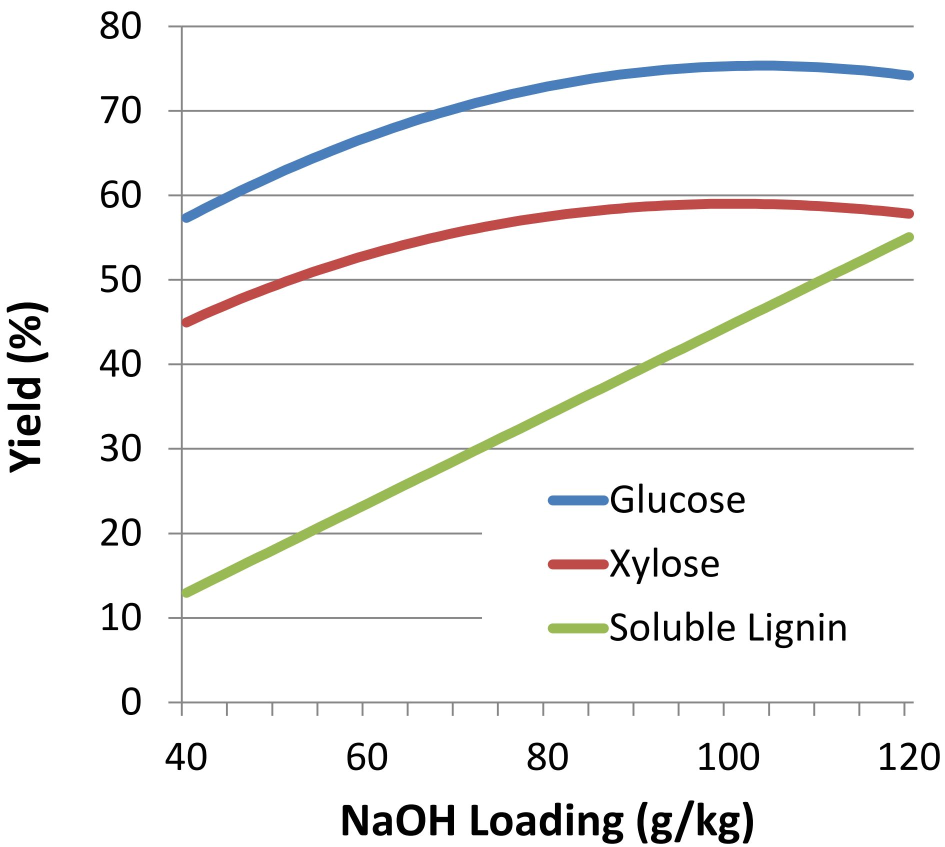
- Process produces a stream (black liquor) containing high molecular weight lignin and low molecular weight aromatic monomer fragments suitable for valorization.
- This work also assessed impact of processing conditions on downstream operations (separations and enzymatic hydrolysis).

Goals:

1) **Determine performance in a 500 kg/d continuous pretreatment reactor.**
2) **Explore downstream processing (solid-liquid separation) and enzymatic hydrolysis yields.**
3) **Provide data for TEA.**

Alkaline Pretreatment Modeling Results

Monomeric glucose/xylose and soluble lignin yields-based components in the raw feedstock



Empirical modeling results displayed above can be used along with TEA to find conditions that minimize cost.

Additional Results

- Temperature and residence time had only a minor effect on yields compared to the effect of NaOH loading.
- Black liquor recovery from these slurries was difficult as centrifugation and filtration processes did not work well and some sugars were loss to the black liquor stream.

Conclusions

- Process development is needed to achieve effective separations.
- Better enzymes might further improve performance.

Recommendations

- Pilot-scale work on alkaline pretreatment on hold until issues associated with performance and economic feasibility (separations, sugar yield/losses) are addressed.

Pretreatment Reactor Scale-Up Study

Can performance in a small bench-scale reactor system be used to predict pretreatment performance in pilot-scale or even commercial-scale continuous reactors?

Collaboration with NREL Pretreatment and Process Hydrolysis, Feedstock Process Interface, and Analytical Development and Support projects.

- Small-scale reactors can be used to quickly and cheaply screen feedstocks and pretreatment conditions.
- Significant differences exist at different reactor scales
  - Heat and mass transfer
  - Mechanical processing (e.g., mechanical grinding, steam explosion)
  - Rheological properties due to different solids loading
  - Residence time distributions.

Bench-Scale Extraction (BSE) System

- Fixed bed, flow-through system
- Three dry grams solids loading
- Indirect conductive heating
- Sixteen conditions/run possible

Goal: Determine if a bench-scale screening system (at right) can be used to predict performance in larger reactor systems (i.e., identify optimum pretreatment conditions).

Other Reactors Tested

- 1-L Batch, steam heated
- 4-L Batch, steam heated, steam explosion
- 500 kg/d continuous, steam heated, steam explosion

Pretreatment Reactor Scale-Up Study Results

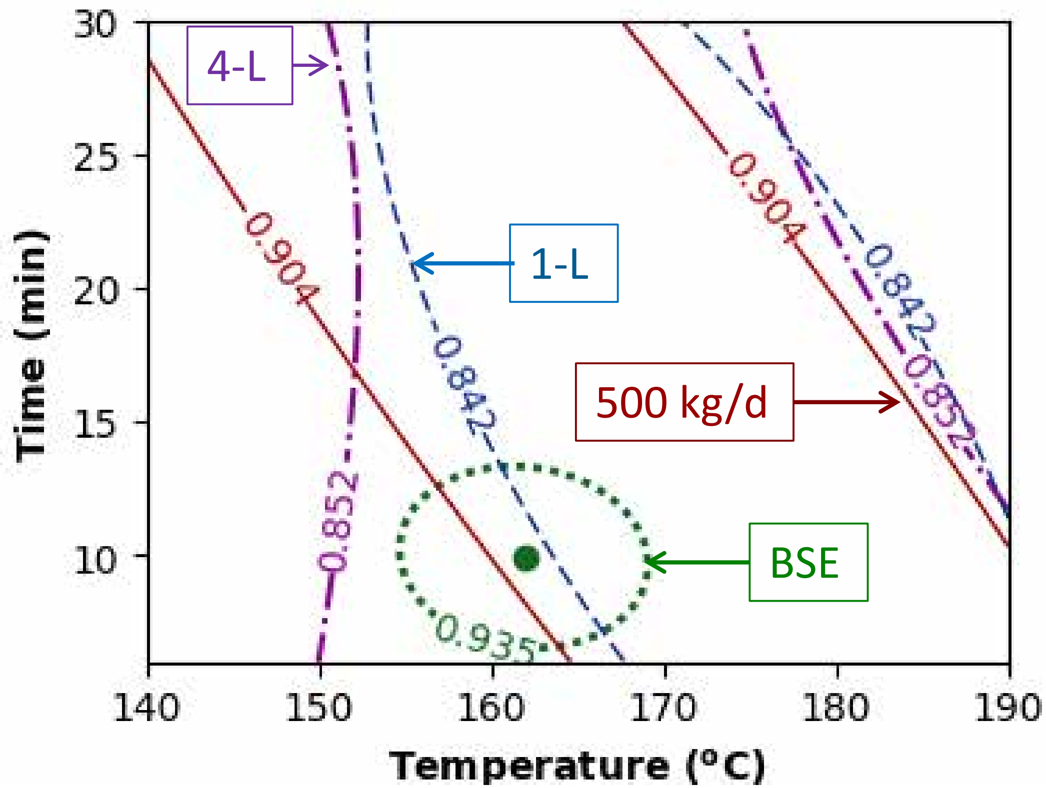
Results

- Except for the BSE reactor, all other reactors displayed a wide range of operating conditions for achieving near optimal performance.
- The optimum total xylose yield predicted by the BSE reactor system is within a two standard deviation envelope of the optimum for the other reactor systems.

Conclusions

- Optimum yields in larger reactor systems can be predicted using results from the small flow-through batch reactor (BSE).
- But optimum operating conditions cannot be predicted in the larger reactor systems.

Empirical Modeling Results for Total Xylose Yield for Each Reactor System



Plot shows the predicted maximum total xylose yield (96%) for the BSE reactor (green point) and yields at two standard deviations from the optimum for all reactor systems.

Lischeske, et al. 2016 “Assessing pretreatment reactor scaling through empirical analysis.” Biotechnol. Biofuels. 9:213.

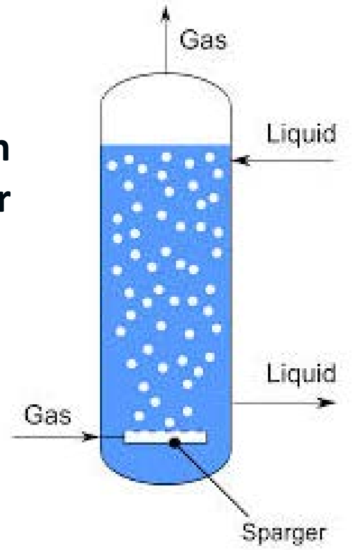
Aeration of Large-Scale Bioreactors

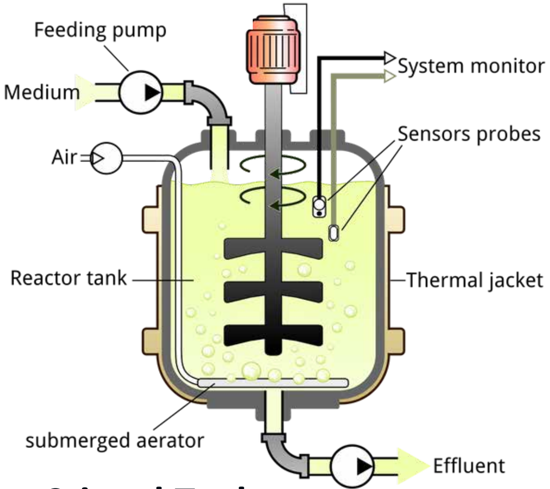
Joint Pilot-Scale Integration, Biochemical Platform Analysis and Process Modeling and Simulation project to better understand aeration in large scale bioreactors.

- Good aeration is essential for biological sugars-to-near hydrocarbon (e.g., triglycerides [TAG]) production.
- Molecular oxygen (O_2) is only sparingly soluble in aqueous media and must be continuously provided to maintain active aerobic culture.
- Volumetric productivity is highly dependent on (and for scale-up limited by) the rate of oxygen mass transfer (OTR).

Goal: Increase confidence in aerobic reactor design and costing (TEA) in consultation with various subcontractors (Genomatica, Katzen International, and Benz Technology).

Bubble Column Reactor (BCR)

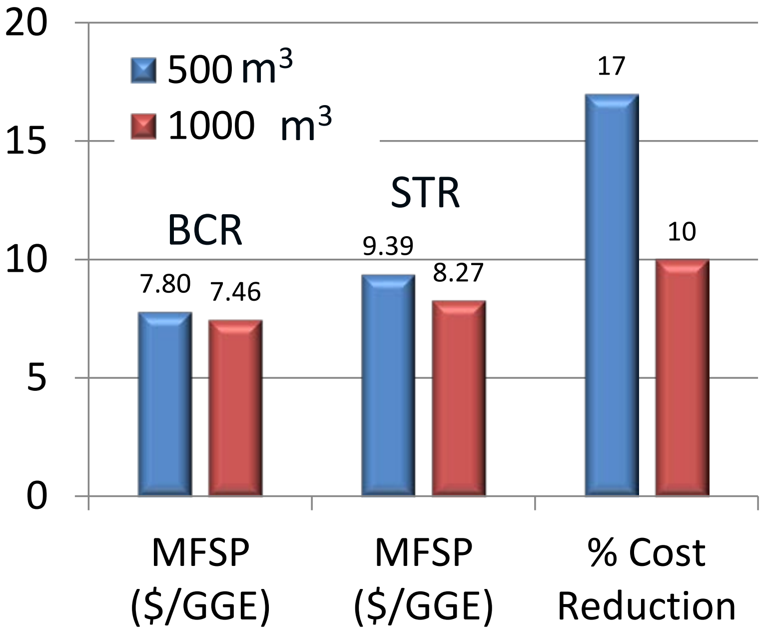




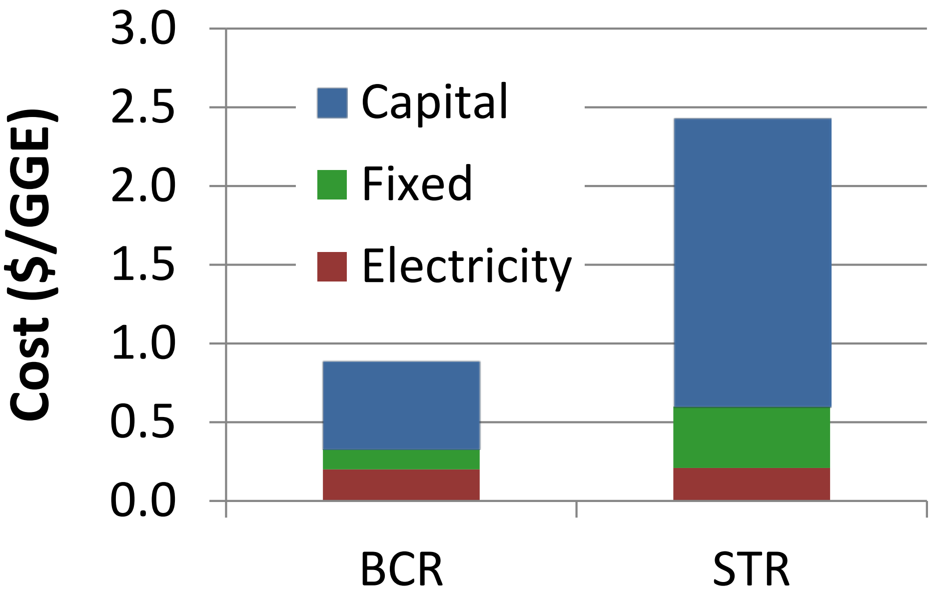
Stirred Tank Reactor (STR)

Proper Aerated Bioreactor Design Can Reduce Costs

Modeled* Minimum Fuel Selling Price (MFSP) and Cost Reduction Achieved by BCR



Cost Contributions for the Aerobic System



Results

- BCRs have good potential to reduce aerobic production cost compared to STRs.
- Process broth viscosity and required OTR must be in BCR operable range.

Recommendation

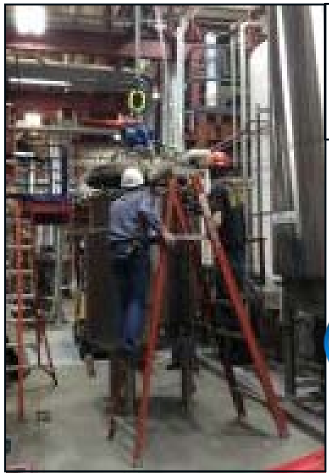
- Develop appropriate pilot-scale aerobic bioreactor design if down-selection occurs on an aerobic biological upgrading process/pathway.

*NREL TEA model for lipid production process with no coproducts

Accomplishments
Pilot Plant Support

- Pilot plant upkeep
- New capabilities
- Plant usage statistics

Pilot Plant Upkeep—Maintaining Plant Operability

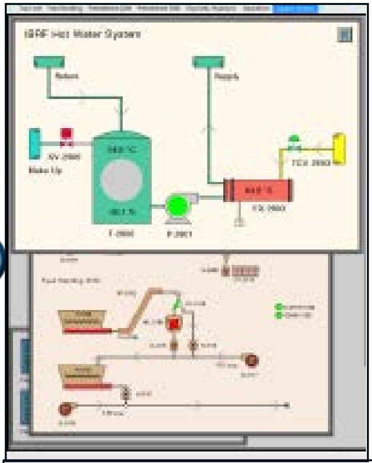


Agitator seal replacement on a 1,500-L fermentor

Equipment maintenance and repair

Major FY16/17 Activities

SCADA system



Upgrading hardware and replacing the Human Machine Interface software

Electrical and utility system repairs

Fermentor control cabinets (8 each) electrical upgrades

Re-tubing the 500-psi boiler

New Capabilities—Installing/Testing New Equipment

FY16/17 focus on acquiring, installing, and testing new equipment for pilot-scale process development of biofuel/bioproduct production technologies

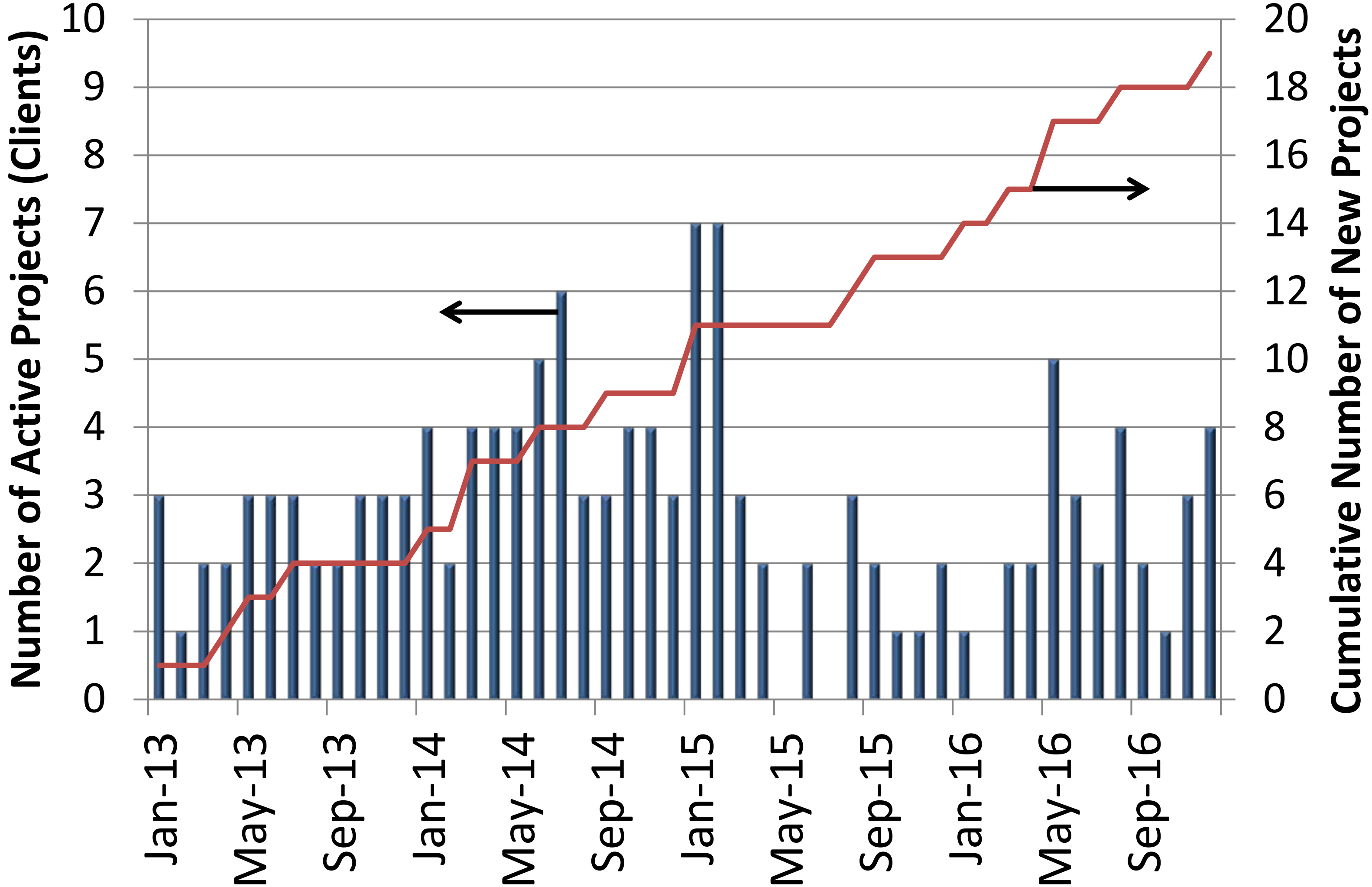
- Rotary Drum Filter—residual (lignin) solids separation from enzymatic hydrolysates
- Disk Stack Centrifuge—cells recovery for cell recycle and bioproduct production technologies
- Force Circulation Evaporator—production of concentrated sugar solutions for aerobic fed-batch fermentations

Acquired and reconditioned a used rotary drum filter; testing on enzymatic hydrolysates is under way.

Existing atmospheric pressure evaporator is being converted to vacuum operation to reduce sugar degradation losses.

Performance testing of a new disk stack centrifuge is under way.

Monthly Use of the Pilot Plant by Industry



Relevance—Pilot-Scale Integration R&D

We investigate process integration and scale-up issues with large or unknown cost impacts and produce integrated pilot-scale performance data for TEA.

This work directly supports BETO's mission to decrease biochemical-based biofuels production cost by

- Generating process relevant research results useful to other BETO program work and to industry.
- Identifying and solving scale-up issues with knowledgeable technical resources and at a cost that is still manageable before deployment to commercial scale.
- Producing data from pilot-scale, integrated demonstration runs for TEA that meets BETO's 2022 $3.00/GGE modeled biofuel production cost target.

DOE/NREL Pilot Plant

Reducing Risk

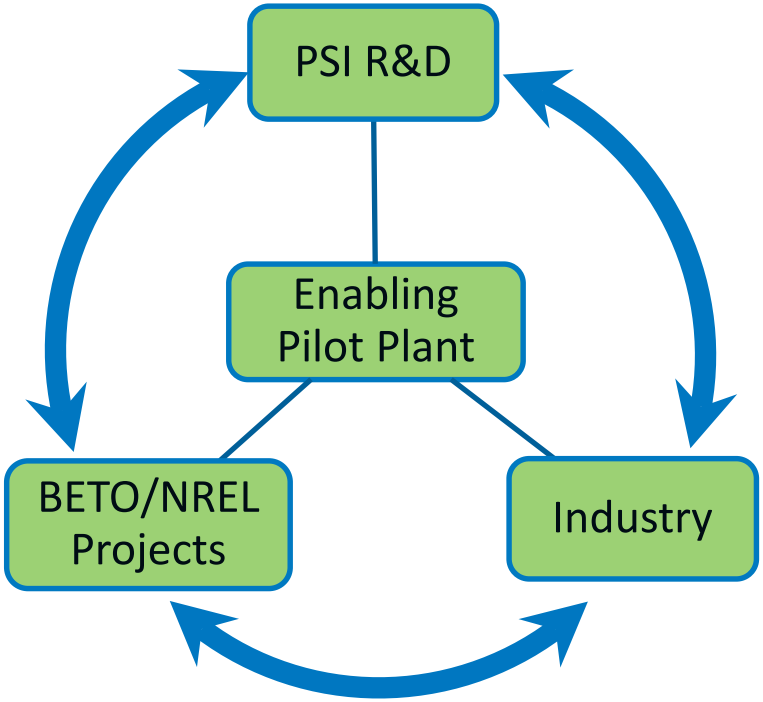
Commercial Biorefinery

Relevance—Pilot Plant Support

We maintain the DOE/NREL biochemical pilot plant to support BETO and industry efforts to commercialize biofuel production technology.

The pilot plant is routinely used for BETO and industry projects. It allows industry to avoid the significant cost of building its own pilot facility.

- Industry can test technology and generate process relevant data and materials, thus reducing its commercialization risk.
- We also freely supply small quantities of process materials produced in the pilot plant to many companies and universities for their technology development efforts (26 shipments made in FY15/16).

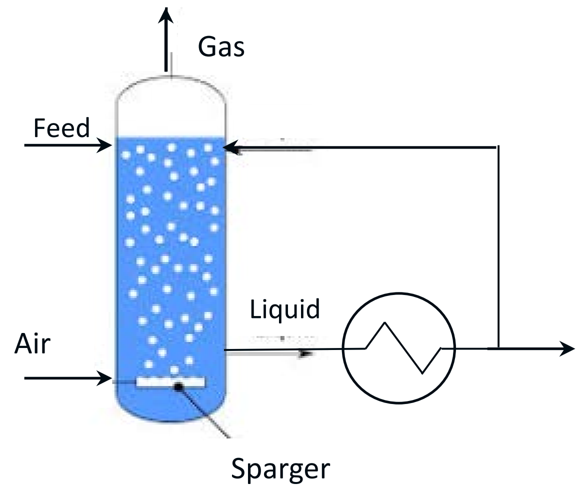


Near Term Future Work (FY17/18)

Pilot Scale Integration R&D

- Code dynamic fermentation model in a more user friendly software.
- Develop tool to measure depth of coke on pretreatment reactor walls as a precursor to future work (go/no-go).
- Develop scale down testing capabilities.

External heating loop testing

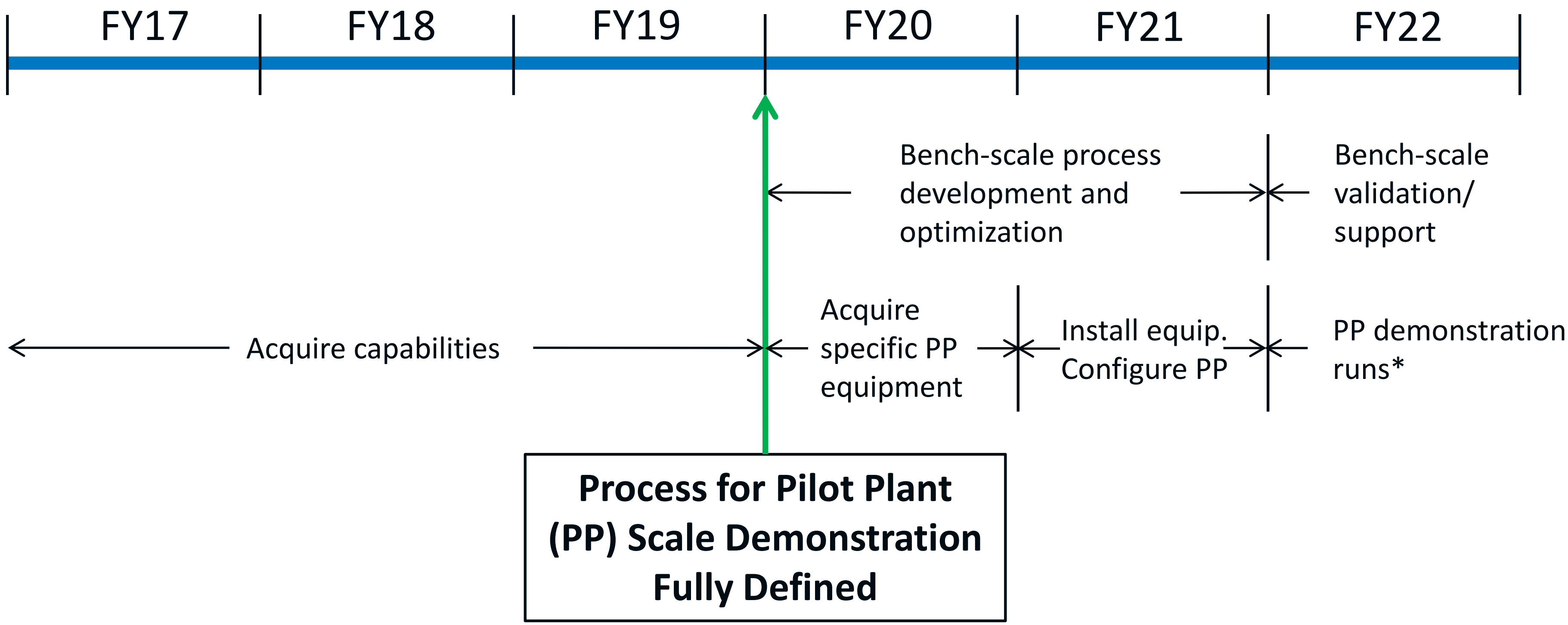


Pilot Plant Support

- Maintain pilot plant equipment/utilities.
- Install/test new separations equipment.
- Identify new equipment needs.
- Design and install (go/no-go) new pilot plant feed process systems.

Additional slides contain more details and Gantt Charts

Future Work—Moving Toward 2022 Demonstration Runs



Process elements to be defined/developed

- Feedstock
- Pretreatment and enzymatic hydrolysis strategy
- Final/intermediate product and production strain
- Product recovery/conversion
- Lignin coproduct process

*Portions of this effort may be in collaboration with other national laboratories or industry partners.

Summary—Pilot-Scale Integration R&D

Technology Development → Process Development → Pilot-Scale Integration R&D

Other NREL Projects → This Project

This project is furthest down the R&D process development pipeline.

Earlier TRL projects identify R&D needs that are better performed at pilot scale, so our work

- Addresses issues/questions that affect integrated performance/cost.
- Identifies and resolves scale-up issues that only manifest at pilot scale.

Recent project work has

- Demonstrated lower cost aeration is achieved in bubble column reactors.
- Showed alkaline pretreatment is effective but with processing challenges (i.e., achieving effective separations).

Near-term future work will continue focus on lowering process cost and uncertainty by

- Developing scaled down fermentation testing capabilities.
- Mitigating pretreatment reactor coking if needed.

Plan on significant shift in project resources beginning in FY20 to demonstration run planning and execution.

Summary—Pilot Plant Support

An important objective is to maintain a functional and relevant biochemical pilot plant for BETO and industry use:

- The pilot plant is used by industry to further its process development goals and to produce process relevant materials.
- The current focus is to identify and acquire new capabilities needed to perform the integrated demonstration runs in 2022 to meet BETO's cost target and general industry pilot-scale needs for de-risking advanced biofuels processes.

Acknowledgments

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Questions

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Additional Slides

Responses to Reviewers' Comments

Reviewers' Comments (from 2015 peer review meeting report):

- The project offers an important function to the program if they want to maintain a relevance to the process designs they make and to be a resource for outside developers to try out processes.
- The NREL pilot plant is an important research vehicle for government and industry parties. Keeping this area up to speed with the latest trend in unit operations and processes is important.
- This is another great industry-supporting project. There is plenty of work performed on in-house projects that supports BETO and MYPP goals, but the availability of the equipment for industry process development work is great. Improvement in pilot-scale testing since the last review is very clear, and integration with techno-economic analysis projects is key.
- The NREL pilot plant has provided valuable scale up data to the biomass conversion community for years. This project has continued to maintain, develop, and deliver on this mission.
- The ability to integrate the various processes at pilot scale is extremely important and invaluable.

PI Response to Reviewer Comments:

- We appreciate the reviewers' comments and their efforts reviewing this project. We will continue to evaluate pilot scale processing needs and acquire capabilities with BETO's support to make the biochemical pilot plant a relevant facility for industry and BETO to develop and test new hydrocarbon fuel production technologies.

Publications

- Kuhn, E., O'Brien, M., Ciesielski, P., Schell, D.J. 2016 "Pilot-Scale Batch Alkaline Pretreatment of Corn Stover." ACS Sustain. Chem. Eng. 4, 944-956.
- Sievers, D.A, Kuhn, E.M., Stickel, J.J., Tucker, M.P., Wolfrum, E.J. 2016 "Online residence time distribution measurement of thermochemical biomass pretreatment reactors." Chem. Eng. Sci. 140, 330-336.
- Schell, D.J.; Dowe, N.; Chapeaux, A.; Nelson, R.S.; Jennings, E.W. 2016 "Accounting for all Sugars Produced during Integrated Production of Ethanol from Lignocellulosic Biomass." Bioresource Technology. 205, 153-158.
- Pannell, T.C., Goud, R.K., Schell, D.J., Borole, A.P. 2016 "Effect of Fed-batch vs. Continuous Mode of Operation on Microbial Fuel Cell Performance Treating Biorefinery Wastewater." Biochemical Engineering Journal. 116, 85-94.
- Lischeske, J.L, Crawford, N.C., Kuhn, E., Nagle, N.J., Schell, D.J., Tucker, M.P., McMillan, J.D., Wolfrum, E.W. 2016 "Assessing pretreatment reactor scaling through empirical analysis." Biotechnol. Biofuels. 9:213.

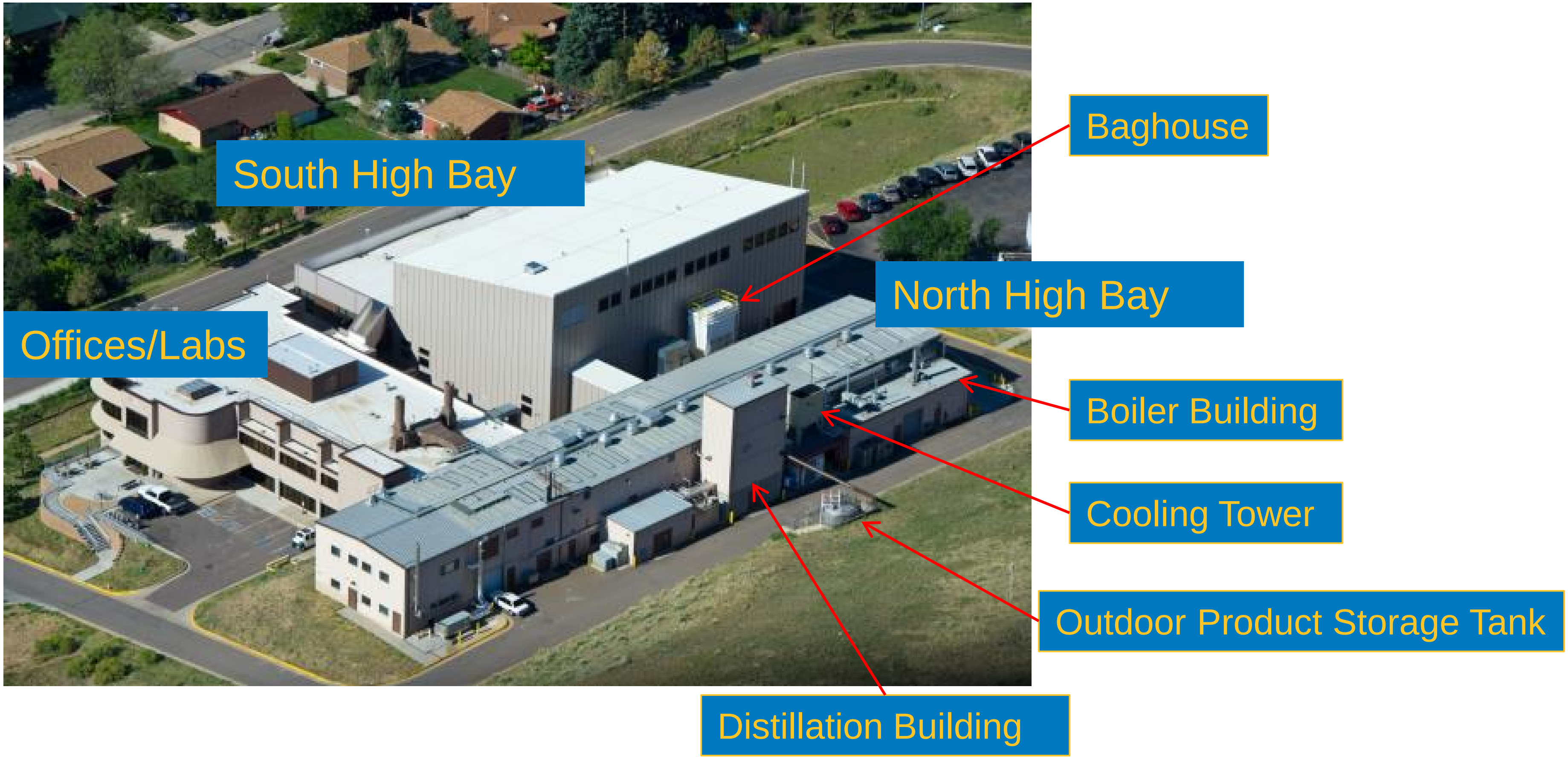
Presentations

- Schell, D.J., Lischeske, J., Shekiro, J., Sievers, D. New pilot plant capabilities for demonstrating biochemical-based technologies for advanced biofuel production from lignocellulosic biomass, Poster, 37th Symposium, San Diego, CA, April 27th-30th, 2015.

- Sievers, D. Residence Time Distribution of a Continuous Pilot Plant Horizontal Pretreatment Reactor at Various Conditions. Oral, 37th Symposium, San Diego, CA, April 27th-30th, 2015.

- Kuhn, E., O'Brien, M., Schell, D.J. Pilot scale alkaline pretreatment in a 1 ton/day continuous, horizontal pretreatment reactor. Poster, 37th Symposium, San Diego, CA, April 27th-30th, 2015.

Biochemical Pilot Plant



Pilot Plant

North High Bay (1994)

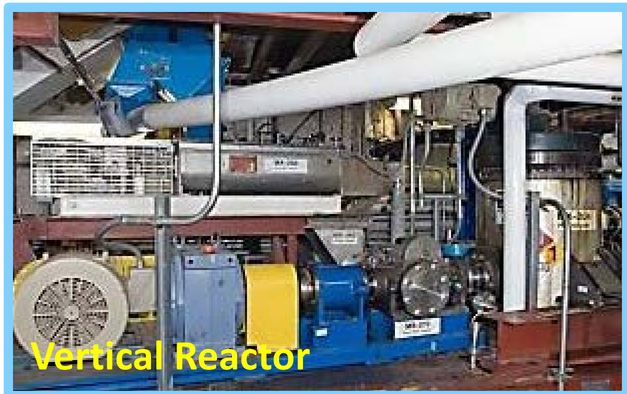
- Integrated 1 ton/d process train
- Feed handling through product separation
- Houses utilities systems

South High Bay (2010)

- Two integrated 0.5–1.0 ton/d process trains
- Feed handling through high solids enzymatic hydrolysis
- Space for expansion

North High Bay Equipment

<u>Pretreatment</u>
- 1.0 ton/d vertical reactor
- 0.2 ton/d horizontal screw reactor
- 160-L batch reactor
- 1-L and 4-L batch reactors



Vertical Reactor

<u>Fermentation</u>
- 30-L seed vessel
- Two 160-L vessels
- Two 1500-L vessels
- Four 9000-L vessels

1500-L Vessels

9000-L Vessels

<u>Separations</u>
- Distillation column (19-sieve trays)
- Perforated 100-L basket centrifuge
- Forced recirculation evaporator

Evaporator

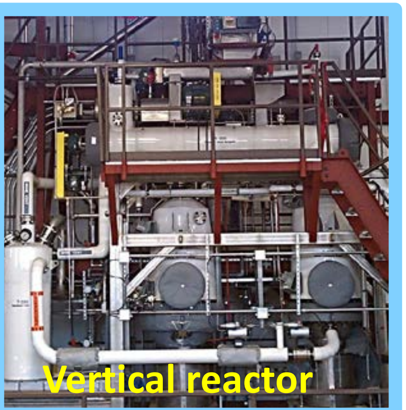
South High Bay Equipment

Feed Handling
- Two knife mills
- Continuous conveyance systems
- Multiple hoppers and weigh belts

Pretreatment
- 1.0 ton/d vertical reactor
- 0.5 ton/d horizontal screw reactor

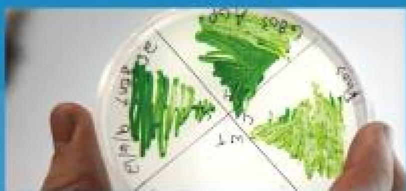
Enzymatic Hydrolysis
- 1900-L paddle reactor
- Four 4000-L paddle reactors

Separations
- Screw presses
- Perforated 450-L basket centrifuge
- Rotary vacuum drum filter



Pilot Plant Utilities Systems

- Steam
  - 500 psi, 3400 lb/h boiler
  - 300 psi, 1200 lb/h backup boiler
  - Distributed in high (up to boiler pressure) and low pressure (35 psi) headers
- Cooling water
- Process water
- Chilled water
- Deionized water
- Hot process water
- Plant compressed air





Information on the Membrane Science, Engineering, and Technology (MAST) Center

NREL is a national laboratory of the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, operated by the Alliance for Sustainable Energy, LLC.

Membrane Science, Engineering & Technology (MAST) Center

NSF Industry-University
Cooperative Research Center Program

Successful Operation Since 1990

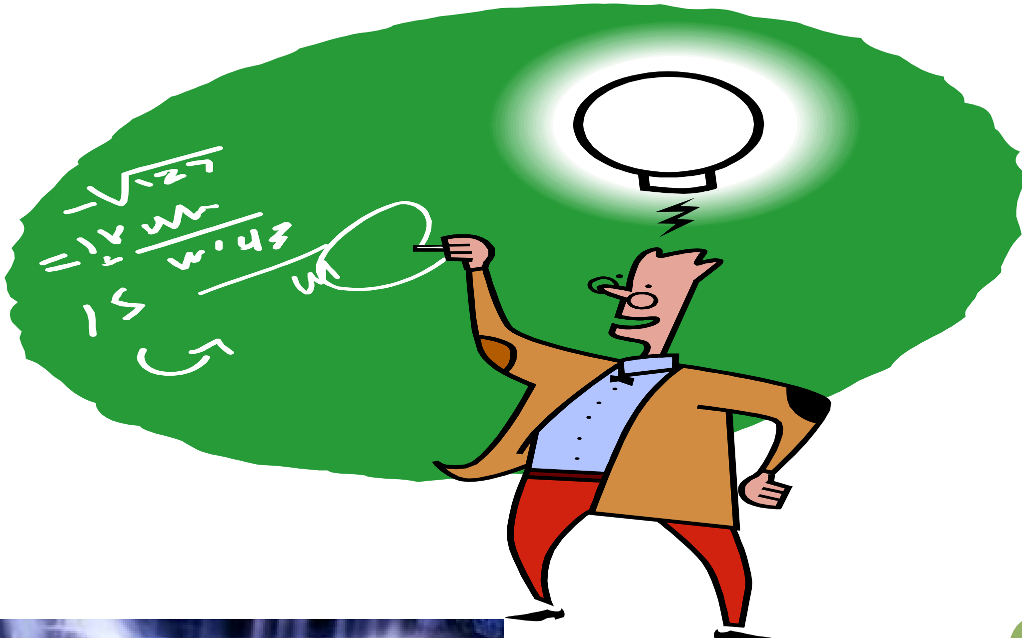
University of Colorado

New Jersey Institute of Technology

University of Arkansas

Fall 2016

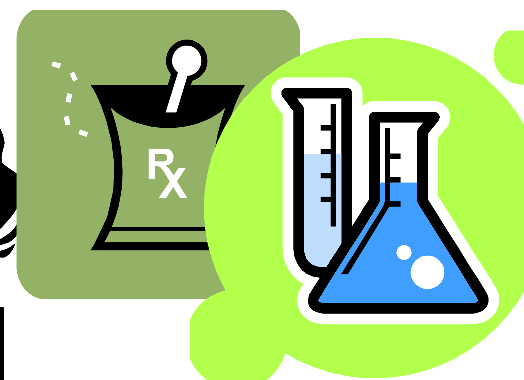
MAST Center Research Program



Fundamental Work in Membrane Materials, Formation & Modification, and Materials & Process Characterization

Water Treatment & Reclamation

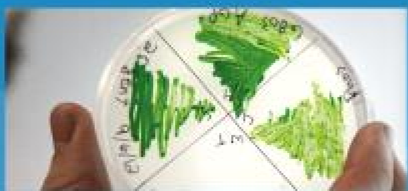
Energy & Chemical

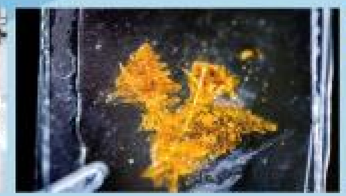


Pharmaceutical & Biomedical

Food & Beverage

Membrane Application Areas Reflect Sponsor Interests!




Technical Accomplishments

Pilot Scale Integration R&D

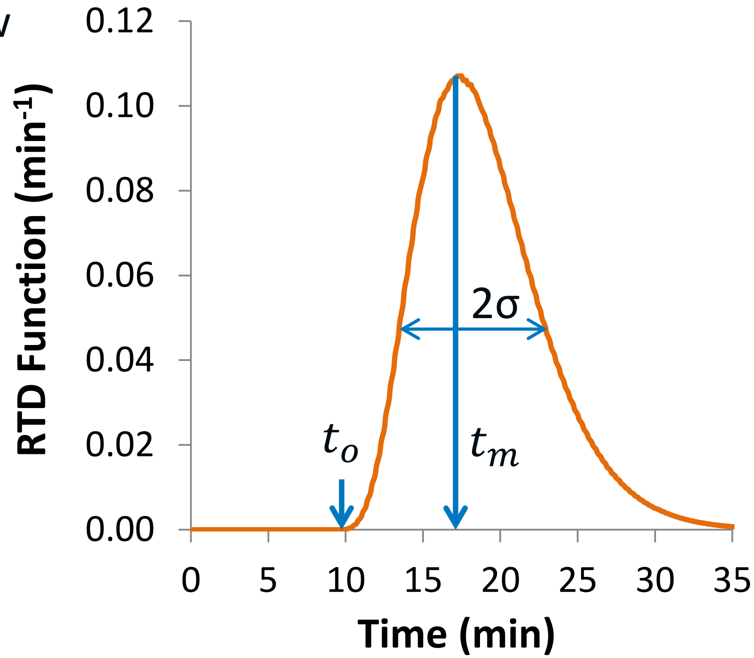
Additional Technical Details

- Residence time distribution study
- Pilot scale alkaline pretreatment
- Pretreatment reactor scale up study
- Aeration of large scale bioreactors

Residence Time Distribution (RTD) Study

Little is known about RTDs during pretreatment in continuous, screw-augered pretreatment reactors being employed in the cellulosic ethanol industry and being sold by several manufacturers (Andritz, Valmet, AdvanceBio).

- These reactors have imperfect flow characteristics producing back mixing and flow stratification leading to variations in mean residence times and particle distributions.
- Tight control of particle distribution is important for optimizing yields.

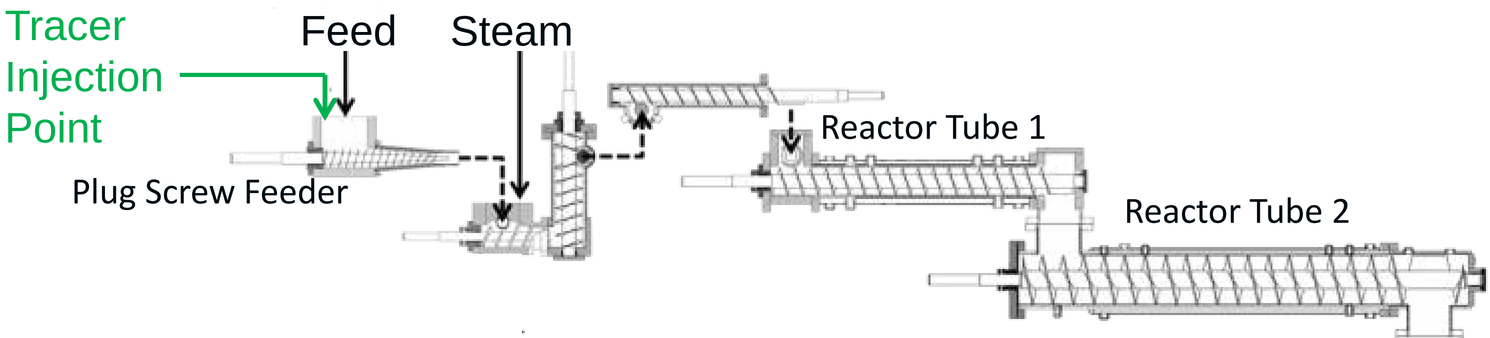


Goals:

1) **Determine mean residence times at various screw speeds during pretreatment.**
2) **Assess impact of dilute acid pretreatment operating conditions on RTD.**

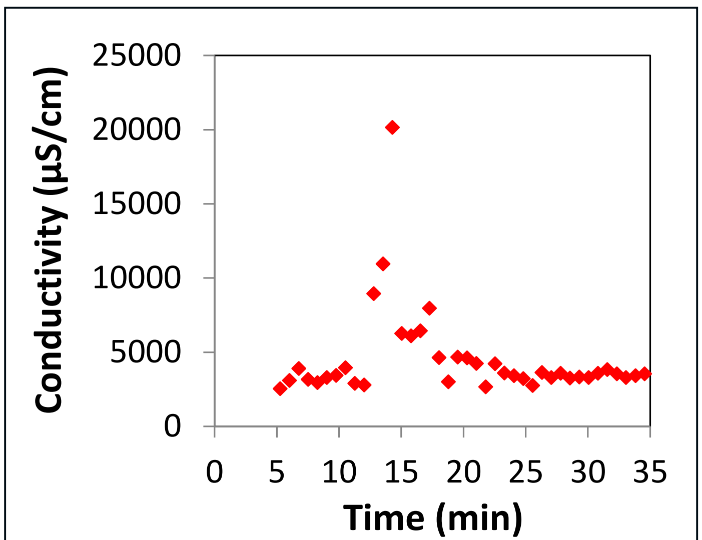
t_o: Theoretical residence time (breakthrough time) based on screw speeds assuming no material slippage
t_m: Mean of the measured RTD
σ: Standard deviation of the measured RTD (particle dispersion)

RTD Measurement in a Horizontal Reactor (500 kg/d)

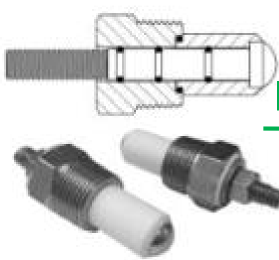


At the last peer review meeting, we reported on the successful development of an on-line technique to measure RTD using an NaCl tracer detected by conductivity.

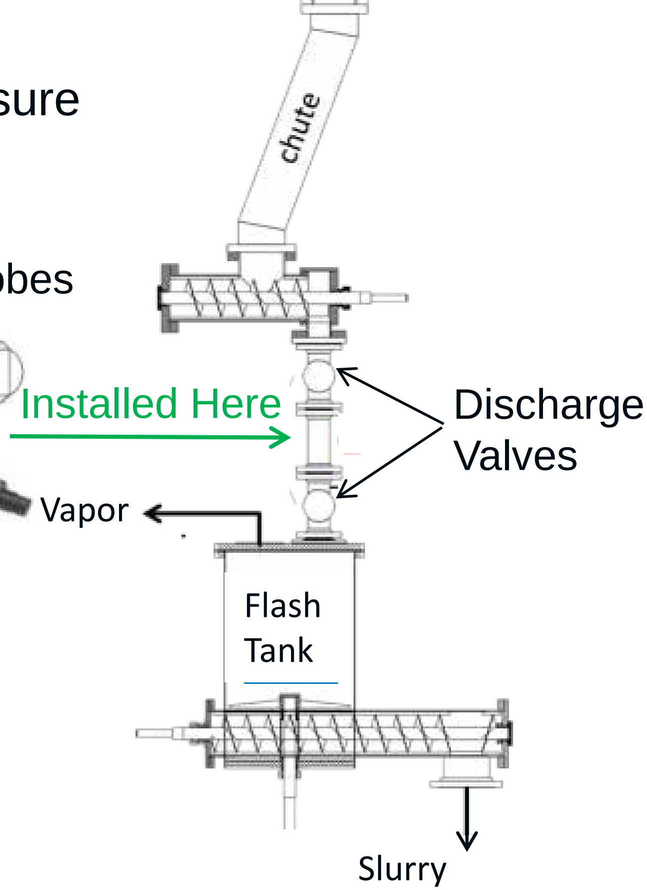
Typical profile for one tracer injection



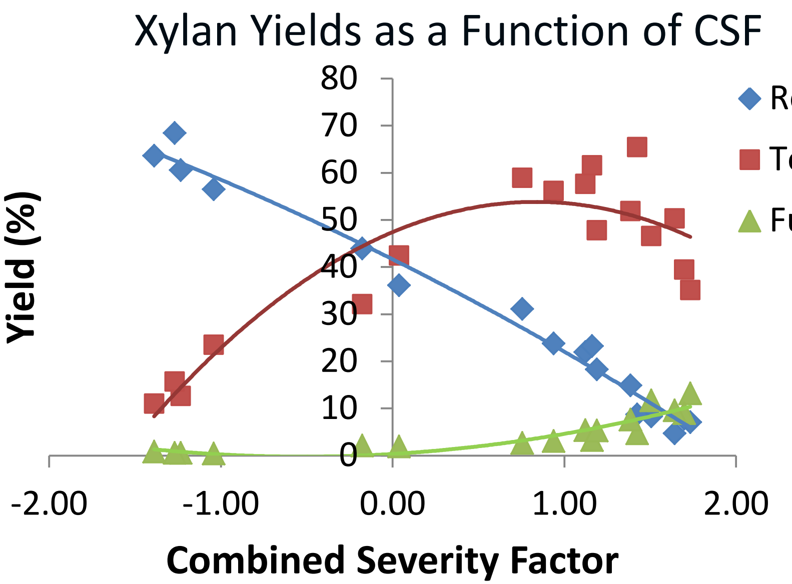
Conductivity Probes

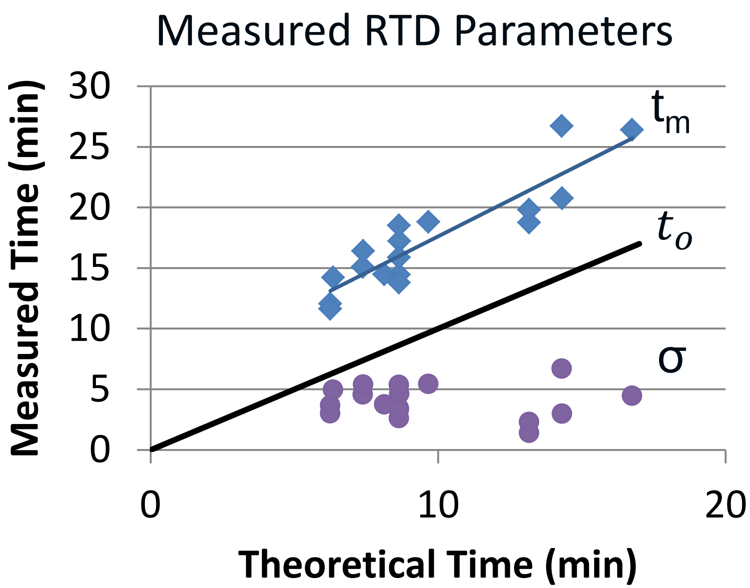


Multiple NaCl injections required to build an accurate profile



RTD Study Results





Results

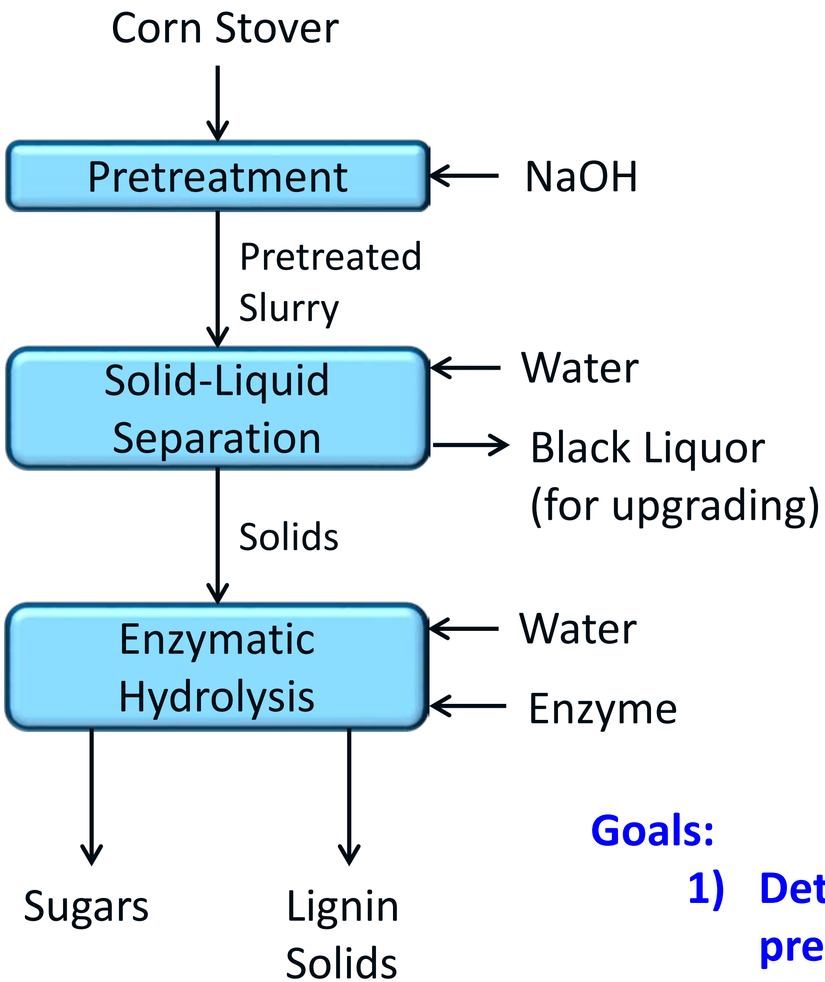
- Regardless of pretreatment severity there is
  - A consistent difference between measured mean and theoretical time.
  - Little change in particle dispersion.
- We understand the RTD for this equipment with dilute acid pretreatment chemistry and so better know potential impacts on reaction kinetics.

Recommendation

- This study has ended unless a clear need to continue the work is identified.

Combined Severity Factor (CSF) $= \log\left(t * e^{\left[\frac{T-100}{14.75}\right]}\right)$ - pH, where t is time (min) and T is temperature (°C)

Pilot-Scale Alkaline Pretreatment



Pilot-scale alkaline pretreatment was researched as one method of obtaining soluble lignin for valorization (with Lignin Utilization project).

- Process produces a stream (black liquor) containing high molecular weight lignin and low molecular weight aromatic monomer fragments suitable for valorization.
- This work also assessed impact of processing conditions on downstream operations (separations and enzymatic hydrolysis).

Goals:

1) **Determine performance in a 500 kg/d continuous pretreatment reactor.**
2) **Explore downstream processing (solid-liquid separation) and enzymatic hydrolysis yields.**
3) **Provide data for TEA.**

Experimental Procedures

Alkaline Pretreatment

Pretreatment in the horizontal reactor

- NaOH loading: 40–120 g NaOH/kg dry stover
- Temperature: 140°–180°C
- Residence time (theoretical): 10–20 min

Solid-Liquid Separation

Solid-liquid separation in a hydraulic piston press

- Recovered black liquor handed off to the Lignin Utilization project
- Solids diluted with water and repressed to produce wash solids for enzymatic hydrolysis

Soluble Lignin

Enzymatic Hydrolysis

Enzymatic hydrolysis in shake flash

- 2% solids loading
- 20 mg protein/g cellulose
- 4:1 cellulase/hemicellulase ratio
- 50°C for five days

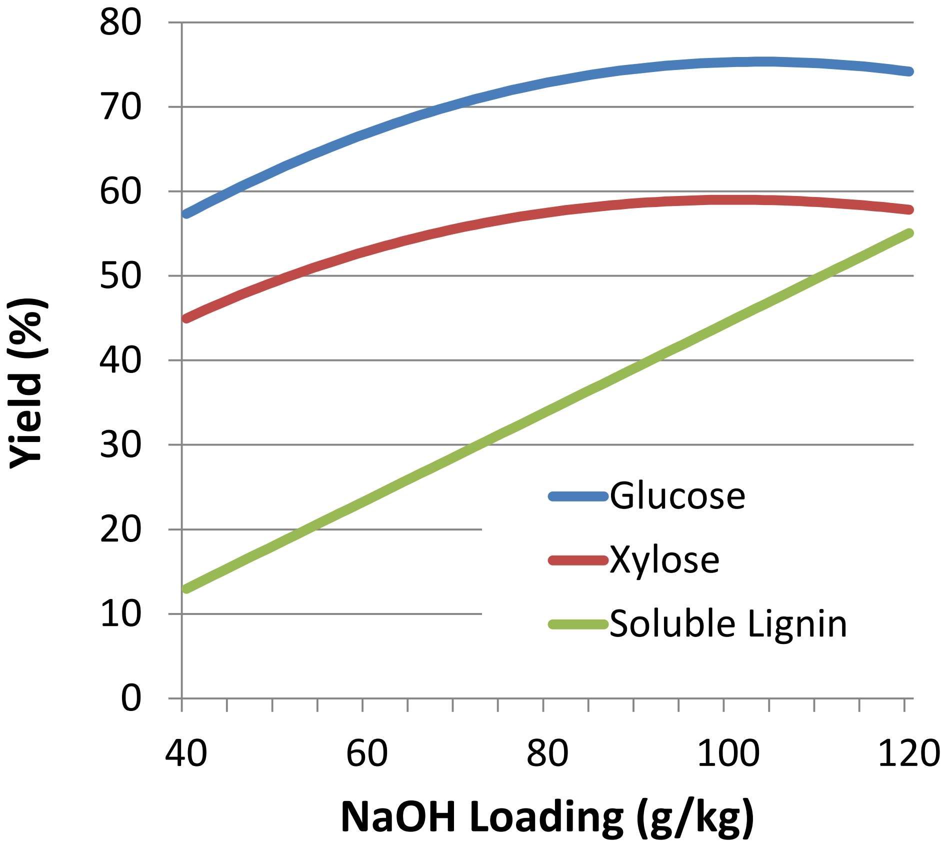
Glucose

Xylose

Output: Empirical models for cellulose/xylan and soluble lignin yields from pretreatment and monomeric glucose/xylose yields from enzymatic hydrolysis

Modeling Results

Monomeric glucose/xylose and soluble lignin yields-based components in the raw feedstock



Empirical modeling results displayed above can be used along with TEA to find conditions that minimize cost.

Additional Results

- Temperature and residence time had only a minor effect on yields compared to the effect of NaOH loading.
- Black liquor recovery from these slurries was difficult, centrifugation and filtration did not work well, and some sugars are in the black liquor stream.

Conclusions

- Process development is needed to achieve effective separations.
- Better enzymes might further improve performance.

Recommendations

- Pilot-scale work on alkaline pretreatment is on hold until economic feasibility has been better determined.

Pretreatment Reactor Scale-Up Study

Can performance in a small bench-scale reactor system be used to predict pretreatment performance in pilot-scale or even commercial-scale continuous reactors?

- Small-scale reactors can be used to quickly and cheaply screen feedstocks and pretreatment conditions.
- Significant differences exist at different reactor scales
  - Heat and mass transfer
  - Mechanical processing (e.g., mechanical grinding, steam explosion)
  - Rheological properties due to different solids loading
  - Residence time distributions.
- No work that we are aware of has directly compared performance in different reactor types, configurations, and sizes during dilute acid pretreatment.

Bench-Scale Extraction (BSE) System
- Fixed bed, flow-through system
- Three dry grams solids loading
- Indirect conductive heating
- Sixteen conditions/run possible

Goal: Determine if a bench-scale screening system (at right) can be used to predict performance in larger reactor systems (i.e., identify optimum pretreatment conditions).

Other Reactors Tested
- 1-L Batch, steam heated
- 4-L Batch, steam heated, steam explosion
- 500 kg/d continuous, steam heated

Methods and Reactor Systems

Bench Scale Extraction Reactor (Flow-Through) Dionex ASE350

1-L Batch Reactor

- Used four different pretreatment reactor systems: fixed bed, flow-through; fixed bed, batch heating with and without steam explosion; and 500 dry kg/d continuous
- Same corn stover feedstock
- Same pretreatment reaction chemistry (1% dilute sulfuric acid)
- Statistically designed experiments spanning different temperature/time combinations producing empirical models
- Measured hemicellulose conversion yields from pretreatment and enzymatic cellulose digestibility

4-L Batch Steam Explosion Reactor

Pilot Scale 500 dry kg/d Continuous Horizontal Reactor

Reactor Systems (cont.)

	BES	1- L Batch	4-L Batch	500 kg/d continuous
Operating mode	Batch	Batch	Batch	Continuous
Biomass amount (kg, dry basis)	0.003	0.07-0.10	0.25	10-25 kg/hr
Biomass impregnation	In situ	Ex situ	Ex situ	Ex situ
Heating	Oven	Steam Injection	Steam Injection	Steam Injection
Minimum Residence Time (min)	4	4	1	10
Solids loading (%)	10%	25%	25 – 30%	30%
Conditions/day	9	9	8	4
Operator Hours Per Condition	0.5	2	2	6
Mechanical Shearing	-	-	X	X
Rapid Decompression	-	-	X	X

Pretreatment Reactor Scale-Up Study Results

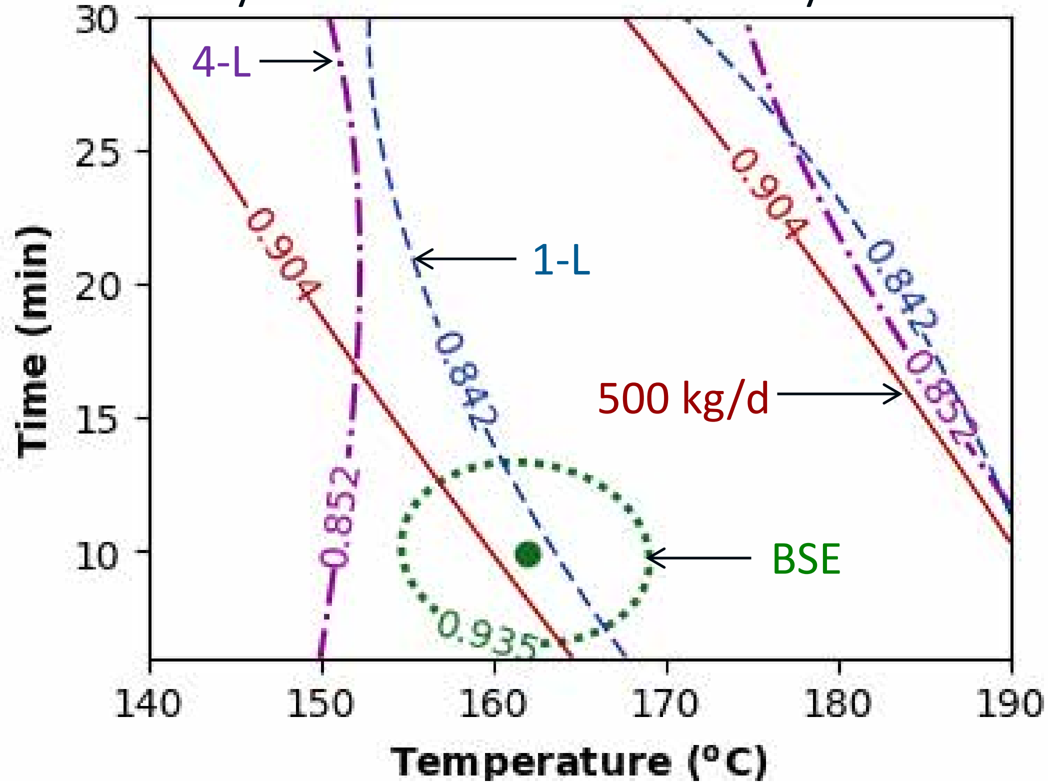
Results

- Except for the BSE reactor, all other reactors displayed a wide range of operating conditions for achieving near optimal performance.
- The optimum total xylose yield predicted by the BSE reactor system is within a two standard deviation envelope of the optimum for the other reactor systems.

Conclusions

- It is possible to predict optimum yields likely achieved in larger reactor systems using results from the small flow-through batch reactor.
- It is not possible to use BSE data to predict operating conditions in the larger reactors that optimize yields.

Empirical Modeling Results for Total Xylose Yield for Each Reactor System



Plot shows the predicted maximum total xylose yield (96%) for the BSE reactor (green point) and yields at two standard deviations from the optimum for all reactor systems.

Lischeske, et al. 2016 “Assessing pretreatment reactor scaling through empirical analysis.” Biotechnol. Biofuels. 9:213.

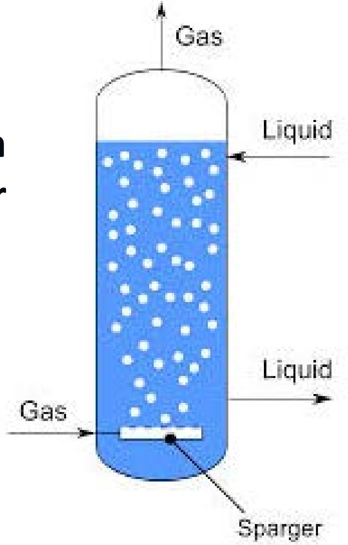
Aeration of Large-Scale Bioreactors

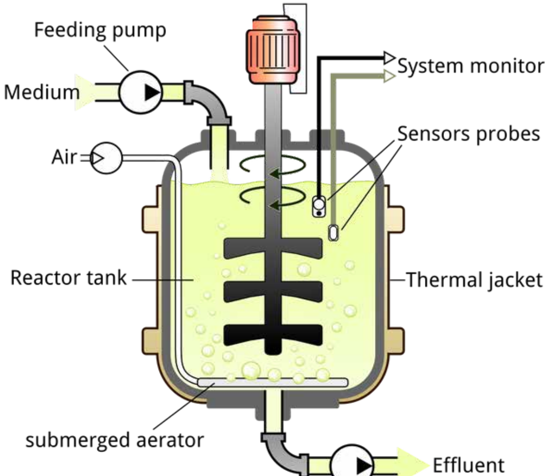
Joint Pilot-Scale Integration, Biochemical Platform Analysis and Process Modeling and Simulation project to better understand aeration in large scale bioreactors.

- Good aeration is essential for biological sugars-to-near hydrocarbon (e.g., triglycerides [TAG]) production.
- Molecular oxygen (O_2) is only sparingly soluble in aqueous media and must be continuously provided to maintain active aerobic culture.
- Volumetric productivity is highly dependent on (and for scale-up limited by) the rate of oxygen mass transfer (Oxygen Transfer Rate, OTR, mMol O_2/L-h = Oxygen Uptake Rate, OUR at pseudo steady-state conditions).

Goal: Increase confidence in aerobic reactor design and costing (TEA) in consultation with various subcontractors (Genomatica, Katzen International and Benz Technology).

Bubble Column Reactor (BCR)





Stirred Tank Reactor (STR)

Examples of Large Scale Commercial Fermentor Installations

Table 2: List of Aerobic Commercial-Scale Fermentors

Company	Location	Reactor Type	Volume (m^3)
Fermic	Lebrija, Mexico	Stirred Tank	190
Novozymes	Ottawa, Canada	Bubble Column	220
Tate & Lyle	Decatur, Illinois	Bubble Column	227
ADM	Clinton, Iowa	Stirred Tank	500
Cargill	Eddyville, Iowa	Bubble Column	500
Cargill	Uberlandia, Brazil	Bubble Column	500
Nutrasweet	Augusta, Georgia	Bubble Column	520
Dupont-Tate & Lyle	Loudon, Tennessee	Bubble Column	600
Solazyme	Moema, Brazil	Stirred Tank	600
Jungbunzlauer	Pernhofen, Austria	Bubble Column	750
Jungbunzlauer	Port Colborne, Canada	Bubble Column	750
Italprotein	Sarroch, Italy	Stirred Tank	1,000
ADM	Southport, NC	Bubble Column	1,000

Source: Genomatica s/c report (2016)

Cost to Aerate

Optimizing OPEX by minimizing power required for mixing and aeration to achieve a particular OUR

Key equation:

$OUR = OTR = k_L\, a\ (C^* - C_L)_{\mathrm{MEAN}}$

For STR

$k_L a = K\,(P/V)^{\alpha}\,(u_S)^{\beta}$

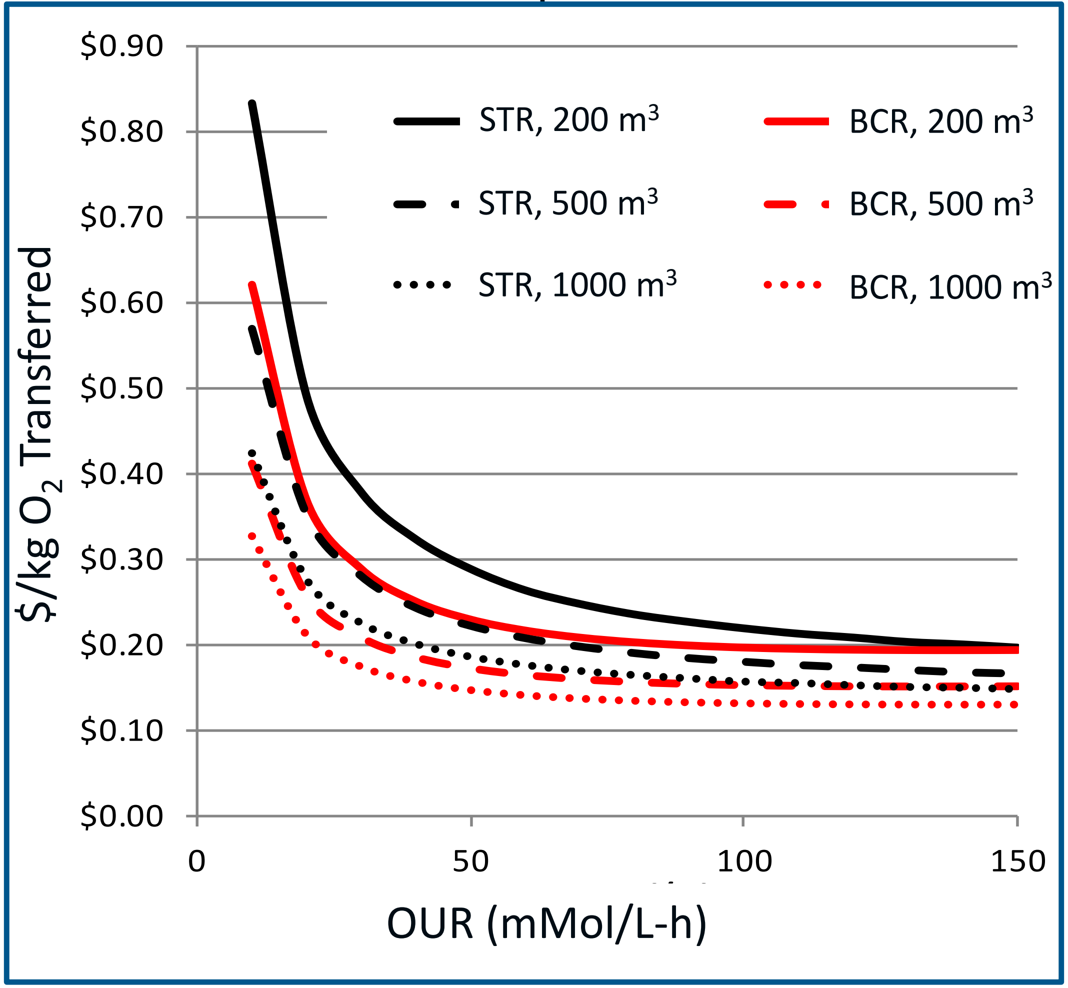
$k_L a\ [\mathrm{s}^{-1}] = 0.002\ (P/V\ [\mathrm{W/m^3}])^{0.7}\ (u_S\ [\mathrm{m/s}])^{0.2}$

For BCR

$k_L a = K'\,(u_S)^{\gamma}$

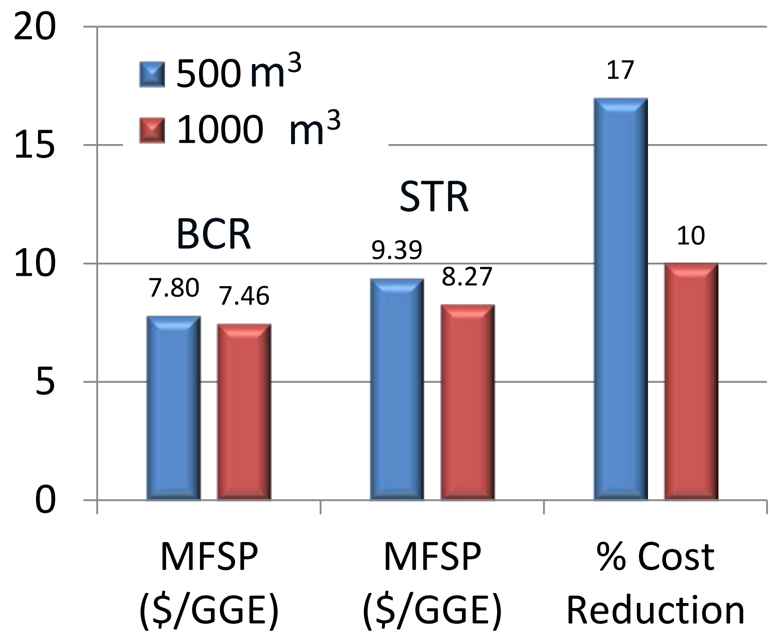
$k_L a\ [\mathrm{s}^{-1}] = 0.32\ (u_S\ [\mathrm{m/s}])^{0.7}\ (\mu_{eff}\ [\mathrm{cP}])^{-0.84} \times 1.025^{(T\,[^{\circ}\mathrm{C}] - 20)}$

Aggregated capital and operating cost to deliver 1 kg O_2 to an STR and BCR as a function of vessel volume and aeration requirements

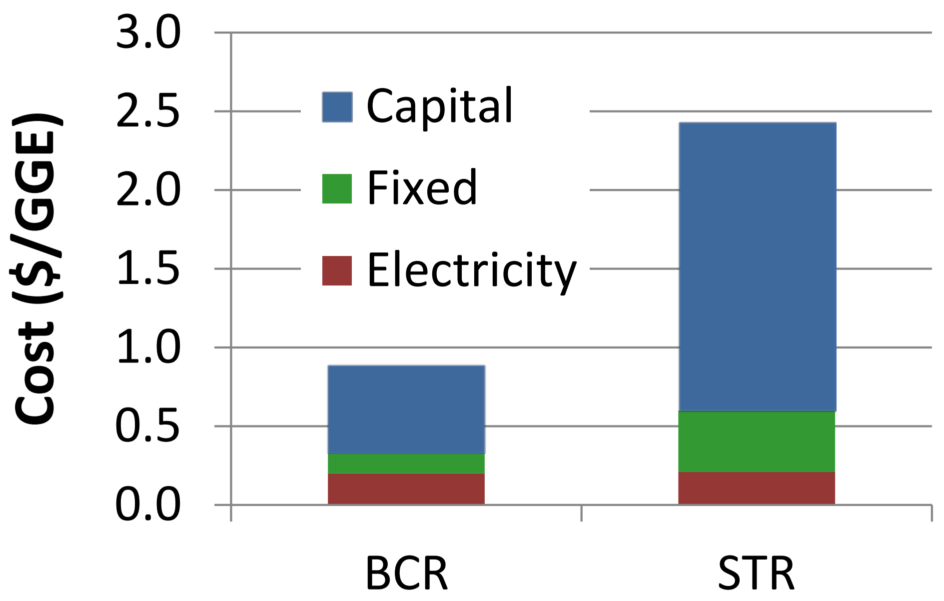


TEA Results

Modeled* Minimum Fuel Selling Price (MFSP) and Cost Reduction Achieved by BCR



Cost Contributions for the Aerobic System



Results

- BCRs have good potential to reduce aerobic production cost compared to STRs.
- Process broth viscosity and required OTR must be in BCR operable range.

Recommendation

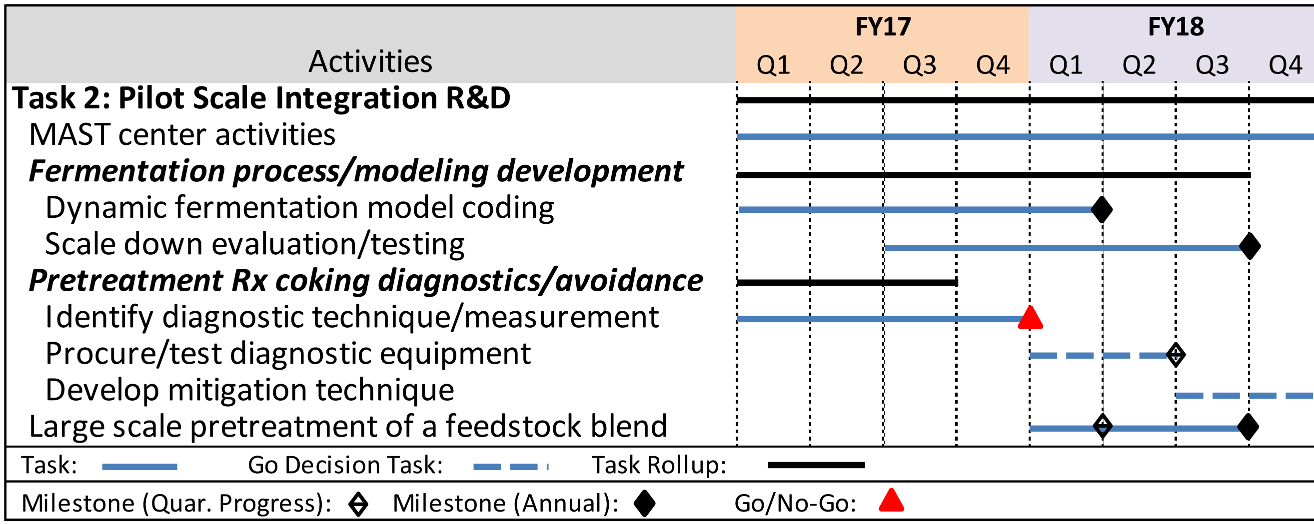
- Revisit (go decision) these results once further down-selection occurs on an aerobic biological upgrading process/pathway.

*NREL TEA model for lipid production process with no coproducts

Future Work Details—Gantt Charts

Future Work—Pilot-Scale Integration R&D (Near Term)

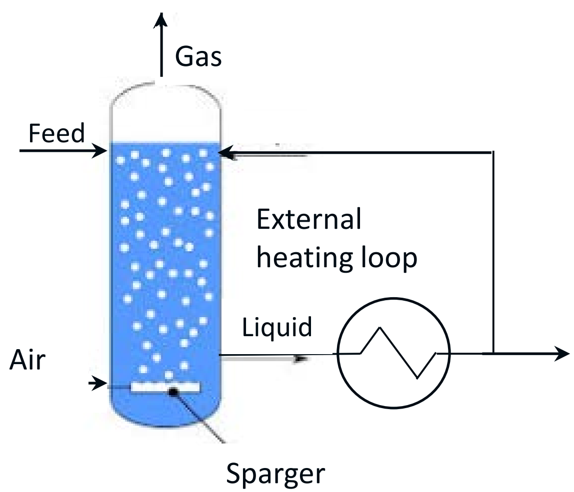
Continue MAST center membership.



Code model in a more user friendly platform and begin developing scaled down testing capability.

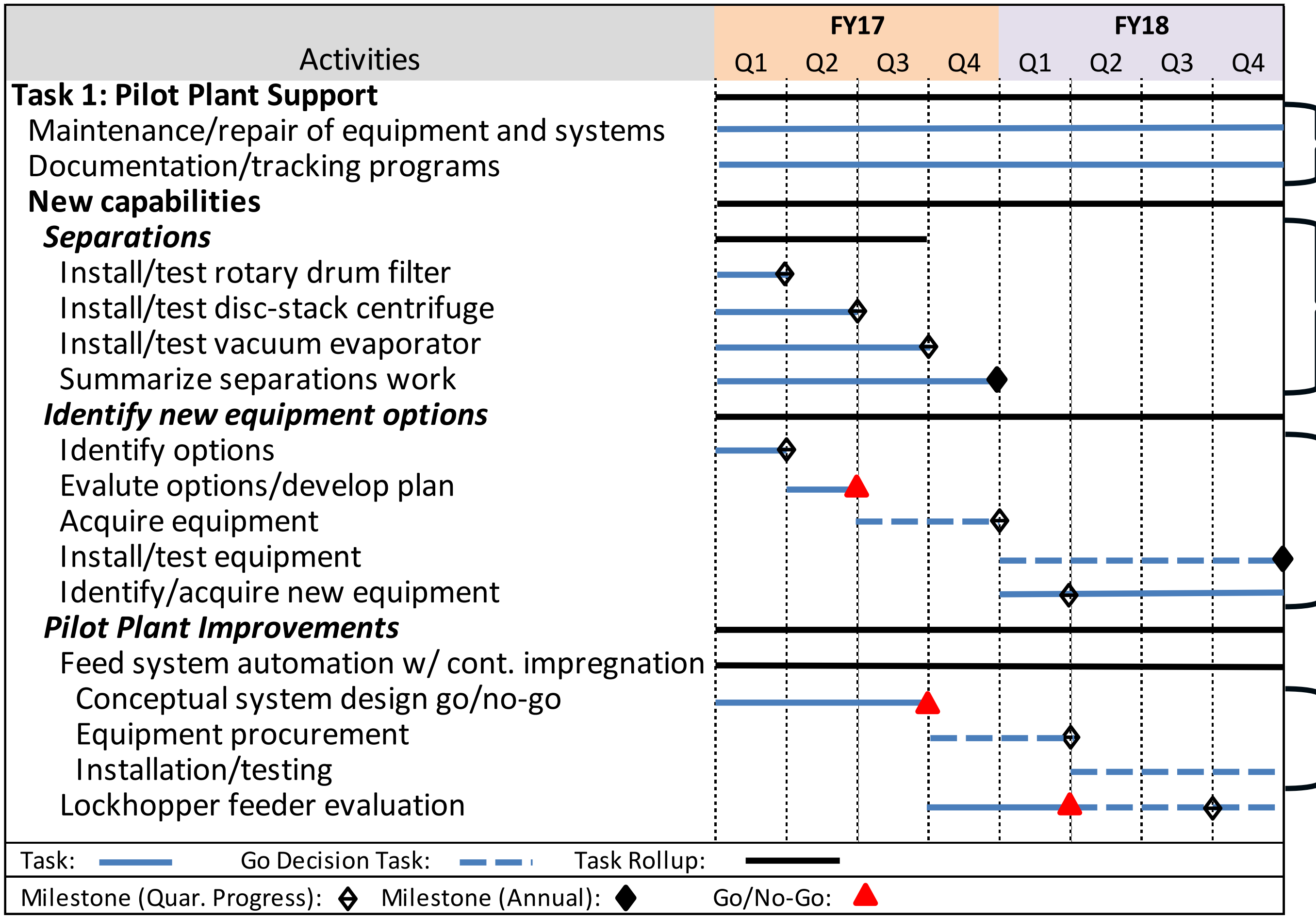
Make go/no go decision based on feasibility and future need.

Effect of External Cooling Loop on Process Performance (Scaled down testing recommended by Genomatica)



Mitigating Reactor Coking

Future Work—Pilot Plant Support (Near Term)



Ongoing work needed to maintain plant functionality

Work to install/test these items ending in FY17

Continuing effort to identify and acquire needed process capabilities

Design effort to upgrade feed systems to improve operations and to meet new process requirements