1.3.3.100: Hydrocyclone Separation of Targeted Algal Intermediates and Products

March 24, 2015

Algal Feedstocks Research and Development

Richard Brotzman

Argonne National Laboratory

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Project Goals

- Evaluate an energy-efficient, separation process
  - Technology: Hydrocyclone separation of components in a fluid mixture
  - Main application: Dewatering of algal cultures
- Program tasks
  - Establish baseline understanding of hydrocyclone separation of algae
  - Develop separations process metrics
  - Identify operational parameters most indicative of optimal performance
  - Develop techno-economic model of hydrocyclone separation
- Metrics
  - Dewatering algae: % concentration
  - Energy input and operation duration
  - Process cost
- Success can lead to cost-competitive algal biofuels that can reduce the nation's dependence on fossil fuels

Quad Chart Overview

Timeline

- Project start date: Oct 1, 2012
- Project end date: Sept 30, 2014
- Percent complete: 100% (FY2014)
- Project Type: Sun-Setting

Barriers

- AFt-B: Sustainable production
- AFt-D: Sustainable harvesting
- AFt-M: Integration and scale-up
- AFt-N: Algal feedstock processing

Budget

- Total project funding
  - DOE: $ 600,000
  - Contractor: $ 0
- Funding received in FY12: $ 0
- Funding for FY13: $ 250,000
- Funding for FY14: $ 332,310

Partners

- George Oyler – University of Nebraska
- REAP production facility at New Mexico State University
- Leveraged activities
  - Industrial experience with hydrocyclones
  - ANL LDRD-funded separations technologies

1- Project Overview

- Uses for large-scale algal culture
  - Biofuels
  - Bioproducts manufacturing
  - Dietary supplements
- Goal

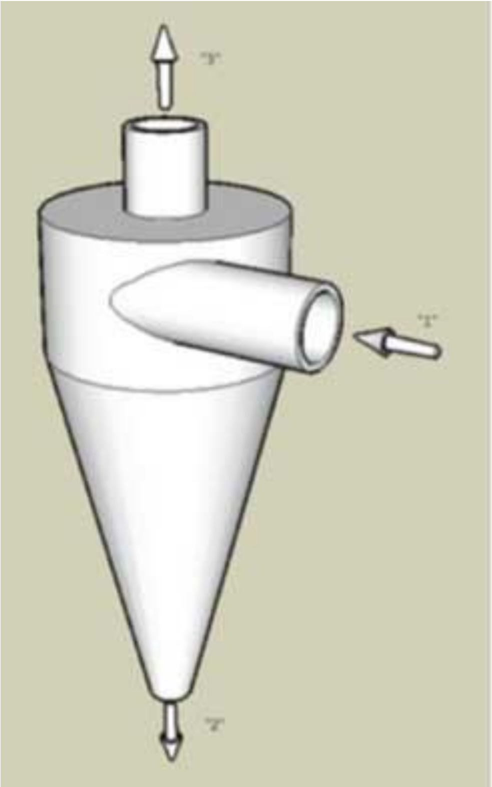
  To evaluate a low-capital, alternative technology to separate algae efficiently from growth medium – the initial dewatering step required to extract neutral lipids from cells for use as fuel precursors

- Competing technologies
  - Settling
  - Filtration
  - Centrifugation
  - Dissolved Air Floatation

capital and energy intensive processes

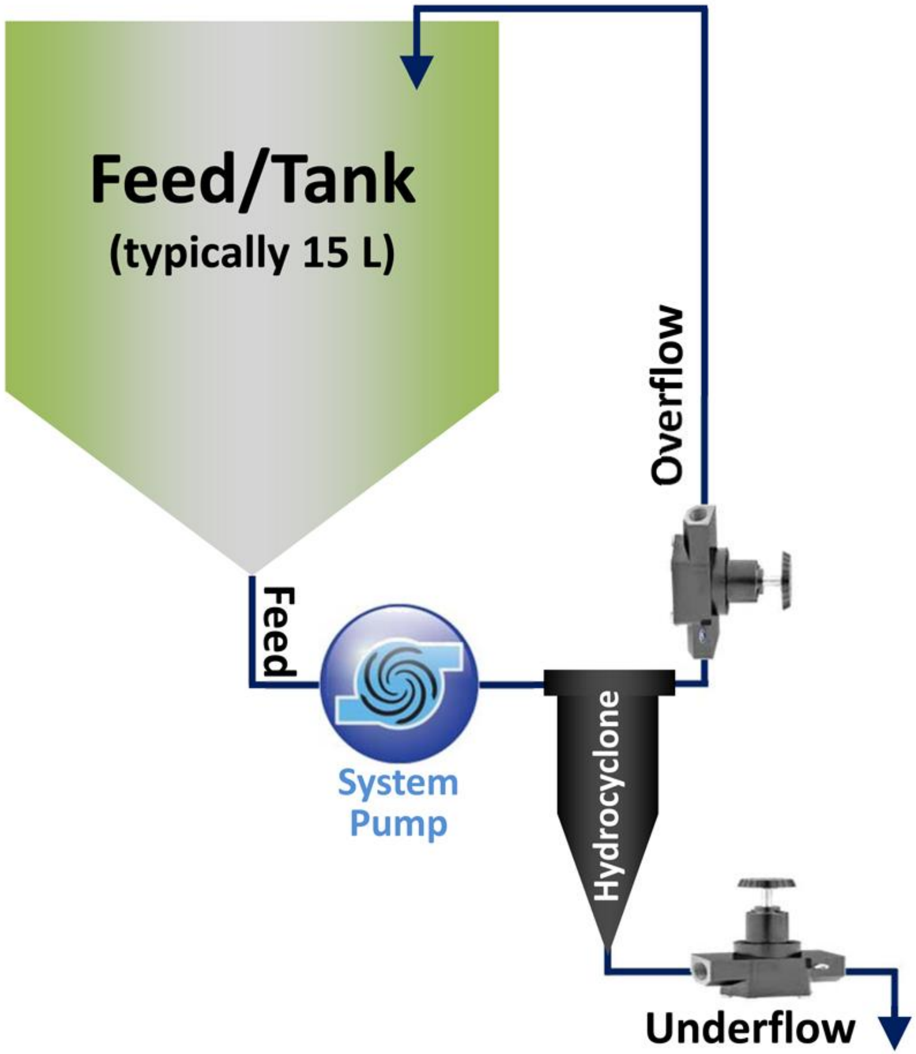
2 – Approach: Hydrocyclone Separation

- Unit Operation
  - Separation of components in fluid mixture – density and/or size
  - Continuous-flow dewatering
  - Replaces several unit operations: centrifugation, filtration, and washing
- Hydrocyclone structure
  - Cylindrical-conical body
  - Conical base
  - Liquid is fed tangentially near the top
  - Two opposite axial exits
    - Top exit (overflow or vortex) – a tube extends into cylindrical section – lighter or finer fraction
    - Bottom exit (underflow) – denser or coarser fraction
- Unique flow dynamics
  - Multi-component vortex fluid motion
    - Tangential, axial, and radial velocities
  - Lowest pressure in center of unit



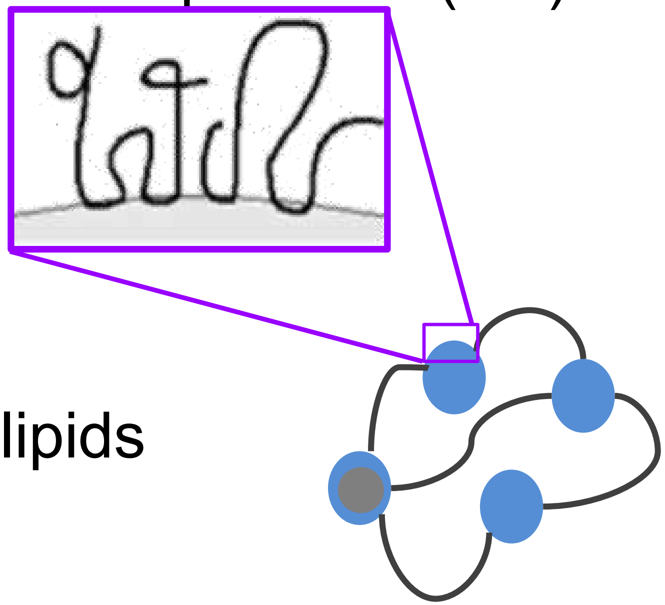
2 – Approach: Abbreviations

- U/F: underflow
- O/F: overflow
- L: hydrocyclone length
- D: hydrocyclone diameter
- LPM: liters per minute
- IQ: installation qualification
- OQ: operation qualification
- PQ: process qualification



2 – Approach: Magnetic-field separations evolved into separate Biochemical Conversion project

- Unique synthesis process: magnetic nanoparticles (NP)
  - Colloidal method
  - Solid-state reaction
- Assembly of magnetic NP
  - Chemically bond NP using polymer chains
  - Forms elastic network – like a rubber band
- Surface treatment on NP to adsorb lipids
  - Heterogeneous gas phase process
  - No process solvents
- Harvest neutral lipids from *Chlorella* species
  - Magnetic
  - Flotation



This approach highlighted in other studies involving separations of biofuels and toxins in bioreactor-integrated separations schemes.

2 – Management: Critical Success (Risk) Factors

Risk	Mitigation Approach
Dewatering: Hydrocyclone separation of algae from growth medium	• Explore entire process space – multiple passes • Explore laminar and turbulent mixing
High algal concentrations limit flow through system	• Turbulent mixing • Increase residence time in hydrocyclone • Decrease outlet tubing diameter
Separation % yield or % concentration low	• Multiple passes through hydrocyclone • Use hydrocyclones in series
Process cost	• Low power pump innovations • Single pass – multistage hydrocyclone

- Potential challenges for success
  - Successful dewatering with low operational costs
  - Conditions for continuous operation with high algal solids
- Structure of approach
  - Explore operating limits of technology (exploratory)
  - Challenging milestones

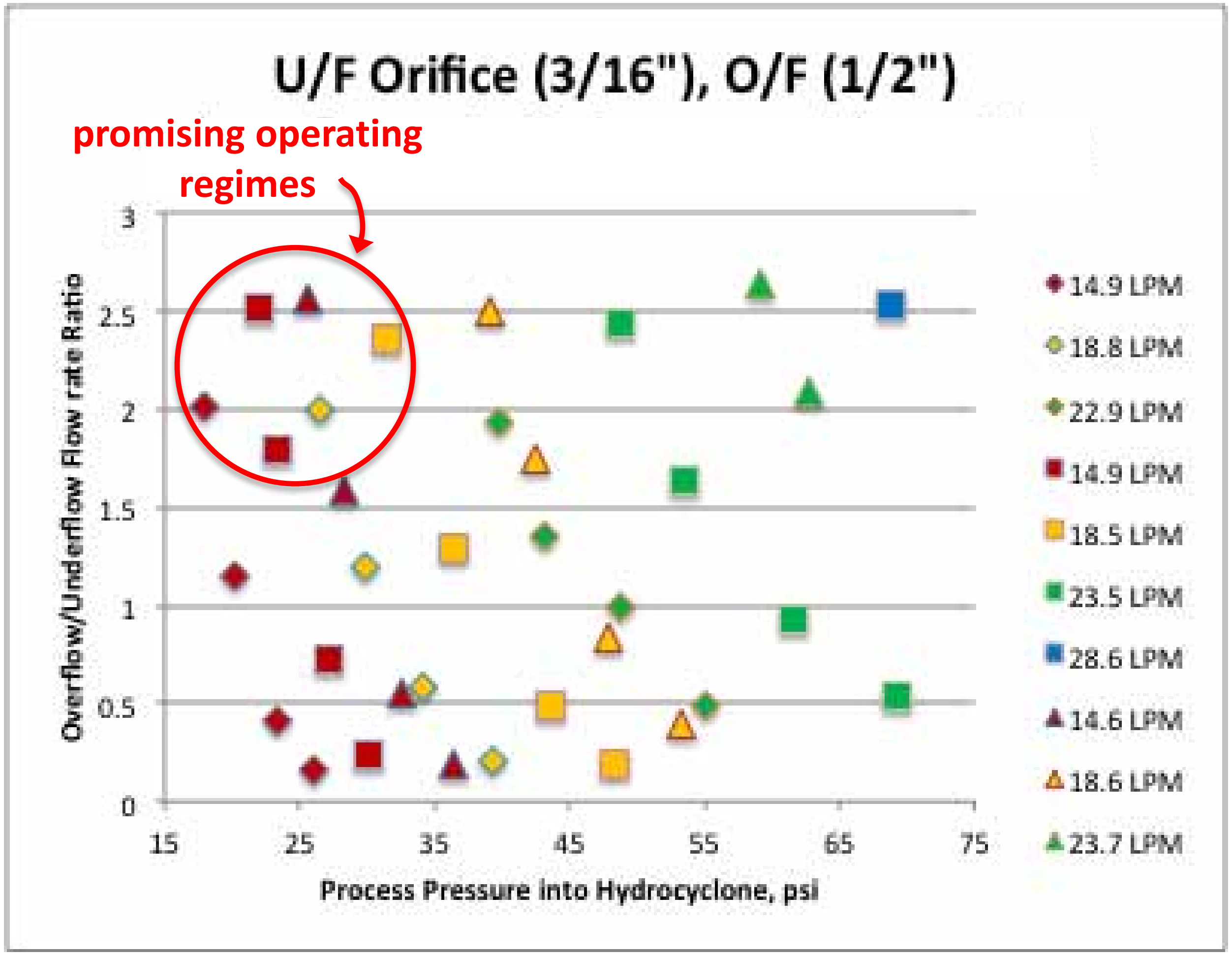
3 - Technical Progress: Schedule

Task/Milestone	Planned Completion	% Completion
Fabricate test system	31-Dec-2012	100
Establish process metrics	31-Mar-2013	100
Optimize process	31-Mar-2013	100
Evaluate technology feasibility	30-Sep-2013	100
Concentrate algae to levels > 15 times the feed stream	31-Dec-2013	100
Design parameters to harvest > 50% biomass	31-Mar-2014	100
Evaluate two-step process for recovery of > 90% biomass	31-Jun-2014	100
Demonstrate favorable cost/performance metrics	30-Sep-2014	100

3 - Fabricated Hydrocyclone Test System

- Hydrocyclone – as installed for operation at ANL

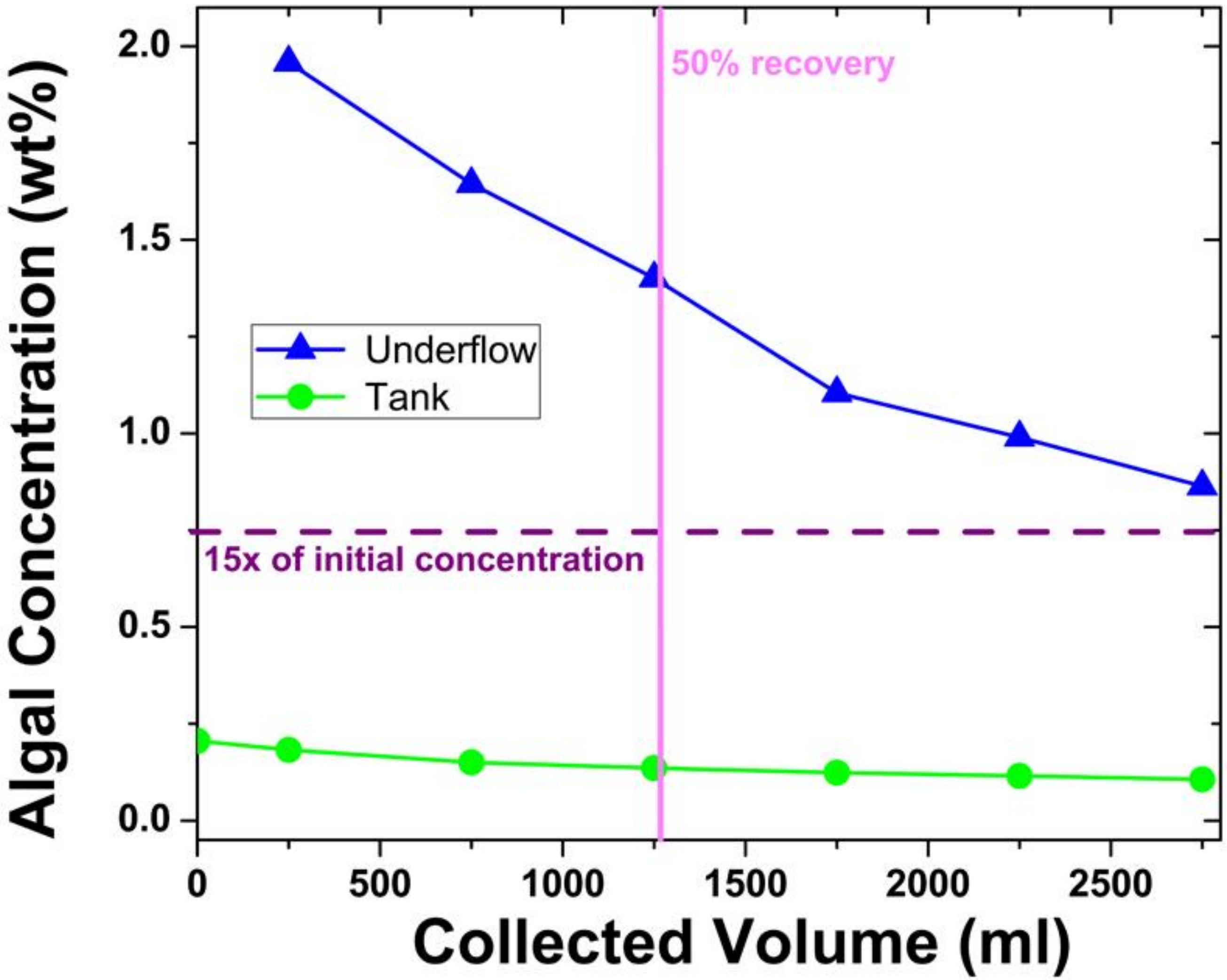
3 – Technical Progress: Separation Process Metrics



- Operational space explored to survey residence times of algae
- Optimized parameters selected for use in dewatering tests at scale

3 – Progress: High-volume, low linear velocity operation

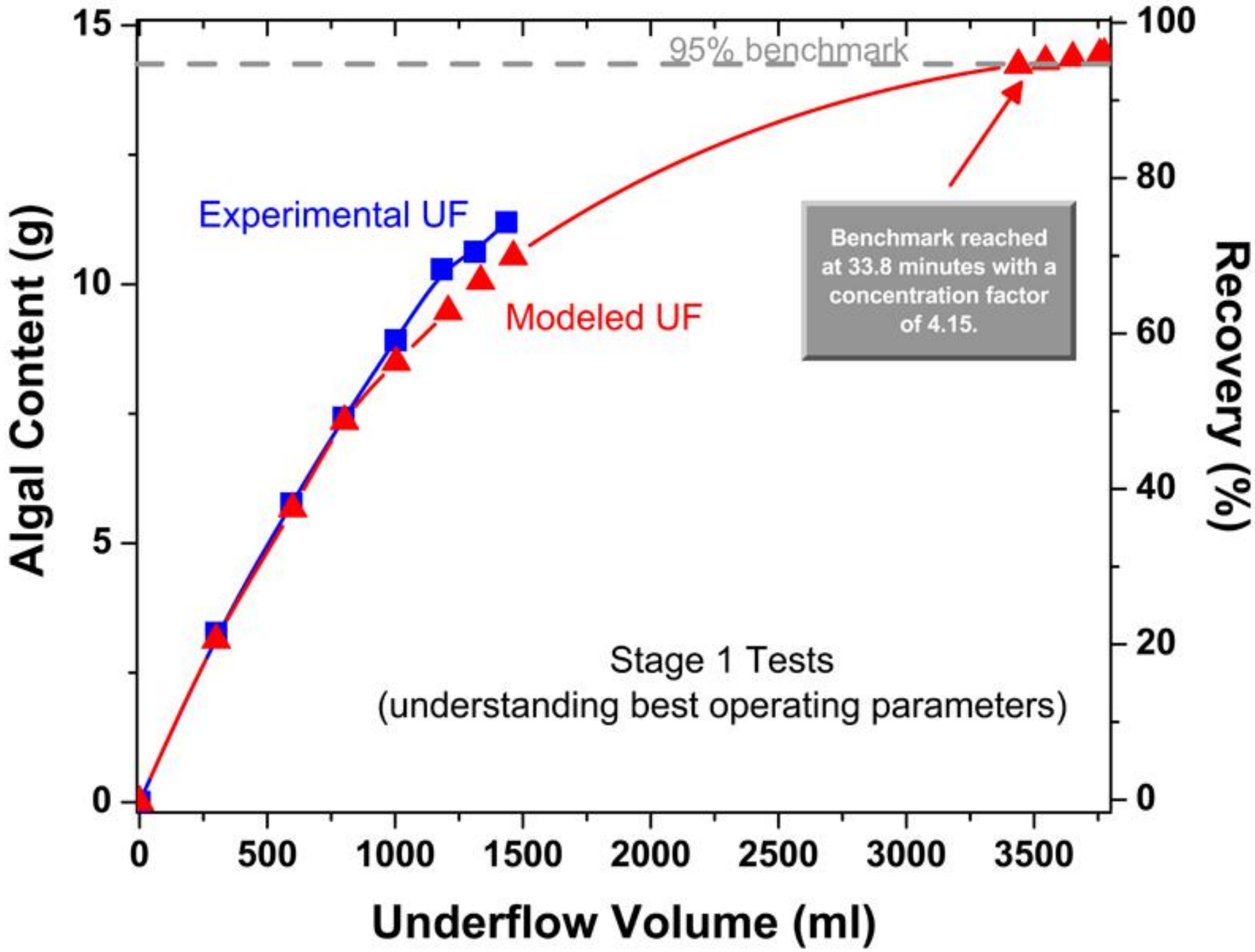
Final of two-stage process for dewatering of 0.05 wt% algal suspension



> 50% recovery achieved; > 15 x concentration; 60W pump power

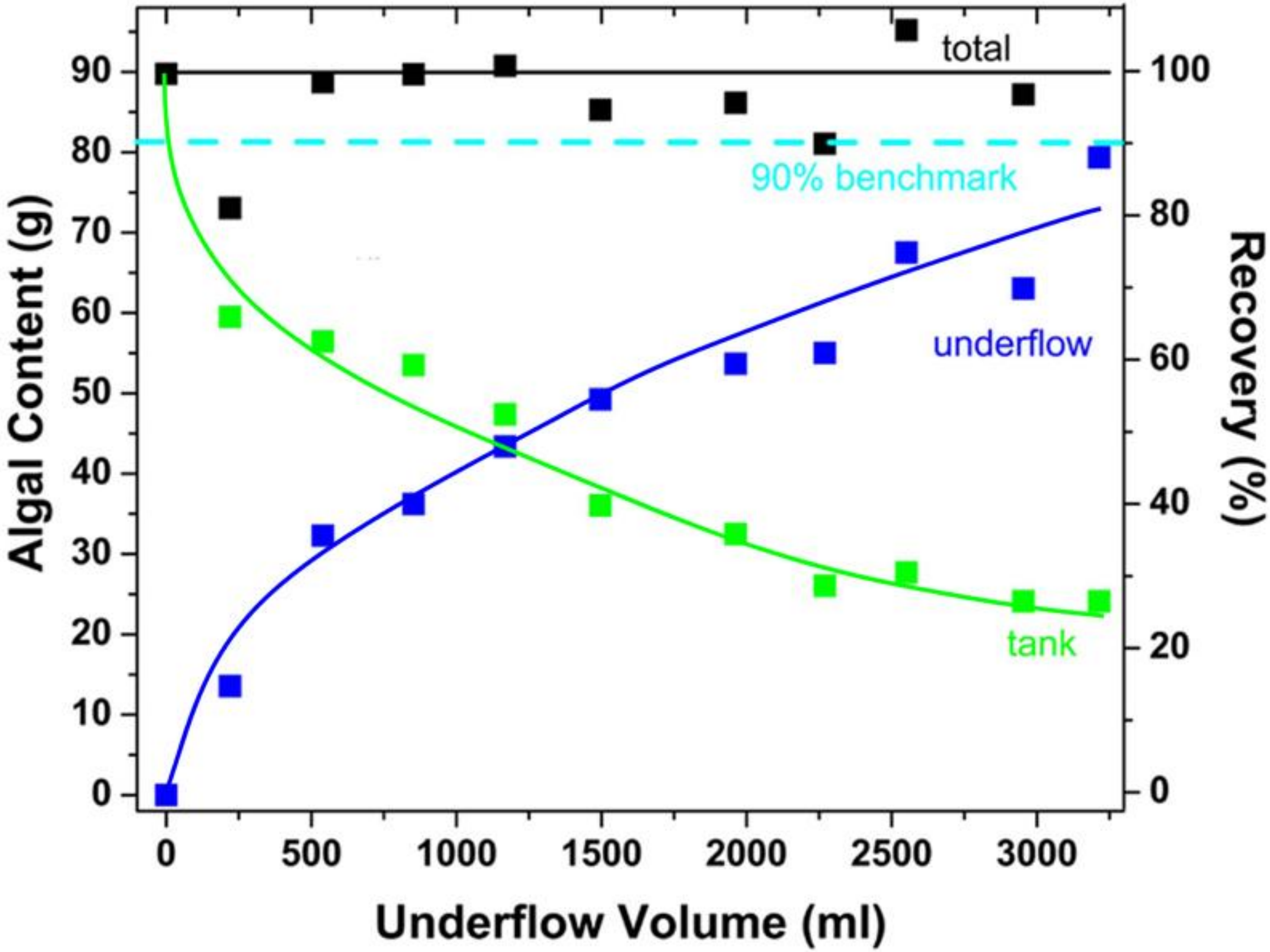
3 – Progress: Modeling hydrocyclone operation

- Hydrocyclone performance with algal cells approaches theoretical limits
- Modeling can predict product recoveries and required operation times
- Results can decrease experimental operation and process energy expended



3 – Progress: Low-volume, high linear-velocity operation

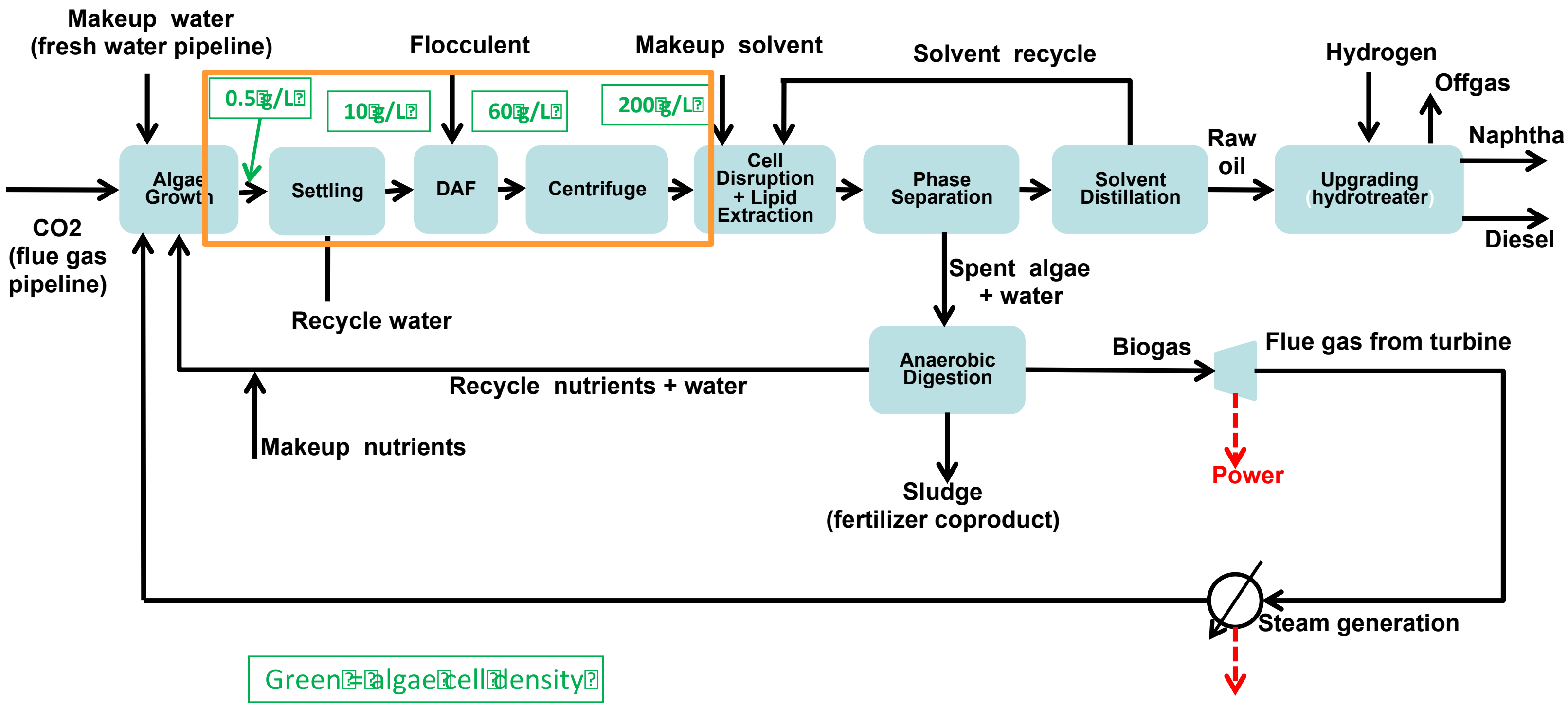
Final of two-stage process for dewatering of algal suspensions



90% recovery can be achieved; > 15 x concentration; 112W pump power

Harmonization Scenario (Renewable Diesel)

3 – ess



- Hydrocyclone process is capable of concentrating cells from 0.5% to ~ 10% solids
- Operating costs on same order of magnitude to competing unit operations
- Capital costs promise gains with
  - Low-cost, simple hardware (no moving parts) and installation
  - Potential for continuous, in-line operation
  - Reduced staffing requirements
  - Complete scalability

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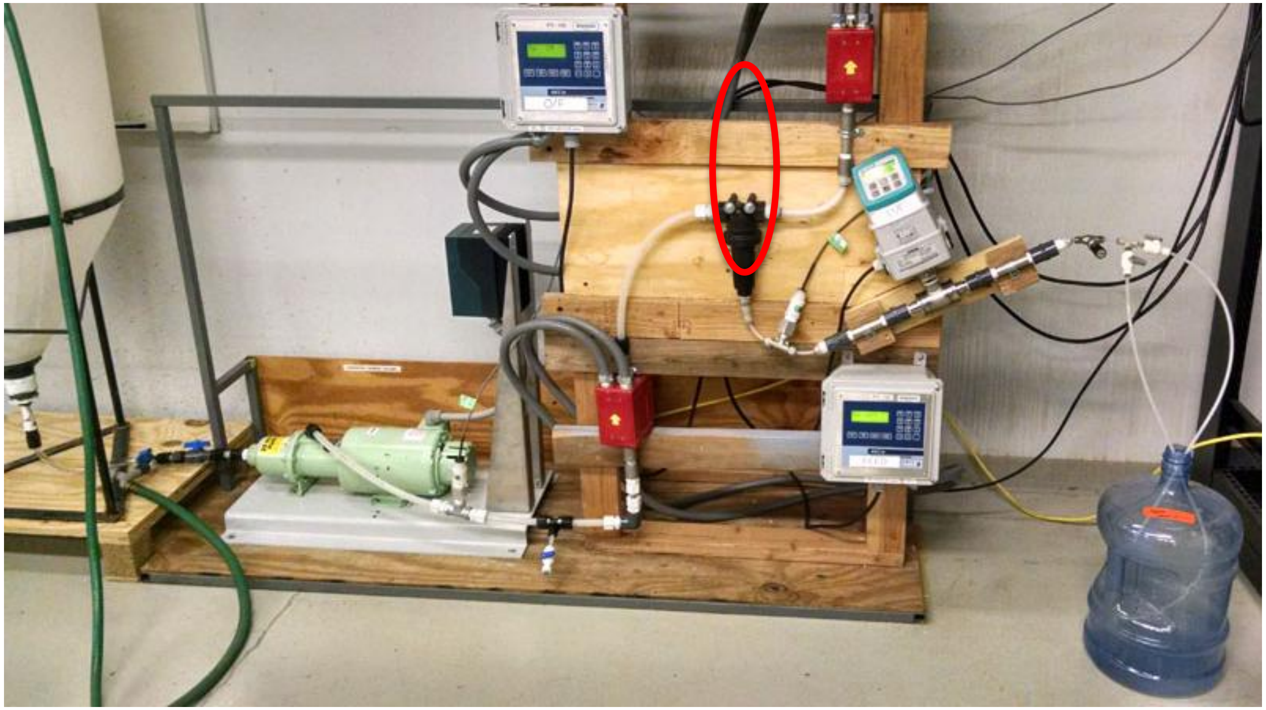
3 – Progress: Integration into production facility at New Mexico State University

(Testing coordinated with the *Realization of Algae Potential* project)



- Facility performance verified
- General applicability increased if parameters also optimized for:
  - Range of algal cell types
    - Size
    - Morphology
  - Cultures with altered buoyancy (varied lipid content)

4 - Relevance

- Algal dewatering by hydrocyclones works near theoretical limits
- Hydrocyclone technology appears to be a viable alterative to existing algal dewatering efforts that can bring down algal harvesting costs
- Hydrocylones are expected to be employed at large-scale outdoor algal facilities to replace settling, dissolved air flotation, and centrifuge unit operations
- Hydrocyclone gains are realized mainly because of the reduced costs of their acquisition and installation
- Hydrocylone operations have been used extensively in other industrial processes and are known to be scalable
- Hydrocyclone operation has been field tested successfully. General applicability to other algal types and lipid content are being explored

Summary

- The project objectives are relevant to BETO’s Algae Technology Area goals through exploration of novel algal dewatering methods – the process critical for cost-competitive utilization of neutral lipids as biofuels precursors
- Hydrocyclones are incorporated into production strategies with efficient, integrated processing steps
- The approach proved effective by exploring a large flow-separation process space
- Hydrocyclone operational costs compare with competing unit operations; lower capital input are where gains may be realized
- Best use of hydrocyclones may be to augment existing technology for early phases of dewatering
- Laboratory-optimized hydrocyclone operation field tested and verified at an outdoor algal production facility

Additional Slides

(Not a template slide - for information purposes only)

- *The following slides are to be included in your submission for Peer Evaluation purposes, but <u>will **not** be part of your oral presentation</u> –*
- *You may refer to them during the Q&A period if they are helpful to you in explaining certain points.*

Responses to Previous Reviewers' Comments

Three similar comments:

- **The project should have better-defined process performance criteria, including mass balance, energetics, and CAPEX and OPEX targets.**
- **Also, the approach seems economically unviable at commercial-scale as presented today.**
- **…………insufficient economic analyses to determine the viable options for use of these technologies, or to set appropriate targets.**
  - Cost/performance analyses and preliminary TEA analyses are included in the current slides and were part of activities post-2013 review.
  - Hydrocylones had never been evaluated for algae dewatering as the initial step towards lipid separation. Literature results indicate a centrifuge outperforms other separation methods for many different types of algal strains. However, centrifugation is energy intensive and is a difficult unit operation to design a profitable biofuel production process; in contrast, hydrocyclones have no moving parts and are significantly less energy intensive. The limits of the hycrocyclone with respect to algal dewatering were investigated; experimental data were required to model the separation process.

Responses to Previous Reviewers' Comments

- **The challenge of disrupting algae cells so that the absorbent can contact oil were not addressed.**
  - Hydroclones were only used to dewater algal cells. Its use in disrupting cells and enriching lipids from extracts is outside of the scope of this project. Early in experiments it was clear that forces inside hydrocyclones would not be sufficient to disrupt cells and plans for lipid separation post cell disruption were rapidly abandoned and effort/milestones redirected.
- **The two very different technologies presented for dewatering algae and recovering oil may be beneficial, but it is not clear how they fit together.**
  - The hybrid approach and use of magnetic nanomaterial absorbents to recover algal was removed from this approach so that effort could be focused on hydrocylone optimization and technology cost/performance evaluation. Magnetic absorbents are used in a separate AOP in the Biochemical Conversion Technology Area.

Publications, Patents, Presentations, Awards, and Commercialization

- Patent Application:
  - Yupo J Lin, Richard W Brotzman, Seth W Snyder. USPTO 20150041400. Application number 13/962480. COMPOSITIONS AND METHODS FOR DIRECT CAPTURE OF ORGANIC MATERIALS FROM PROCESS STREAMS. Filed August 12, 2013. Published February 12, 2015.

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