9.5.1.9: Hydrocyclone Separation of Targeted Biochemical Intermediates and Products

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Department of Energy Bioenergy Platform – Algae Technology Area

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Goals

- Evaluate hydrocyclone separation to harvest algal lipids
  - Employing turbulent mixing with concomitant heat transfer
  - Nanostructured adsorbent materials based on magnetic separations and/or flotation
- Couple hydrocyclone with nanostructured adsorbents to yield efficient separation process
- Technology spans between biomass processes
  - Applicable to targeted products and intermediates
  - Enables investigation of quality requirements of intermediates and products

Abbreviations

- IQ: installation qualification
- OQ: operation qualification
- PQ: process qualification
- NP: nanoparticle
- NA: nanostructured adsorbent
- U/F: underflow (hydrocyclone)
- O/F: overflow (hydrocyclone)
- L: hydrocyclone length
- D: diameter (NP)

Quad Chart Overview

Timeline

- Project start date: Oct 1, 2012
- Project end date: Sept 30, 2015
- Percent complete: 55% (FY2013)

Barriers

- Hydrocyclone selection, IQ, OQ, PQ
- Nanostructured adsorbents
- Acquisition of Algae

Budget

- Total project funding
  - DOE: $ 250,000
  - Contractor: $ 0
- Funding received in FY12: $ 0
- Funding for FY13: $ 250,000
- ARRA Funding: $ 0

Partners

- George Oyler – University of Nebraska / Synaptic Research
- Leveraged activities
  - PI experience with hydrocyclones
  - BETO project to adsorb sugars
  - ARPA-E nanostructured magnets
  - LDRD magnetic nanostructures

Project Overview

- Separation process combines two innovative technologies
  - Hydrocyclone: separates components in fluid mixture – density and/or size
  - Nanostructured adsorbents (NA): harvest lipid
- Program tasks
  - Establish baseline understanding of hydrocyclone separation of lipid products from algal growth media
  - Develop separation process metrics based on lipid products from algal growth media
  - Optimize hydrocyclone separation of algal growth media. Test impact of nanostructured adsorbent materials
  - Demonstrate feasibility of hydrocyclone separation of lipids from algal growth media. Develop techno-economic model of hydrocyclone separation.
- Metrics
  - Dewatering algae: % concentration
  - Harvest lipid: % harvested and % concentration
  - Process cost

1.1 Hydrocyclone Separation Approach

- Hydrocyclone unit
  - Classification of components in fluid mixture – density and/or size
  - Continuous-flow dewatering
  - Replaces: centrifugation, filtration, and washing
- Hydrocyclone structure
  - Cylindrical-conical body
  - Conical base
  - Liquid is fed tangentially at 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: 3-dimensional vortex fluid motion
  - Tangential (m/s), axial (m/s), and radial velocity (1 - 0.1 m/s)
  - Low pressure in center of unit

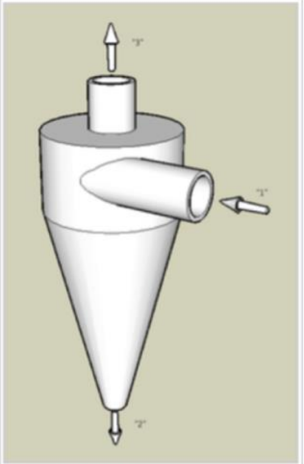
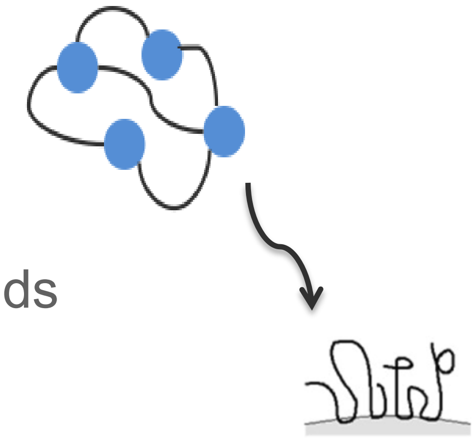


Diagram of a hydrocyclone:
1. the liquid-solid mixture enters,
2. heavy solids leave,
3. cleaned liquid leaves.

Hydrocyclone

1.2 Adsorbent Separation Approach

- Unique synthesis process: magnetic nanoparticles (NP)
  - X Colloidal method
  - – Solid-state reaction
- Assembly of magnetic NP
  - Chemically bond NP using polymer chains
  - Forms elastic network – like a rubber band
- Unique: surface treatment on NP to adsorbs lipids
  - Heterogeneous gas phase process
  - No process solvents
- Harvest lipids from the algal strain: Chlorella sorokiniana UTEX 1230
  - Magnetic
  - Flotation

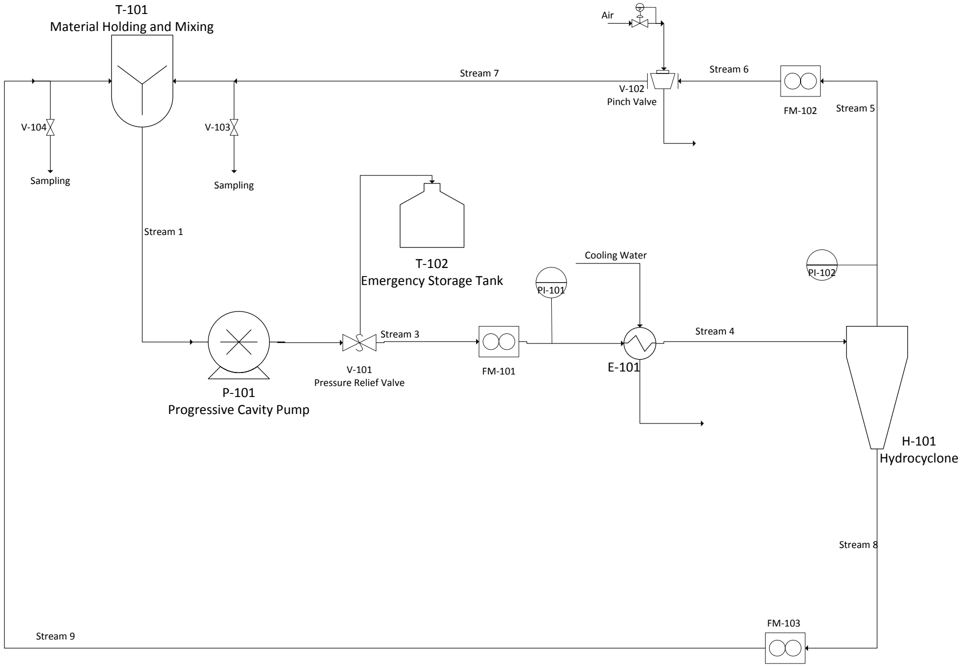


2. Technical Progress: Schedule

Task, Milestone, Deliverable	Title - Tasks, Subtasks, Milestones, Deliverables including Go No/Go Decision Pts	Planned Completion Date	% Actual Completion
A	Hydrocyclone	31-Dec-12	100
A.1	Baseline	1-Dec-12	100
A.2	Fabricate Test System	31-Dec-12	100
A ML 1	Test System Complete	31-Dec-12	100
B	Separation Process Metrics	31-Mar-13	100
B ML 1	Separation Process - 50%	31-Mar-13	85
C	Separation of Algal Growth Media / Adsorbents	31-Jul-13	35
C ML 1	Optimized Process	31-Jul-13	
D	Feasibilityof Hydrocyclone/Adsorbent Process	30-Sep-13	
D.1	Turnover Cycle Number	30-Sep-13	
D.2	Techno-economic model	30-Sep-13	
D ML 1	Technology Feasibility	30-Sep-13	

2.A.1 Hydrocyclone Accomplishments

- Hydrocyclone:[1] 5 to 10 GPM; top diameter of 1", adjustable length 4-8"
  - Feeds of 1- and 0.75-L/min – separation efficiencies for algae solids of 78% and 37% [2]
- Process flow diagram



[1] ChemIndustrial Systems, Inc.

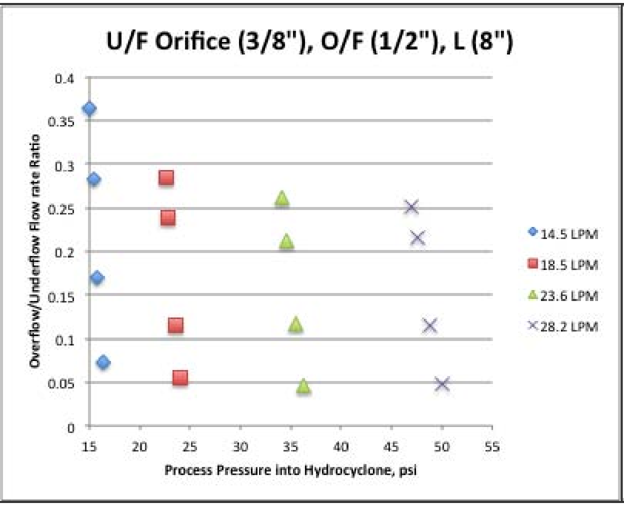
[2] Towler G & Sinnott R (2008) *Chemical Engineering Design: Principles, Practice and Economics of Plant Process Design* (Elsevier, Burlington, MA).

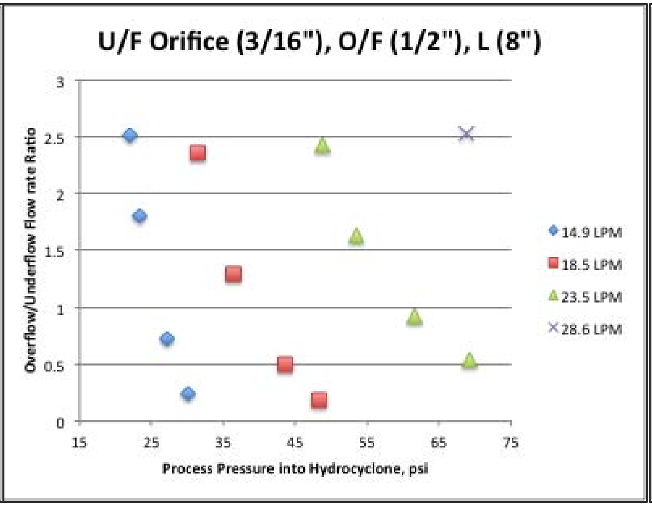
2.A.2 Fabricate Hydrocyclone Test System

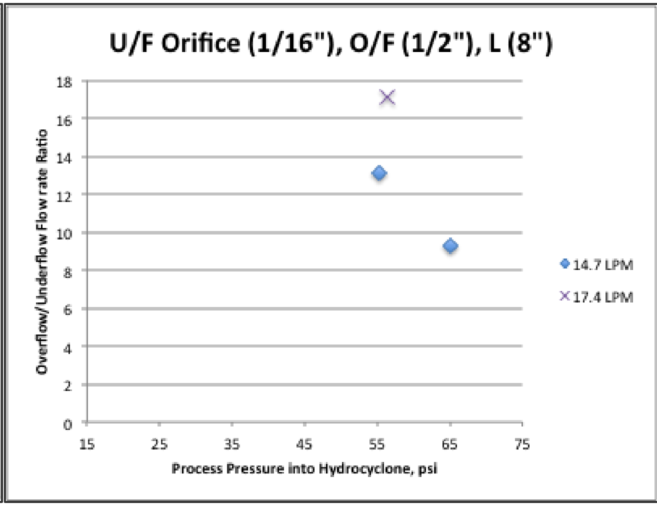
- Hydrocyclone – IQ and OQ completed

2.B Separation Process Metrics – PQ Completed

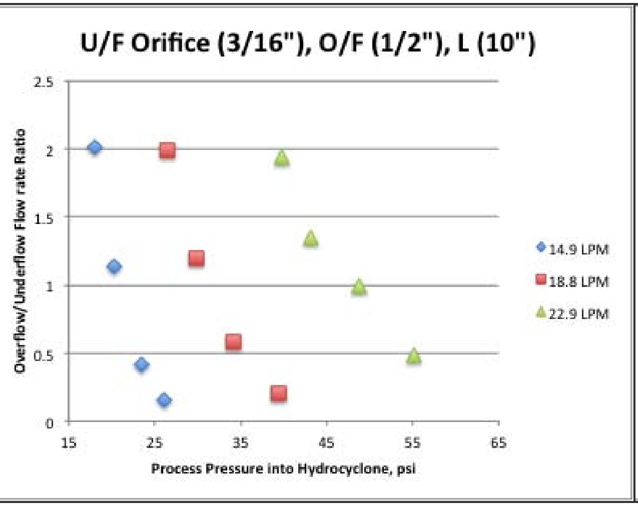
- O/F stream pressure applied by restriction valve
- Residence time is also controlled by hydrocyclone volume

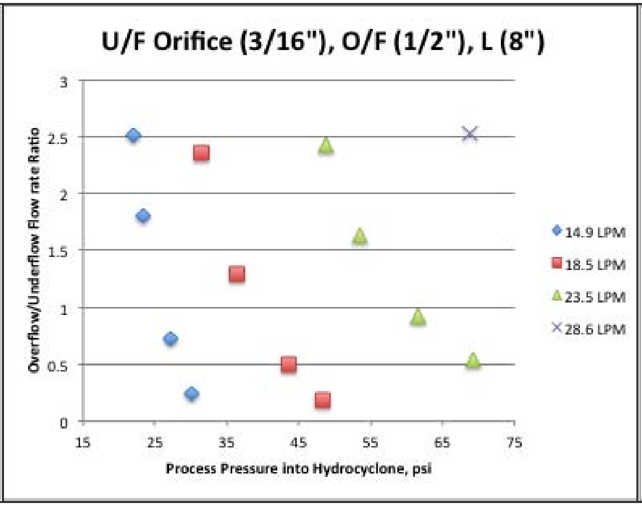


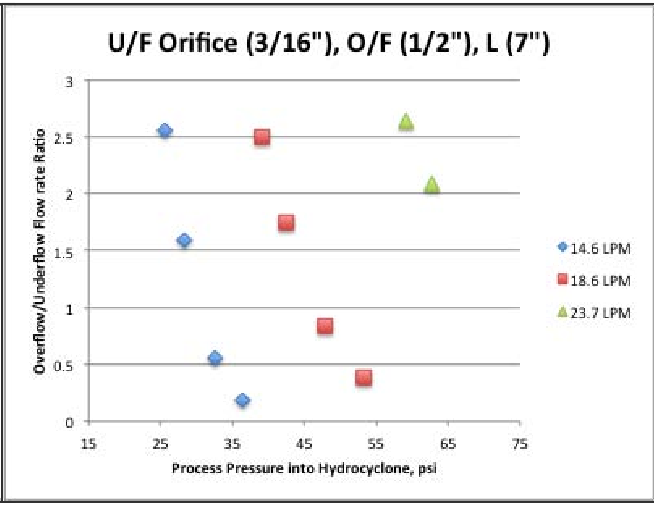


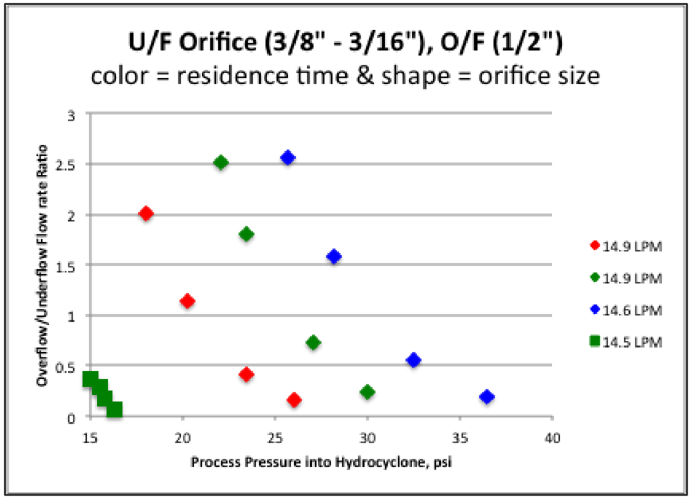


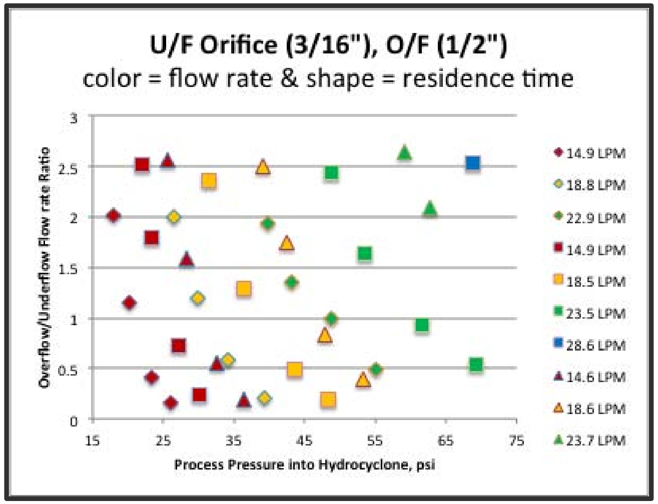
2.B Separation Process Metrics – Residence Time and Process Space



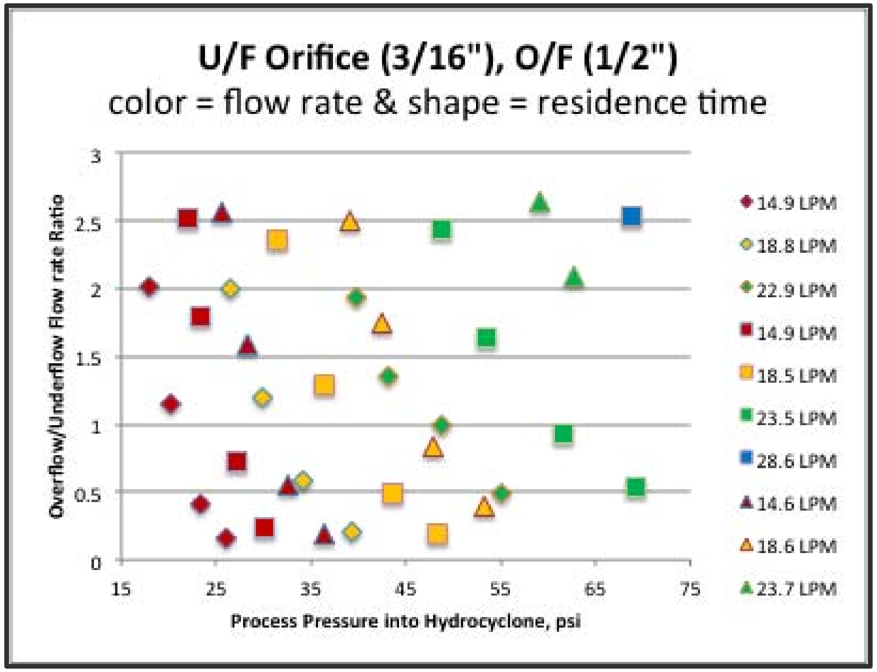


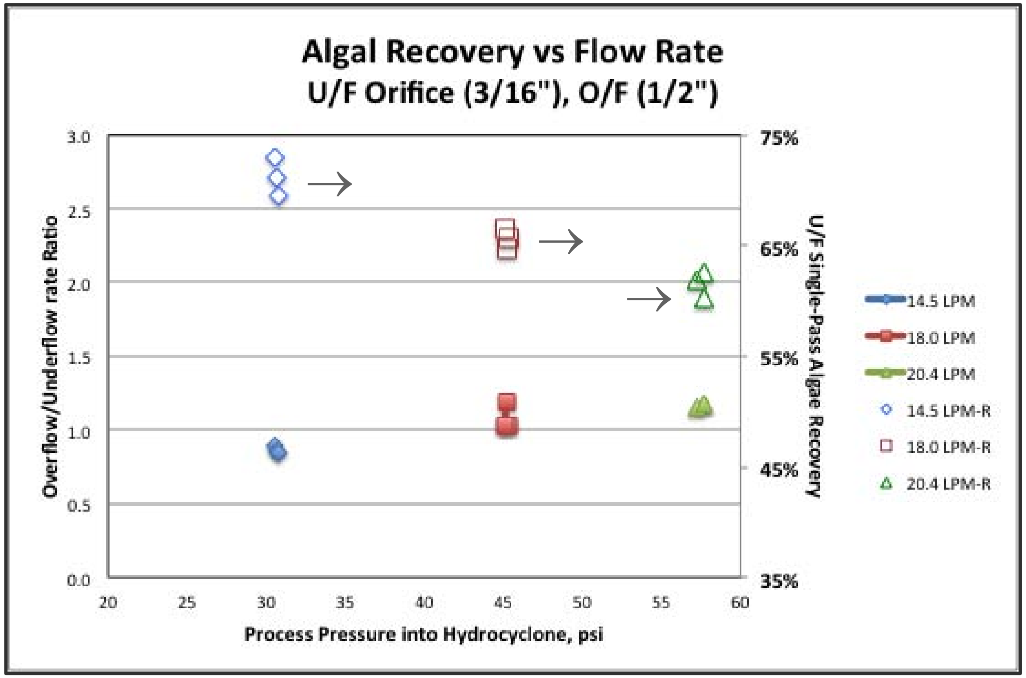






2.B Algal Dewatering – Preliminary Data





Turbulent flow: R_e = 38,900 for 14.5 LPM and 54,600 for 20.4 LPM

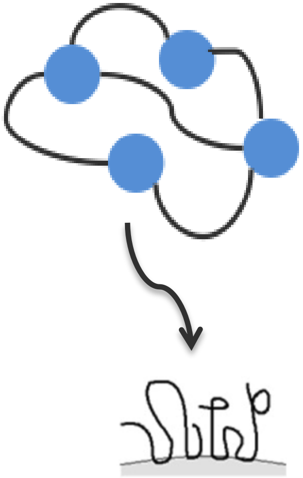
2.C Adsorbent Accomplishments: NP Synthesis

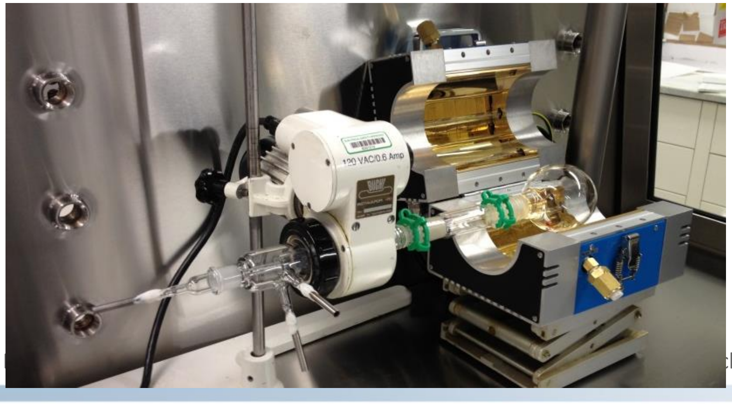
- Superparamagnetic Fe_2Co
  - X Colloidal:[1]
    D ~10-nm; iron chloride and cobalt acetate by polyol reduction at 130°C in ethylene glycol using sodium hydroxide and $H_2PtCl_6 \bullet 6H_2O$ (~ 2.4×10-5 mol/L)
    $15,000/kg
  - – Solid-state reaction of $Fe(NO_3)_3$ and $Co(NO_3)_2$
    Fe_2Co cylinders (D ~10-nm, L ~30-nm) – $350/kg
- Al_2O_3 NP
    (D ~30-nm, spheroidal)

[1] *Adv. Mater.* (2006) **18**: 3154-3159.

2.C Adsorbent: Network and Surface Treatment

- Nanostructured adsorbent (NA) network
  - Chemically bond NP using polymer chains – bi-functional coupling
    - 1,8-bis(triethoxysilyl)octane
    - bis(3-triethoxysilylpropyl)poly-ethylene oxide.
  - Forms elastic network – junction functionality ~ 2.2 to 2.5
- Nanoparticle (NP) surface treatment
  - Heterogeneous vapor-phase polymerization – hydrocarbon adsorption
  - Lyophilic: octyl (C_8), octadecyl (C_{18}), phenyl ($-C_6H_5$)
  - Hydrophilic: hydroxyl (-OH), amino ($-NH_2$), carboxyl (-COOH)
  - ST characterized by TGA, MAS, and CP MAS solid-state NMR techniques
- Pilot process





R. Brotzman, "Industrial Scale Processes for Nanomaterials," Presented at Northwestern, (May 2005).

2.D Feasibility of Hydrocyclone / Adsorbent Process: Next Steps

- Complete dewatering of algal growth medium – determine process conditions to separate algae
  - Metric: > 10%
- Determine effectiveness of hydrocyclone process to harvest lipid
  - Metric: > 50% separation
- Determine effectiveness of hydrocyclone / adsorbent process to harvest lipid – flotation versus magnetic flow separation
  - Metric: > 98% separation
- Complete techno-economic analysis

3. Relevance

AI-B Algal Fuel Production

M.8.1 Algal feedstock production

- M.8.1.1 Development of technically viable, sustainable and cost effective algae production

M.8.2 Algal conversion technologies

- M.8.2.1 Development of technically viable, sustainable and cost effective fuel production from algae

- Applications of expected outputs
  - Dewatering algal growth medium
  - Harvesting algal lipids

4. 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 • Explore NA
NA become entangled with algae cells	• Apply turbulent mixing • Increase residence time in hydrocyclone • Flotation separation
Separation % yield or % concentration low	• Multiple passes through hydrocyclone • Modify adsorbents
Process cost	• Flotation separation • Single pass – multistage hydrocyclone

Future Work

- Through September 30, 2013
  - Complete algae medium dewatering
  - Complete flow specification for flotation/magnetic separation
  - Complete adsorbed product separation
  - Complete techno-economic analysis
- Through September 30, 2014
  - Integrate 2013 separation performance with LCA to establish cost/performance goals
  - Optimize flow and adsorption technology, and incorporate lipid recovery and adsorbent recycling strategies to achieve cost/performance goals
  - Leverage magnetic nanostructure programs to determine adsorbent scale-up metrics
  - Go/No-Go: cost/performance of hydrocyclone/adsorbent versus conventional algal separation processing (coagulation-flocculation-centrifuge-lipid recovery)

Summary

- The objectives are relevant to BETO's Algae Technology Area by exploring novel methods to dewater algae growth media and harvesting algal lipids
- The approach is effective by accessing a large flow separation process space which may be coupled with NA
- The work has many technical accomplishments – mapping complete hydrocyclone process space, NP synthesis, NP surface treatment, and adsorbent network formation
- The work has leveraged technology from ARPA-E, Argonne LDRD, and BETO programs
- Critical success (risk) factors were identified along with mitigation strategies
- Scale-up processing methodologies identified

Additional Slides

- This is a new project
- No prior comments are available