

# Goal Statement

This project focuses on a number of innovative research areas where more development is necessary to make algae derived biofuel an attractive option.

The overall goals of the project are to optimize lipid yield for select algae; conduct feasibility studies (lab and pilot scale) for the use of hollow fiber membranes for delivery of CO<sub>2</sub> for algae growth and to conduct energy and LCA studies for the algae separation processes.

***These goals fall within the Sustainability, Strategic Analysis and Demonstration/Deployment areas of the BETO goals.***

[http://www1.eere.energy.gov/biomass/pdfs/replacing\\_barrel\\_overview.pdf](http://www1.eere.energy.gov/biomass/pdfs/replacing_barrel_overview.pdf)

# Quad Chart Overview

## Timeline

- Project start date: September 1, 2010
- Project end date: December 31, 2013
- Percent complete: 75%

## Budget

- Total project funding \$937,523
  - DOE \$750,000.000
  - Rowan University \$187,523
- Funding received in FY12: \$261,493
- Funding for FY13: \$74,673
- ARRA Funding

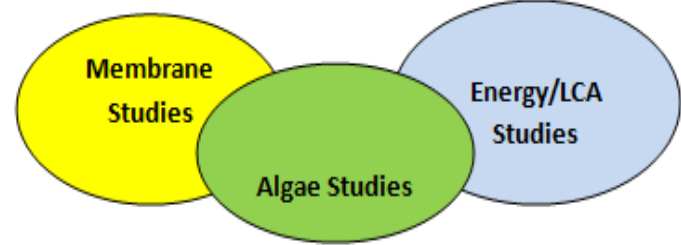
## Barriers

- Barriers addressed
  - Eliminating contamination of alga species
  - Eliminating oxygen buildup during membrane delivery of CO<sub>2</sub> gas

## Partners

- Rowan University
- Garden State Ethanol
- Algaedyne

# Project Overview



Research was conducted in three major areas:

- Algae Growth Studies for Optimizing Lipid Yield
- Use of Membrane Technology for CO<sub>2</sub> Transfer
- Energy and LCA studies

## Objectives

- Determine algae species and optimize lipid yield under various cultivation conditions
- Evaluate carbon dioxide and oxygen gas transfer characteristics of hollow fiber membrane modules
- Evaluate the design criteria of continuous flow algae reactors
- Evaluate the environmental footprint of the downstream processing for algal feedstocks conversion to biodiesel



# 1 - Approach

## MEMBRANE STUDIES

### ALGAE STUDIES

- Thorough literature review to select robust algae strains for lab-scale studies
- Batch studies for optimizing algae growth rate and lipid yield under varying light, CO<sub>2</sub> concentration, carbon source, and nutrient concentrations

- Select microporous hollow fiber membranes (HFM)
- Test hydrophilic and hydrophobic membranes for CO<sub>2</sub> gas transfer
- Evaluate membrane performance under varying pressure, flowrate and surface area
- Provide design data for pilot scale studies

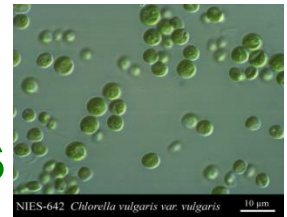
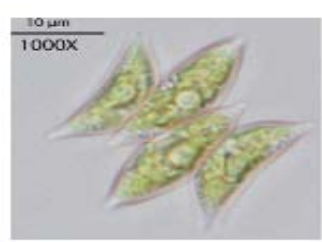
### LCA STUDIES

- Apply life cycle analysis (LCA) to the biodiesel manufacturing processes with emphasis in the separation processes
- Investigate various alternative separation schemes and determine the most efficient process from a carbon-footprint perspective

### MANAGEMENT

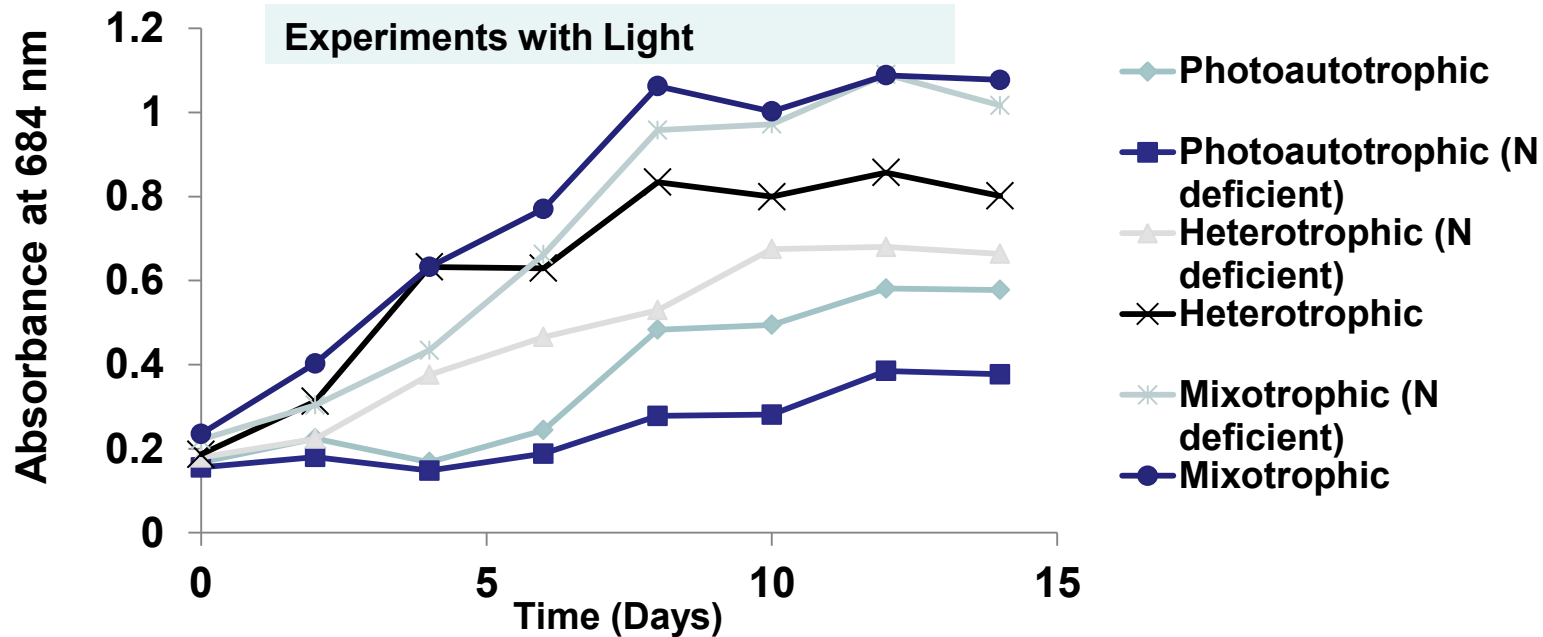
- Weekly meetings with research team
- Communication with sub-awardees
- Quarterly meetings with students and participating faculty
- Information dissemination

## 2 - Technical Accomplishments/ Progress/Results for Algae Studies



- *Scenedesmus dimorphus* and *Chlorella vulgaris* were selected for the study after a thorough literature review
- These were selected due to their easy availability, high chlorophyll content, fast growth rate, easy cultivation and high lipid content
- Select strains of *Chlorella Vulgaris* have shown to respond well under heterotrophic and mixotrophic conditions
- Algae obtained from UTEX (**UTEX 395 and UTEX 1237**)
- Bold's Basal media (BBM) was used for algae cultivation
- Nitrogen source in BBM was sodium nitrate at 1.75 g/L
- Light intensity varied from 200 to 1500 foot candles
- Pure CO<sub>2</sub> concentration varied from 5% to 25%
- Nitrate concentration was reduced by 75% (0.4375 g/L) for the nutrient deficient studies
- Glucose added as organic carbon source

# Results for Algae Studies

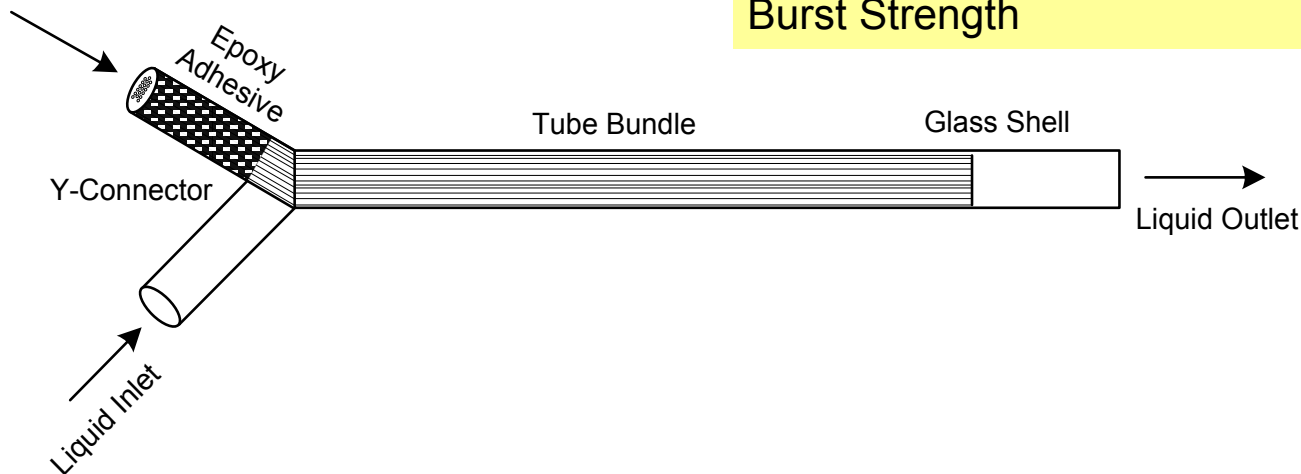


LIGHT			DARK		
Cultivation Conditions	$\mu$ (d <sup>-1</sup> )	Lipid%	Cultivation Conditions	$\mu$ (d <sup>-1</sup> )	Lipid%
Photoautotrophic	0.0415	18.627	Autotrophic	0.0128	9.41
Photoautotrophic (N deficient)	0.0187	23.90	Autotrophic (N deficient)	0.0211	8.74
<b>Heterotrophic</b>	<b>0.2003</b>	<b>22.12</b>	<b>Heterotrophic</b>	<b>0.1451</b>	<b>16.19</b>
<b>Heterotrophic (N deficient)</b>	<b>0.1694</b>	<b>34.02</b>	<b>Heterotrophic (N deficient)</b>	<b>0.1204</b>	<b>16.66</b>
<b>Mixotrophic</b>	<b>0.2177</b>	<b>29.81</b>	<b>Mixotrophic</b>	<b>0.1941</b>	<b>14.85</b>
<b>Mixotrophic (N deficient)</b>	<b>0.1821</b>	<b>33.11</b>	<b>Mixotrophic (N deficient)</b>	<b>0.1339</b>	<b>19.06</b>

# 2 - Technical Accomplishments/ Progress/Results for Membrane Studies

CO<sub>2</sub> transfer experiments were performed using membrane modules constructed with microporous hydrophobic (polypropylene) hollow fiber membranes (Celgard X40-200, Hoechst-Celanese, Charlotte, NC).

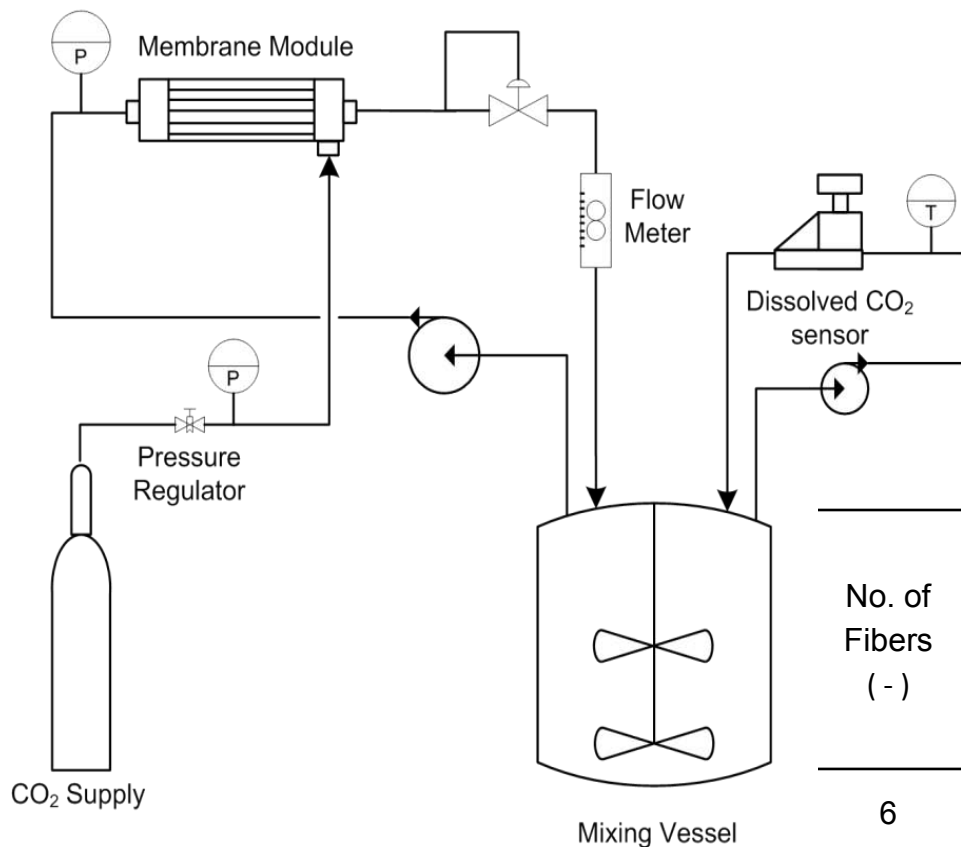
Membrane modules were operated in a sealed-end, parallel flow configuration



## Membrane Characteristics

Characteristic	Value
Fiber outer diameter	300 $\mu\text{m}$
Fiber inner diameter	200 $\mu\text{m}$
Fiber wall thickness	50 $\mu\text{m}$
Pore dimensions	0.04 $\mu\text{m}$ - 0.10 $\mu\text{m}$
Effective pore size	0.04 $\mu\text{m}$
Nominal Porosity	25%
Burst Strength	400 psi

# Results for Membrane Studies



- All of the experiments were performed for  $50 < Re < 2700$ .

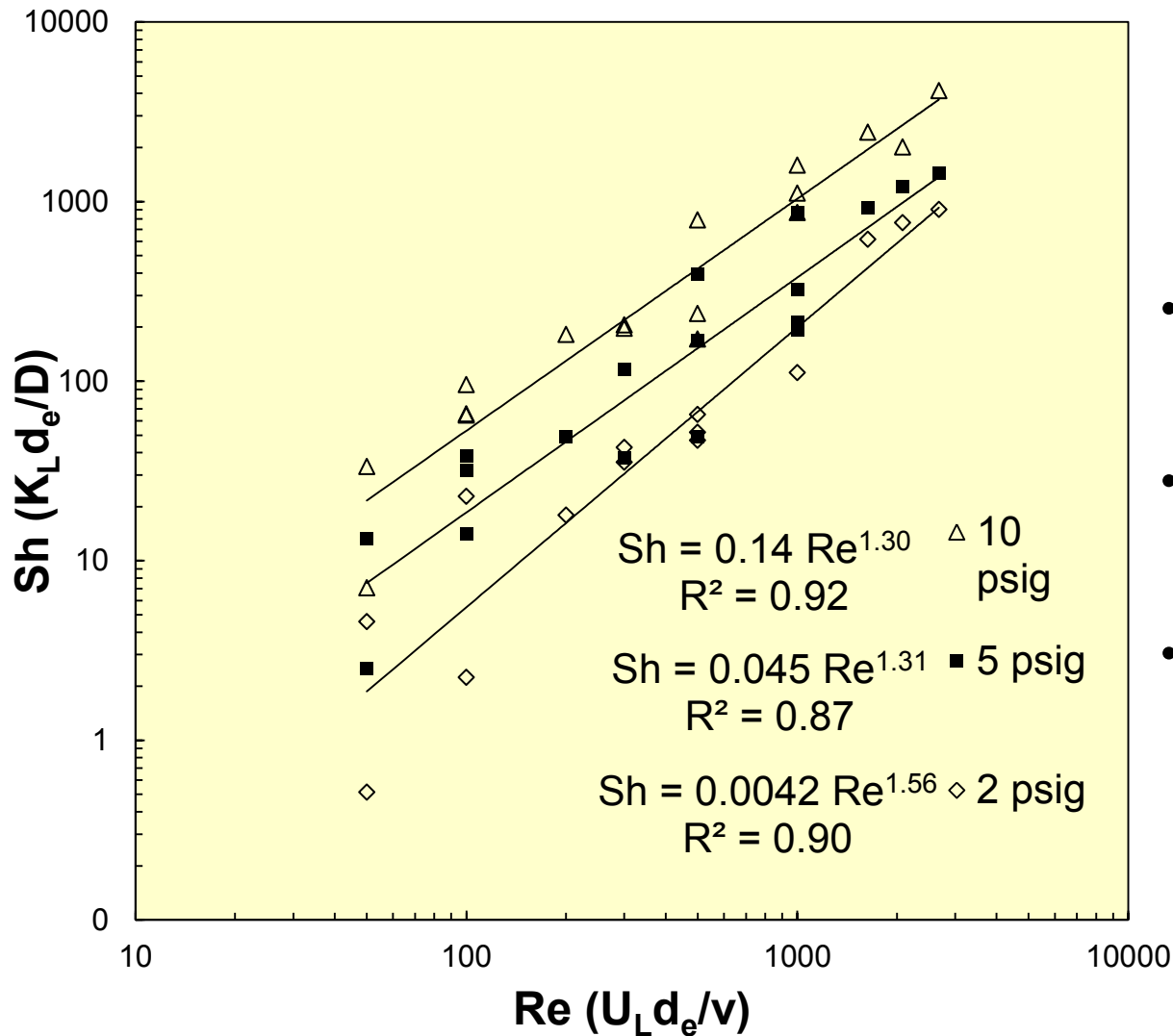
- The overall mass transfer coefficients were expressed in their dimensionless form as the Sherwood number.

No. of Fibers (-)	Fiber Diameter (cm)	Void Frac. (-)	Fiber Length (cm)	Equivalent Diameter (cm)	Surface Area (cm <sup>2</sup> )	Shell ID (cm)
6	0.03	0.966	115	0.267	64.7	0.4
12	0.03	0.933	126	0.196	142.5	0.4
18	0.03	0.899	80	0.153	135.7	0.4
24	0.03	0.865	126	0.124	284.3	0.4
54	0.03	0.924	127	0.244	646.4	0.8
60	0.03	0.916	118	0.225	667.8	0.8

**Parameters Varied:**  
 # of fibers  
 Membrane Length  
 Shell Diameter  
 Flowrate  
 Operating Gas Pressure



# Results for Membrane Studies



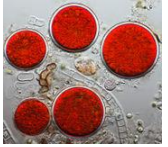
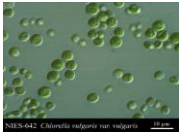
$$Sh = 0.14 Re^{1.30} \quad \triangle$$

$$Sh = 0.045 Re^{1.31} \quad \blacksquare$$

$$Sh = 0.0042 Re^{1.56} \quad \diamond$$

- $Sh$  dependent on  $Re$  as expected
- Significant pressure dependence apparent
- Increasing pressure by 25% increases  $Sh$  by factor of 3

## 2 - Technical Accomplishments/ Progress/Results for Garden State Ethanol (GSE) Studies

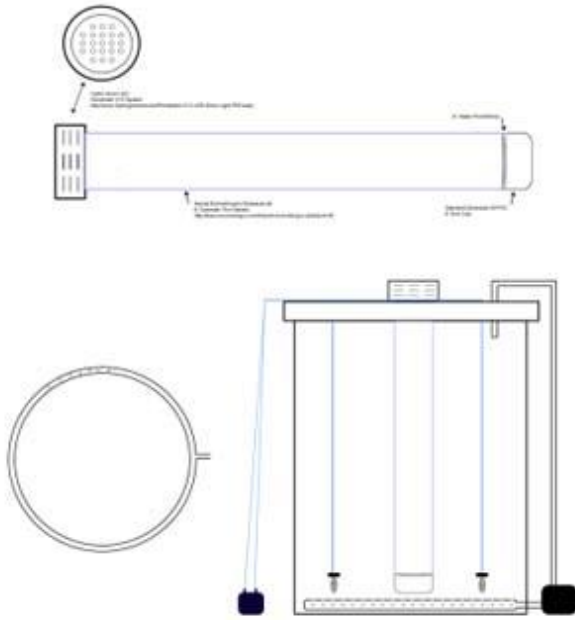


- GSE worked with Algaedyne, its technology company to determine dewatering of harvested algae using various available commercial technologies. GSE directed Algaedyne to conduct several studies of harvesting techniques from its photo-bioreactors.
- *Chlorella Vulgaris* (CV) and *Haematococcus Pluvialis* (HP) were grown in two different sized bioreactors rated by both volume of working fluid, the growth media, and the length of photon injection into the depths of the tanks. The optical and system technology matched the emission wavelengths of LEDs with absorption wavelengths of primary and accessory pigments in autotrophs to maximize growth, and yield for algal cultivation.
- Three methods of dewatering were employed and compared: low temperature evaporation, centrifuge, and Phyco BioSciences' Algaeventures HDD capillary action dewatering system.

## 2 - Technical Accomplishments/ Progress/Results for Garden State Ethanol Studies

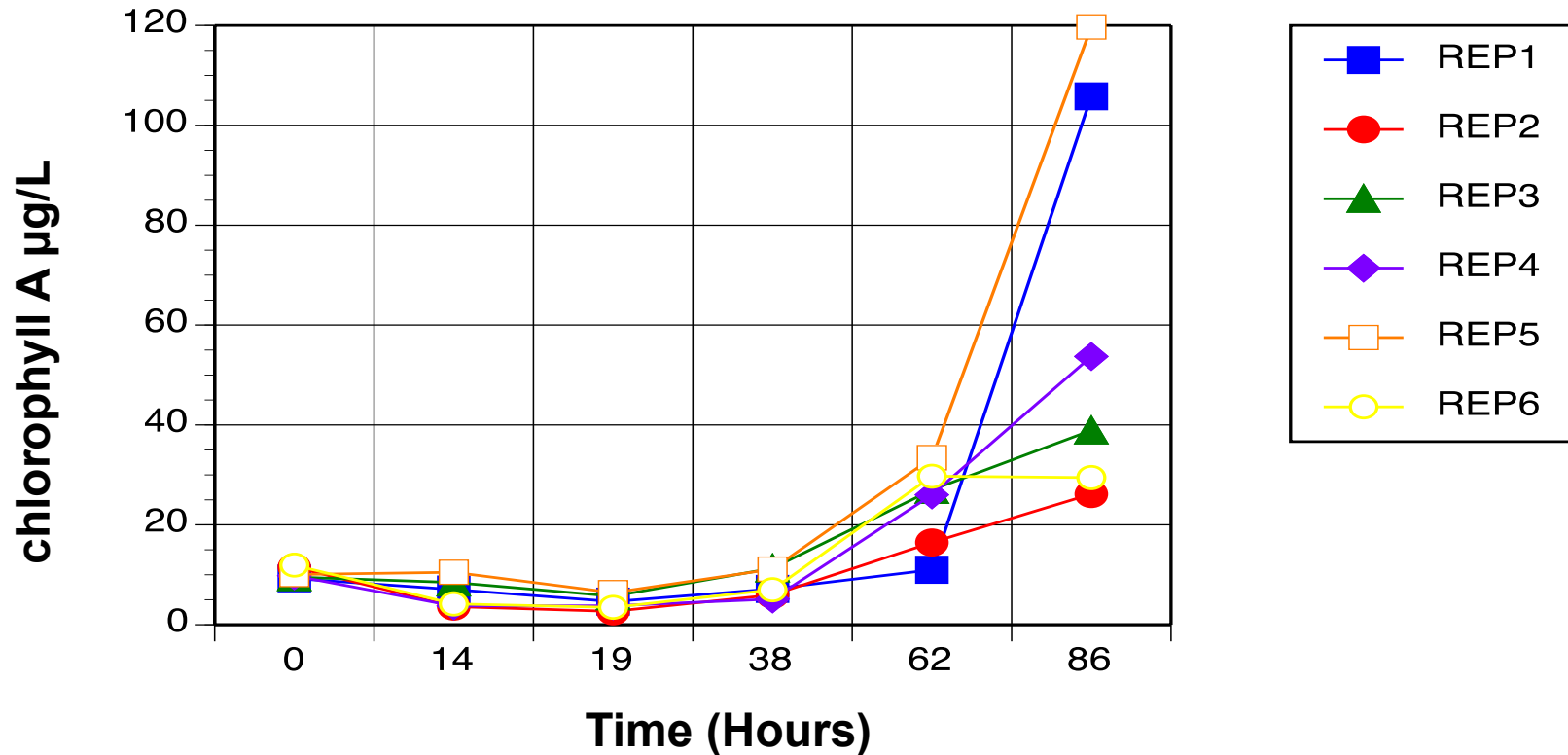
- Harvesting and dewatering algal cultures requires taxa specific procedures.
- Using Ax-10 bioreactors from Algaedyne and the HDD water separation system from Algaeventure, algal biomass was successfully cultivated, harvested and dewatered to a 4-5% dry weight.
- Cultivating HP (*Haematococcus Pluvialis*) requires a harvest method that is low energy but effective in transferring samples with the highest densities of suspended alga.
- A low power pump proved effective to remove settled layers of algae once circulation and aeration was suspended.
- The HDD system provided a practical solution to dewatering the algae to flake and powder achieving the 4-5% moisture content suitable for vacuum packaging and rail transport.

## 2 - Technical Accomplishments/ Progress/Results for Algaedyne Studies



- The AX-50 is a laboratory 50 Gallons (non commercial) scale version of Algaedyne's light injection bioreactor.
- The AX-50 systems are equipped with an onboard pH system attached to a CO<sub>2</sub> cylinder.
- Carbon dioxide is delivered on an as needed basis using a solenoid hardwired to the pH system.
- The system can be converted to discharge atmospheric gasses through the membrane delivery system with control regulated by the pH system.

## 2 - Technical Accomplishments/ Progress/Results for Algaedyne Studies



Replicate paired growth experiments of *Scenedesmus dimorphus* in AX-50 Photobioreactors.

Replicates 1,3, and 5 were equipped with hollow fiber membrane CO<sub>2</sub> delivery and replicates 2, 4, and 6 were sparged with CO<sub>2</sub> via airstone.

## 2 - Technical Accomplishments/ Progress/Results for Algaedyne Studies

- In paired experiments for each of the experimental strains, hollow fiber gas delivery proved superior to standard sparging.
- Average growth for experimental replicates were approximately double for fiber gas delivery systems compared to those with sparge gas delivery.
- This state is valid for both *Chlorella vulgaris* and *Scenedesmus dimorphus*.
- Growth rates for the AX-500 experiment was slower than that seen in the AX-50 system. This is attributed to rate limitation of the fiber system being used in the AX-500, specifically the volume of water was an order of magnitude greater and the fiber system and gas flow rate were unmodified. Despite these modifications, post experimental harvest values were just above 1 g/L dry mass (1.13g/L).

# Technical Accomplishments/ Progress/Results for LCA Studies

- Selection of “base case”
  - 52,300 ton BD/yr “commercial” plant <sup>1</sup>
  - Design based on existing technologies
  - *Scenedesmus Obliquus* - High lipid yields (61.3%) <sup>2</sup>
- Develop life cycle inventories (SimaPro<sup>®</sup> and EcoSolvent<sup>®</sup>)
  - Raw materials
  - Process energy
  - Waste disposal
- 1 metric ton BD/yr basis
- Identify major challenges to processing from LCA perspective



<sup>1</sup> National Biodiesel Board, 2011

<sup>2</sup> Shovon, Mallick, "Microalga *Scenedesmus Obliquus* as a potential source for biodiesel production," *Applied Microbiology and Biotechnology*, 84, 81-291, 2009.

# Technical Accomplishments/ Progress/Results for LCA Studies

- Water removal is required to effectively perform extraction
  - Algae culture is highly dilute (< 2.5% dry algae)
  - Dewatering needs to achieve moisture contents of 15 to 5% <sup>1</sup>
- Achieving this moisture content is major bottleneck in biofuel production
- Algae cells can contain 40 to 80% bound intercellular water <sup>1</sup>
  - Thermal drying required to remove this water



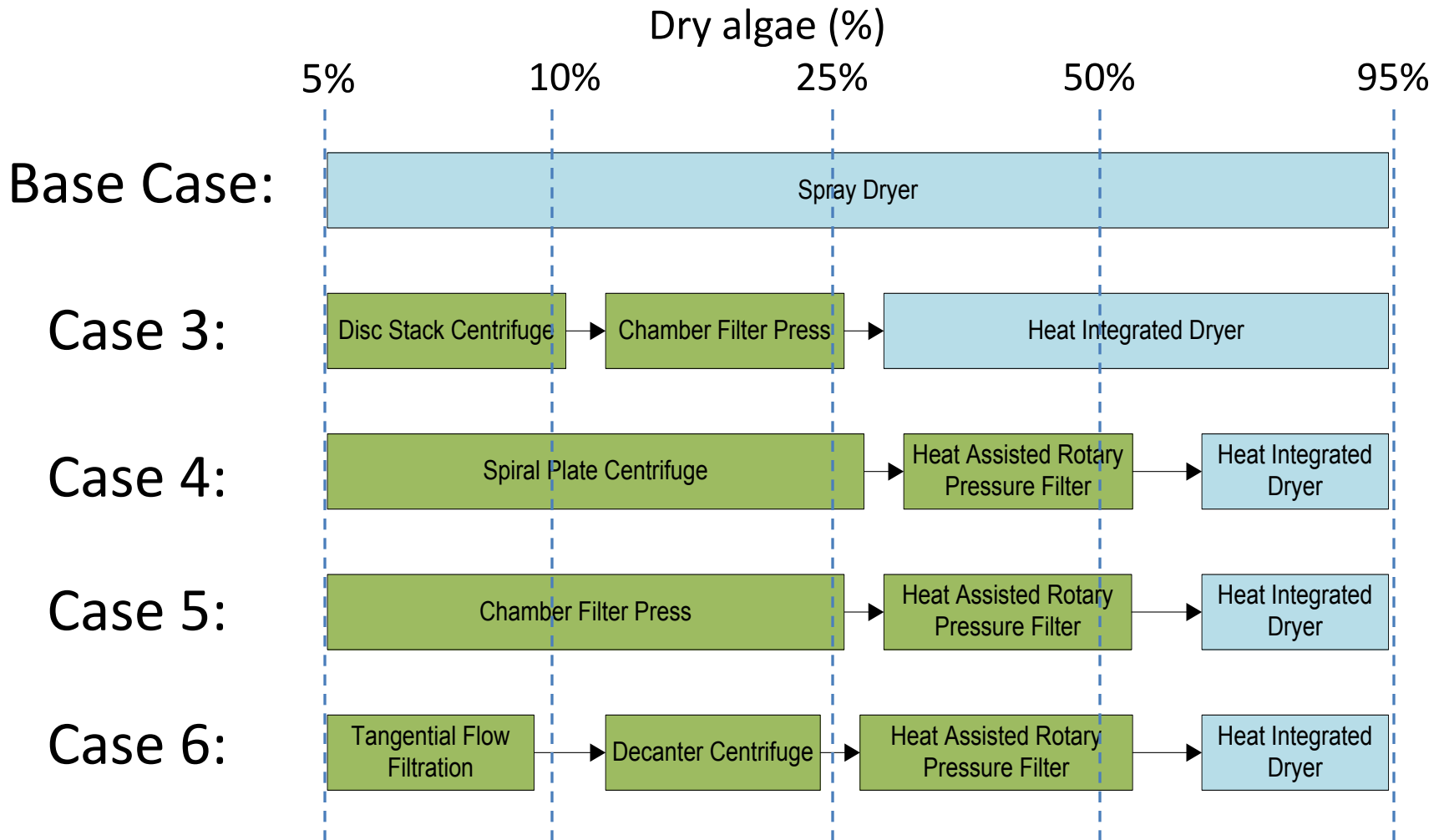
# Technical Accomplishments/ Progress/Results

- Algae dewatering methods

Dewatering Equipment	Initial Water Content	Final Water Content	Energy Consumption	Equipment Capacity
Flocculation	Very High	High	Very Low	Very High
Centrifugation	High	Medium	Medium	High
Filtration	High	Medium	Medium	Medium- High
Thermal Dryers	Medium	Very Low	Very High	High

- Proper thermal/mechanical process selection and sequencing is key to reduce energy consumption

# Technical Accomplishments/ Progress/Results for LCA Studies



# Technical Accomplishments/ Progress/Results for LCA Studies

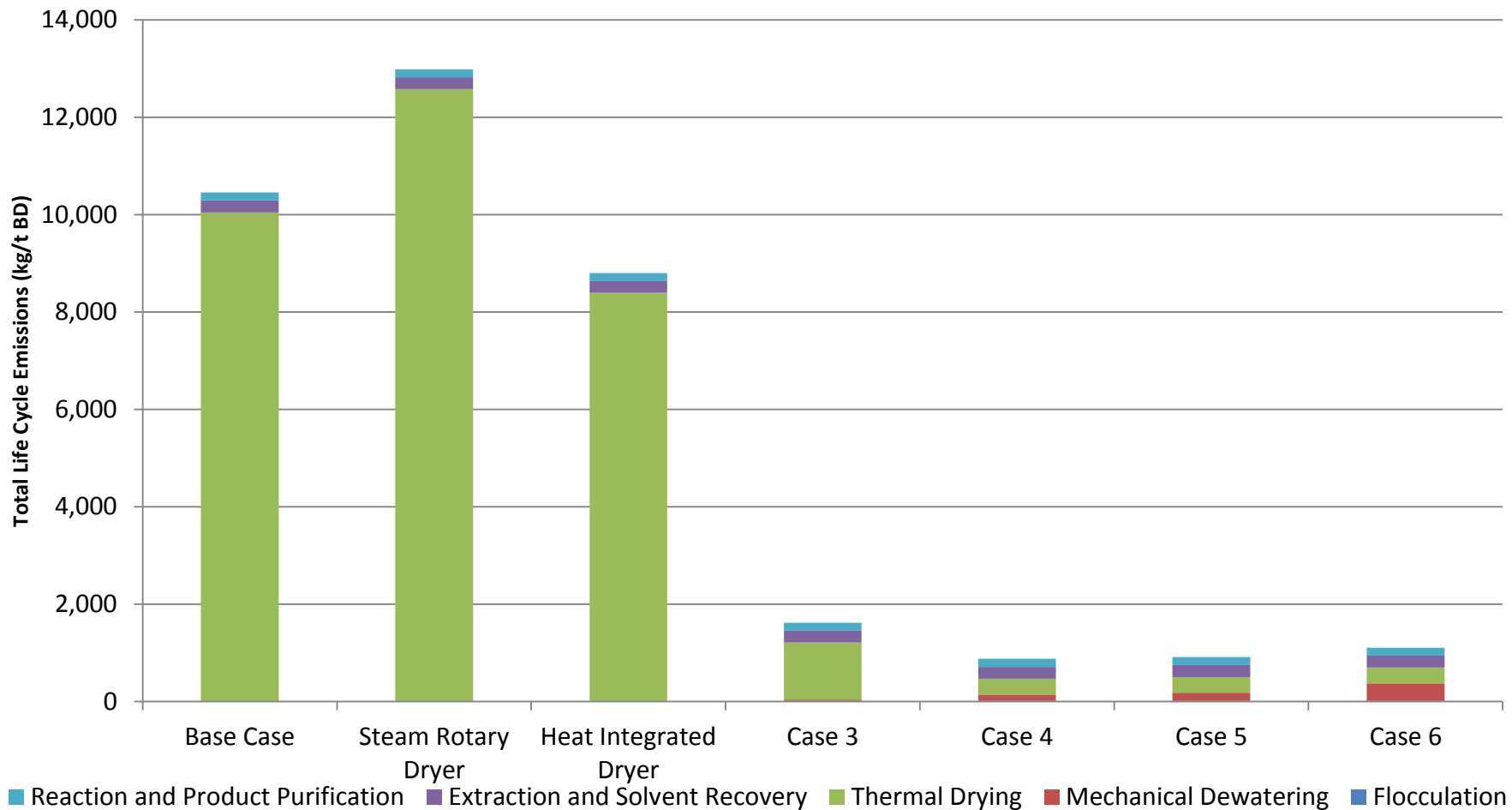
- Evaluation of life cycle emissions of processes

Operation	Dewatering Capability (Dry Weight Algae)	Total Emissions (kg/t water removed)
Disc Stack Centrifuge	12%	0.69
Spiral Plate Centrifuge	31.5%	1.14
Decanter Bowl Centrifuge	22%	10.7
Tangential Flow Filtration	8.8%	1.58
Chamber Filter Press	27%	0.83
Heat Assisted Rotary Pressure Filter	56%	36.9
Spray Dryer	95%	287
Steam Rotary Dryer	95%	230
Heat Integrated Dryer	95%	345

# Technical Accomplishments/ Progress/Results for LCA Studies

- Minimize use of processes like thermal dryers that consume the most energy/generate most life cycle emissions
- Optimize the use of separation technologies
- Evaluate energy of novel downstream processes
- Model developed to confirm design approach
- Outcomes can help guide decision makers in sustainable design of commercial facilities

# Technical Accomplishments/ Progress/Results for LCA Studies



## 3 - Relevance

- The goals of this project fall within the Sustainability, Strategic Analysis and Demonstration/Deployment areas of the BETO goals and objectives.
- The project demonstrated via batch and pilot scale studies the application of membrane technology for efficient CO<sub>2</sub> delivery.
- The project also identified strains that can use organic carbon and allowed identifying optimum conditions for enhancing algae lipid yield.
- The project outcomes help guide sustainable commercial design by reducing the life cycle emissions of downstream dewatering processes.

## 4 - Critical Success Factors

- Algae cultivation experiments indicated that lipid yield is enhanced under nutrient deficient conditions at 400 foot candles in the presence of an organic substrate
- Not all algae species can use organic carbon
- Mixed culture studies may not result more lipid yield
- Membrane delivery of CO<sub>2</sub> enhanced algae growth in pilot scale studies
- Harvesting of algae is critical to avoid membrane biofouling
- Minimize use of processes like thermal dryers that consume the most energy/generate most life cycle emissions
- Optimize the use of separation technologies
- Evaluate energy of novel downstream processes

## 5. Future Work

### Current Studies

- Complete current membrane pilot-scale studies
- Prepare final report for the DOE
- Complete journal submissions and continue with technical presentations

### Future Studies

- Results of membrane pilot-scale studies at Algaedye will influence the use of membrane technology in commercial algae cultivation
- Investigate membrane technology for algae separation
- Investigate the use of materials that will allow temperature control, provide light for growth and attract algae during the harvesting period eliminating major dewatering costs- an AlgaeMag
- LCA Conversion R&D - Telescoping alternatives – techniques that perform multiple operations in one step
- Sustainability – Apply LCA approach to new technologies investigated



# Summary

## Relevance of objectives

Optimize cultivation conditions  
Test new gas delivery method  
LCA of downstream processes  
Pilot scale testing

## Approach

- Batch studies for optimizing cultivation conditions
- Lab scale membrane studies
- Pilot scale testing for dewatering and gas delivery
- LCA of downstream processes

## Technical Accomplishments

- Identified optimum light and nutrient deficient conditions
- Developed a membrane module for efficient CO<sub>2</sub> delivery to promote algae growth
- Identified to minimize use of processes that consume the most energy/generate most life cycle emissions

## Future Work

- Testing of new materials
- Membrane for algae separation
- Telescoping processes to optimize energy utilization

## Success Factors & Challenges

- Avoiding contamination
- Avoid oxygen buildup during membrane assisted CO<sub>2</sub> delivery
- **High algae growth rate with membrane assisted CO<sub>2</sub> delivery**
- **LCA reduction**

## Technology Transfer

**Feasibility of membrane delivery of CO<sub>2</sub> gas to promote algae growth looks promising in pilot scale studies**