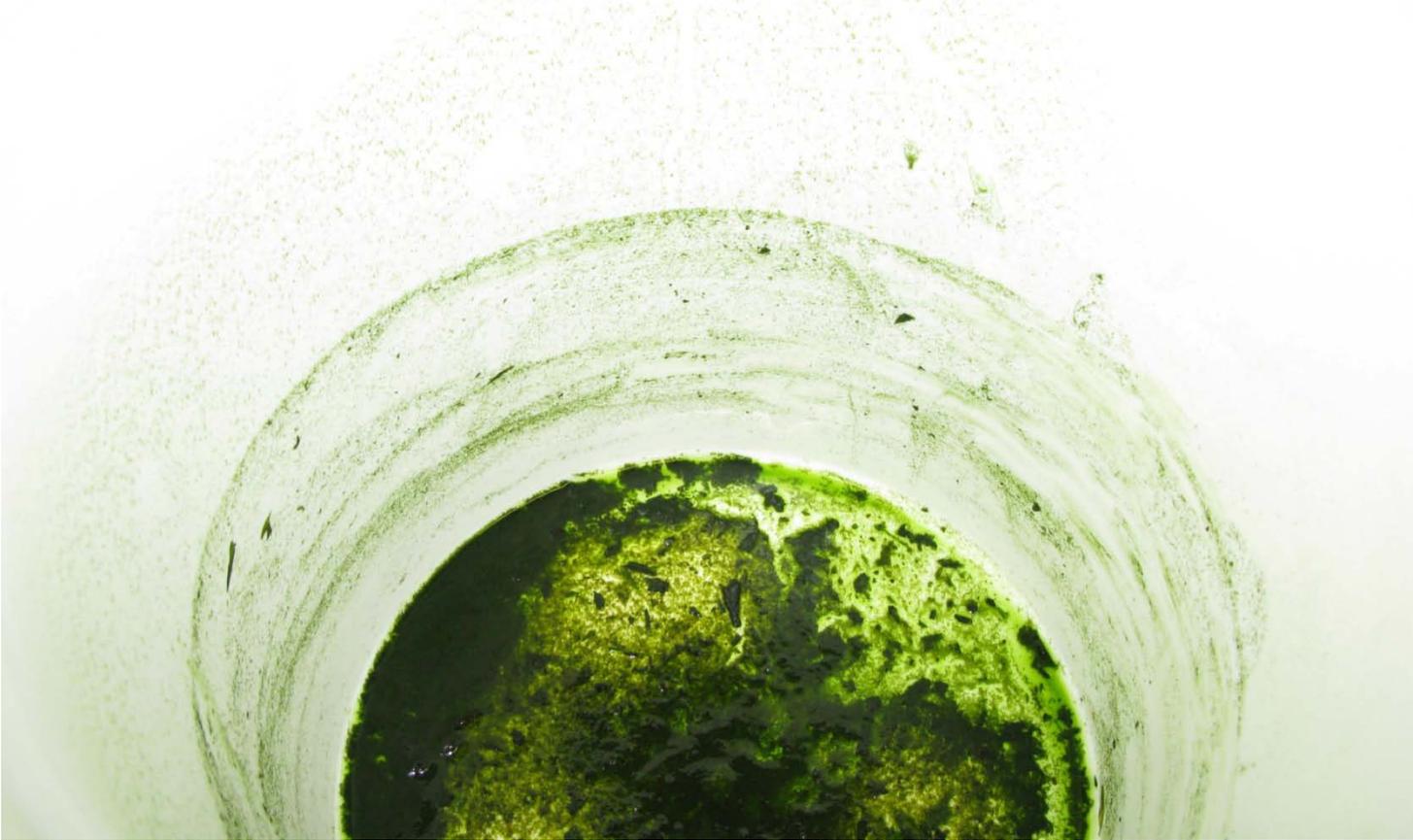


# Oil Extraction from Microalgae



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## Abstract

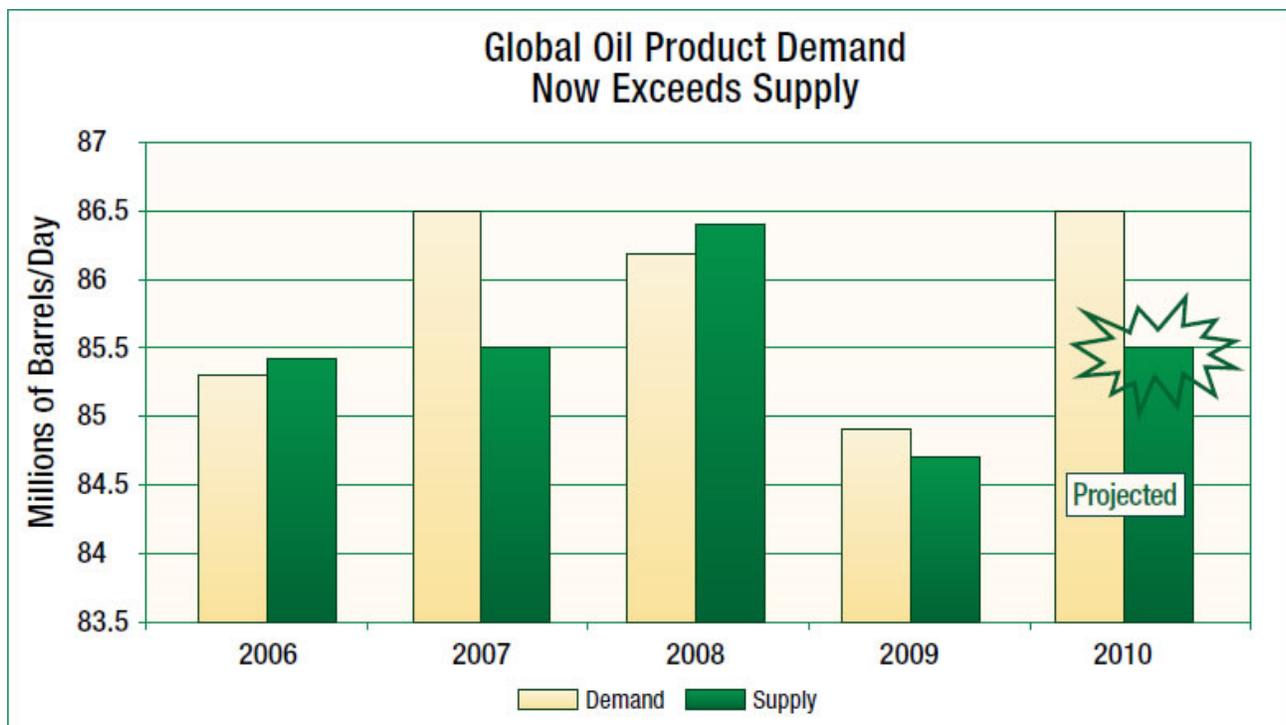
Ideally, alternative fuel sources would be utilized to their full potential and bridge the gap between the current supply and demand of oil. Unfortunately, many alternative fuels have not been researched extensively and are not being fully utilized. Alternative fuel has become one of the largest growing areas of research in recent history because of the environmental and economical impacts. It has been the goal of the algae oil extraction capstone to research a potentially extremely viable source of alternative fuel, biodiesel from microalgae. The goal of the capstone algae group has been to develop a low energy input process to bridge the technological gap between the well known process of growing algae and the process of conversion of the algae's vegetable oil to biodiesel. By developing a concept scale

algae belt dryer in conjunction with plans for a hexane extraction unit, the capstone algae group has started paving the way for the University of Maine to enter a new era of alternative fuel research and development.

## 1.0: Project Importance: The Need for Alternative Fuels

### 1.1 Background

The United States of America is the largest consumer of oil in the world. As of 2008, the US consumed 19.5 million barrels of oil per day, on average, with a production of only 8.154 million barrels per day. (Central Intelligence Agency, 2008) As shown in Figure 1, in recent history, 2008 was the last year that global oil supplies were greater



Source: IEA Oil Market Report – 11 February 2010 © OECD/IEA 2010  
Source of 2010 Supply Estimate: OriginOil, Inc.

**Figure 1: Global Oil Product Demand. (The Need For A New Oil, 2010)**

than the demands globally. As of 2009 around 84.9 million barrels of oil were consumed daily, with a supply of 84.6 million. For 2010, staggering predictions were made that the oil demand will exceed availability by nearly 1 million barrels of oil per day. This difference has a great affect on oil prices and certainly contributes to the reason prices have skyrocketed in recent history. Such high oil consumption has many implications. The first major global implication is that, since astronomically high amounts of oil are being consumed, there is an ever increasing release of polluting emissions to the atmosphere. The second concern is how the oil demand gap can be met in order to keep providing affordable energy. Many companies worldwide are currently addressing these two issues. What if it were possible to create a carbon neutral fuel source? Though this may never be fully achievable, there are biological alternative fuel sources currently being researched and produced, called *biofuels*. A biofuel is any fuel derived from biomass, organic matter that has some form of stored energy. Many high yield crops such as corn, soy, rapeseed, sunflower, and most recently algae have been researched for the feasibility as a fuel source. Many of these sources, in particular corn, have become discredited after further investigation of a life cycle analysis, as they have an overall greater environmental impact than they solve. Biofuels derived from algae, however, appear to be the most feasible at present date. Certain species of algae have a particularly high growing rate, which allows for frequent harvesting, and subsequently conversion to biofuel. A comparison of several biofuel sources and

their oil production potential on a per acre basis is shown below in Figure 2. Figure 2: Crop potential of algae.

Plant	Production (gal/acre)
Algae	20,000
Corn	13
Soy	48
Safflower	83
Sunflower	102
Caster	150
Rapeseed	171
Jatropha	192
Jojoba	192
Coconut	290
Palm	640
Algae (Autotroph)	3,000-6,000

**Figure 2: Crop potential of algae.** (Algaeventure Systems, a Univenture, Inc. company, 2010)

Algae is clearly a promising solution for bridging the gap between the supply and demand in the oil industry. In addition, its production might be relatively environmentally sustainable, as long as the oil production processes needed to create the fuel are not energy intensive.

## 1.2: The Technological Challenge

Although approximately 30,000 distinct species of algae exist in a variety of sizes and colors, only a select few are able to be coaxed into producing a high yield of lipids. The clear choice at the University of Maine with the expertise of Dr. Xinfeng Xie was *Chlorella photothecoides* and *Chlorella sorokiniana* microalgae.

Both *C. photothecoides* and *C. sorokiniana* are single-celled green algae with an approximate diameter of 2-10 microns. A microalga, such as *C. photothecoides*, is of particular interest, because of its ability to contain up to 15% lipids by mass when grown under phototrophic conditions; while the lipid yield can reach 55% under heterotrophic grown conditions (Wu, 2006). Algae can either be grown in a photo-bioreactor utilizing the process of photosynthesis (phototrophic), or in a light absent environment with addition of sugars through a heterotrophic process. Currently, in Nutting Hall on the University of Maine campus, three 100-gallon photo-bioreactors are producing *C. sorokiniana* on an 8-day cycle. *C. photothecoides* is also being grown, but at a small level. The growth of the algae itself is a well defined and published science; however, extraction of the lipids through the 0.5 micron pores continues to be a secretive, problematic, and not well understood process.

The extraction of oil from crops such as soy is relatively easy due to the macroscopic size of the media being used. Simple inexpensive grinding devices with relatively imprecise machining tolerances can be used and will still extract oil. The microscopic nature of *chlorella spp.* and the tough flexible cellulose cell wall, however, make extraction extremely difficult.

## 2.0 Project Overview

This report describes the three key stages of our project to develop a viable extraction process. First, we needed to experiment with various extraction processes to determine

which process held the most promise for future development. Second, once it was discovered that a dry extraction process using hexane as a chemical medium would be worth pursuing, we then needed to begin work on a system for drying the algae. Finally, we needed to design an efficient system for processing the algae to release its oils.

## 3.0 Experimentation: The difficulties of microalgae oil extraction

### 3.1 Two Methods of Extraction

Over the course of the first semester, extensive experimentation and research was conducted to determine the best extraction method. The experiments themselves consisted of two types, wet extract processes that focus on disrupting the algae cells in solution, and dewatering methods which remove the algae from aqueous water solution and then mechanically or chemically disrupt the cells. The two *Chlorella spp.* that were used grows in solution at 99% water by mass, which makes a wet extraction process extremely advantageous; however, achieving results proved difficult.

Ultimately, Dozens of wet extraction experiments were conducted, including 4 techniques and various combinations of each of these techniques.

## 3.2 Wet Extraction Methods

### 3.2.1 Freezing

#### *Theory*

Ice is 8% greater in volume than liquid water. The desired effect is that the expansion of the water inside the cell as well as the water around the cell will cause the cell walls to rupture from the inside out or be disrupted by the compressive forces.

#### *Procedure*

1. Two algae solutions of high and low concentration were dispensed into two 10 ml beakers and placed within a freezer until the solutions became frozen.
2. The solutions of algae were then thawed by placing the beakers into warm water.
3. A sample of the lower concentration of algae was collected and placed under a microscope for inspection.
4. The remainder of the less concentrated solution as well as the high concentrated solution was placed back into the freezer for a second session.
5. The two solutions were then thawed and samples of each were taken and inspected under a microscope.
6. The remainders of the two solutions were then placed back into the freezer for a third session.
7. Samples of the two solutions were thawed for a third time and were then inspected under a microscope.

#### *Results*

After inspecting the two samples under the three freeze cycles, it appeared as though the algae cells began to cluster together. However, when the samples were compared to the control sample (the algae sample that did not undergo any freezing) it seemed

evident that the cell walls were still intact without experiencing rupture.

### 3.2.2 Homogenization

#### *Theory*

Homogenization is the process of reducing the size of particles in a mixture so that the media is uniform throughout. The process is usually done by expelling mixtures through small valves at high pressure. To test the viability of homogenization as an option for extracting fuel oil from the chlorella algae, two different tissue grinders of different homogenizing capabilities were tested, similar to the one shown below in Figure 3.



**Figure 3: Tissue Grinder**

#### *Procedure*

Two tissue grinders were borrowed from Nancy Kravit in the Biological Engineering Department at the University of Maine. The grinders are comprised of glass pestles that fit tightly inside glass cylinders. The spacing between the pestles and cylinders are tightly toleranced. Both grinders were designed to homogenize mammalian cells: one to break up the cells and one to break up nuclei. Because the homogenizers were

designed for use with mammalian cells, which, unlike algal cells, do not have cell walls, the effectiveness was uncertain.

### **Results**

Using two different concentrations of decanted aqueous solution of algae, referred to as slurry, in the two separate tissue grinders, what appeared to be homogenized samples of algae were produced. After examination under microscope, the non-homogenized algae cells looked the same as the homogenized algae cells. It was clear that the mammalian cell homogenizers were ineffective in breaking the cell walls, and no oil was released.

During the grinding process, it was noticeable that small flakes of algae were escaping past the grinding pestle in the glass stator. The problem could be that the tissue grinders used were not of fine enough tolerancing to destroy the algae cells. Homogenizers having nano-tolerancing are a potentially viable source of extracting oil on a lab scale; however, they be extremely difficult to reproduce on an industrial scale.

### **3.3.3 Sonification**

#### **Theory**

Ultrasonic waves could be used to induce cavitation bubbles adjacent to the algae cell wall. The cavitation bubbles should create a pressure gradient great enough that the cell wall will collapse and release its' contained oil.

#### **Procedure**

Algae is dispensed into six separate beakers, each consisting of 40 ml of algae. Each of the six tests vary within time and output

from a Branson Sonifier 450, while keeping the duty cycle at a constant setting.

Test 1:

*C. photothecoides* algae are kept in the sonifier for 1 minute with an output of 1.

Test 2:

*C. photothecoides* algae is kept in the sonifier for 1 minute with an output of 5.

Test 3:

*C. photothecoides* algae is kept in the sonifier for 1 minute with an output of 10.

Test 4:

*C. photothecoides* algae is kept in the sonifier for 5 minutes with an output of 1.

Test 5:

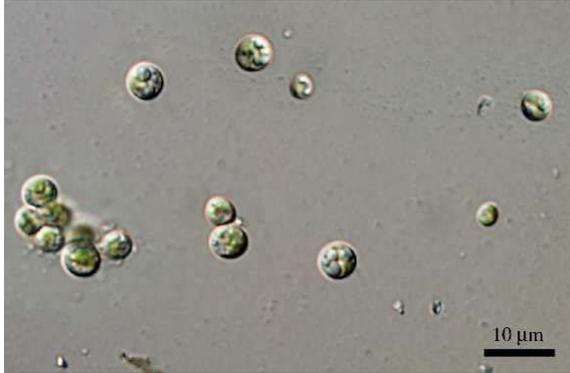
*C. photothecoides* algae is kept in the sonifier for 5 minutes with an output of 5.

Test 6:

*C. photothecoides* algae is kept in the sonifier for 5 minutes with an output of 10.

#### **Results**

Using a 100X power microscope, the sonified algae specimens were observed in conjunction with a control sample. [Figure 4](#) shows algae similar to that observed in the experiment. The specimens looked identical to those of the control, indicating that the cells were neither destroyed nor disturbed.



**Figure 4: Undisturbed *Chlorella* spp. Cells**

### **Conclusions**

*C. photothecoides* algae cell walls are too malleable and do not rupture when subjected to the cavitation bubbles created by the Branson sonifier 450 at the power levels capable by this device. A lower concentration of algae could be used to test if an increase of cavitation bubbles per algae cell might cause the cells walls to collapse or break. Additionally, other sonifiers capable of different frequencies than that of the Branson 450 potentially could achieve the proper frequency to disturb *C. photothecoides* cell walls.

### **3.3.4 Osmotic Shock**

#### **Theory**

Cell membranes are permeable to many solutes. If a cell is placed in solution, the solute will diffuse across the cell membrane, provided the solute is small enough. If the solutes were suddenly removed from the water surrounding the cell, there would be a net transfer for water into the cell. At the same time the solutes will also diffuse across the cell membrane into the water in order to equalize the concentration inside and outside the cell. If the concentration of solutes in the water is dropped quickly

enough, water can engorge the cell causing cell lysis. This process is called osmotic shock and this procedure was tested extensively during the fall semester in hopes the lysing of cells would liberate contained oils.

For osmotic shock to work on a large scale, an economical solute and dilution technique is required. Dr. Xienfeng Xie suggested the use of carbon dioxide, a relatively inexpensive and readily available commodity. Introducing pressurized CO<sub>2</sub> to an algae solution, similar to the carbonation process used for soft drinks, would allow the gas to dissolve into the solution and form the carbonate ion, CO<sub>3</sub><sup>2-</sup>, and diatomic hydrogen, H<sub>2</sub>. These solutes drive the osmotic shock process. Having found a way to introduce a solute to the algal solution, a method of rapidly decreasing its concentration was required for the osmotic shock process to have a chance of rupturing the algal cells. Two methods of diluting the solution of the carbonate solute were tried:

- Ultrasonic pulsation
- Throttling the solution through a needle valve

The ultrasonic pulsation involved placing the solution inside of a beaker, which was then placed into a water bath. The beaker once in the bath was bombarded with ultrasonic waves, causing a visible liberation of the carbonate ions, similar to rapidly shaking a soda to release carbonation. The throttling technique consists of slightly opening the throttling needle valve, such that the velocity increases to the point where the algal cells atomize. The throttling

technique was employed, because it was thought that it would increase the dilution process rate.

### ***Experimental Device and Procedure***

The device used to carbonate the algal solution is shown in Figure 5 and 6 below. The body was constructed of a section of black iron pipe, a black iron pip reducing tee, 2 reducers, and various Swagelok fittings.



**Figure 5: Osmotic shock throttling valve**

### ***Carbonation Procedure***

Algal solution is added via syringe through the top port. The port is then capped and pressurized with the CO<sub>2</sub> between 20 and 40 psi and connected to the other port. The device is shaken to promote diffusion of CO<sub>2</sub> throughout the solution.

### ***Throttling Procedure***

In order to liberate the CO<sub>2</sub> from solution by throttling, a small needle valve was used. The valve was connected to the algae port on the pressure device. The valve was fed by a tee fitting, with one inlet from the algal solution in the pressure device and another inlet from a compressed air tank. The pressure device was pressurized with CO<sub>2</sub> to the same pressure as the compressed air, to ensure that the algal solution would exit. When the needle valve was opened, the algal

solution was allowed to drain from the pressure device through the valve. The solution became entrained in the compressed air flow and was forced through the valve at high velocity. The resulting turbulence caused the liberation of CO<sub>2</sub> from the device, as shown in Figure 6.



**Figure 6: Throttling procedure using rapid pressure release**

### ***Ultrasonic Pulsation Procedure***

The ultrasonic pulsation was supplied by an ultrasonic cleaner, which consisted of a water bath vibrated at a high ultrasonic frequency. The algal solution was placed inside a beaker and was then placed into the bath. This caused the liberation of CO<sub>2</sub> from the solution.

### ***pH Tests***

Prior to thorough testing of the osmotic shock device, it was necessary to determine if it was probable that osmotic shock was capable of occurring with the algae cells. Since dissolving CO<sub>2</sub> forms H<sub>2</sub> molecules, it raises the acidity of the solution. The corresponding rise in acidity results in a lower pH value than initial. Therefore the amount of CO<sub>2</sub> dissolved can be qualitatively gauged by testing the pH of the solution before and after carbonation. The carbonation procedure was performed on several samples of water and the pH was tested before and after using a litmus test. It was found that in the carbonated water the pH dropped approximately 1 point of the 14 point scale. This test was repeated for samples of algae and yielded the same results. The same water samples were then subjected separately to the throttling procedure and ultrasonic pulsation procedure. It was found that the pH of the solution was raised to the original level within a value of 0.1, after both throttling and ultrasonic procedures. This indicates that H<sub>2</sub> molecules were formed during the carbonation process lowering the acidity, and the process could undergo osmotic shock by returning to the initial pH value.

### ***Experiments and Results***

The osmotic shock protocol was tested for a variety of concentration of algal solution. Ultrasonic pulsation and throttling were employed separately and sequentially on samples to test the effectiveness of each dilution technique. The following concentrations were tested:

- Several preliminary tests were performed on algal solutions approximately 0.1% by mass
- A decanted solution was used to produce four different concentrations:
  - Full concentration (1% algae by mass)
  - ¾ concentration
  - ½ concentration
  - ¼ concentration

Samples of each concentration tested were used as a control to compare under microscope in order to gauge the level of cell disruption caused by osmotic shock.

After performing the osmotic shock protocol, slides of the samples were taken to be examined under a 100X microscope. The remainder of each sample was centrifuged so that any liberated oil would be visible.

### ***Results***

Of all experiments run with the osmotic shock protocols, only one seemed to show liberated oil. These were only preliminary tests and were not highly controlled. Although this single test was the cause of much excitement, optimism soon fell due to inability to duplicate results. The samples treated with ultrasonic pulsation showed little disruption to the algal cells. Samples treated with the throttling procedure showed moderate disruption and those treated with both showed significant disruption. After the samples were centrifuged, it was apparent that despite cell disruption, oils were not liberated from the cells. Increasing the algal concentration appeared to decrease effectiveness of both procedures.

### 3.3 Dry Extraction using Hexane

#### 3.3.1 Hexane Extraction

The aforementioned experiments all required a wet extraction method; however, none of them yielded the desired results, release of oil from the *C. photothecoides* algae cells. As a result, one final method was tested, Hexane extraction. This method unlike the other requires dry algae flakes. Therefore, a drying system would have to be created in conjunction with a hexane extraction system.

#### **Theory**

Oil that is present inside of the single cell algae is trapped by the cell wall and plasma membrane, which inhibits its ability to easily be exported from the cell. When the algae cell is dried, the plasma membrane is degenerated and weakens the cells ability to retain the oil. When the hexane, an organic solvent, is introduced to the dry algae sample, the cell wall is penetrated by the hexane and the oil within the cell is dissolved. When the hexane is removed from the algae sample, the oil dissolved in the hexane is transported through the cell wall and effectively removed from the Algae cell. The collection of the oil is done by evaporating the hexane off, which will leave the algae oil behind.

#### **Procedure**

The algae used in the experiment has been dried under a heat lamp and crushed into a fine powder. The algae powder has been dispensed into a paper container and enclosed to withstand any solid algae discharge. The container has been arranged within an extraction chamber and successfully prepared for hexane extraction.

The extraction process follows these systematic steps.

1. Hexane is heated within the miscilla tank, creating vapor rising to the condenser.
2. The hexane then condenses and is released into the extraction chamber with the algae.
3. The hexane begins to break down the cellular wall, releasing lipids into the extraction chamber
4. The hexane/ lipid mixture then reaches a critical height level within the extraction chamber. This initializes the siphoning process.
5. Once siphoned back into the miscilla tank, the process starts over, turning the hexane into vapor under specified temperature and pressure while retaining the algae oil within the miscilla tank.
6. Steps 1-5 run for roughly 2.5 hrs, until the cellular wall has been completely broken down.
7. The hexane/lipid mixture is then heated once more, converting the liquid hexane into vapor.
8. The hexane vapor is run through a condenser and released into the hexane chamber.
9. Steps 7 and 8 run for a subsequent amount of time until all hexane is released from the chamber leaving only algae oil

#### **Results**

The experiment above was run using 10g of oven dried algae, which produced 1.4g of oil, a yielding of 14% of the overall algal mass.

### 3.3.2 Post experimental conclusions

Based on the results of the wet and dry extraction methods it is clear that hexane extraction is the most feasible extraction method given the time constraints and financial limitations. Repeatability is crucial to the success of the capstone project and hexane extraction was the sole experiment, which provided any liberation of oil from the *C. photothecoides* algae.

## 4.0: Dryer Project

### 4.1: Design Objectives

The belt dryer concept was established to bring algae from aqueous solution form to dry flakes in preparation for the extraction process. One of the underlying objectives for the dryer, besides dewatering the algae, was to have the process that could be easily modified in the future. The project is in the early stages of development and changes and/or optimization of the belt dryer in the future is necessary.

### 4.2 Design Constrictions

The final design of a small, prototype belt dryer was a significant change from the original charge the group was given. Due to funding constraints, the dryer was scaled down dramatically in effort to conserve funds and changed from a full-scale dryer to a tabletop proof of concept. The same general principles govern the belt dryer on both the large-scale and the small-scale design. The difference, however, lies in the ability to fully dry algae. The dimensions of the full scale dryer allows for a longer capillary belt to enable more water removal from the algae before final air drying. Not

only can the capillary belt be longer, but the drying section after the capillary belt can also be longer, allowing the algae more time to dry before it is removed from the belt.

The smaller, tabletop belt dryer isn't as effective at completely drying the algae due to dimensional limitations. A complete process re-design may have enabled the algae to exit the dryer in dry flake form, but time constraints forced a scale down of the original design.

## 4.3 Drying Process Concept Design

### 4.3.1 Performance Targets

Early on in the design process, goals were set in order to evaluate concepts generated by the drying group. The five main goals considered during design development were as follows:

- Dewatering of algae sufficient for extraction process
- Low energy requirements
- Reproducible results
- Affordability
- Ability to process large batches of algae solution

The five main goals allowed the drying group to quickly move from design to design in the feasibility assessment.

### 4.3.2: Alternative Designs

The three main alternatives for drying processes were centrifuge, evaporative dryers, and belt dryers. Each of these methods has multiple variations in their designs.

Continuous centrifuges can be efficient at dewatering but are very complicated,

expensive, and energy intensive. More simple centrifuges are very inefficient due to the large acceleration of the massive liquid. Due to these factors the centrifuge drying process was not pursued.

Evaporative driers are energy intensive due to the increase in water temperature and the phase shift required to drive off water. Low-pressure systems can be made, but those too require significant energy input.

Belt dryers that use capillary action to draw water out are an attractive option due to their low energy input. A belt dryer requires no large accelerations or heat transfer rates, and the design can be refined into a simple and manageable arrangement.

#### 4.3.3: Review of Alternatives

It was decided that a belt drying method using a fine pore algae membrane and absorbent capillary belt would be the best option to pursue. Various belt arrangements and confinements were researched and discussed during the concept generation process. Two arrangements for the belt drying process were a cylindrical confinement or a conveyer belt style arrangement supported by rollers. After encouragement from advisors to not “reinvent the wheel,” the group decided to pursue the simpler conveyer style. Additional options for the conveyor belt dryer process were discussed and could be easily integrated into the system post initial construction. Some of these options included vacuum boxes and blowers to increase the amount of drying. Due to the energy requirements and the project timeframe, these features were not pursued.

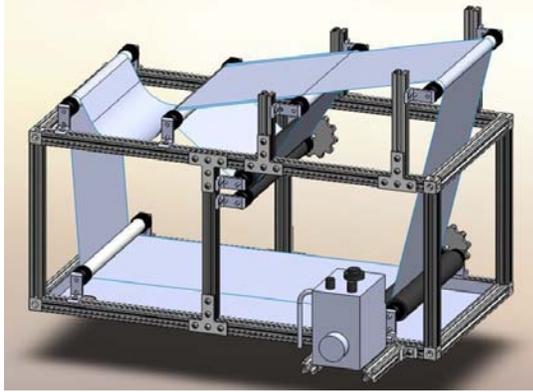
#### 4.3.4: Concept Testing

A key challenge in testing the belt dryer concept was to find an appropriate membrane to test. After testing countless membrane materials, a small disc of expanded PTFE used for chemistry filtration and a piece of absorbent cloth was used. The PTFE was wetted with the algae solution and moved across the cloth. The water was drawn through the membrane and retained in the cloth. From visual inspection, none of the algae had been able to pass through the membrane. Two underlying concepts were tested in this experiment: the passage of water through the membrane into the absorbent cloth via capillary action and proper pore sizing of the membrane, such that algae does not pass through. With this proof on concept, manufacturers of expandable hydrophilic PTFE were contacted to produce a continuous sheet of 0.45 micron nominal pore diameter material.

#### 4.4: Final Concept and Rationale

The team decided to pursue a conveyer style belt dryer, shown below in Figure 7, due to its lower energy requirements and manageable construction requirements. The proof of concept on a small scale provided assurance that a scaled system would offer reasonable repeatable performance. It was also apparent that the system could be built for relatively little money when compared to an algae centrifuge, and especially those required for highly toleranced pressing devices similar to ones used in seed oil extraction industry. Reproducibility was one

of the original goals of the project.



**Figure 7: Belt dryer final product**

#### 4.5: Final Dryer Design Description

The small-scale belt dryer described below is shown in Figure 8. The small-scale belt dryer skeleton is made from 1" 80/20 framing. The rollers are made from 1" PVC tubes and end caps, joined together by friction. No PVC cement was used. The four drive rollers were coated in Plasti-Grip, a liquid plastic made for coating tool handles in a shop. The capillary belt is made from Sham-Wow super-absorbent chamois cloth. Lastly, the belt itself is made from a 0.45-micron hydrophilic PTFE membrane that allows water to pass through, but not algae cells. Half-inch nylon straps were glued to the membrane with HH-66 vinyl cement to give it some rigidity.



**Figure 8: Belt Dryer Final Product**

A manual crank drives the membrane belt. On the opposite side of the drive roller there is a large 18 tooth sprocket. The capillary belt has a drive roller with a smaller 9 tooth sprocket on one end, which is connected the drive gear with a series 25 chain. The difference in sprocket sizes allows the capillary belt to move faster in relation to the algae membrane.

#### 4.6: Analysis of Dryer Belt Design

The belt dryer is still in its initial design process. This project is in its first year, so the capstone algae team has pioneered a new process. Part of the design of the belt dryer was to create a skeleton for a dryer that future design teams can modify as needed.. If algae oil extraction is a capstone project for future years, the dryer can be modified and/or optimized to do its job more effectively. Currently, there are four different sized sprockets that allow variation in the relative speed of the capillary belt to the membrane. The materials used in the frame construction allow extensive changes to be made to the overall drying process design in the future.

### 5.0 Hexane Extractor Project

#### 5.1 Concept Generation

For the extraction team, a set of six goals were created as a guideline for concept generation. The extraction unit needed the following abilities:

- The unit should successfully remove oil from *C. photothecoides* algae.
- The unit should be simple, requiring only one operator.

- The unit should be safe enough for the average homeowner to use without serious risk of injury.
- The unit should remove oil on a time scale compatible with the growing cycle of the algae.
- The unit should be energy efficient
- The unit should be able to process large amounts of algae solution or dry algae.

These criteria were the main design goals during the concept generation process.

## 5.2 Current Chemical Extraction Systems

The basis of the extraction system design is an existing laboratory extraction device, called a Soxhlet extractor, which was used to test the method of chemical extraction for removing the algal oil. The system, shown in Figure 9, recirculates hexane by constantly boiling and condensing it. Dried algae is placed in the *Algae Reservoir* and liquid hexane is then added. The hexane fully immerses the algae and dissolves a small amount of it. When the hexane fills the reservoir to a certain level, a siphon is created, and the hexane, along with whatever oil it has dissolved, drains into the bulb, labeled *Hexane/Oil Reservoir*. Here, a hot plate heats the hexane and oil mixture. The hexane is boiled to vapor and rises through the tubes indicated by the dashed path in the diagram; because oil has a higher boiling point, it does not vaporize. When the vapor hexane reaches the *Condenser Tube*, cooling water that encases the tube removes heat from the hexane, causing it to condense. The condensed hexane drains to the *Algae Reservoir*, where it immerses the algae and dissolves more oil. The

recirculation continues in this way until sufficient oil extraction has occurred.

At the end of this extraction process, a mixture of hexane and oil is left in the *Hexane/Oil Reservoir* and the leftover algae,

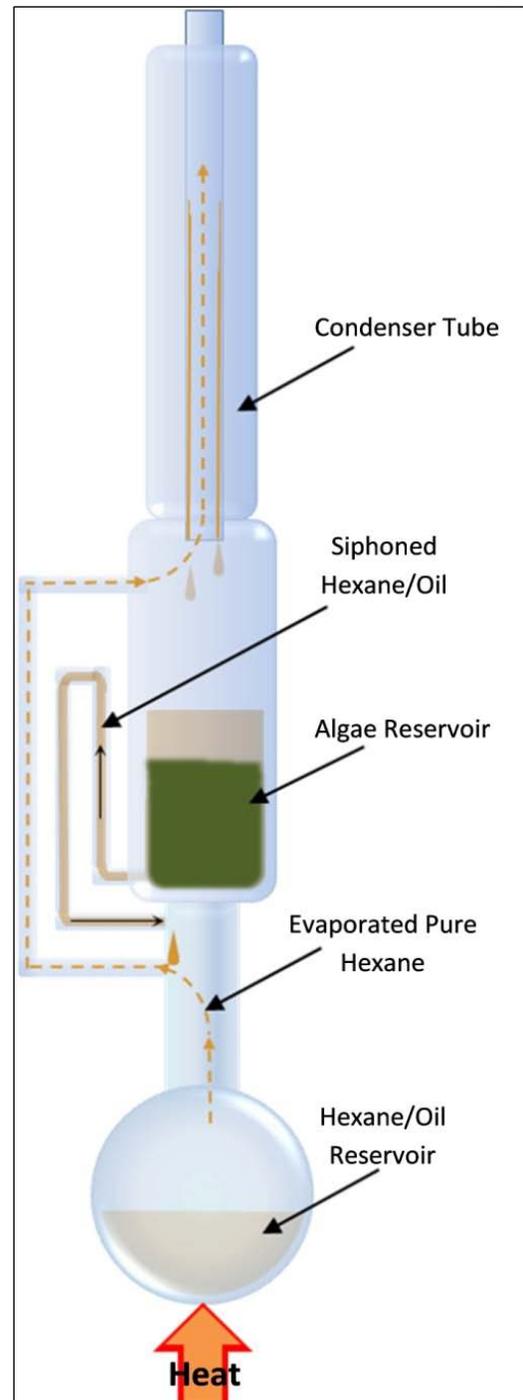
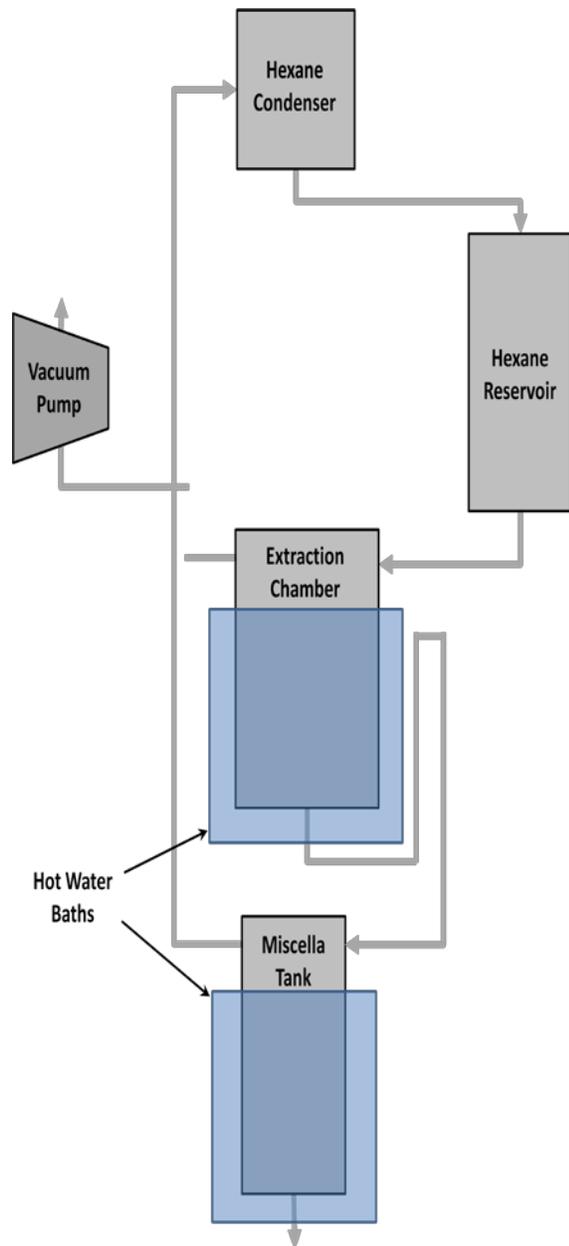


Figure 9: Soxhlet extractor used to extract oil from sample of algae

called *mill*, is left in the *Algae Reservoir*, soaked with residual hexane. This leaves two problems: separating the hexane from the oil, and recovering the hexane from the dried algae. In the laboratory setup, the hexane and oil mixture is exposed to a vacuum and then heated in order to remove



**Figure 10: Extraction system schematic**

the hexane. The vaporized hexane passes

through a condenser so that it can be recovered. However, there are high hexane losses in this process, and there is no laboratory process to recover the hexane contained in the mill.

## 5.3 Hexane Extractor Final Design Concept

### 5.3.1 Overview

Our team's extractor design uses the same principles for extraction as are used in the Soxhlet extractor, but incorporates processes to recover the hexane so that it can be reused in the system.

A schematic diagram of the hexane extraction process is shown in [Figure 10](#) below.

### 5.3.2 Oil Extraction

Like the Soxhlet process, the extraction system shown in the diagram processes a single batch of dried algae at a time. The dried algae is placed inside the *Extraction Chamber* and liquid hexane is placed in the *Hexane Reservoir*. Prior to circulating the hexane through the dried algae, the system must be evacuated. The hexane is sealed inside the *Hexane Reservoir* during evacuation (to prevent hexane loss by evaporation), and the *Vacuum Pump* is used to bring the pressure of the system down to approximately 2.5 psia.

After vacuum has been achieved, the entire system will be sealed to maintain it. At this point, the *Hexane Reservoir* is opened so that the hexane will drain into the *Extraction Chamber* and begin dissolving oil from the algae. Like the Soxhlet setup, the *Extraction Chamber* will drain when a siphon is

created. The mixture of hexane and oil, called *miscella*, drains to the *Miscella Tank*. A hot-water bath surrounds the *Miscella Tank*. Because of the low operating pressure of the system, the boiling point temperature of the hexane is lower (approximately 85°F), which means that domestic hot water (110-120°F) can be used to boil the hexane.

The hexane vapor rises through a tube to the hexane condenser, where the hexane is condensed into liquid. This hexane condensate drains back to the *Hexane Reservoir* and then back to the *Extraction Chamber*. The hexane is circulated in through the system until adequate oil extraction is achieved.

### 5.3.3 Hexane Recovery from Miscella

Extracting the oil from the algae is only half of the job of the extraction system. After extraction, the hexane must be recovered from the miscella and the algae mill. This is done by using the same heating and cooling system used during the extraction phase.

To recover the hexane from the miscella, the hot water bath is used again to boil the hexane from the *Miscella Tank*. However, unlike the extraction phase, the condensed hexane does not flow to the *Extraction Chamber*. Instead, a valve at the outlet of the *Hexane Reservoir* causes the hexane to collect here. At the end of this recovery process, oil is left in the *Miscella Tank*, and the recovered hexane is in the *Hexane Reservoir*.

### 5.3.4 Hexane Recovery from Algae Mill

Recovering the hexane from the algae mill is done in a similar way to the recovery from the miscella tank. A hot-water bath

surrounds the *Extraction Chamber*, and during the hexane recovery from the mill, hot water enters the bath and heats the chamber. The hexane contained in the mill is evaporated, and it flows to the condenser. The hexane condensate then drains to the *Hexane Reservoir*, where it is stored for use in the next extraction batch.

## 5.4 Progress toward Final Design Description

### 5.4.1 Deliverables: Thermal Condenser Construction

Due to the complexities of the hexane extraction system, the final design was not completed until midway the second semester of work because much of the first semester was required for researching potential extraction techniques. It was not until November that, having exhausted all potential mechanical methods for oil extraction, the chemical extraction method was chosen. Because research consumed so much time, designing the concept of the system could not proceed until late in the first semester. By the end of the first semester, the overall system operation was defined, and engineering calculations were underway. The first several weeks of the second semester were spent finishing calculations and selecting equipment for the extraction machine. Once a budget for the proposed design was assembled, it was proposed for approval. However, due to the high budget, the number of parts that would need fabrication, and the time required to test the final system, it was determined that it would be unfeasible to attempt to construct the entire extraction system by the end of the year.

Rather than attempting to build the entire system, it would be more feasible to select a single component to construct and test. One of the crucial parts of the system is the hexane condenser. Without proper operation of this part, the system would not run efficiently. Hence, it was decided to spend the remainder of the semester focusing on the operation of the thermoelectric condenser. Consequently, the milestones of the project were revised to reflect this goal.

#### **5.4.2 Deliverables: Design Packet**

In addition to constructing the condenser, another deliverable added to the milestones was an overview of the design of the extraction system. The goal would be to produce a document outlining the entire design, including concept design, technical calculations, and equipment selection. This document would serve to educate any future groups on the design considerations and provide a template for continuing work and constructing the entire system. The design document includes SolidWorks images of the design, as well as machine drafts for producing the parts and assemblies. Also included are pertinent design calculations and annotations, which help readers understand the decisions made throughout the design process.

#### **5.4.3 Deliverable: MTI Grant**

Another deliverable added to the revised milestones was a proposal for a research grant from Maine Technical Institute (MTI), to be used to construct and test the entire hexane extraction system. The grant proposal was written with the help of team advisor, Xinfeng Xie. Pending the approval of the grant, several team members will

work to construct the extraction system and test its operation.

## **5.5 Technical Design Considerations**

### **5.5.1 System Sizing**

Much of the information that the group had obtained on algae culturing had come from Steve Crawford, and the estimates for the amount of algae that could be grown were based on his algae bioreactors. It was determined that it would be feasible to grow about 2000 gallons of algae solution every three to four days. At a concentration of 0.1% algae concentration by volume, this corresponds to 2 gallons of dried algae that the extraction system must process per batch.

### **5.5.2 System Vacuum Operation**

The design of the hexane extraction system includes the operation of a vacuum pump. By incorporating the vacuum, the extraction system will become more efficient due to the lower levels of energy needed to heat the system. Given that the hexane within the extraction system must change to vapor, the hexane must reach a boiling temperature. By including the vacuum within the design, the total pressure within the system will drop to an approximate 3.5 psia, which in turn will drop the boiling temperature to 84.5 degrees Fahrenheit, allowing the phase change to take place.

### **5.5.3 Material Selections**

The proper material used for the design of the hexane extraction system was chosen under the criteria that the material could not be corrosive when in contact with hexane. Given that the properties pertaining to

stainless steel permitted contact with hexane without the possibility of corrosion, stainless steel became the primary material used for the design of the hexane extraction system.

#### 5.5.4 Thermoelectric Element

The thermoelectric element acts to create a temperature difference by means of converting an electric voltage to a change in temperature, known as the Peltier effect. The element has been constructed as a thin, ceramic square object with two opposite surfaces. Within the two surfaces, the temperature change is clear where one side becomes hot and the other cold.

#### 5.5.5 Thermoelectric Condenser Construction

A thermoelectric device operates by the thermoelectric effect, whereby a temperature difference is produced across a material when a voltage is applied to it. The thermoelectric condenser operates by placing the cold side of the thermoelectric device in contact with the hexane vapor. To accomplish this, an aluminum slab has been machined with hollow tubes running through it. Within the tubes, hexane vapor will be present, which will be condensed due to the temperature drop produced by the cold side of the thermoelectric device. To ensure that the hot side of the thermoelectric device does not heat the cold side, a cooling block has been machined out of aluminum and placed in contact with that of the hot side. Within the cooling block, hollow tubes run through it, with a constant flow of cold water passing through. With this presence of cold water, the aluminum block will experience a drop in temperature, in turn

dropping the temperature of the hot side of the thermoelectric device.

### 5.6 Testing & Evaluation of Thermoelectric Condenser

After completion of the thermoelectric condenser, the team ran preliminary tests to verify its performance and provide a proof of concept. In order to test the operation of the condenser, two thermistors were attached to the condenser, one on each of the hot and cold side blocks. A digital display showed the readout of each thermistor in degrees Celsius. The thermoelectric elements were turned on and water was piped through the water cooling block, although no hexane was put through the condenser. It was found that the steady state temperature of the water cooling (hot-side) block was approximately 12.5° C (54.5° F), and that the steady state temperature of the hexane condenser block was about 13.5° C (56.3° F). However, when the water flow was turned off, the water cooling block temperature approached 40° C (104° F) while the hexane cooling block temperature hovered around 25° C (77° F). This, of course, would be unacceptable for condensing the hexane in the extraction system, since the temperature of the hexane in the system would be at approximately the same temperature.

In the case of the water cooling block being utilized, though, an adequate temperature of the hexane condenser block was achieved. Because the hexane condenser block remained at a temperature of approximately 56° F and the condensation temperature for the hexane in the extraction system would be approximately 80° F, the hexane

condenser block would easily perform its function. The question that begs to be answered is that if the hexane condenser block and the water cooling block are so close in temperature with the use of water flow, why not simply eliminate the use of the thermoelectric elements? This is a reasonable question, and a test was performed to verify the performance of the condenser without the use of the thermoelectric elements. It was found that without the thermoelectric elements being run, the temperatures of both blocks were nearly the same as with the elements on. This suggests that using a single aluminum block with both water and hexane flow passages could be the most effective, and energy-efficient means of condensing the hexane.

All testing was performed at standard atmospheric pressure, as it was not possible to replicate the vacuum of the extraction system with the available materials and budget. It was originally planned to test the condenser using hexane, boiled in an Erlenmeyer flask on a hot plate. Plastic tubing was assembled to route the hexane vapor from the flask to the condenser, and a tube was attached to the condensate drain for collection of the hexane. This testing would not provide performance data that would be true to its operation in the actual system, since the hexane in the system would be at a far lower pressure and temperature (approximately 2.5—3 psia and 75—80°F). Because the tests would be performed at atmospheric pressure, the temperature of the boiled hexane would be approximately 156°F. This would be a far more demanding test of the condenser than

is required for system operation. However, these tests had to be abandoned, because the power supply for the thermoelectric elements broke. Other power supplies were searched for in Crosby Laboratory, but one could not be found that could supply the amperage required. Due to the timing of this unforeseen equipment failure, the testing with hexane was unable to be performed. However, this was a non-critical part of the testing. The more valuable data was the temperature data that was obtained for the condenser operation, and which is provided above.

### 5.7 Analysis of Design

Due to time and budget limits, it was unfeasible to attempt to completely construct the entire hexane extraction system. Since the hexane condenser was chosen as the focus of the latter half of the second semester, following the design of the entire extraction system, its strengths and downfalls should be examined. Because the thermoelectric condenser was the first prototype of the condenser, there were several design flaws which should be addressed in a future condenser. Firstly, the condenser succeeded in cooling the hexane condenser block to a temperature (approximately 56° F) that would be able to condense the hexane in the operation of the hexane extraction system.

The hexane condenser block was machined from a 1½ inch thick aluminum block, although the water cooling block was machined to a thickness of 1 inch. The hexane condenser block should be machined to a smaller thickness, in order to eliminate the thermal mass of the hexane block, and

decreasing the time that is required to cool the hexane condenser block.

Another issue with the current condenser is that the hexane passages are cut horizontally into the hexane condenser block, which causes the hexane to pool up in the passages. If the passages were cut at an angle, sloping inward toward the center of the block and the vertical condensate drainage passage, then the hexane would drain more effectively.

It seems that the thermoelectric elements could be removed from the condenser without a loss in performance. If this were to be done, the hexane and water passes would be combined in the same aluminum block. Although this might be possible, it may require a faster flow rate of water, and may effectively entail running water continuously for the duration of the system operation. However, the thermoelectric elements require a significant power input to run.

A balance must be struck between the resource of water used and the cost of power to operate the thermoelectric condenser. This should be a focus of continued work on the hexane extraction system. It is suggested that future groups build a single condenser block, which contains both hexane and water passages in a single block, and compare its performance to the existing condenser. It should be decided whether or not water cooling alone is adequate, or if the thermoelectric elements are required to provide suitable condensation rates.

## 6.0 Conclusions and Recommendations

The design process to extract oil from algae ultimately yielded a complete design of both a dryer and extractor along with a proof of concept scale dryer and thermoelectric condenser. This design and hardware development has advanced the capacity to further research the utilization of algae as a feedstock for biofuels. Continued scientific development would support the engineering of further advanced devices and processes for algal oil extraction.

The main concern with continuing with the existing extraction system and belt dryer designs is that ultimately, a mechanical method may prove more effective. It is advised that more research be done to explore new methods of oil extraction. This is a constantly growing field, and several companies utilize proprietary extraction methods. There exist mechanical means of oil extraction, and more time and research is needed to discover them. Ultimately, this project belongs more in a research stage, since much of the group's time was spent researching the technology rather than design.

Given the time restrictions and goals of the capstone project, it made sense for the group to go forward with a design using a chemical extraction method. However, future efforts should not be limited to this method. It would be more beneficial to allow more research to be done so that there is a well-grounded technology that can be used for a design. Otherwise, much time will be spent on research, when it should be spent on design.

### **6.1 Strengths of the Design Process**

A collaborative spirit and a fresh starting point strengthened the design process of the dryer. Starting off with no prior hardware allowed our team to assess alternatives freely without being constrained. This resulted in multiple possibilities and arrangements being discussed in a collaborative environment. This environment was another key strength as team members shared and refined ideas to generate final concepts for the design.

### **6.2 Weaknesses of the Design Process**

Not having well defined drying or extraction processes in addition to not having extensive prior work and experience in the field were two challenges to the design process. Since there was not an established facility producing or consuming the algae during the design process, it was difficult to create design specifications. Also, the project scope shifted halfway through the school year from an operational unit to a demonstration scale unit. The lack of a currently operational algae drier was another challenge. Not having prior work to rely on for design decisions made the design much more open ended and experimental.

## Bibliography

Algaeventure Systems, a Univenture, Inc. company. (2010). *Algaeventure Systems-Algae vs. Other Biomass*. Retrieved April 13, 2010, from Algaeventure Systems, a Univenture, Inc. company: <http://www.algaevs.com/the-wonders-of-algae/algae-vs-other-biomass>

Beyond Petroleum. (2010). *Biofuels Fact Sheet*. Retrieved April 13, 2010, from BP- Beyond Petroleum: [http://www.bp.com/liveassets/bp\\_internet/globalbp/STAGING/global\\_assets/e\\_s\\_assets/e\\_s\\_assets\\_2008/downloads/bp\\_biofuels\\_biofuels\\_factsheet\\_BP\\_Final.pdf](http://www.bp.com/liveassets/bp_internet/globalbp/STAGING/global_assets/e_s_assets/e_s_assets_2008/downloads/bp_biofuels_biofuels_factsheet_BP_Final.pdf)

Central Intelligence Agency. (2008). *CIA - The World Factbook--United States*. Retrieved 4 13, 2010, from Central Intelligence Agency: <https://www.cia.gov/library/publications/the-world-factbook/geos/us.html>

*Chlorella*. (2010, March 19). Retrieved March 24, 2010, from Wikipedia: <http://en.wikipedia.org/wiki/Chlorella>

Converti, A., Casazza, A. A., Erika Y. Ortiz, P. P., & Borghi, M. D. (2009). *Effect of temperature and nitrogen concentration on the growth and lipid content of Nannochloropsis oculata and Chlorella vulgaris for biodiesel production*. Genoa, Italy: Department of Chemical and Process Engineering University of Genoa.

*The Need For A New Oil*. (2010). Retrieved March 17, 2010, from Origin Oil: <http://www.originoil.com/market/the-need-for-a-new-oil.html>

*Why Biofuels? BP Biofuels*. (2009). Retrieved April 13, 2010, from bp alternative energy: <http://www.bp.com/sectiongenericarticle.do?categoryId=9030039&contentId=7055156>