

Old Dominion University

## ODU Digital Commons

---

Civil & Environmental Engineering Theses & Dissertations

Civil & Environmental Engineering

---

Fall 2019

# Challenges of Designing and Operating a Pilot Scale Short Residence Time Continuous Hydrothermal Flash Hydrolysis Reactor for High Slurry Load Biomass Processing

Mason James Martin

*Old Dominion University*, [mason121912@gmail.com](mailto:mason121912@gmail.com)

Follow this and additional works at: [https://digitalcommons.odu.edu/cee\\_etds](https://digitalcommons.odu.edu/cee_etds)



Part of the [Environmental Engineering Commons](#)

---

### Recommended Citation

Martin, Mason J.. "Challenges of Designing and Operating a Pilot Scale Short Residence Time Continuous Hydrothermal Flash Hydrolysis Reactor for High Slurry Load Biomass Processing" (2019). Master of Science (MS), Thesis, Civil & Environmental Engineering, Old Dominion University, DOI: 10.25777/zh93-wv22  
[https://digitalcommons.odu.edu/cee\\_etds/109](https://digitalcommons.odu.edu/cee_etds/109)

This Thesis is brought to you for free and open access by the Civil & Environmental Engineering at ODU Digital Commons. It has been accepted for inclusion in Civil & Environmental Engineering Theses & Dissertations by an authorized administrator of ODU Digital Commons. For more information, please contact [digitalcommons@odu.edu](mailto:digitalcommons@odu.edu).

**CHALLENGES OF DESIGNING AND OPERATING A PILOT SCALE SHORT  
RESIDENCE TIME CONTINUOUS HYDROTHERMAL FLASH  
HYDROLYSIS REACTOR FOR HIGH SLURRY LOAD BIOMASS  
PROCESSING**

by

Mason James Martin  
B.S December 2016, Montana State University Northern

A Thesis Submitted to the Faculty of  
Old Dominion University in Partial Fulfillment of the  
Requirements for the Degree of

MASTER OF SCIENCE

ENVIRONMENTAL ENGINEERING

OLD DOMINION UNIVERSITY  
December 2019

Approved by:

Dr. Sandeep Kumar

Dr. Mujde Erten-Unal

Dr. James W. Lee

## ABSTRACT

### CHALLENGES OF DESIGNING AND OPERATING A PILOT SCALE SHORT RESIDENCE TIME CONTINUOUS HYDROTHERMAL FLASH HYDROLYSIS REACTOR FOR HIGH SLURRY LOAD BIOMASS PROCESSING

Mason James Martin  
Old Dominion University, 2019  
Director:?

Increasing demand for renewable energy, fuels, and bioproducts has resulted in a push for increasingly efficient and economically favorable biomass pretreatment methods. Flash Hydrolysis, (FH) a promising biomass pretreatment method, has been extensively studied at a laboratory scale. FH employs a continuous subcritical hydrothermal process capable of fractionating microalgae for lipid and protein recovery. FH is unique in that the residence time is very short (~ 10 s). Maintaining this residence time was key in designing a scaled pilot flash hydrolyzing unit (FHU). We have developed a one of its kind pilot FHU capable of increasing the processing output of the laboratory scale 160 times, while maintaining the residence time and increasing heating rate in a continuous flow reactor.

This study tests a range of microalgae slurry concentration (1-10 wt%) in the pilot continuous FHU for fractionating algae components. The FH products mainly contain biofuels intermediate in solids (most of lipids) and hydrolyzate (soluble proteins and carbohydrates). Different concentration of microalgae can affect the heating rate and the products yield. It can also limit the pressure controlling ability of the backpressure regulators used in the process. Determining the algal slurry weight percentage in which the FHU can operate effective and efficiently is a vital step in the process of scaling FH. Two different microalgae species *Scenedesmosus* and *Chlorella* were used in the range of 150-220°C under subcritical water

conditions. Besides the microalgae concentration effect, the study shares the experiences of designing, and operating one of its kind FHU for processing microalgae slurry.

## **NOMENCLATURE**

ESIA	Environmental and Social Impact Assessment
FH	Flash Hydrolysis
MFHU	Mobile Flash Hydrolysis Unit
s	seconds
IH	Induction Heat
BPR	Back Pressure Regulator
FS	Factor of Safety
I.D.	Inside Diameter
Wt%	percent weight
mL	milileter
TOC	Total Organic Carbon
TN	Total nitrogen
ppm	parts per million
GLM	General Linear model
psi	pounds per square inch
DI	Deionized
CI	Confidence Interval

## TABLE OF CONTENTS

TABLE OF CONTENTS.....	5
LIST OF TABLES .....	8
LIST OF FIGURES .....	9
CHAPTER	
CHAPTER 1 .....	1
INITIAL SCALE-UP OF FLASH HYDROLYSIS PROCESS TECHNOLOGY .....	1
1.1.0 Introduction.....	1
1.1.1 Background.....	1
1.2.0 Methods.....	2
1.2.1 Scale up requirements .....	2
1.2.2 Algal Slurry Delivery Under High Pressures.....	3
1.2.3 Rapid Heating .....	5
1.2.4 Mobility Design .....	9
1.2.5 System Design .....	10
1.2.6 Pumping System .....	11
1.2.7 Process System Design .....	13
1.3.0 Results.....	15
1.3.1 Fabrication .....	15
1.3.2 Operation and Testing.....	17

1.4.0 Conclusion .....	19
CHAPTER 2 .....	20
MOBILE FLASH HYDROLYSIS UNIT OPTIMIZATION .....	20
2.1.1 Background .....	20
2.1.2 Literature Review .....	21
2.2.0 Methods .....	22
2.2.1 Objective .....	22
2.3.2 Test of Hypothesis .....	22
2.3.3 Source of Data .....	24
2.2.4 Validation of Data .....	24
2.2.5 Assumptions and Limitations .....	25
2.2.6 Statistical Analysis .....	26
2.2.7 Procedure and Analysis .....	26
2.3.0 Results .....	30
2.3.1 Pressure Effect on Hydrolyzate Quality .....	30
2.3.2 Discussion .....	32
2.3.3 Improved Pressure Regulation Testing .....	34
2.4 Conclusion .....	36
REFERENCES .....	37
APPENDIX I .....	38

Appendix II: MFHU shutdown checklist.....	46
Appendix III: Pressure Study Sample Data .....	46
Appendix IV: SAS outputs .....	47



## LIST OF TABLES

Table	Page
Table 1: Pumping Options .....	4
Table 2: Heating Options .....	6
Table 2: Heating Options .....	9
Table 3: TOC normality tests.....	24
Table 4: TN normality tests .....	25
Table 5: Total Nitrogen GLM SAS results .....	31
Table 6: Total Carbon GLM SAS results .....	32
Table 7:TN Duncan Grouping .....	34
Table 8: TOC Duncan Grouping.....	34

## LIST OF FIGURES

Figure	Page
Figure 1: MFHU mock design .....	10
Figure 2: Pilot Layout .....	11
Figure 3: Pump System Layout.....	12
Figure 4: HiP Catalogue Reference .....	14
Figure 5: Chilling Zone Design .....	15
Figure 6: Fabrication Progression .....	17
Figure 7: Collected Hydrolyzate.....	27
Figure 8: Centrifuged Hydolyzate .....	28

## CHAPTER 1

### INITIAL SCALE-UP OF FLASH HYDROLYSIS PROCESS TECHNOLOGY

#### 1.1.0 Introduction

##### 1.1.1 Background

There is a growing need for alternative fuel sources. In 2007 the US the Energy Independence and Security Act (EISA) required that by 2022 a substantial portion of the US liquid fuels be from renewable sources[1]. Numerous biofuel production pathways have been studied. A major problem with biofuels is their cost compared to traditional petroleum fuels. It has been modeled that using a production pathway capable of producing high-value coproducts can help offset the cost of biofuel production and make the pricing comparable to petroleum fuels[2].

Flash Hydrolysis (FH) is a biomass conversion technology capable of fractionating microalgae to be further processed into high-value products and biofuels[3]. FH is a continuous hydrothermal process operating under subcritical conditions. Subcritical water conditions allow for water to be heated above its boiling point (100°C) through the application of pressure on the system of water[4]. Utilizing subcritical water conditions allows for the primary biomass, microalgae, to be processed with little or no change to how algae is naturally harvested. Microalgae is typically harvested in a water algae mixed slurry[5]. By processing the microalgae as a slurry, the overall biomass conversion process will increase in efficiency. While utilizing subcritical water conditions is beneficial for processing the algae slurry, it makes scaling more challenging, especially when scaling the FH technology from laboratory to pilot plant scale. This study will examine the challenges faced when scaling a new technology from lab to pilot scale.

FH fractionates the microalgae into two fractions, an aqueous and solid fraction. The aqueous fraction of the hydrolyzate contains peptides, amino acids, micronutrients, and carbohydrates. The solids portion is lipid rich, and these lipids can be converted into a biofuel.

## **1.2.0 Methods**

### **1.2.1 Scale up requirements**

When transitioning from laboratory to pilot scale FH algae processing, key aspects were considered when evaluating unit operations. In order for a reaction to occur and the algae to fractionate inside the FH reactor, certain conditions must be met regardless of scale. Reaction temperatures must be capable of reaching 280°C rapidly while maintaining subcritical water conditions. The reaction conditions were divided into different areas of concentration when scaling the FH process. The key aspects of scaling considered are as follows:

**Pump Selection:** capable of delivering the algae slurry at appropriate flow rates, as well as create appropriate pressures for subcritical water conditions (~2000psi).

**Heating Unit:** Heat generation must be capable of heating the slurry inside the reactor to the reaction temperature of 280°C with a heating rate of greater than 30°C/s.

**Quench:** the slurry must be cooled below the 100°C boiling point of the slurry before it reaches atmospheric conditions to avoid gas formation and degradation of products. The final all-encompassing aspect of the design was that the FH pilot unit must be mobile. This will allow for testing and demonstration at various facilities and laboratories.

### 1.2.2 Algal Slurry Delivery Under High Pressures

The first challenge to overcome when designing the mobile Flash Hydrolysis Unit (MFHU) is the capability to pump a viscous algae slurry with a relatively high solid weight percentage (wt%). This must be done while simultaneously being capable of reaching pressures of up to 2000 psi. With these stringent qualifications, every pumping option was thoroughly evaluated. Table 1 shows the options considered for pumping options. The advantages and disadvantages are listed based on the desired end use of the pumps.

The primary types of pumps considered were “positive displacement” pumps, due to the characteristics of the fluid being pumped. Centrifugal pumps were ruled out due to the nature of pump’s construction. The impeller used in a centrifugal pump limits the viscosity of the fluid being pumped. Impellers would increase the likelihood of clogging the pump with a high wt% algae slurry, limiting the effectiveness of operation. Positive displacement pumps operate by moving a volume of fluid through mechanical means. This allows for higher viscosity fluids to be moved at high speeds and under high pressure. The limitation of a positive displacement pump is that due to it moving a certain volume of fluid at a time to produce a flow, the flow is in turn not as consistent. This inconsistency is referred to as a pulsed flow.

It was ultimately determined that a diaphragm metered pump was ideal for the flow and pressure requirements of the proposed MFHU. A LEWA EcoFlow Diaphragm metering pump was chosen because of its technical qualifications as well as the LEWA company’s eagerness and guarantee to deliver a quality product. Major specifications of the LEWA pump can be seen in Table 1. The major benefits of the LEWA pump were its ability to produce precise flowrates of up to 3.5 gph due to its submillimeter piston stroke length capabilities. The pump was also capable of reaching pressures of up to 3000 psi. The main drawback of using a diaphragm metered pump,

pulsating flow, can be attenuated by the use of a pulsation dampener. A pulsation dampener is a nitrogen charged unit where fluid flows into a membrane. This membrane is calibrated to a pressure 200psi below the system operating pressure. As the fluid flows into the membrane, the membrane absorbs the pulsed flow caused by the pump, greatly reducing the pulsation of the flow. This results in a smooth continuous flow for the rest of the system, which leads to a continuous and precise flow leading into the FH reactor.

Table 1: Pumping Options

OPTION	ADVANTAGES	DISADVANTAGES	REMARKS
<b>SYRINGE PUMP</b>	Suitable for slurries with very high viscosity Already been used (published studies)	Very expensive Limitation on scale up Manual piston feeding	Rejected due to budget limitation and no further scaling up potential
<b>HYDRAULIC CYLINDER</b>	Not expensive Can operate at very high pressure Not affected by viscosity of slurry Produces pulse free flow No valves to clog.	A batch process (each batch equals volume of cylinder) Potentially difficult to precisely control flow	Rejected because it is a batch process and the potential difficulty in controlling flow
<b>GEAR PUMP</b>	Relatively inexpensive Can operate at very high pressure Produces pulse free flow No valves to clog	Requires minimum viscosity due to slip High horsepower motor; thus-3-phase Has minimum flow that exceeds pilot criteria	Rejected due to minimum viscosity requirement and the inability to meet the minimum flow criteria
<b>ROTARY VANE PUMP</b>	Produces pulse free flow No valves to clog	Cannot meet pressure requirement	Rejected because it cannot meet the minimum pressure requirement
<b>PROGRESSING CAVITY PUMP</b>	Produces pulse free flow No valves to clog	Cannot meet pressure requirement	Rejected because it cannot meet the minimum pressure requirement

<b>HYDRA-CELL METERING PUMP</b>	Can produce high pressure. Multiple-diaphragms produces virtually pulse free flow Not as expensive as other metering pumps	Vendor was slow to respond with answers to ODU's questions	Rejected because supplier was slow in responding and there was not enough time to evaluate
<b>CONVENTIONAL DIAPHRAGM METERING PUMP</b>	Can produce high pressure Can meet the flow criterion that ODU needs Flow rate can be precisely controlled	Expensive Produces a pulsed flow; however, this can be attenuated with a pulsation dampener	Selected due to vendor guarantee that if the pump did not work, ODU could return it for a full refund.

### 1.2.3 Rapid Heating

To reach the required reaction temperatures of 280°C with very high heating rate, various heating methods and technologies were evaluated. The first step in evaluation was to determine if the preheated water mixing utilized in the laboratory scale FH unit could be reasonably implemented at pilot scale. The design criteria used to evaluate all heating potentials were as follows:

#### Design Criteria

- Maximum Flow: 195 mL/min (3.1 gph)
- Reactor residence time: 10 s
- Heating rate: 30°C/s
- Ambient temperature of algal slurry:  $\pm 20^\circ\text{C}$
- Temperature of slurry inside reactor: 280°C
- Time to achieve  $\Delta T(260^\circ\text{C})$ : 10 s

- Reactor material used: 316 Stainless Steel

It was determined the mixing chamber was not feasible at scale, so other possibilities were evaluated. Upon investigation, it was found that the high reaction temperature requirement severely limited the heating options available for use. Table 2 summarizes the options investigated. The first option involves the use of a fluidized bed reactor. A fluidized bed reactor is able to produce high heat capability through the interaction of gas-solid reaction rates[6]. The main principals of a fluidized bed consist of a chamber containing a solid, typically sand. This chamber is filled with air. Air is pressurized and moves through the chamber until the weight of each sand particle is supported by the flow of the air. When the sand is supported by the flowing air the air/sand mixture acts as a fluid. When external heat is applied to the air/sand fluid, the sand absorbs the heat and stores it. Due to the small nature of sand particles, this creates a very uniform heat transfer for the fluid. The heat from the air/sand fluid is then able to transfer to the reactor, capable of reaching the 280°C reaction temperature. While the reaction temperature could be reached, a fluidized bed is not capable of heating the slurry at the rate required (30°C/s). Design criteria would need to be altered for this option to be viable.

Table 2: Heating Options



OPTION	ADVANTAGES	DISADVANTAGES
<b>FLUIDIZED BED</b>	Helical coil in the bed Maximum temperature 600°C	Coil length would exceed residence time Cannot meet 30°C/s rapid heating
<b>TUBE/CERAMIC FURNACE</b>	Maximum temperature 1100°C	Cannot meet 30°C/s rapid heating through ambient heat Would require preheated water line
<b>INDUCTION HEAT</b>	Capable of reaching 280°C in 1-5s 1s start up time Consistent heat	3 phase power required External chilling unit required Expensive

The next option evaluated will be classified as “conventional” heating units. These units are ceramic heaters, tube furnaces and industrial ovens that utilize convection heat. The heat transfer mechanism used by conventional heating units is radiant heat, where the unit generates heat and the air is used as the medium to transfer heat to the reactor and in turn the slurry within. When discussing possibilities MHI, Inc. a reputable vendor proposed a radiant heating chamber capable of reaching 1700°C. This option could achieve the reaction temperature but would miss the heating rate by a large margin. The electricity required to power a unit of that capability would also likely be very inefficient energy use.

The final heating option considered was induction heating (IH). Induction heating utilizes basic principles of electricity. If an alternating current is applied to a transformer, and alternating magnetic field is created[7]. This magnetic field created can then be used to create heat through the circulating magnetic fields. In short, an alternating field is applied to a transformer, creating the magnetic field. An object with natural electric resistivity can be placed within the magnetic field. This is where heat will be generated. Heat is generated as the currents of the magnetic field flow against the electric resistivity of the material inside the field. Due to the “joule effect” which states that when an electric current flows through an object with electrical resistivity the electric loss within the material will then be converted to heat. The collision of electrons within the material collides with that of the electric/magnetic current creating the energy needed for heat generation[8]. The secondary method in which heat is produced in the induction heating process is due to the internal friction caused by the changing magnetic fields passing through the inducted material. This process is referred to as hysteresis[9]. The resistance from the indicated material’s natural properties to the changing magnetic fields surrounding it cause friction, which in turn produces heat. The inductive material used by the vendor GH Induction Atmospheres, is 316 stainless steel. This is the material to be used to construct the reactor and FH system, discussed further in section 1.2.7. IH is capable of meeting all design criteria, most importantly the 30°C/s heating rate. The main drawback of implementing induction heating in the FH unit, is the power requirement of the induction heating system. The induction heating system capable of meeting the design criteria would require a 5kW power supply. Not only is this energy intensive, but would require 3-phase power on site, limiting the potential mobility of the FH unit. The power supply for IH also requires a chiller unit to prevent overheating. This is an added capital, as well as operating cost.

Table 3: Heating Options

After careful evaluation it was determined that IH was the most appropriate option for meeting the design criteria. The rapid heating capabilities of the IH was unparalleled. The chiller unit required by the heating supply can also be slightly oversized and the excess cooling capacity will be used in the quenching phase of the process further discussed in section 1.2.7. The IH utilizing 316 stainless steel for the reactor material allows for more flexibility in system design as well. The only material flowing through the reactor in the IH case is the feed algal slurry. This allows for other fluids to be utilized in the future without needing to redesign the reactor or heat source. It also allows for easier cleaning and maintenance on the system as a whole.

#### **1.2.4 Mobility Design**

A key aspect of the FH unit was its ability to be mobile and taken to different locations to be operated as a demonstration its capabilities. The mobile design must be capable of facilitating all necessary process operations. All aspects of the FH unit must be contained within the framework of the mobile design. This means all large components must be on the cart and able to perform their functions. The proposed cart was designed to fit through standard interior doorways of 32 inches. The mobile cart was designed to accommodate the weight of all the components of the mobile flash hydrolysis unit. The double deck design can support all the individual components of the system. It was also designed to allow for flexibility in the component layout, so that design

iterations could easily be made during testing of the MFHU. Figure 1 shows the mock design used which includes the pump, IH, and chilling unit.

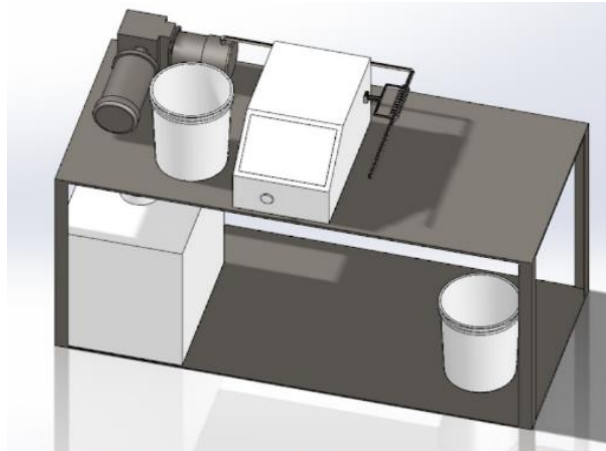


Figure 1: MFHU mock design

### 1.2.5 System Design

The system design encompasses all equipment used to meet the process conditions. This includes all the equipment from the pump delivery system to the pressure regulation. A process diagram is shown in Figure 2 to describe the overall process flow. For clarity, the system design will be broken up into two sections, Pumping System and Process System.

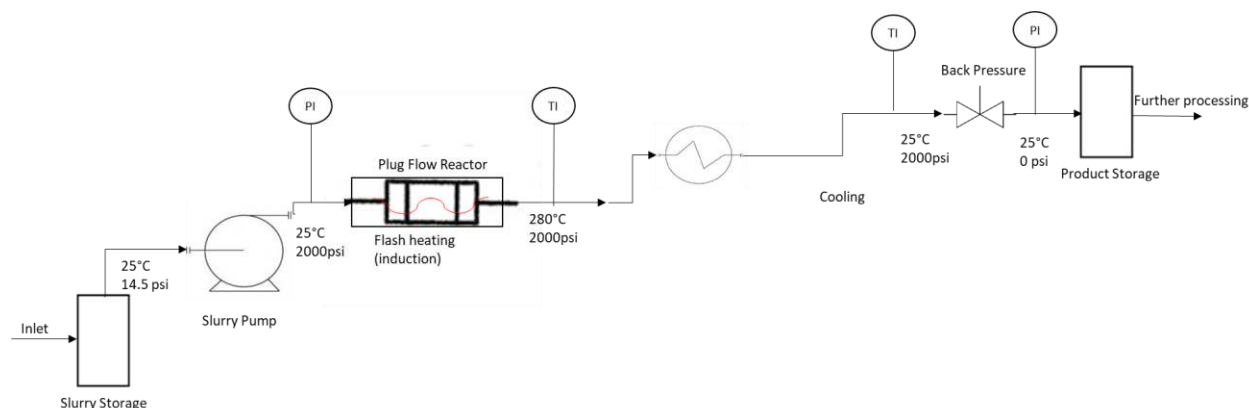


Figure 2: Pilot Layout

### 1.2.6 Pumping System

The pumping system had to be designed based on the specifications of the LEWA pump used. The pumping system designed was based upon the supplier's recommendation seen in Figure 3. Slight modifications were made to better facilitate the needs and limitations of the MFHU. Key components of the pumping system included the Calibration Column, Pulsation Dampener, and Back Pressure Regulator (BPR). The Calibration Column is located upstream of the pump inlet and is used to adjust the flow rate of the pump to meet the residence time of the Process System. The calibration column is filled with a known volume of water and while the pump is pulling from the calibration column time is measured, allowing for the calculation of the flow rate. The pulsation dampener is located downstream of the pump outlet. The pulsation dampener is designed to greatly reduce the pulsed flow caused by the metered diaphragm pump. The dampener is "charged" with nitrogen gas pressurized to a pressure that is 80% of the process pressure. The nitrogen gas' pressure is applied to a specially designed diaphragm that reduces the pulse of the fluid's flow as it passes through the dampener. The back pressure regulator (BPR) is located at the end of the pumping system, as it transitions into the process system. The BPR controls the pressure of the pumping system. The BPR controls pressure through a spring that applies force to a membrane or

seal. Fluid flows into the BPR through a small orifice where it comes in contact with the seal. The flow of the fluid builds pressure as the spring and seal resist the force generated by the flow until the force of the flow exceeds the force of the spring, allowing flow to pass through the BPR. The pressure created can be adjusted precisely by tightening or loosening the tension placed on the spring. Due to the high pressure conditions, all tubing, fittings, and valves used must be capable of exceeding process pressure requirements for a factor of safety (FS). Figure 3 shows the pump system layout.

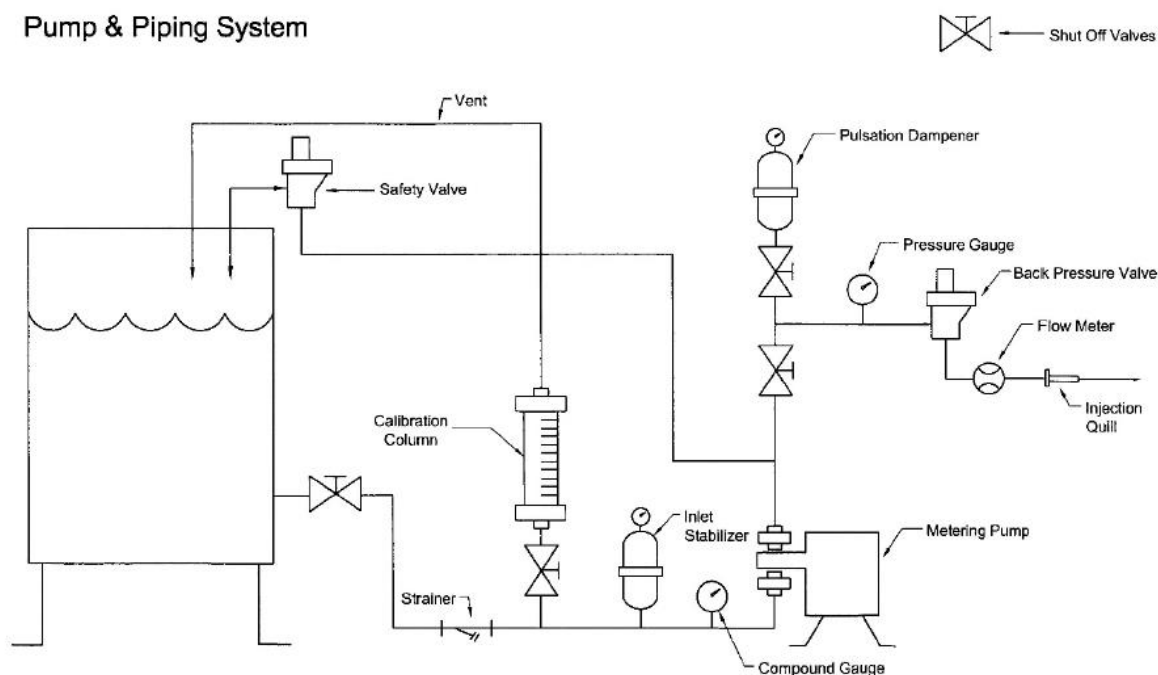


Figure 3: Pump System Layout

### 1.2.7 Process System Design

The process system is located downstream of the pumping system and consists of all the tubing, reactor, reaction heating, quenching zone and overall system pressure regulation. For ease of construction and time, the reactor and tubing were made of the same high pressure pipe. The diameter of the piping selected was determined by the reaction residence time using *equation 1[10]*:

$$t = \frac{V}{\frac{\rho_{\text{pump}}}{F(P_1, T_1)}} \quad (1)$$

where  $t$  is reactor residence time (s);  $V$  is reactor volume (ml);  $F$  pump volumetric flow;  $\rho_{\text{pump}}$  is the density of water at pump conditions (g/mL); and  $\rho(P_1, T_1)$  is the density of water at reaction conditions (g/mL). Substituting the volume of a cylinder we can solve for the required diameter with the known length of the reactor coming from the coil of the IH of 12in. Based on the calculations and the available diameters from the chosen tubing supplier (HiP High Pressure Equipment) seen in Figure 4 an internal diameter of 0.312 in was selected to meet the required residence time.

<b>9/16"</b>	9/16" O.D. x 0.359" I.D.	10,000	9/16" MEDIUM PRESSURE (LF9)	316 SS
	9/16" O.D. x 0.312" I.D.	20,000		304 SS
				316 SS
<b>3/4"</b>	3/4" O.D. x 0.516" I.D.	10,000	3/4" MEDIUM PRESSURE (LF12)	316 SS
	3/4" O.D. x 0.438" I.D.	20,000		
<b>1"</b>	1" O.D. x 0.688" I.D.	10,000	1" MEDIUM PRESSURE (LF16)	316 SS
	1" O.D. x 0.562" I.D.	20,000		

Figure 4: HiP Catalogue Reference

The maximum pressure the pipe is rated for is 20,000psi well within the FS for our 2,000psi reaction pressure required. Ten linear feet (LF) of pipe were ordered to serve as the piping for the Process System with appropriate fittings and adaptors to allow for a variety of layouts. An Omega TJ36 thermocouple located at the end of the reactor was used to measure reaction temperature.

The quenching zone was designed to utilize a chilled liquid to lower the algal slurry from reaction temperature to below 100°C so the output hydrolyzate would remain in liquid phase upon reaching atmospheric conditions. Figure 5 shows the design of the chilling zone. The loop shown is designed to be able to be easily modified as the MFHU is tested and improved upon. The size of the loop can be changed to increase surface area contact with the cooling fluid and or the volume of cooling fluid can be increased.

Just like the pumping system the process system regulates pressure through the use of a spring loaded BPR. The pressure of the system builds until the set pressure is reached and then the fluid is allowed to pass through. Unlike the pumping system the fluid or hydrolyzate passing through is open to atmospheric conditions and then collected in an appropriate container.



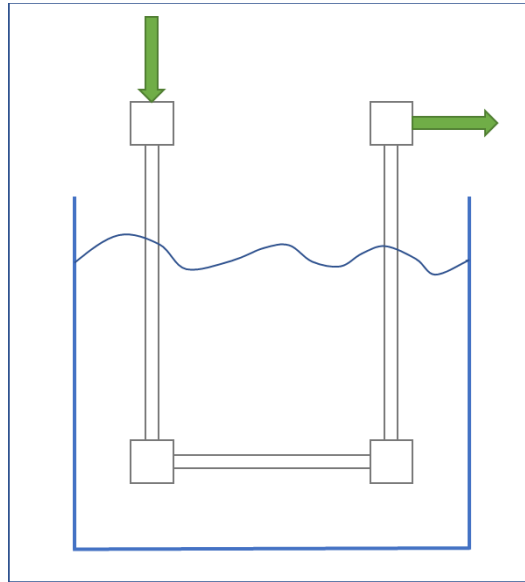


Figure 5: Chilling Zone Design

### 1.3.0 Results

#### 1.3.1 Fabrication

The cart of the unit was built using 3/4" steel angle iron and 3/4" marine plywood. The angle iron was fastened together using bolts instead of welding to allow for disassembly of the cart if needed in the future. Figure 6 shows some progress pictures of the fabrication of the cart.

The tubing for the pump accessories was built using prefabricated high pressure tubing and fastenings to connect the various pump accessories. This involved using a pipe cutter to cut the tubing to the required lengths and then attaching and tightening the fittings to ensure a proper seal. After the accessories were assembled and attached to the pump, the pump was attached to the upper deck of the cart. With the first portion of assembly completed fabrication of the larger diameter reactor tubing began. This tubing needed to be cut to length, then threaded and coned in

order to fit into the appropriate fitting connections. The threading and coning were done with the use of a lathe and a dye with the assistance of ODU's machine shop.

The overall layout of the pilot unit has been an iterative process. With changes and improvements made during and after fabrication. For example, the induction heating coil positioning was changed from horizontal (picture 5) to vertical (picture 6) to better handle the thermal expansion of the tubing due to the rapid heating of the induction heating. While testing continues more changes to the layout will be made until a suitable final layout is chosen. Additional fabrication may be needed to achieve the final layout.



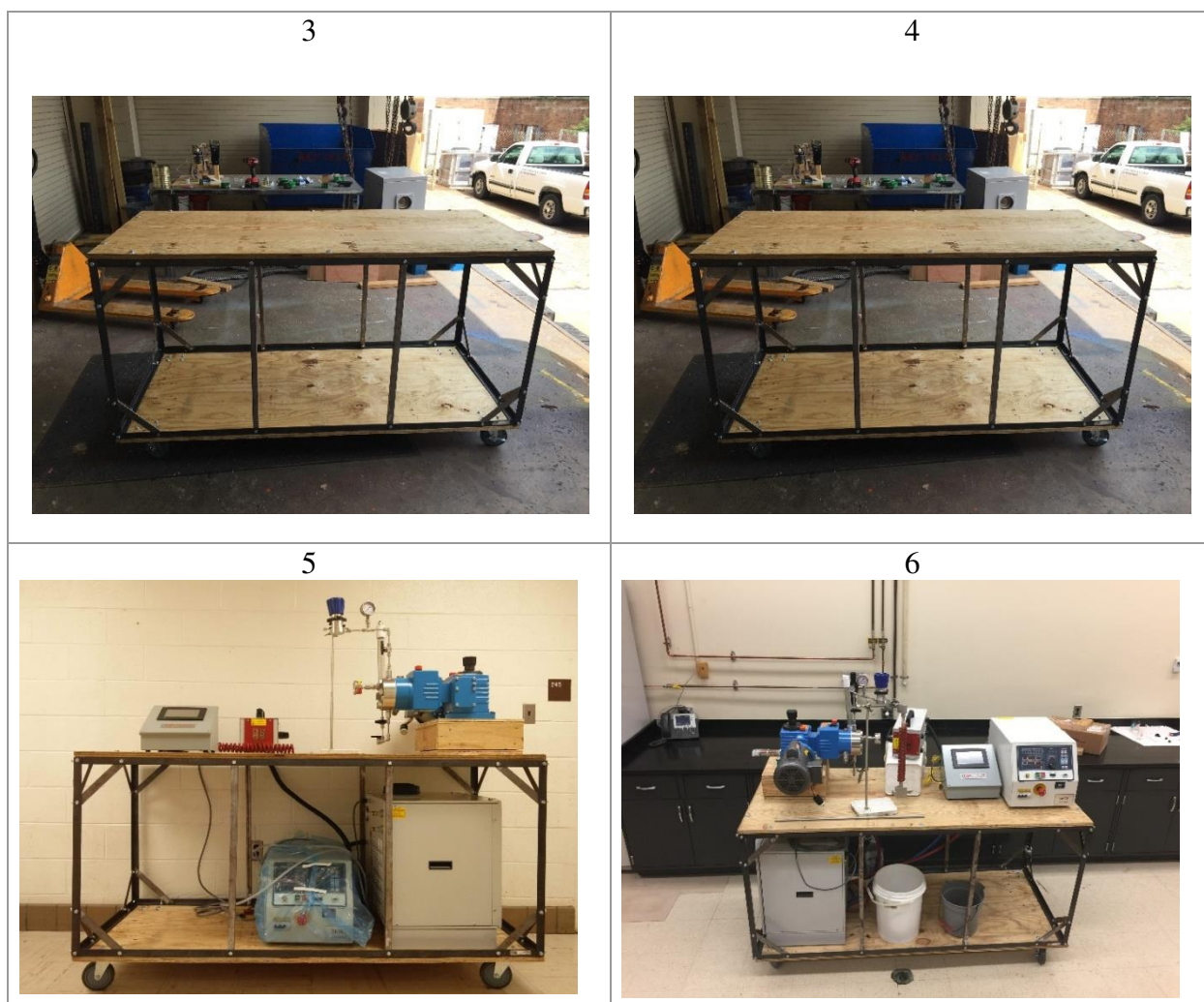


Figure 6: Fabrication Progression

### 1.3.2 Operation and Testing

Initial testing of the MFHU was performed using water to evaluate the pressure and heat regulation of the system. With a flow rate of 160 mL/min the pump and BPR was able to easily reach operating pressures of 2000psi. After it was shown that the system could maintain flow and pressure, the heat was introduced via induction heating. For the first attempt, the heating rate was

slow. It was programmed to reach operating temperatures by steadily increasing heat for two minutes until it reached 280°C and then temperature was maintained.

After operating conditions were met, algal slurry of 1wt% was used as the fluid. The resulting hydrolyzate from the effluent of the system showed that the flash hydrolysis reaction had occurred, fractionating the algae. Flow was maintained for 10 minutes to ensure that the process could remain continuous. The next test for the system was to increase the solid weight percentage of the algal slurry to 3%. The testing process was identical to the previous test, only using a 3 wt% slurry instead of 1 wt%. The initial flow of the slurry was consistent, but slight clogging began to occur within 3 minutes of operation. These slight clogs limited flow through the BPR, causing an increase in pressure. As the pressure of the system built up (2500-2800psi) the solid material causing the clog in the BPR was forced through and continuous flow resumed.

The objective of the MFHU was to be capable of processing an algal slurry of at least 6 wt% with a desired outcome of 8 wt% or greater. To further tests the limits of the initial design of the MFHU a slurry with 4 wt% was tested. This was done to determine if the clogging observed at 3 wt% would be amplified with increasing slurry solid weight percentages. The operating procedure was the same as the previous two tests. Similar to the previous 3 wt% slurry the 4 wt% slurry initially had a stable and continuous flow. However, clogging occurred within the first two minutes of operation. During this test the pressure began to rise and as it approached 3000 psi the system was shut off as a safety concern. It was observed in this test that despite the increased pressure the solids causing the clog could not be forced through the BPR. The system was allowed to cool to safe temperatures and the clog further investigated. Upon further investigation it was determined that no clogging occurred in the plumbing of the system. After this the BPR was taken

apart to verify that the clog indeed occurred in this piece of equipment. This showed that the solid portion of the hydrolyzate built up in the inlet orifice of the BPR, causing the clog in the system.

#### **1.4.0 Conclusion**

The objective of this study was to design and build a pilot scale MFHU capable of maintaining the continuous flow and short residence time of the laboratory experimental set up, while increasing the solid weight percentage of the algal slurry used to at least 6 wt%. The key aspects of scaling up, heat supply, high pressure slurry delivery, and mobile operation were clearly met. The heat is delivered via induction heating, capable of heating the slurry to 280°C in 5 seconds. This is a vast improvement over the laboratory set up and can be easily scaled further. The one downside to IH is the need for a 3-phase power supply, which can limit where the MFHU can be operated. The pump selected is capable of reaching operating pressure and can easily pump the viscous algal slurries used. Using two decks to support the different pieces of equipment allowed to keep the footprint of the MFHU small and allow for easy maneuverability. Overall the MFHU is more than capable of meeting the design criteria and meeting the reaction conditions for FH.

While the MFHU meets the reaction conditions at pilot scale, it still falls short of being able to process high slurry loading. Clogging of the BPR is the only factor limiting the slurry loading, as no clogs were observed in any other part of the system. Further options for pressure regulation should be examined to alleviate clogging and allow for higher slurry loading to be processed. As with all new designs, iterations and improvements after testing are common and expected. Further improvements and optimization regarding pressure regulation and slurry clogging will be investigated further in Ch 2 of this study.

## **CHAPTER 2**

### **MOBILE FLASH HYDROLYSIS UNIT OPTIMIZATION**

#### **2.1.1 Background**

During the design and implementation of a new process, improvements and iterations must be made at any scale. Despite careful planning and design, not all circumstances can be accounted for. Therefore, an important step in implementing a new technology such as the Mobile Flash Hydrolysis Unit this study focuses on, is continued improvement and optimization after testing. This study will examine the steps taken to improve some of the issues found during testing of the initial design of the MFHU in order to meet the technology's end goal. The major goal of optimizing the MFHU is to reach the primary target of processing an algal slurry containing 6% solids or greater.

The limiting factor in increasing slurry solids percentages found in Chapter 1 was the pressure regulation equipment used in the MFHU. This study will focus on the way in which pressure regulation can be managed. One of the influencing principals was observations taken from operation of the laboratory scale Flash Hydrolysis experiments. It was observed that clogging still occurred at laboratory scale but was typically alleviated by allowing the system pressure to build, forcing the clog through the BPR. At lower pressures, these clogs were either forced through very quickly, allowing minimum pressure build, and thus minimum disruption to system flow, or fewer clogging occurred overall. Lowering the pressure requirements for operation my help alleviate clogging, but it also allows for different pressure regulation options that may also reduce the chances of clogging occurring.

In order to reduce the potential for clogging to occur during operation of FH, lowering the operating standard operating pressure of 2000 psi to as low as 1200 psi has been investigated. This could potentially reduce capital costs in future scaling of the FH technology. In order to confidently reduce the pressure requirements, the quality of the product (hydrolyzate) must be maintained or improved. This study aims to determine if there is a relationship between operating pressure and the hydrolyzate Total Organic Carbon (TOC) and Total Nitrogen (TN) levels.

### **2.1.2 Literature Review**

Flash Hydrolysis is a relatively new biomass treatment process. Because of this, there is not much literature on the subject. Hydrothermal Liquefaction (HTL) is a process of similar conditions, so studies involving HTL were used to make inferences on FH. The effect of temperature has on TOC and TN of hydrothermal process has been studied. There has been evidence that temperature may play a role in which phase of the product nitrogen is stored in (4). The researchers in the study observed that, at higher operating temperatures, the majority of the nitrogen was hydrolyzed in the liquid phase. This is important because the separation of proteins in liquid phase can easily be done through the use of membrane filtration. A study involving the effects of pressure on a hydrothermal process was not found. This shows that a comprehensive study will be useful for future works.

## 2.2.0 Methods

### 2.2.1 Objective

This study will investigate if pressure influences the nitrogen recovered in the liquid phase of the hydrolyzate. Nitrogen is an indication of the presence of proteins, which are high-value products. In order to gain a better understanding of the effect of operating pressures on the FH hydrolyzate, the study will also consider the organic carbon recovered. The objectives of the study are as follows:

- 1) Determine if operating pressures have an effect on Total Nitrogen in the liquid phase of the hydrolyzate
- 2) Determine if operating pressures have an effect on Total Organic Carbon in the liquid phase of the hydrolyzate

### 2.3.2 Test of Hypothesis

For this hypothesis testing, the dependent variable will be the TN or TOC respectively. The independent variable will be pressure, dilution level of the analysis, diluted concentration results, category of dilution, and the overall TOC or TN concentration.

Performing a General Linear Model (GLM);

Confidence Interval (CI)=95%

$$y = X\beta_i + \varepsilon$$



where:

$y$  = TOC/TN concentration with respect to (i)

$\beta$  = column vector: consisting of  $k$  coefficients (dilution level of the analysis (dilution), diluted concentration results (dTC/dTN), category of dilution (dTNsg/dTCsg))

-assuming  $(X'X)$

$X$  = variable: Pressure

$\epsilon$  = error

Hypothesis 1: Total Nitrogen

$$H_0: \beta_1 = \beta_2 = \beta_i = 0$$

Null: pressure has no effect on the TN concentration

$$H_1: \beta \neq 0$$

Alternative: pressure has an effect on the TN concentration

Using F-test

Hypothesis 2: Total Organic Carbon

$$H_0: \beta_1 = \beta_2 = \beta_i = 0$$

Null: pressure has no effect on the TOC concentration

$$H_1: \beta \neq 0$$

Alternative: pressure has an effect on the TOC concentration

Using F-test

### 2.3.3 Source of Data

Sample data was collected after each Flash Hydrolysis trial run. Each run was performed under the same temperature of 280°C, varying the pressure each run. After a FH run was conducted the liquid portion of the hydrolyzate was analyzed using a Shimadzu TOC-VCSN TC/TN instrument. In order for TOC/TN concentrations to be accurately tested, the hydrolyzate sample was diluted before being analyzed in the TC/TN machine. The dilutions were performed using a pipet and a volumetric flask at ratios of 1/25 and 1/10. The output concentrations of the TOC/TN analysis must then be multiplied by the respective dilution factors in order to determine the overall TC/TN concentrations.

### 2.2.4 Validation of Data

Testing Normality:

Using the univariate function in SAS the normality of the dependent variable can be determined.

Tests for Normality				
Test	--Statistic--		-----p Value-----	
Shapiro-Wilk	W	0.95904	Pr < W	0.5249
Kolmogorov-Smirnov	D	0.099375	Pr > D	>0.1500
Cramer-von Mises	W-Sq	0.030458	Pr > W-Sq	>0.2500
Anderson-Darling	A-Sq	0.237579	Pr > A-Sq	>0.2500

Table 4: TOC normality tests

### Tests for Normality

Test	--Statistic---		-----p Value-----	
Shapiro-Wilk	W	0.787612	Pr < W	0.0006
Kolmogorov-Smirnov	D	0.309772	Pr > D	<0.0100
Cramer-von Mises	W-Sq	0.343506	Pr > W-Sq	<0.0050
Anderson-Darling	A-Sq	1.924852	Pr > A-Sq	<0.0050

Table 5: TN normality tests

With a confidence interval of 95%, Table 3 shows that the dependent variable Total Organic Carbon (TOC) is normally distributed. This is due to the Shapiro-Wilk p-value being greater than  $\alpha(0.05)$ . Table 4 displays the normality tests for the dependent variable Total Nitrogen. The Shapiro-Wilk p-value for TN is not large enough to confirm normality. Normality of TN dependent variable can be approximated by using the p-value of its ANOVA table discussed in a later section. No data was filtered for the purpose of this study. There were no significant outliers.

### 2.2.5 Assumptions and Limitations

For this study it is assumed that the operating temperature of the FH run is constant at 280°C. Although it is assumed that the operating temperature remains at a constant temperature, in practice that does not occur. The temperature can see a range of values from 280°C±10°C. This can limit the accuracy of the results, as previous studies have observed that temperature can affect TOC/TN output recovery. A major limiting factor is the detection limits for the Shimadzu instrument. Therefore, dilution of the sample was required for analysis. By having to dilute the sample, it is easier for error to be propagated.

### 2.2.6 Statistical Analysis

To determine the effects that the independent variables had on the dependent variable, a General Linear Model (GLM) was used. In general, the GLM is used to describe the relationship of several independent variables used to determine or predict the outcome of the dependent variable (5). In this case the dependent variables considered are Total Nitrogen and Total Organic Carbon. The independent variables used to determine if they have an effect on TOC are pressure, diluted TOC concentration, and category of dilution concentration. Similarly, the independent variables used to determine if they have an effect on TN are pressure, diluted TN concentration, and category of dilution concentration.

### 2.2.7 Procedure and Analysis

#### Flash Hydrolysis

- A proprietary microalgae species with a solid weight of 22 wt% was loaded into a piston used to deliver the algae-water slurry into the reactor
- A water line surrounding the reactor inside the furnace is heated until 280°C is reached at a flow rate of 13.85 ml/min
- System pressure is set and regulated via a backpressure regulator
  - The regulator was set to the various pressures for each run
- Once operating temperature is reached, the piston begins to pump, mixing the algae slurry with the preheated water line inside the reactor
  - Piston flow rate is 1 ml/min
- The slurry flows through the reactor with a residence time of 9 seconds

- After leaving the reactor the slurry flows through a cooling loop to reduce the temperature to below 100°C
- The output hydrolyzate is collected and stored for analysis (see Figures 7&8)



Figure 7: Collected Hydrolyzate



Figure 8: Centrifuged Hydrolyzate

#### TC/TN determination

- The hydrolyzate is centrifuged in order to separate the solid and liquid portions (see Figure 2)
- The liquid portion is then diluted to be analyzed
  - 1/25 ratio: 1 ml of hydrolyzate is pipetted into a 25 ml volumetric flask. The flask is then filled with MiliQ water

- 1/10 ratio: 1 ml of hydrolyzate is pipetted into a 10 ml volumetric flask. The flask is then filled with MiliQ water
- The diluted samples are then analyzed using a Shimadzu TOC-VCSN instrument to determine the concentration of TC and TN
- A standard solution of Organic Carbon 500 ppm C was prepared and analyzed to ensure accuracy of TC results
- A standard solution of Nitrate Nitrogen 100 ppm N was prepared and analyzed to ensure accuracy of TN results

#### Data Analysis

- Results of the TOC/TN analysis were brought into an excel spreadsheet to be organized (see appendix i)
- The data was then imported to SAS to be statistically analyzed
- Normality was tested using univariate
- GLM was utilized because of the normality of the data
- A 1<sup>st</sup> order equation was used because of the use of multiple independent variables

#### **2.2.8 Improved Pressure Regulation**

Depending on the results of the pressure study, it may be possible to use a different type of BPR previously unavailable for use due to operating pressure of the system. The BPR investigated is a dome loaded regulator. It works similar to the spring loaded BPR discussed in section 1.2.6. The major difference is the manner in which force is applied to the membrane or seal creates

pressure as the fluid flows into it. In a dome loaded BPR a flexible membrane resists the fluid flow through compressed air applied to the membrane. This allows for more flexibility in flow path as the fluid meets the membrane as compared to a static seal used in a spring loaded BPR. It also allows for multiple inlet and outlet orifices to be utilized. Both factors could be beneficial in allowing continuous flow with no clogging.

The reason a dome loaded BPR was initially ruled out during design of the MFHU, was because the maximum pressure capacity was 2000 psi. This pressure capacity would not be viable under the initial operating conditions. If the results of the pressure study indicate that lower operating pressures can be use without compromising the quality of the hydrolyzate product, then a dome loaded BPR can be used to increase slurry solid percentage and reduce clogging potential.

### **2.3.0 Results**

#### **2.3.1 Pressure Effect on Hydrolyzate Quality**

Total Nitrogen:

The results of the GLM model used in SAS are displayed below (Table 7. It can be seen that the p-value for the overall model is very low ( $<0.0001$ ). When comparing this to the TN hypothesis test with the confidence interval of 95% it can be seen that the null hypothesis is correct. This means that pressure does not affect the TN output from the FH process.

The other factors involved (dilution, diluted concentration, and category of dilution) have significant p-values. This means that these factors do play a role in the TN concentration of the hydrolyzate. This will be examined further in the discussion section.



---

### The SAS System

#### The GLM Procedure

Dependent Variable: TN

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	13	2064372.404	158797.877	84.26	<.0001
Error	6	11307.659	1884.610		
Corrected Total	19	2075680.063			

R-Square	Coeff Var	Root MSE	TN Mean
0.994552	2.549341	43.41209	1702.875

Source	DF	Type I SS	Mean Square	F Value	Pr > F
Pressure	9	2049829.375	227758.819	120.85	<.0001
Dilution	1	12078.613	12078.613	6.41	0.0446
dTNsg	2	144.771	72.386	0.04	0.9626
dTN	1	2319.645	2319.645	1.23	0.3097

Table 6: Total Nitrogen GLM SAS results

Total Organic Carbon:

The results of the GLM model used in SAS are displayed below (Table 5&6. It can be seen that the p-value for the overall model is very low (0.0001). When comparing this to the TOC hypothesis test with the confidence interval of 95% it can be seen that the null hypothesis is correct. This means that pressure does not affect the TOC output from the FH process.

The other factors involved (dilution, diluted concentration, and category of dilution) do not have significant p-values. This means that those factors played no role in the TC concentration of the hydrolyzate.

## The SAS System

### The GLM Procedure

Dependent Variable: TC

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	13	4753110.492	365623.884	163.55	<.0001
Error	6	13413.558	2235.593		
Corrected Total	19	4766524.050			

R-Square	Coeff Var	Root MSE	TC Mean
0.997186	0.912402	47.28206	5182.150

Source	DF	Type I SS	Mean Square	F Value	Pr > F
Pressure	9	3379133.300	375459.256	167.95	<.0001
Dilution	1	1115336.450	1115336.450	498.90	<.0001
dTCsg	2	40744.800	20372.400	9.11	0.0152
dTC	1	217895.942	217895.942	97.47	<.0001

Table 7: Total Carbon GLM SAS results

### 2.3.2 Discussion

While it the p-value of the model for both TOC and TN reveals that pressure does not affect the concentration of TOC/TN recovered, some insights can still be seen. For the case of TN, it appears that the dilution, diluted concentration, and category of dilution have an effect on the model. This is likely tied to the necessity to dilute the sample in order to analyze the TN concentrations.

Utilizing the Duncan test some observations about the data can be made. For the TN model (Table 7), it can be seen that pressures of 1200 psi for both trial 1 and 2 appear to have the largest mean TN concentrations, while trials for 2000 psi have the lowest. It has already been shown by the

model's p-value that pressure does not directly influence the TN concentration. The variations between pressure trials could be due to one of the limitations of the study. Temperature cannot be maintained precisely at 280°C. It is possible that lower pressures allow for easier flow through the backpressure regulator, leading to more consistent temperatures, which would benefit the TN concentration of the hydrolyzate. Looking at the Duncan test for TC (Table 8) we see much more variation between pressure groupings and TOC mean concentration giving credence to the observation about the limitations of this study.

Means with the same letter are not significantly different.			
Duncan Grouping	Mean	N	Pressure
A	2041.88	2	1200-1
A			
A	2019.63	2	1200-2
B	1904.00	2	1600-2
B			
B	1878.25	2	1400-1
B			
B	1861.38	2	1400-2
B			
B	1830.13	2	1600-1
B			
B	1817.13	2	1800-1
C	1238.63	2	2000-2
C			
C	1223.75	2	2000-3
C			
C	1214.00	2	2000-1

Table 8:TN Duncan Grouping

Means with the same letter are not significantly different.			
Duncan Grouping	Mean	N	Pressure
A	5847.00	2	1200-1
B	5701.50	2	1200-2
C	5397.00	2	1800-1
C			
D	5333.50	2	1600-2
D			
D	5256.50	2	1400-1
D			
D	5220.75	2	1400-2
E			
E	5108.25	2	1600-1
F	4765.00	2	2000-1
F			
F	4728.50	2	2000-3
G	4463.50	2	2000-2

Table 9: TOC Duncan Grouping

### 2.3.3 Improved Pressure Regulation Testing

Based on the findings discussed in section 2.3.1 & 2.3.2, it was determined that operating pressures could be lowered without negatively impacting hydrolyzate quality. This allows for the potential implantation of a dome loaded BPR. Due to the complex nature of dome loaded BPRs, few manufacturers are available. Though discussion with various manufactures around the

industry Equilibar was selected for their quality and willingness to facilitate testing options for research purposes.

Although Equilibar is capable of manufacturing a dome loaded BPR with a maximum pressure capacity of 2500psi, it would be costly and time consuming to produce. It was decided that a standard version of the exact type of BPR would be tested to evaluate if it is capable of handling slurry loading of greater than the 4 wt% previously reached. The maximum pressure capacity of the test BPR was 500 psi. Due to the decreased pressure capacity, the heat of the process must also be reduced to maintain subcritical conditions.

Testing of the standard Equilibar dome loaded BPR was performed to evaluate its potential to reduce clogging and increased slurry solid percentage. The results of this testing were used to determine if a custom BPR would be procured and used for the continued operation of the MFHU. The reaction conditions used for these tests were a pressure of 400 psi and a temperature of 180°C. The flow rate remained the same 160 mL/min as operating conditions to try to maintain as many operating conditions the same as previous MFHU testing. Slurries of 1 wt%, 3 wt% and 4 wt% were tested continuously for 15 minutes. No blockages or clogs were observed in slurries of 1 wt% & 3 wt% as flow was continuous and there was very little pressure fluctuation ( $\pm 20$  psi). The 4 wt% slurry saw slightly larger pressure fluctuations ( $\pm 50$  psi) but did not clog and flow remained continuous. It was observed in the hydrolyzate of all test runs that it appeared that full fractionation of algae did not occur since large particle sizes seen in the liquid. This is due to the reduced temperature of the testing.

## 2.4 Conclusion

This study's aim was to determine if pressure influenced the Total Nitrogen and Total Organic Carbon concentrations of the liquid hydrolyzate the Flash Hydrolysis process produces. By analyzing the results using a General Linear Model it was determined that pressure does not affect the TOC or TN concentrations of the liquid hydrolyzate. This allows Flash Hydrolysis to operate at lower pressures. Lowering operating pressures can ease the constraints of scaling Flash Hydrolysis to become an industrial process, leading to increased success for Flash Hydrolysis as a whole.

With lower pressures still being favorable for hydrolyzate quality, the MFHU can now be fitted with a custom dome loaded BPR. Based on the test runs of a standard BPR at lower pressures, it is expected that this will allow the slurry loading goal to be reached.

## REFERENCES

1. *Energy Independence and Security Act of 2007. In H.R. 6 100th Congress. 2007.*
2. Andrew P. Bessette, A.T., Mason J. Martin, Ben J. Stuart, Eleazer P. Resurreccion, Sandeep Kumar, *Life Cycle Impacts and Techno-economic Implications of Flash*. 2018.
3. Jose Luis Garcia-Moscoso, W.O., Sandeep Kumara, Patrick G. Hatcher, *Flash hydrolysis of microalgae (Scenedesmus sp.) for protein extraction and production of biofuels intermediates*. 2013.
4. Hartonen, K., *Greenfluids*. 2009, The Association of Finnish Chemical Societiesw.
5. John J Milledge, S.H., *A review of the harvesting of micro-algae for biofuel*. 2013. Rev Environmental Science Biotechnol(12:165-1784).
6. Natarajan, E., A. Nordin, and A.N. Rao, *Overview of combustion and gasification of rice husk in fluidized bed reactors*. Biomass and Bioenergy, 1998. **14**(5): p. 533-546.
7. *What Is Induction Heating?* 2019 [cited 2019 10/15/2019]; Available from: [http://www.gh-ia.com/induction\\_heating.html](http://www.gh-ia.com/induction_heating.html).
8. Inc, C. *The Joule Heating Effect*. 2019; Available from: <https://www.comsol.com/multiphysics/the-joule-heating-effect>.
9. Britannica, T.E.o.E. *Hysteresis*. September 22, 2013 [cited 2019; Available from: <https://www.britannica.com/science/hysteresis>.
10. Kumar, S. and R.B. Gupta, *Hydrolysis of Microcrystalline Cellulose in Subcritical and Supercritical Water in a Continuous Flow Reactor*. Industrial & Engineering Chemistry Research, 2008. **47**(23): p. 9321-9329.

## **APPENDIX I**

### **MFHU OPERATION CHECKLIST**

1. Ensure all units are plugged into the appropriate power supply
  - a. Chilling Unit: standard 115v outlet
  - b. LEWA pump: standard 115v outlet
    - i. Note: pump turns on automatically when plugged in. A power strip can be used as an on/off switch
  - c. Induction Heat power supply: 230v outlet
  - d. Induction Heat digital display: standard 115v outlet
2. Turn on Chilling Unit: must be turned on prior to IH power supply to prevent IH power supply from overheating.
3. Prime Lewa Pump: If the pump has not been used for several days priming may be necessary.

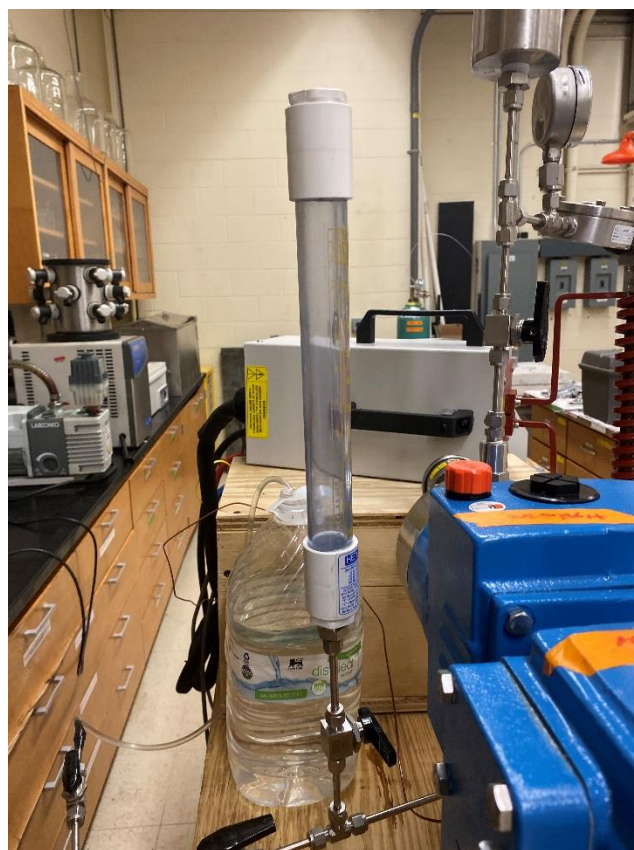


- a. To prime the pump, close the inlet valve so water is only pulled from the calibration column



- b. Ensure pressure regulators are set to zero as not to increase pressure created by the pump

- c. Turn on the pump and allow water to be drawn from the calibration column that is elevated above the pump inlet

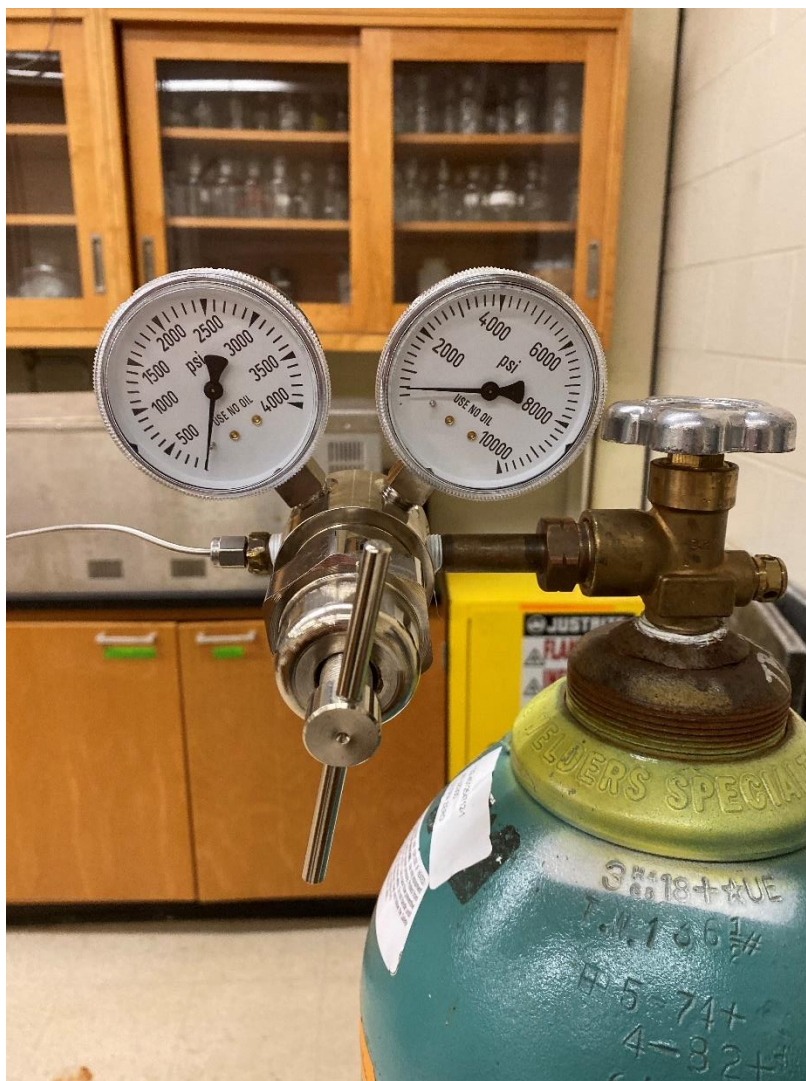


- i. Ensure no air pockets or bubbles are formed. Pump outlet may have to be disconnected from the system for this to occur.
4. Once the pump is primed flow of water needs to be slowly transitioned from the calibration column to DI water storage.

- a. This can be done while slowly opening the valves to allow water to flow from the water storage using the following two valves



5. Set BPR to operation pressure (1400 psi): This is done by adjusting the regulator gauge on the compressed air cylinder that is connected to the dome loaded BPR



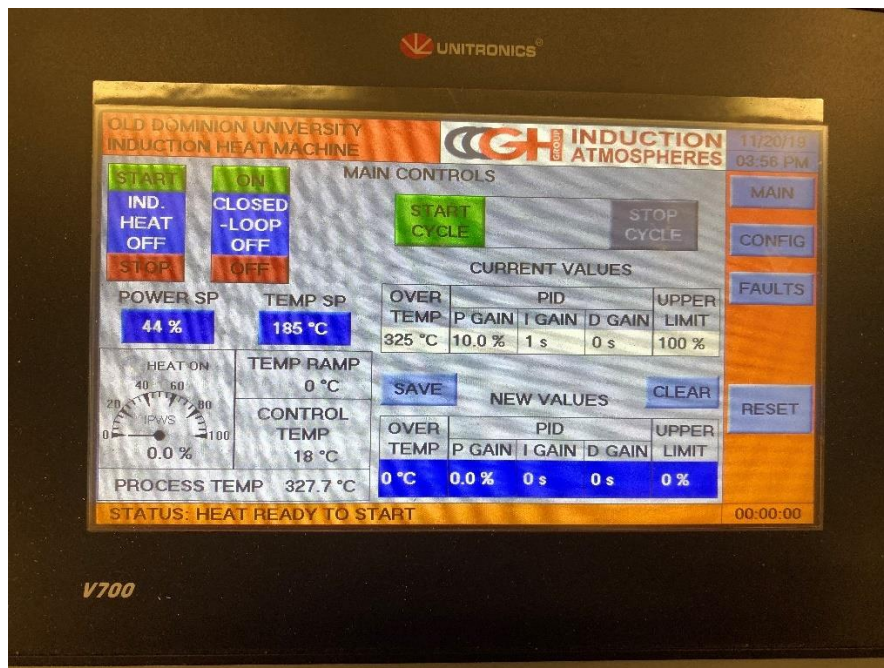
- a. Verify that the pressure gauge on the MFHU reads the 1400 psi. If not adjust the compressed air regulator until the MFHU gauge reads 1400 psi.
6. After reaching operating pressure, ensure flow is continuous from the outlet of the dome loaded BPR.
7. Once flow is established the Induction heating can be turned on.



a. First turn both switches on the power supply



- b. Once the power supply is on you can access the digital display



- i. There is a preprogrammed start up and heating rate to 280°C that can be used.
  - ii. Parameters can be adjusted via the digital display as need. Please refer to the manufatrures manual to do so.
8. Allow system temperature to reach 280°C
  9. Once reaction temperature is reached flow can be transferred from DI water to algal slurry.

10. Similar to priming the pump slowly adjust the DI water inlet valve to closed while simultaneously adjusting the algal slurry inlet valve to open.



11. Once the algal slurry inlet valve is completely open ensure flow is occurring by examining the algal slurry storage.
12. Algal slurry should now be flowing through the system and processing
13. Watch system pressure to check for clogging. If pressure stays elevated above operating pressure a clog has likely occurred and system should be shut down.
14. Collect hydrolyzate in an appropriate container.

## Appendix II: MFHU shutdown checklist

1. Switch flow from algal slurry to DI water as described in step 10 of Appendix I.
2. Allow water to flow through the system for 5 minutes to ensure algae is cleared
3. Shut off induction heating from the digital display.
4. Allow system to cool to 60°C
5. Turn off pump

## Appendix III: Pressure Study Sample Data

Operating Pressure	Dilution	dil TC	dil TN	TC (mg/L)	TN (mg/L)	
1200-1	1/25	222.8	83.55	5570	2088.75	1200
1200-1	1/10	612.4	199.5	6124	1995	1200
1200-2	1/25	214.8	82.13	5370	2053.25	1200
1200-2	1/10	603.3	198.6	6033	1986	1200
1400-1	1/25	207.6	78.46	5190	1961.5	1400
1400-1	1/10	532.3	179.5	5323	1795	1400
1400-2	1/25	195.5	74.67	4887.5	1866.75	1400
1400-2	1/10	555.4	185.6	5554	1856	1400
1600-1	1/25	189.5	72.57	4737.5	1814.25	1600
1600-1	1/10	547.9	184.6	5479	1846	1600
1600-2	1/25	199.6	77.2	4990	1930	1600
1600-2	1/10	567.7	187.8	5677	1878	1600
1800-1	1/25	205.6	72.57	5140	1814.25	1800



1800-1	1/10	565.4	182	5654	1820	1800
2000-1	1/25	183.6	49.52	4590	1238	2000
2000-1	1/10	494	119	4940	1190	2000
2000-2	1/25	178.2	50.73	4455	1268.25	2000
2000-2	1/10	447.2	120.9	4472	1209	2000
2000-3	1/25	181.2	49.58	4530	1239.5	2000
2000-3	1/10	492.7	120.8	4927	1208	2000

## Appendix IV: SAS outputs

```

data proj;
INPUT Pressure $ Dilution $ dTC dTCsg $ dTN dTNsg $ TC TN @@;
CARDS;
1200-1      1/25    222.8 dTCsg2      83.55 dTNsg2      5570    2088.75
1200-1      1/10    612.4 dTCsg4      199.5 dTNsg4      6124    1995
1200-2      1/25    214.8 dTCsg2      82.13 dTNsg2      5370    2053.25
1200-2      1/10    603.3 dTCsg4      198.6 dTNsg4      6033    1986
1400-1      1/25    207.6 dTCsg2      78.46 dTNsg2      5190    1961.5
1400-1      1/10    532.3 dTCsg3      179.5 dTNsg4      5323    1795
1400-2      1/25    195.5 dTCsg1      74.67 dTNsg2      4887.5    1866.75
1400-2      1/10    555.4 dTCsg3      185.6 dTNsg4      5554    1856
1600-1      1/25    189.5 dTCsg1      72.57 dTNsg2      4737.5    1814.25
1600-1      1/10    547.9 dTCsg3      184.6 dTNsg4      5479    1846
1600-2      1/25    199.6 dTCsg1      77.2  dTNsg2      4990    1930
1600-2      1/10    567.7 dTCsg3      187.8 dTNsg4      5677    1878
1800-1      1/25    205.6 dTCsg2      72.57 dTNsg2      5140    1814.25
1800-1      1/10    565.4 dTCsg3      182   dTNsg4      5654    1820
2000-1      1/25    183.6 dTCsg1      49.52 dTNsg1      4590    1238
2000-1      1/10    494   dTCsg3      119   dTNsg3      4940    1190
2000-2      1/25    178.2 dTCsg1      50.73 dTNsg2      4455    1268.25
2000-2      1/10    447.2 dTCsg3      120.9 dTNsg3      4472    1209
2000-3      1/25    181.2 dTCsg1      49.58 dTNsg1      4530    1239.5
2000-3      1/10    492.7 dTCsg3      120.8 dTNsg3      4927    1208
;

proc univariate data=proj;
    var TC;

Proc GLM data=proj;
    Class pressure dilution dTCsg;
    Model TC=pressure dilution dTCsg dTC;
    Means Pressure Dilution dTCsg/Duncan;

```

```
proc univariate data=proj;  
    var TN
```

```
Proc GLM data=proj;  
    Class pressure dilution dTNsg;  
    Model TN=pressure dilution dTNsg dTN;  
    Means Pressure Dilution dTNsg/Duncan;
```