



# WPI

## **Waste to Energy: Optimization of Biofuel Production from Hospital Food Waste**

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## Abstract

Despite the disastrous effects of climate change, renewable energy sources are still neglected in favor of the more harmful alternative, fossil fuels. Hydrothermal liquefaction is a sustainable technology that allows for renewable energy to be produced from biomass. Conversion of biomass to useable and storable fuels, biogas and bio-crude provides a sustainable answer to humanity's nonrenewable fuel crisis. This project utilizes hospital food waste as feedstock to the hydrothermal liquefaction process, to both produce sustainable renewable sources of energy and develop a sustainable, environmentally friendly waste management method. In this study we experiment with the solid loading percent and reaction time to decipher the optimal condition. Our findings highlight the conditions that produce the maximum amount of oil and the highest quality oil.

## Acknowledgements

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## Chapter 1. Introduction

The world today is overly reliant on fossil fuels and other nonrenewable sources of energy. Nonrenewable energy sources are finite, difficult to extract and are continually diminished as human population and energy consumption increases. In addition to the diminishing sources, non-renewable energy also exacerbates the problem of pollution. Most non-renewables energy resources produce carbon dioxide as a byproduct, which remains trapped in the atmosphere and is a leading cause of climate change. Non-renewable energy also pollutes the air and reduces the quality of the air that people breathe in each day. Polluted air is the main cause for aggravated respiratory diseases and several other health-related problems. The US Office of Energy Efficiency & Renewable Energy (EERE) even goes as far as to say that a transition to renewable energy sources could save 25,000 lives (Solar energy technologies office, 2016). One of these renewable energy sources is biofuels, which are fuels produced from biomass rather than the slow natural process involved in the formation of fossil fuels. Unlike other renewable energy sources such as solar, wind or geothermal power, biomass can be converted directly into liquid phase biofuels which help address transportation fuel needs. The two most common types of biofuels in use today are ethanol and biodiesel, both which are described by the EERE as first-generation biofuel technology (BETO, 2022). The Bioenergy Technologies Office (BETO) is collaborating with industry to develop next-generation biofuels made from waste, cellulosic biomass, and algae-based resources. BETO is focused on production of hydrocarbon biofuels that could serve as petroleum substitutes in existing refineries, pumps, and vehicles (BETO, 2022).

Our group, like BETO, strives to develop next-generation biofuels using Hospital Food Waste (HFW). Food waste is just one of many sources of biofuels, but its benefits extend beyond reducing fossil fuel consumption as far as waste management. Landfills can contribute to a number of environmental issues, including climate change, habitat destruction, and soil, air, and groundwater pollution. According to the EPA, 56% of all food waste in the United States was diverted to landfills in 2018. During this same year in the institutional sector, which covers hospitals, none of the generated food waste was used for bioproducts, and was instead sent to landfills, incinerators, or composting plants (EPA, 2020). One of the goals of using HFW as a source of biofuel generation is to reduce the amount of waste diverted to landfills, and at the same time use this waste to produce an environmentally friendly substitute for fossil fuels.

One of the primary methods of converting biomass to bio-crude oil is hydrothermal liquefaction (HTL), which is also the mechanism used in this project. This process involves superheating a mixture of biomass and water to produce aqueous, char, and bio-crude products. These products are then separated and undergo several analyses.

Through HTL experimentation with multiple variables, using HFW as a feed, and analysis of the products, this project provides evidence for most ideal conditions for generating the most viable bio-crude product. The ideality of the product is measured by carbon analysis, gas chromatography/mass spectroscopy, and Karl-Fischer Titration, as well as elemental analysis.

## Chapter 2: Background

### 2.1 The Need for Renewable Energy

Renewable energy's value is growing exponentially. People are burning fossil fuels faster than it can be naturally generated, and therefore the amount available for use will eventually run out. According to the U.S. Energy Information Administration, 134.8 billion gallons of gasoline were burned in the United States in 2021 alone (Frequently asked questions). According to WorldMeters, the world has approximately 47 years of oil consumption left until natural reserves are depleted (World Oil Statistics). Within that time, the world needs to discover, develop, refine, and market a new product that can replace fossil fuels. There have been several different products presented throughout the years such as electric cars, solar and wind power, and biofuels, but most are still in the early stages of implementation.

Aside from the depletion of the world's supply of fossil fuels, climate change is another reason why the world desperately needs a new, cleaner source of energy to replace fossil fuels. Burning fossil fuels contributes to a phenomenon called the greenhouse effect. Two of the major greenhouse gasses are carbon dioxide ( $\text{CO}_2$ ) and methane ( $\text{CH}_4$ ), which are produced by the burning and extraction/transport of fossil fuels, respectively. When these compounds are produced and remain in the atmosphere, they block heat energy arriving from the sun from being released back into space. This effect causes more and more heat to be retained by the earth over time, increasing global temperatures (US EPA, O. Basics of Climate Change). While global temperature change is not unnatural, and has happened before in Earth's long history, the current

global increase in temperature is far from natural. The increase in global temperature from 1986-2016 was 1.2 °F higher than the entire increase from 1901–1960 (Usgcrp). This indicates that the increase in global temperature is not linear and is likely being affected by the emission of gaseous human waste and pollution.

This increase in temperature has a secondary effect as well. As the earth warms, the hot seasons in each region also become more extreme. This causes ice caps and glaciers to become more prone to melting during their warm seasons. Furthermore, as the earth warms so do her oceans, and as seawater warms it thermally expands to a greater volume. As a result, sea levels have been rising in tandem with the increase in global temperature. Since 1880, sea level has risen 8-9 inches, resulting in a loss of surface land area. This loss of land area could result in mass displacement of the coastal human and wildlife population. Given that 30% of the population in the United States lives in an area prone to effects from sea level rise (Lindsey, R.), this is an issue that could have devastating effects to not only the U.S. population, but also people around the world.

Due to the depletion of fossil fuels and the unnatural warming of the earth, society has started to discover alternative ways of producing and harnessing energy in a manner that is sustainable. Below are some of the methods by which humans are beginning to establish ways of producing renewable energy.

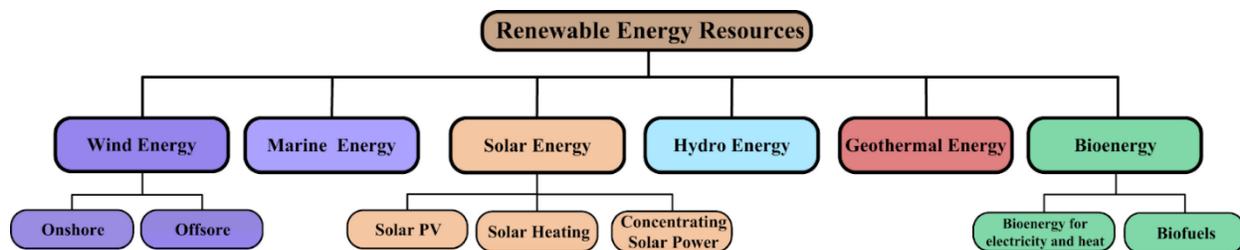


Figure 1: Branches of some of the most popular sources of renewable energy (Ellabban et al, 2014).

Many of these methods have to do with the movement or natural occurrences of the earth by converting a certain type of energy into electricity. Wind energy converts the kinetic energy of air movement, geothermal energy converts the earth’s core’s heat energy, and solar energy converts radiation emitted by the sun. Biofuels on the other hand operate differently. Biofuels

take advantage of the waste produced by humans and are a resource capable of solving multiple problems at the same time.

## 2.2 Waste as an Energy Source

Waste, often referred to as trash or garbage, is not perceived by most people as having any value. Municipal solid waste (MSW) in particular refers to solid waste generated by people's everyday lives in homes, schools, hospitals, businesses and other urban areas (EPA, 2013). MSW refers to the everyday items people use and throw away, such as product packing, newspapers, appliances, clothes, bottles and food scraps. As mentioned before, many people view waste as a problem. This perception is due to the waste management methods that are currently in place. Waste management refers to the processes that are employed to manage waste from its perception to its final disposal. The most commonly utilized methods of waste disposal currently, are Sanitary Landfills, High temperature composting and Incineration. These methods are not sustainable and are often harmful to the surrounding environment. Sanitary landfills control landfill leachate and gas, however as land and space become increasingly scarce, there is often not enough space for MSW (EPA, 2011). Sanitary landfills do try to control landfill leachate and gas by employing an antiseptic treatment to MSW. This forms an independent drainage to prevent the underwater from being contaminated. However, it is inevitable that there will be high volumes of carbon dioxide, methane and other pollutants in the landfill. The pollutants damage the surrounding environment, posing health risks to the people in the area. High temperature composting, which is heavily utilized in Chinese cities, is low cost but releases a large amount of pollutants to the environment. Lastly, incineration has the potential for energy recovery with 80-90% waste reduction (GreenBeston, 2018). However, the process releases acidic gasses, ashes and other pollutants that damage the environment. Nonetheless, people continue to use these methods due to its convenience and cost. For most, when their waste is removed from their house, they tend not to think of the harmful environmental effects that their waste causes.

While many view waste as a problem, others see waste's potential as a solution to other problems. Deficiencies in current waste management methods call for future sustainable and environmentally friendly methods. Sustainability is even more important because as energy demands increase and energy sources diminish, we will need to generate energy from waste. This is the idea behind the United States Environmental Protection Agency's Food Hierarchy (EPA,

2022). The food recovery hierarchy orders several waste management actions that can be used to prevent and divert waste. These actions are ordered from most preferred to least preferred. The tiers on the food recovery hierarchy highlight different waste management strategies for food waste. The top tiers of the hierarchy suggest strategies that prevent waste from accumulating because these are the most beneficial to the environment, society and the economy (EPA, 2022). The bottom tiers of the hierarchy suggest strategies that manage waste but are not sustainable or environmentally friendly and should only be used as a last resort.

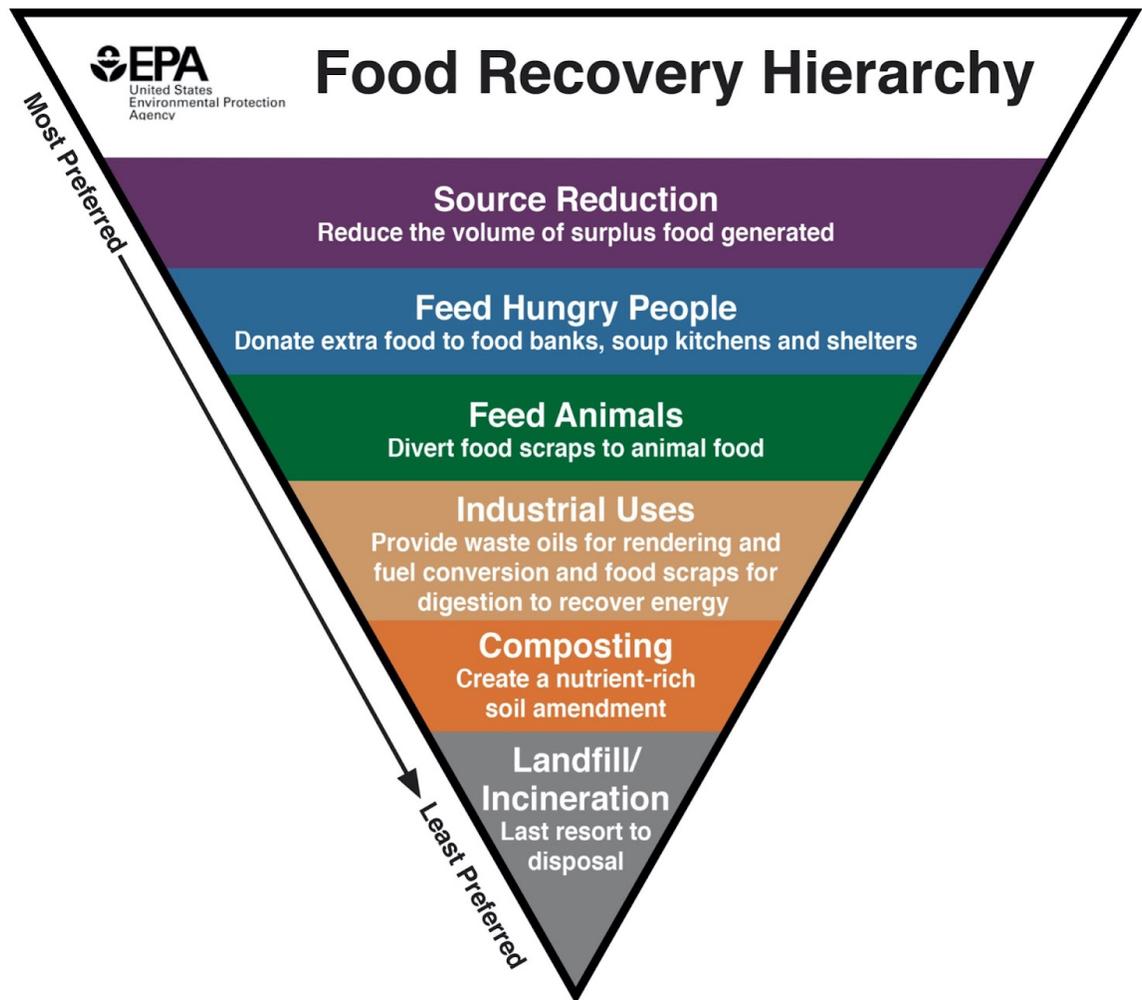


Figure 2: Food recovery hierarchy (EPA, 2022)

The largest part of human daily trash is food. The EPA reports that 133 billion pounds of retail food in the United States does not get eaten (EPA, 2019). Therefore, it is advantageous to use waste as a feedstock because it is readily available and an effective waste management

technique. Our Project implements the fourth tier of the Food Recovery Hierarchy, Industrial uses, by using hospital food waste as a feedstock for the hydrothermal liquefaction process.

### 2.3 Hydrothermal Liquefaction

The increase in attention towards renewable sources of energy has encouraged research into alternative pathways for fuel products. Conversion methods for agricultural and organic waste to fuels are still being explored today. The conversion of biomass into usable and storable fuels, biogas and bio-crude, are considered the most viable solution to humanities sustainable fuel issues (Scarsella et al, 2020). The study conducted in this paper explores the thermochemical pathway for biomass conversion into fuel.

There are a few processes that can convert biomass into fuel, each with their own limitations. Pyrolysis and hydrothermal liquefaction, HTL, are the two processes that were considered for this study. Several factors led to choosing HTL over pyrolysis. Both pyrolysis and HTL can produce fuel-grade biocrude from biomass feed, but pyrolysis has a more severe impact on the environment according to a life cycle assessment done by Yi Heng Chan (Chan et al, 2016). According to Chan's study, pyrolysis had almost 50% more global warming potential impact than HTL. The impact categories considered in this study included climate change, acidification, eutrophication, photo-oxidant forming, and human toxicity. When studying the methodology behind pyrolysis, these issues come as no shock: pyrolysis is a picky, intense process compared to HTL.

Hydrothermal liquefaction's reaction kinetics are not completely understood. The process is complex and involves a variety of chemical reactions, making it hard to track each step of the conversion. Because of this, the exact kinetic model of HTL's mechanism is unknown (Xu & Li, 2021). Despite this, HTL still generates a lot of attention: HTL has the potential to solve the growing waste management issue by pairing it with the constant need for sustainable fuels. Though pyrolysis gave similar promise, HTL is often the frontrunner due to its relatively low reaction temperatures, high energy efficiency, and ability to react in the presence of oxygen and water (Sandquist et al, 2019). A major difference between HTL and pyrolysis is the required process energy: the reaction alone requires the system to be maintained at temperatures exceeding 1000 °C (Trubetskaya et al, 2016). The feed to a pyrolysis process must be dried before it begins reacting. This energy cost, paired with the extremely high reaction temperature,

raises the operating cost of any pyrolysis process far above that of HTL. Lastly, the energy density of pyrolysis biocrude is “significantly lower” than that of HTL biocrude (Xu & Li, 2021).

Though the exact steps to HTL are not clear, the general stages have been noted. First, the lignocellulose is depolymerized. Now in monomer form, the peptides are further decomposed through cleavage, dehydration, and decarboxylation into smaller fragments. Once the reactor is quenched, the reaction intermediate cools, recombining into hydrothermal products such as syngas, biocrude, and aqueous phase products (Xu & Li, 2021). To further understand HTL’s mechanisms, researchers are exploring the natural degradation pathways of lignocellulose.

Hydrothermal liquefaction is already an efficient process, still, catalysts’ effects have been tested on the process and product quality. Different catalysts can be applied to favor different products. For example, the presence of alkaline earth metals greatly improves the oil yield, but in doing so lower its product quality (Scarsella et al, 2020). The lignocellulose molecule is very complex, but the study of lignocellulose conversion to biofuel is aided by catalytic research. Catalytic studies on lignocellulose conversion aim to inform the community further about the finer process details, while also attempting to lower the production cost of “second generation biofuels.”

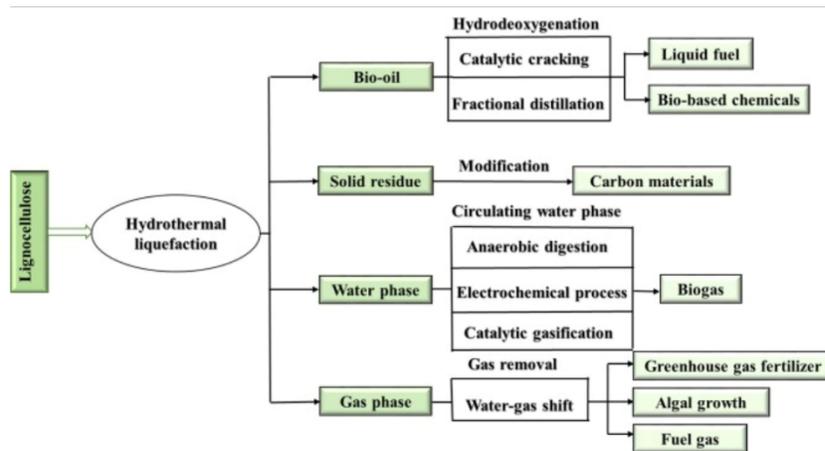


Figure 3: Hydrothermal liquefaction product tree (Xu & Li, 2021)

## 2.4 Hospital Food Waste

According to the United States Department of Agriculture, food is the largest category of material placed into municipal landfills, acting as the third largest source of human related methane emissions in the United States. This emission was roughly 14.1% of all methane

emissions in 2017 (USDA). A large portion of this food waste comes from hospitals and can be split up into two categories of waste: preparation and leftover. Preparation waste consists of solid waste including peels, seeds, etc. or liquid waste such as pickle juice, canned fruit liquid, etc. Leftover food waste is unprepared food that expired, fully prepared food that was not served and plate waste which was not eaten (Alshqaqeeq et al). When this food is wasted at the consumption side of the food supply chain, all of the previous energy inputs, such as water, labor, energy, time, and cost, are wasted. According to Cook et al, one-third of all food that is wasted costs the global economy one trillion US dollars annually and contributes to the continuing problem of food insecurity in both developed and developing countries. The top contributions to food waste in hospitals are food spoilage, food trimmings, large portions, meal quality, patient appetite, and medical interruptions (Cook et al).

There are currently 6,210 hospitals in the United States, each of which produce a staggering amount of food waste. One study found that the amount of food produced in one hospital that serves 6,640 patient meals per week discards more than 48,000 pounds of food annually (Alshqaqeeq et al). Since the average number of beds per hospital in the US is 227 serving around 4768 meals per week, the average food waste produced per hospital annually is 34,465 pounds, which is over 100,000 tons of waste per year (American Hospital Directory). Moreover, several states, including Connecticut, Massachusetts, Rhode Island, and Vermont have enacted laws effectively banning organic waste (Hegwer). With the large quantity of food waste available and effective laws that restrict hospitals from disposing of organic waste with traditional waste, hospital food waste becomes a viable energy source option.

## 2.5 Food Waste Compositions

Through several studies on the starting materials used to create biocrude oil with hydrothermal liquefaction, it was observed that larger compositions of fat in the starting material would produce more oil with higher energy potential. The first observed report compared microalgae, *D. tertiolecta*, with different compositions of fat content as starting materials for the creation of biocrude through hydrothermal liquefaction (Shuping et al 2014). They reported that liquefying *D. tertiolecta* with higher protein content produced a biocrude oil with shorter optimal holding times and optimal bio-oil yields about 10% less were observed relative to microalgae of the same species with higher fat content. They explain this by stating that crude fat components

are easier to liquefy and contribute more to the biocrude yield from hydrothermal liquefaction than proteins. In relation to hospital food waste starting materials, higher fat content should yield a larger amount of biocrude.

The next study examines animal fatty wastes as a starting material for the generation of biocrude oil due to its large content of triglycerides (fat), with high energetic value (Ben Hassen-Trabelsi et al 2014). They mention that the starting materials have high carbon contents (around 75%), moderate oxygen contents (around 20%), high hydrogen contents (around 12%), and low nitrogen/sulfur contents (around 0.6% and 0.2% respectively) which are optimal for the production of biocrude [source]. After the oil was collected from the reactor, they measured the higher heating value (HVV) for the produced oil using animal fat and compared it to several other studies. The HVV for the organic phase of the produced oil was 9,600 kcal/kg, which is slightly less than that of petroleum fuel, which is typically on the range of 11,000 to 15,000 kcal/kg. The following table provides the calorific value for oils produced with different starting materials referenced in this report:

<b>Starting Material</b>	<b>HVV (kcal/kg)</b>
Animal Fatty Waste	9600
Fish Fatty Waste	9314
Macroalgae	5565-8025
Microalgae	6687
Woody Biomass	3822-4538

*Table 1: Higher heating values for various starting materials*

As shown in the table above, the oils produced from starting materials with a higher fat content had higher calorific values, meaning that they were more energy dense (Ben Hassen-Trabelsi et al 2014). This study also found that the oil yield in suitable conditions with the animal fatty waste was on the range of 58-77.9 weight percent. Finally, through FTIR, they found that the oil characterization revealed a higher presence of straight carbon, high oxygenated groups content, and a low quantity of aromatic groups. In other words, a higher fat content in the starting material produces a larger quantity of oil that is higher quality.

## Chapter 3. Methods

The study aims to understand the impacts of the influential variables on the yield and composition of bio-oil produced using the hydrothermal liquefaction process. In order to achieve this, first the group carried out experiments as stated in the Procedure, then the group further analyzed the resulting bio-oil using Proximate elemental analysis, KF titration, and Gas chromatography mass spectrometer (GCMS). Additionally, the group calculated the Moisture Content, Higher heating values and the mass balance. Lastly, the group carried out the experiment at varying solids weight percent, run times and reactor temperatures.

### 3.1 HTL Reactor Safety Considerations

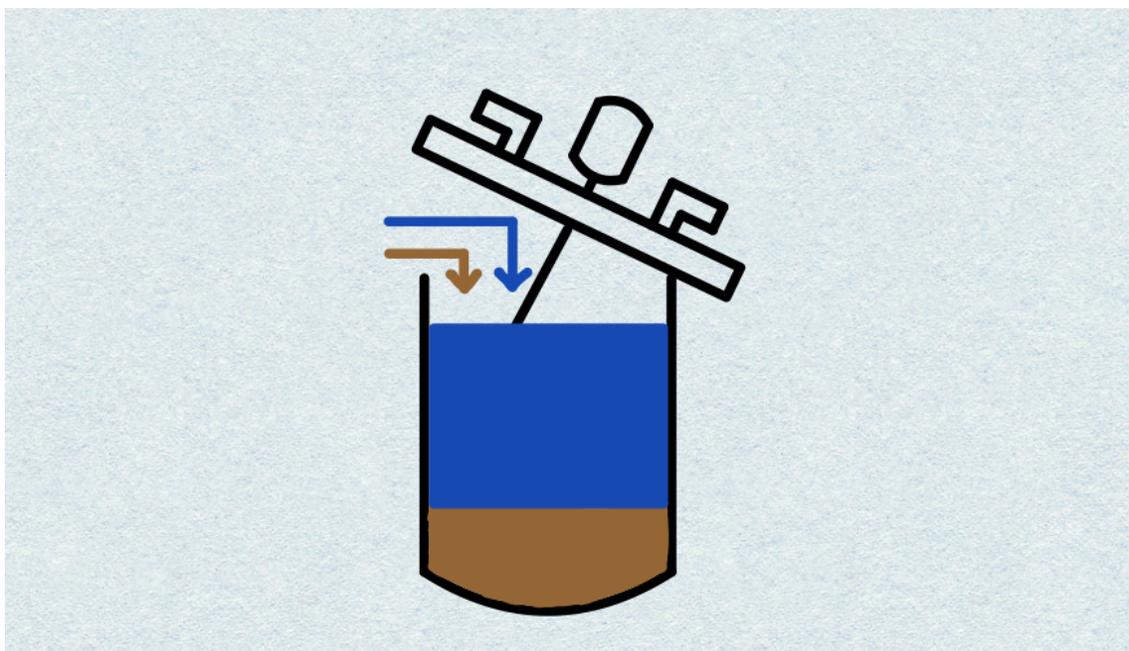
Before each reaction, the operating team ensured the reactor was safe to operate. To start, the team inspected the reactor top and bottom for visible damage. This inspection checked the O-ring, thermocouple, stir rod, and cooling jacket for any sign of fatigue. Had any of the pieces shown wear beyond continuable use, the operators would replace the piece before starting the reaction. Even with the visual checking stages, the rupture disk's age was tracked and replaced every 100 runs to prevent a failed reaction due to metal fatigue.

Along with the reactor, the accessory equipment was checked for functionality before the reaction was initiated. The cooling lines were checked for loops and the water level was maintained above a set threshold. The heater and thermocouple were checked for error using the thermocouple reading against an independent thermometer.

During the entirety of the reaction operators wore proper lab attire. Gloves and goggles were required, and lab coats were of frequent use when preparing the reactor and extracting the products.

### 3.2 Hospital Food Waste HTL Reaction Procedure

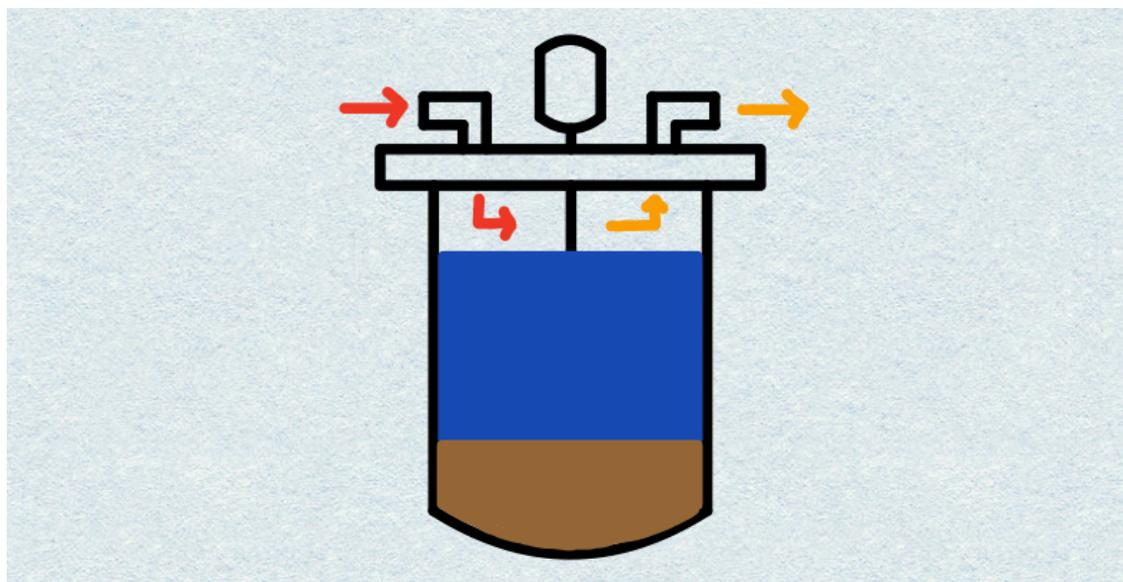
To begin the procedure, the reactor, its pieces, and all the required glassware are washed to verify nothing from the last reaction carries over to the current reaction. From here, initial weight measurements are taken for all glassware as well as the reactor top and bottom. This is recorded on the data sheet for reference during the extraction stages. Once everything is recorded, solid waste and deionized water are loaded into the reactor in accordance with the reaction parameters.



*Figure 4: Solid and liquid reactants are loaded into the reactor*

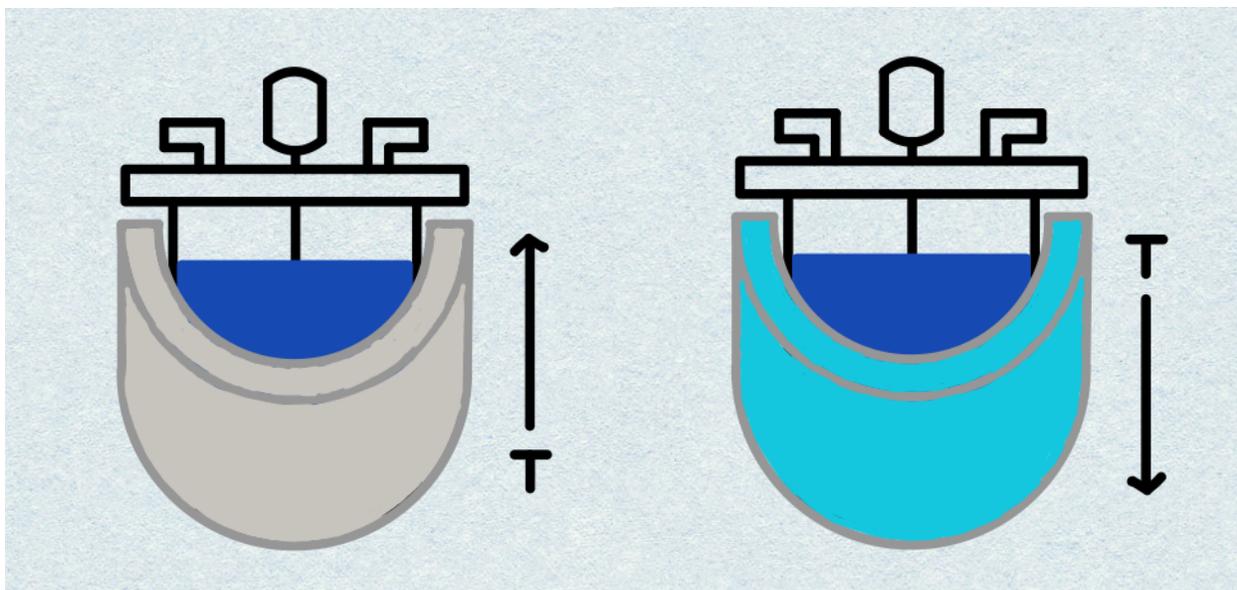
The initial solid and liquid loading adds to 100 grams total, no matter the initial parameters. This allows the team to assume a 100 gram mass balance for product yield analysis. Once the reactants have been inserted, the full reactor is weighed before the clamps are added to seal the reactor. The clamps are tightened in a star pattern for uniform sealing.

Once the clamps are adequately tightened, the reactor is brought into the fume hood. The thermocouple, stir bar, and water cooling lines are attached to the reactor. The reactor is then flushed with nitrogen gas to remove any oxygen in the ambient air to maintain the oxidation levels from run to run. After the first flush, the reactor is held at the set pressure for 15 minutes to verify the reactor seal is holding.



*Figure 5: Oxygen is flushed from the reactor using nitrogen gas*

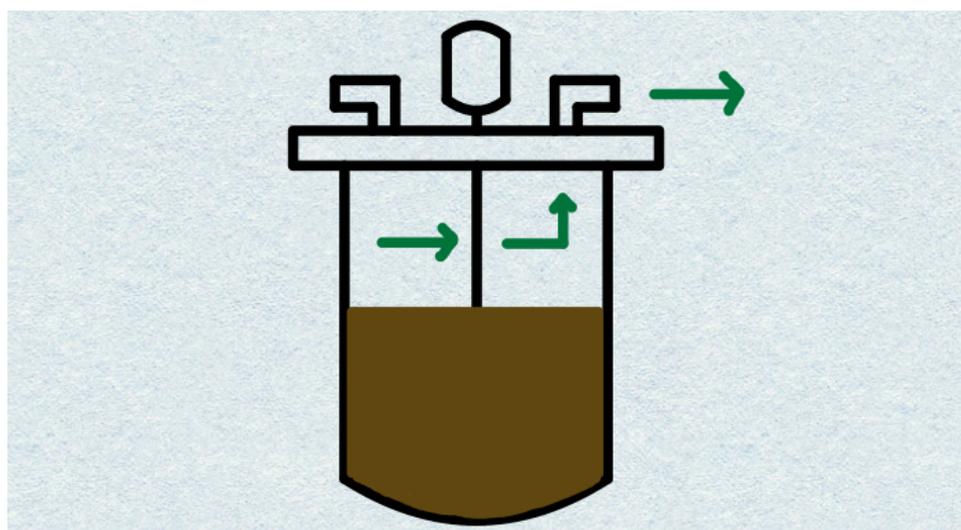
Once the reactor is confirmed capable of maintaining pressure, it is flushed two more times before being set to 900 psi starting pressure. When the reactor is at the set pressure, the gas line is disconnected from the reactor. The heating mantle is raised up and turned on. The stirring rod is set to the third tick and the cooling pump is plugged in. To track the run conditions, the temperature and pressure are noted every three minutes until the operating temperature is reached. To increase the ramp up speed, the set point is initially set to 20°C higher than the operation temperature. Once the reactor reaches within 25°C of this temperature, return the heater's set temperature to the operation temperature.



*Figure 6: The heating mantle and ice bath regulate the reactor temperature*

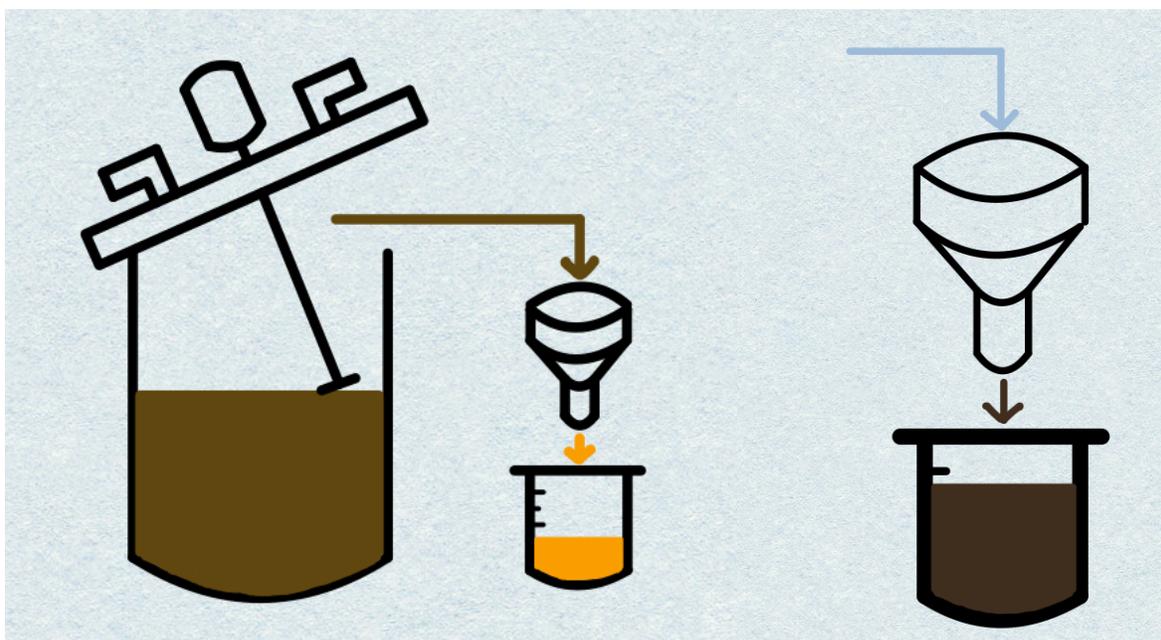
Once the designated reaction time is up, the reactor is immediately quenched with an ice bath. The temperature and pressure of the reactor are noted at the time of the quench and again when the reactor temperature returns to 23°C.

Once the reactor is quenched, the reactor is prepared for the extraction stages. First, the reactor is depressurized. This is done slowly so none of the reactor contents are ejected through the nozzle. The reactor is then put into a vice where the clamps are taken off.



*Figure 7: The quenched reactor is depressurized*

After the clamps are removed, the reactor is dried of any water from the quench bucket. The entire reactor is then massed and compared with the initial weight. Next, the reactor is opened and the vacuum pumped connected to the appropriate glassware. The char product is removed from the reactor using a spatula to scrape any char stuck to the walls and top. When the char has been sufficiently collected, and the vacuum pump is no longer pulling any aqueous product from the char, the first extraction stage is complete. The vacuum flask containing the aqueous product is swapped for a new, larger flask and a larger rubber valve is added under the vacuum filter. The char product is next washed with 1 liter of acetone to remove the organic phase from the char product. Some of this acetone is used to wash the reactor bottom and stir rod.



*Figure 8: The aqueous and solid products are extracted using a vacuum filter*

Now, the acetone and oil solution is brought to a rotovap to separate the two species. The rotovap is prepared with cyclohexanol and frozen carbon dioxide in the cooling tower. The reactor bath is set to 50°C and acetone is selected in the solvent library. Once the tower has the cooling species and the heating bath has reached the set temperature, pour the acetone and oil solution into a round bottom flask until it fills to 3/4 volume. If needed repeat this until the acetone oil solution is exhausted.

Finally, all products are collected in labeled vials. The char product is scooped into a vial and massed. Once the mass is recorded, place the vial in the oven to be weighed once it has dried. The aqueous and oil products are collected in multiple vials to be sent for elemental analysis.

### 3.3 Proximate Elemental Analysis

To perform proximate elemental analysis, samples of the oil phase and aqueous phase were sent to Midwest Micro Lab (Indianapolis, IN, USA) where analyzed using CHONS analyzers.

### 3.4 KF Titration

To determine the water content of biocrudes a Mettler Toledo Karl Fisher titration instrument was used. Biocrude is dissolved in methanol (99.95% Fisher) to achieve approximately <1wt% water solution (0.02g biocrude per 2 g methanol). The dilute solution is injected into the titrator (0.5 mL) per run and each sample is run in triplicate. Water in methanol solutions are used as standards to assess the instrument calibration. Biocrude water content is back calculated from the dilution factor and the KF titrator wt% value.

### 3.5 Gas Chromatography Mass Spectrometry

To characterize the composition of the light fraction of the biocrude samples were diluted in acetone (Fisher 99.95%) to approximately 1wt%. A Shimadzu GC 2021 Plus/MS was used with a Shimadzu SH-RXI-5Sil MS column. The GC-MS method uses a temperature starting at 30°C, 4 minute hold, followed by 3°C/min ramp to 290°C and hold for 5 minutes. A 16 mL/min column flow rate was used with a 20:1 split ratio. A 1-3 microliter sample injection was typically used.

### 3.6 Moisture Content

To calculate moisture content of food waste the weight after placing it in a drying oven at 65°C for hours was subtracted from the weight before drying.

### 3.7 Higher Heating Values

To determine the energy content higher heating values (HHV) were measured. HHV of the biocrude product was measured using a semi-micro calorimeter (25720, Parr, Moline, IL, USA). The instrument was given a measured amount of oil which is combusted within the apparatus. Using the known reactor temp and the rise in temperature post combustion, the energy content of the combusted sample can be determined. The instrument was calibrated using benzoic acid. There are two instruments, one located at Worcester Polytechnic Institute and a second is located at Mainstream Engineering, in Florida and operated by Mainstream staff.

### 3.8 Total Organic Carbon Testing

Total organic carbon (TOC) testing is a method used to determine the concentration of organic carbon in a given sample. To test for TOC, the sample is run through a device that converts all carbon into carbon dioxide gas by either catalytic combustion or wet chemical oxidation, the mechanisms of which are shown below.

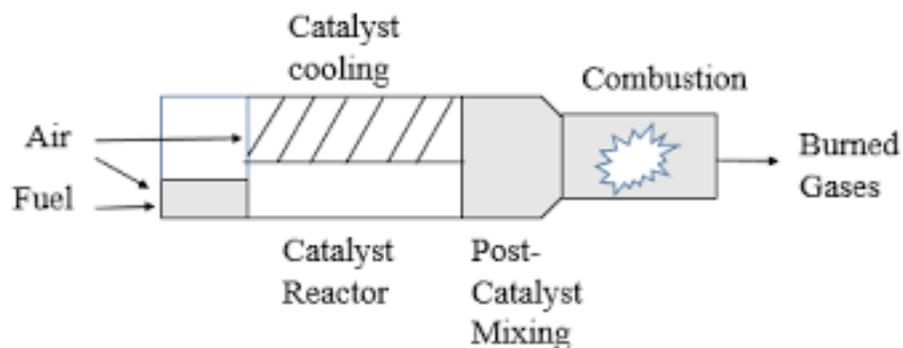


Figure 9: Mechanism of catalytic combustion. Air enters the chamber as well as fuel, where contents are mixed with catalyst supply. The mixture then enters the combustion chamber, and product gasses are released (Catalytic Combustion).

# Wet Air Oxidation - Process Flow Diagram

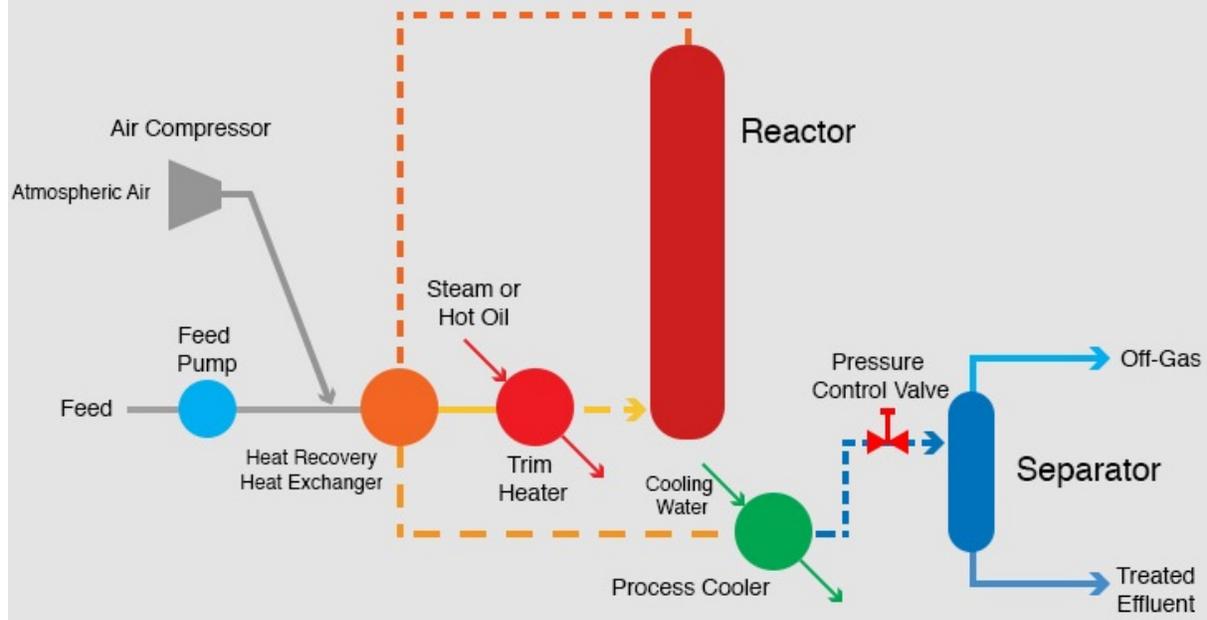


Figure 10: Wet air oxidation sample. Prepared feed is introduced to a compressed air environment. Sample is heated to reactor temperature and enters the reactor. Reaction takes place and products are cooled and collected (Wet Air Oxidation).

This testing was used in order to determine the amount of organic carbon present in the aqueous phase of the product of the hydrothermal liquefaction process.

## Chapter 4. Results and Discussion

In this section, the data obtained through experimentation will be analyzed and plotted. Effects of changes in reaction time and solids loading will be observed in terms of the total organic carbon, moisture content, carbon yield, oil quality, moisture content, and visual appearance.

## 4.1 Total Organic Carbon Testing

Samples taken from experimentation were sent to a lab where samples went through one of these processes to be analyzed for TOC. Data on concentration and the total mass of carbon in each sample were taken. Using these results, the percentage of carbon supplied from the feed that was present in the aqueous product was tracked and plotted. Results from each experiment were plotted as a function of solids loading and reaction time.

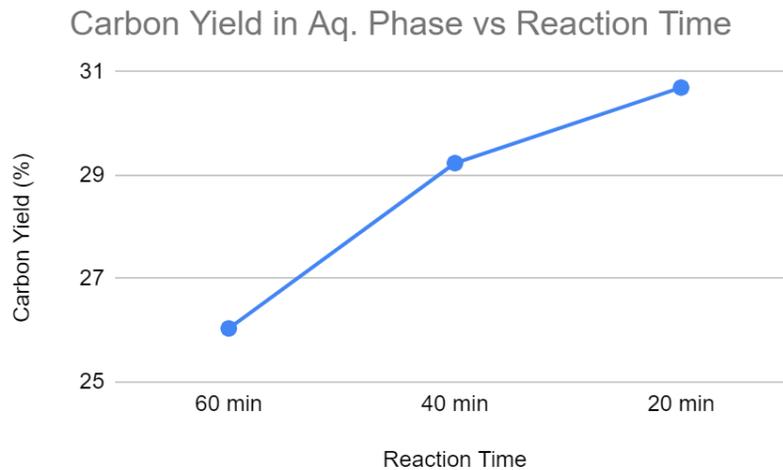


Figure 11: Carbon yield in the aqueous phase as a function of reaction time. As reaction time decreases, more carbon from the feed remains in the aqueous phase.

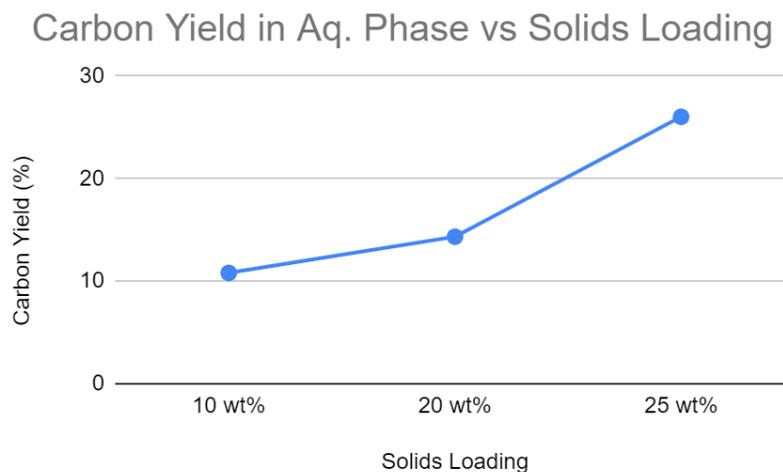


Figure 12: Carbon yield in the aqueous phase as a function of solids loading. As solids loading increases, proportionally more carbon from the feed remains in the aqueous phase.

These trends show the effects that the reaction time and solids loading have on the carbon yield in the aqueous phase. The longer the reaction is allowed to proceed, the less carbon from the feed that remains in the aqueous phase. It is hypothesized that during the reaction mechanism, carbon from the feed first converts to an aqueous phase, then to oil, and then to char. This would explain why more carbon remains in the aqueous phase with less time to react. Furthermore, the higher solids loading used for the experiment, the more carbon remained in the aqueous phase. This can also be explained by the previous hypothesis. This hypothesis implies that the more feed available for reaction, the longer it will take for the entire sample to be converted. Since reaction time remains the same when testing for solids loading, the highest weight percent will take the longest to fully react, and therefore contain a higher carbon yield in the aqueous phase.

#### 4.2 Moisture Content

The purpose of the Karl-Fischer (KF) titration was to determine the moisture content in the oil phase. This determination was necessary to calculate the carbon yield accurately. If omitted, the mass of the oil and water phase would cause the carbon yield in the oil to appear higher due to more mass. A sample calculation based on the value produced by the titrator and the diluted sample with methanol can be found in the appendix. The following tables display the results from the KF Titration:

*Table 2: Moisture content at various solids loading (on the left) and reaction times (on the right)*

Trial Condition	Moisture Content
10%	4.5%
15%	6.1%
20%	6.5%
25%	5.69%

Trial Condition	Moisture Content
20 min	5.595%
40 min	5.1%
60 min	5.69%

As shown in the tables above, there are no clear trends for moisture content in the oil samples. From the table on the left, it appears that loading the reactor at 10% will produce oil with the least amount of moisture. As for the table on the right, it appears that the reaction time does not have a significant effect on the moisture content. As oil is hydrophobic, the moisture content is expected to be relatively low as shown in the data above. One factor that can affect the moisture content is not completely rotovaping the acetone out, which can explain the discrepancy between the trial conditions.

### 4.3 Carbon Analysis

This section is concerned with how the carbon from the hospital food waste source was distributed among the products by the end of the reaction. By analyzing this data, the amount of carbon in the desired phase was able to be tracked from one experiment to the next. Then each experiment can be compared, and the ideal conditions can be identified.

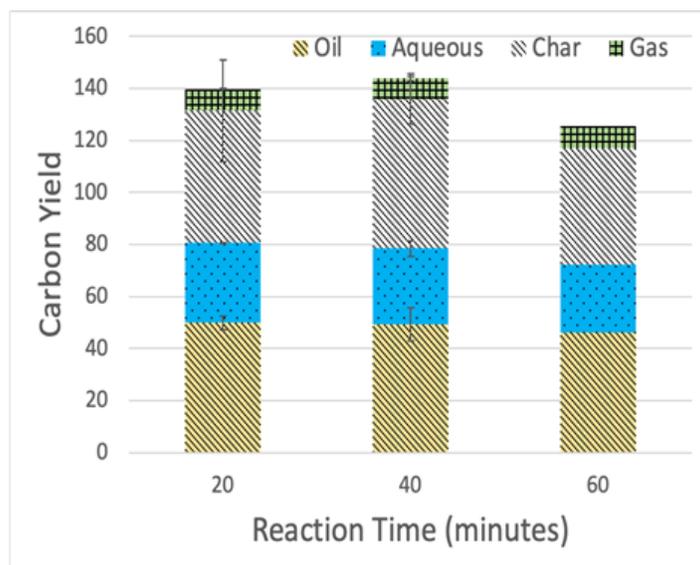


Figure 13: Carbon yield graphs for 25 percent solids by weight at various run times

As shown in the graph, the carbon distribution in the oil phase is low, meaning that the time of the reaction does not have much effect on the quantity or quality of the oil. This is not expected; as the reaction occurs, the carbon is expected to go into the oil phase or from the oil phase into the char phase if it runs for too long. However, as shown in the HHV section, the energy contained in the oil samples at the different reaction times is relatively consistent. In other

words, the reaction time does not appear to have any effect on the quantity or quality of the oil produced. The graph also shows that the carbon in the aqueous phase is relatively consistent throughout the different reaction times. This is not expected as explained in the image matrix section because the aqueous phase for the lower reaction times is darker signaling more carbon is present. Therefore, varying the reaction times will change the amount of carbon pushed into the char phase or left in the aqueous phase.

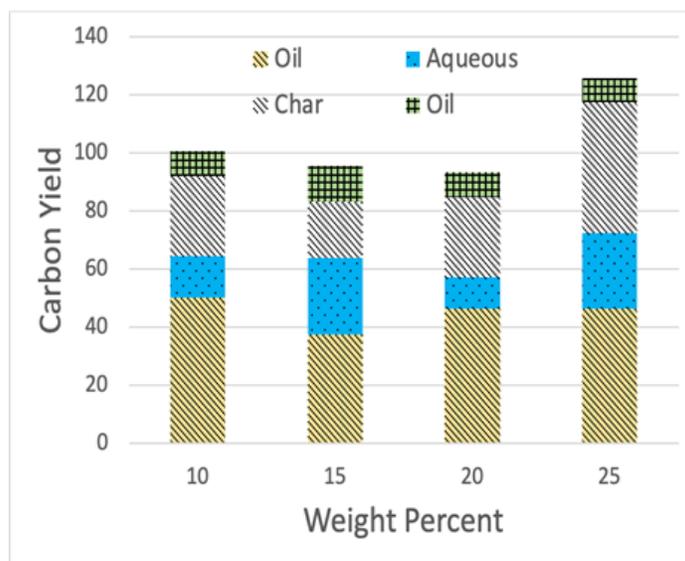


Figure 14: Carbon yield graphs for varying weight percentages at a run time of 60 minutes

The key takeaway from this graph is that the amount of uncertainty increases as the weight percent increases. When producing a carbon yield plot, it is best if the produced yield is at or around 100. For the lowest weight percent, this was the case; however, as the weight percent was increased, the yield moved further away. In terms of where the carbon would be at the end of the reaction, there is no clear trend. In other words, it is more beneficial to run this reaction at lower weight percentages because the carbon distribution is relatively similar at the other weight percentages, but the certainty in the quantity and quality should be more accurate at the lower solids loading.

#### 4.4 Oil Quality

To test the quality of the biocrude product multiple analytical instruments examined samples from valid trials. The team used a semimicro bomb calorimeter to test the heat of

combustion for different oil samples. To analyze the composition of the oil product, the team used gas chromatography to isolate the present molecules.

The group determined the higher heating values (HHV) of the bio-oils produced after the HTL process, using a bomb calorimeter. Table 3 shows the HHV of the bio-oils produced at the different HTL conditions.

*Table 3: HHV values at varying weight percentages and reaction times*

Weight %	Reaction time (minutes)	HHV (MJ/kg)
25	60	32.77
25	40	33.24
25	20	33.6
25	0	34.08
10	60	34.91
15	60	32.8
20	60	35.15
25	60	32.77

The heating value is the amount of energy obtained when fuel or some other substance of a specific unit quantity is combusted, hence a larger higher heating value represents a higher energy content fuel. The HFW produced HHV's as high as 35.15 MJ/kg which is high compared to the bio-oils obtained from other biomass (Ravikumar et al, 2017). This HHV value of 35.15 MJ/kg is approximately 80% the HHV of gasoline fuel oil (42 MJ/kg) (Yu et al, 2007). The table also displays displays a slight trend as the reaction time is increased.

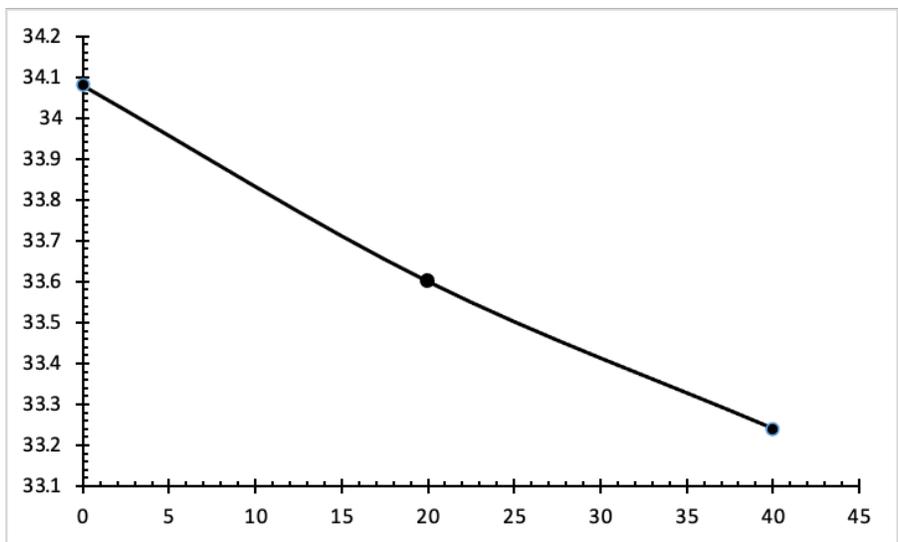


Figure 15: Oil quality study of sample energy content vs sample reaction time

The oil analysis done by gas chromatography was inconclusive. For much of the year, the team experienced issues with the gas chromatograph (GC). The team was only able to test a few of the oil samples in the chromatography system before having to focus attention elsewhere. Below is a graph showing the chromatograph data for both a sample produced by the team and a reference sample for hospital food waste.

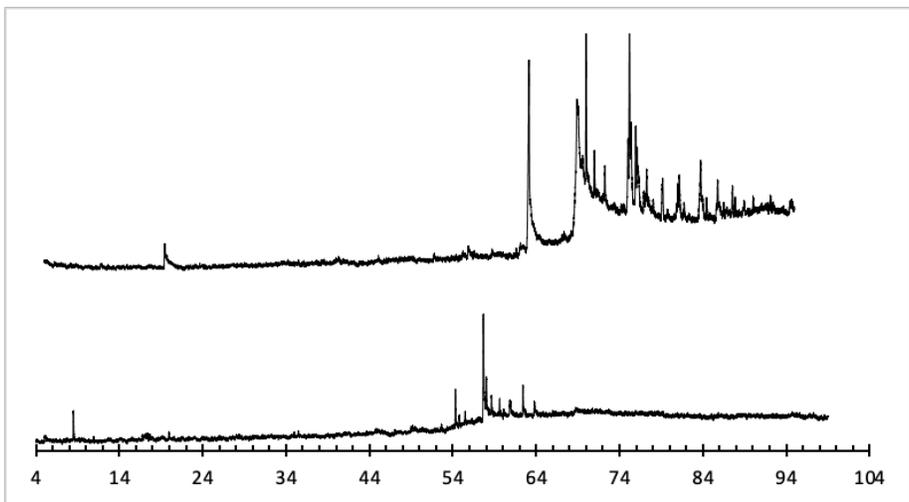


Figure 16: Gas chromatography sample comparison

The referenced hospital food waste sample is shown on top of the sample tested by the team. Immediately, one notices the flat trend of the team's oil sample. In this case, the lack of information is very indicative of some unknown issue. The observed peaks in the sample correspond to impurities that prove difficult to remove from the instruments within the GC,

blending the concentration from run to run. When troubleshooting, the method instructions for the gas chromatography instrument were found to be very similar. The main difference being our team had a 1°C per minute higher heating rate. However, had this been the real factor, our peaks would appear further left on the x axis instead of not appearing at all.

With the high lipid concentration in the food waste feed, the team expected comparable levels of fatty acid compounds in the oil. The lack of data in the team's sample beyond  $x = 64$  on Figure 16 above is indicative of a larger problem. The team believes the issue rests in the long refrigeration period between oil production and gas chromatography. The difference in curvature of the above data trends suggests the long refrigeration period allows for the potential recombination of compounds into larger, undetected compounds. Future research should be done to test the oil quality shortly after it has been produced against the same sample after long storage periods. The second theory is that the prepared samples were too low in concentration for major detection of individual species. The group diluted the samples that were run to 1% and 2% weight percentage of oil in methanol solvent. A higher oil to methanol ratio increases the individual species' concentrations, thus for future research the team also recommends increasing the oil to methanol ratio to 5%.

## 4.5 Image Matrix

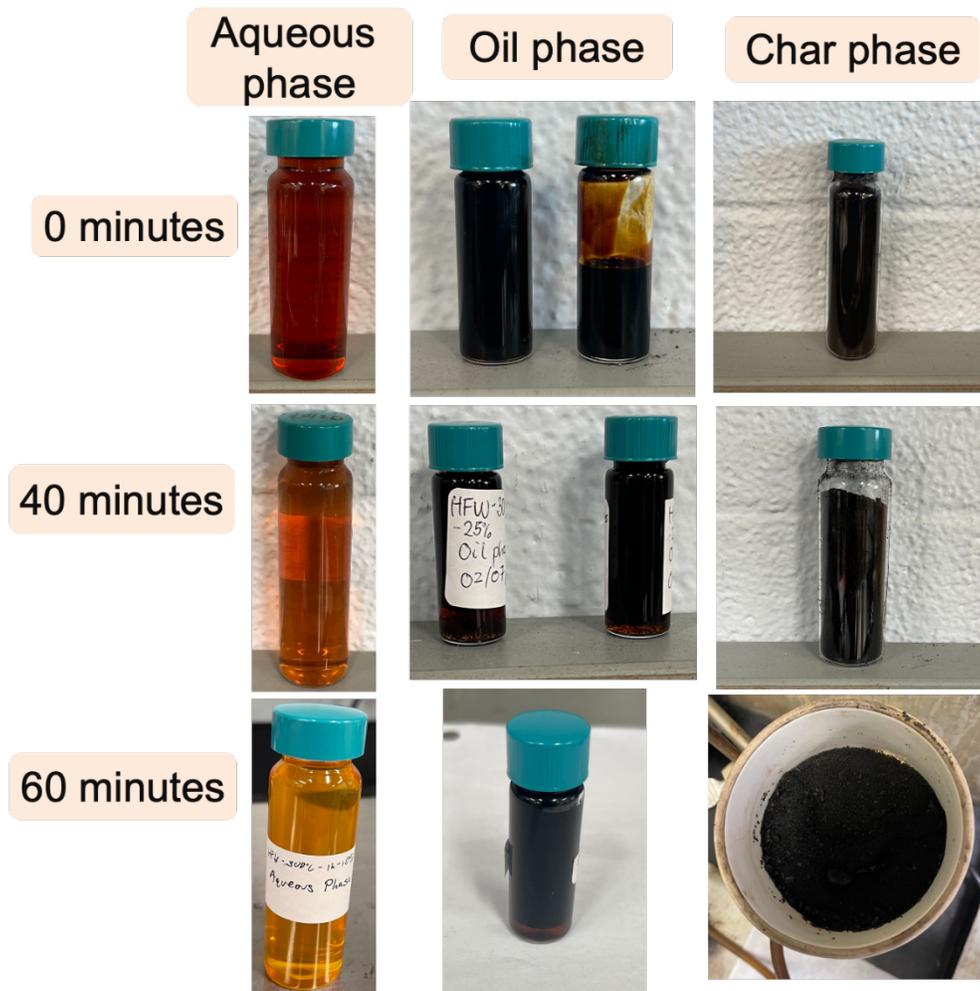


Figure 17: Image Matrix at a solids loading of 25 percent

The group created an image matrix to visualize the different phases of the bio-oil and check if there is a difference in appearance as the reaction conditions were changed. The group did not notice any visual change in phases as the solids loading weight percentage changed but there was a noticeable difference as the reaction time changed. The image matrix shown in Figure 17 visualizes the three phases of the bio-crude after undergoing the hydrothermal liquefaction reaction. The matrix shows that as the reaction time for the experiment is increased, the oil and solid phase do not change visually, however, the aqueous phase becomes more transparent. This can be explained by the fact that as the reaction went on longer more carbon was in the oil phase, leaving the aqueous phase clearer.

## Chapter 5. Conclusion

The issues caused by the consumption of fossil fuels will continue to exponentially grow as society continues to be reliant on this resource. Hydrothermal liquefaction is being widely studied as a method of resolving this issue. Furthermore, using food waste as a feed source for this process presents solutions to several other problems as well. However, in order to create usable product, the amount of carbon distributed to the oil phase of the product must be optimized. Finding the right conditions to optimize this product is key to making the process industrial and the product marketable.

Through experimentation of the hydrothermal liquefaction process using hospital food waste as a feed source and analyzation of the products, several key trends have been identified. First, based on the carbon yield data, changing the reaction time of the process does not greatly affect the carbon distribution between the products. There is a slight increase in the amount of carbon in the aqueous phase as reaction time decreases, but not an appreciable amount. However, the uncertainty of the carbon distribution also increases with increased reaction time, indicating that more experimentation is needed to concretely determine the effect of reaction time on the carbon distribution. Qualitative results such as the image matrix suggest that a lower reaction time results in a higher carbon yield in the aqueous phase.

On the other hand, there is a significant change in the carbon distribution when the solids loading is changed. Based on the carbon yield data, the lowest solids loading of 10 wt% resulted in the highest carbon yield in the oil phase. Furthermore, a lower weight percent also results in the lowest ratio of char to oil, optimizing oil production proportional to the amount of feed that is being reacted. Based on this experimentation, the ideal operating conditions to optimize both the carbon yield in the oil phase and the ratio of char to oil are a solids loading of 10 wt% and a reaction time of 60 mins. However, more experimentation is needed to concretely determine the viability of these conditions.

## Chapter 6. Recommendations for Future Work

The main goal of this project was to vary the trial conditions for producing bio-crude oil with hydrothermal liquefaction from hospital food waste to determine the optimal conditions for

carbon yield in the oil phase and oil quality. Through experimentation, the solids loading weight percentage and reaction time were varied. In future iterations of this project, it could be beneficial to vary the reaction temperature and ramp-up time as they may affect the carbon content in the oil and the amount of char produced. Further, it may be beneficial to perform an energy analysis on the energy lost during heating and produced by the oil at varying weight percentages to determine energy efficiency. Collectively, optimizing these conditions would help with transformation into a continuous process.

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## Appendices

### Appendix A. Data for carbon yield graph

Solids loading%	Date	Aqueous	Oil	Char	Gas
20	11/15	10.78647966	46.33086207	27.70074052	8.620689655
10	11/30	14.31636207	50.04474425	27.7545931	8.359456635
25	12/7	26.03102498	46.26764769	44.94662621	8.359456635
25 (20 min)	1/24	30.67671418	52.61532819	70.22068966	8.568443051
25 (20 min)	1/31	30.71730452	47.30674828	31.27613793	8.150470219
25 (40 min)	2/2	32.22376766	42.95806667	47.63420276	6.896551724
25 (40 min)	2/7	26.24297318	55.62688943	67.16524138	8.777429467

### Appendix B. KF Titration data and sample calculation

Run name	Oil Weight	Methanol Weight (g)	Ratio	Syringe 1 Full (g)	Syringe 1 Empty	Solution 1 Weight	Output	Moisture Content of Sample (%)	Syringe 2 Full	Syringe 2 Empty	Solution 2 Weight	Output	Moisture Content	Average
25% - 20 min - 1/24	0.0395	2.0049	51.75696203	3.0109	2.6131	0.3978	0.12%	6.37%		2.5906	0.3733	0.14%	7.02%	6.69%
10% - 1 hr - 11/29	0.0419	2.0026	48.7947494	3.0616	2.6419	0.4197	0.08%	3.83%	3.0893	2.6369	0.4524	0.08%	3.95%	3.89%
25% - 20min - 1/26	0.041	2.0095	50.01219512	3.0956	2.6309	0.4647	0.12%	5.80%	3.058	2.6245	0.4335	0.12%	6.00%	5.90%
20% oil 1	0.043	2.0148	47.85581395	3.0376	2.648	0.3896	0.11%	5.23%	3.0848	2.637	0.4478	0.11%	5.17%	5.20%
10% Oil 2 - 11/30	0.0413	2.0045	49.53510896	3.0239	2.6293	0.3946	0.09%	4.46%	3.0597	2.6249	0.4348	0.09%	4.54%	4.50%
25% - 60 min	0.0386	1.926	50.89637306	2.9989	2.6317	0.3672	0.11%	5.66%	3.0355	2.6214	0.4141	0.11%	5.71%	5.69%

- 12/7														
20% - 60 min														
- 11/1 5	0.04 14	2.090 9	51.50483 092	3.038 3	2.610 1	0.428 2	0.13 %	6.45%	3.046 8	2.605 5	0.441 3	0.13 %	6.55%	6.50 %
25% - 60 min														
- 12/6	0.04 65	2.258 2	49.56344 086	3.000 2	2.608 3	0.391 9	0.11 %	5.23%	3.034 8	2.604	0.430 8	0.12 %	5.99%	5.61 %
20% - 60 min														
- oil	0.04 03	2.000 1	50.63027 295	2.984 8	2.613 4	0.371 4	0.18 %	9.01%	3.045 8	2.608 5	0.437 3	0.19 %	9.62%	9.32 %

### Sample calculation

$$\text{Moisture Content} = \text{Output \%} * 1/(\text{oil weight}/(\text{oil weight} + \text{methanol weight}))$$

If oil weight =0.0413g, methanol weight = 2.0045g and output % = 0.09

$$\text{Moisture Content} = 0.009 * \frac{1}{\frac{0.0413}{0.0413 + 2.0045}} = 4.46\%$$

### Appendix C Step wise procedure and example run sheet

#### HFW HTL Reaction Procedure

1. Mass the top and bottom of the reactor
2. Combine 20% hospital food waste and 80% di-ionized water
  - a. This allows us to assume a 100g basis for calculations
3. Mass reactor with reactants inside
4. Clamp reactor with star pattern
5. Set-up reactor in the hood
  - a. Attach the stirrer, thermocouples, cooling water lines, and gas line
  - b. Raise the heater
6. Wash reactor 3 times with Nitrogen at 1000 psi [leave pressurized for 5 mins of the first
  - a. washing to ensure that pressure tightness
  - b. Set tank regulator to 1000psi
  - c. Open line to reactor making sure that the valve on the reactor is closed
  - d. Open valve to reactor and close it once pressurized
  - e. Loosen nitrogen line to bleed pressure down to 100-200 psi
  - f. Repeat 3 times
7. Prepare the reactor for HTL
  - a. Remove the nitrogen line
  - b. Turn on the heater and set to 320°C
  - c. Reduce heater to 300C when temperature reaches 275C

- d. Turn on stirrer to the 3rd tick mark
- e. Plug in the cooling pump
8. After 1 hour, quench with ice bath until reactor temperature reaches 30°C and record pressure
  - a. Wait 15 minutes after quenching and record temperature and pressure
9. Set up rotavap
  - a. Set the bath temperature to 50°C
  - b. Set the vacuum to 370 mPa
  - c. Use the library function to select acetone
  - d. Fill cooling system with ethanol and dry ice
10. Disassemble reactor
  - a. Turn off stirrer and remove it
  - b. Remove the thermocouples
  - c. Unplug the cooling water pump and remove cooling lines
  - d. Slowly bleed reactor
    - i. Do not remove it from fume hood if it is still pressurized
  - e. Remove clamp
11. Mass the entire reactor with products
12. Remove aqueous layer
  - a. Lay down paper towels in the hood
  - b. Place the reactor on paper towels
  - c. Set up vacuum filter
  - d. Scrape everything into the reactor and pour into filter
  - e. Scrape remaining residue into the filter
13. Mass aqueous layer in vacuum flask; then put into vials
14. Mass solid and oil mixture in funnel
15. Remove oil layer with 1000 mL of acetone
  - a. Set up filter with larger attachment into 1000 mL collection flask
  - b. Use approximately 1/3 of acetone to wash reactor
  - c. Wash and scrape reactor with acetone including the reactor top, bottom, stirrer, and thermal line
  - d. Use remaining acetone to wash oils from solid layer through vacuum filter
16. Mass washed solid in funnel; then put into vials
17. Rotavap acetone out acetone/oil mixture
  - a. Fill round bottom flask 3/4th of the way with mixture and attach it to the rotavap
  - b. Turn on vacuum
  - c. Set rotator to 5
  - d. Lower the round bottom flask into the bath
  - e. Repeat until all acetone is removed from oil
18. Mass oil in the round bottom flask; then put into vials

Appendix D. Example run sheet

H<sub>2</sub>O: 74.998 - 0.132g

Date 02/14/22

Run Name HFW-300°C-0 mins-25%

Food	24.964g - 0.019g
Catalyst	—
Conditions	300°C - 0 mins - 25%

Start	10:57
Reach	11:34
End	11:34
Quench	11:39

Before Reaction

Bottom	2225.2g
Top	3015.9g
Total	5340.5g

Initial P	950 psi
Initial T	29°C
Start P	2450
Start T	300

After Reaction

Bottom	2225.2g
Top	3016.2g
Total	5337.6g

Hot P	2450
Hot T	300
Cool P	1250
Cool T	30

AFTER 15 MINS  
P-1100  
T-17

Processing Measurements

Empty Aqueous Flask	162.746g
Full aqueous flask	234.267g

Empty acetone/oil	524.6g
Full acetone/oil	1251.7g

Funnel + stopper + FP	337.8g
& oils and solids	
& solids only	349.9g

Empty RB flask	408.2g
Full RB flask	417.6g

348.3g 343.7g

Oil Yield	9.4
Aqueous Yield	71.521
Solid Yield	12.1
Gas Yield	2.9

Total: 95.921g

Notes:	
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