

# Design of a Flow-Through Pretreatment Biomass Reactor and Examination of

# **System Fluid Mechanics**

A Major Qualifying Project Report

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### Abstract

Biofuels such as ethanol represent an increasingly important part of the global energy portfolio. Ethanol is produced by pretreating biomass to breakdown recalcitrant structures and fermenting the remaining sugars into fuel. Flow-through (FT) pretreatment is currently being explored as a more efficient alternative to batch processes. However, very little research has been done on the behavior of biomass in FT systems. Biomass swelling in these systems, and the associated change in pressure drop over the length of the reactor, need to be characterized in order to produce accurate models for industrial-scale processes. In this report, we analyze the pressure drop characteristics of a small-scale FT pretreatment reactor using pine sawdust and corn stover biomass. While further work is needed to fully understand the system that we created, the data that has been generated should prove useful for future modelling of FT pretreatment systems.

# Acknowledgements

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### **Chapter 1: Introduction**

Most aspects of our societies and everyday lives involve energy. Since the 1990s, the world's energy consumption has increased greatly and demand is expected to grow by 1.3% each year to 2040 [1]. This is due to both the industrial development of more nations, especially in Africa, as well as the rise of China as a global manufacturing leader. This increase in energy production and energy demand has led to the need for more fuel. The main energy source for the world is fossil fuels, which are both limited and negatively affecting the global climate. As we continue to see the devastating effects of climate change emerge throughout the world, alternative energy sources are being explored to reduce our dependence on these fuels.

One alternate fuel source being explored is biomass. Renewable energy accounted for a total of 11% of primary energy use in the U.S. in 2017. 45% of the renewable energy was from biofuels derived from biomass [1]. Ethanol, produced by corn, is one of the most widely used biofuels that is currently commercially available. Ethanol has been blended into gasoline for many years in the United States.

When using biomass to produce biofuels, there are structural components of the biomass that hinder the ability to produce fermentable sugars. In order to overcome these structural factors, it is necessary to pretreat the biomass [2]. There are acid/base pretreatment options as well as pretreatment with water. Using water in pretreatment removes the need to neutralize and condition chemicals, because acid is not added to the system [2]. There are also flow through (FT) and non-FT pretreatment methods. Flow through methods involve a stream of acid/water passing over a stationary bed of lignocellulosic biomass. Each method has different effects on the structure and composition of the biomass. For example, FT "typically achieves higher solids reactivity, higher xylan removal, less sugar degradation and substantially higher removal of lignin compared to pretreatment in non FT configurations at the same temperature and residence time" [3]. However, FT uses a greater amount of water which dilutes sugar stream and increases energy consumption.

In order to develop and understand efficient FT biofuel reactors, the fluid mechanics of the system must be taken into account along with the chemical and physical factors. One factor that is present within a packed flow-through reactor is pressure drop. As the water flows over a packed bed of biomass, the pressure decreases from what was present at the inlet of the reactor. Pressure drop depends on many different characteristics of the feedstock, as well as the reactor set-up. One contributor to pressure drop that is important to develop an understanding of is biomass swelling. Being able to model the effect that biomass swelling has on a flow-through reactor system can help to understand and optimize the production of biofuels from biomass.

### **Chapter 2: Background**

### 2.1 Ethanol as Biofuel

Ethanol is the most widely used biofuel in the United States. Biofuels such as ethanol offer several advantages (and some disadvantages) relative to conventional fuels like gasoline. From an economic standpoint, ethanol is a cheaper alternative to gasoline. Common ethanol blends like E85, which contains between 51% and 83% ethanol (depending on geography and season), are almost always cheaper than pure gasoline. Currently, E85 is around \$0.40 cheaper than gasoline on a per-gallon basis [4]. Unfortunately, these cost advantages are made up for by gasoline's higher energy density. In fact, based on Gasoline Gallon Equivalent (GGE), E85 is actually around \$0.10 more expensive per gallon than gasoline. Ethanol's lower energy density also means that cars using blended fuels will experience somewhat worse gas mileage. E85 that contains 83% ethanol has 27% less energy per unit volume compared to gasoline. If combustion engines were better optimized for ethanol use, perhaps this energy difference could be mitigated. Unfortunately, almost every car on American streets will see their mileage take a hit when using ethanol blends [5]. However, these tradeoffs are offset by the economic benefits associated with domestically producing biofuels.

Economic factors such as job creation and energy independence are generally improved by bioethanol production. The bioethanol industry currently uses corn as its primary feedstock. Many farmers are able to make a living due to the demand created by ethanol producers. In 2018 ethanol production directly accounted for over 70,000 jobs nationwide [5]. A large portion of these jobs are in rural areas where employment opportunities are often scarce. According to the Department of Energy, ethanol production alone accounted for \$46 billion in GDP and \$25 billion in household

income. Producing biofuels also helps to improve American energy independence. Almost all biofuel consumed in the U.S. is also produced in the U.S. Petroleum, on the other hand, is highly dependent on imports from around the world. 11% of the oil used in America is imported, and this number would likely be higher without the ethanol industry. Increased production of ethanol and other biofuels could lessen American dependence on foreign energy.

The environmental impact of ethanol and other biofuels has received a lot of publicity in the last 20 years, and for good reason. Because the energy in ethanol is derived from plant life, the emissions in carbon dioxide created by burning it as fuel are offset by the carbon dioxide captured to grow the feedstock. Conversely, no carbon is captured in the production of gasoline. As a result, ethanol produced from corn cuts emissions by around 34% when taking into account the full life cycle of the fuel. Ethanol derived from cellulosic feedstocks is even more promising, with up to 108% lower emissions than gasoline [5].

#### 2.2 Lignocellulosic biomass

Utilizing biomass as a source of energy through the production of ethanol and other biofuels has been an area of interest since the mid-20th century. Specifically, the process of biofuel production generally revolves around the fermentation of sugars from lignocellulosic biomass into energy dense compounds. Lignocellulosic biomass refers to plant biomass that is composed of the aromatic polymer, lignin, and the carbohydrate polymers, cellulose and hemicellulose, as shown in Figure 1. However, the proportions of lignin, cellulose, and hemicellulose vary between different sources of biomass. For example, corn stover contains less lignin but more hemicellulose than coir, a type of cellulosic textile fiber [6] [7].

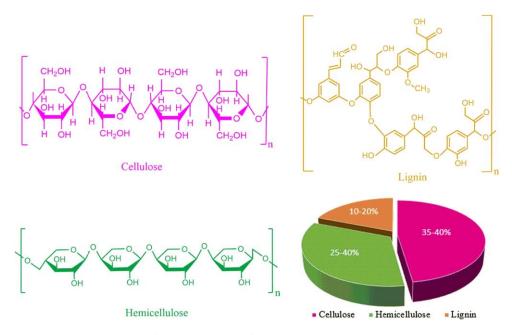


Figure 1: Composition of Lignocellulosic Biomass [7]

Biofuels from lignocellulosic biomass are an attractive alternative to fossil fuels due to the wide availability of lignocellulosic biomass. Waste from the paper and agricultural industry serve as sources of lignocellulosic biomass in the form of waste paper, corn stover, or residue from sunflower oil pressing [6][8].

### 2.2.1 Lignin

Lignin is composed of complex heterocyclic phenols and makes up 15-30% of lignocellulosic biomass. Within lignocellulosic biomass, crosslinking occurs between the polysaccharides (cellulose and hemicellulose) and lignin through ether and ester linkages. These linkages are the main contributors to recalcitrance within biomass, and therefore must be broken in order to access fermentable, simple sugars. Within lignin, there are also no specific subunit

patterns, making it difficult to characterize and decompose further. The challenges presented by the lignocellulosic matrix within biomass is a research focus for many within the biofuels field [9] [10] [11] [12].

### 2.2.2 Cellulose

Cellulose is the most abundant compound within lignocellulosic biomass, accounting for 40-60% in weight. Cellulose is a polymer consisting of thousands of  $\beta$  linked D-glucose units that form a linear chain. These chains are arranged to form microfibrils, which are further packed together to form cellulose microfibrils. The cellulose microfibrils are embedded into the lignocellulosic matrix, contributing to its rigidity [9] [10] [11] [13].

### 2.2.3 Hemicellulose

Hemicellulose is a relatively amorphous polysaccharide consisting of multiple types of monosaccharide. These monomers include 5-carbon sugars, 6-carbon sugars, and uronic acids. These sugars can form long, branched carbon chains, although individual hemicellulose fibers are generally only 100-200 monomers long. Hemicellulose does not contain significant cross-linkage, and the constituent sugar monomers are relatively easily accessible for fermentation and breakdown [9] [10] [11].

### 2.3 Pretreatment

Before processing the lignocellulosic biomass for biofuel production, it must be pretreated in order to break down the rigid lignin structures that naturally inhibit the conversion of cellulose and hemicellulose into usable alcohols. There are many different options for pretreatment through physical, chemical, physicochemical, and biological means. The recalcitrant nature of the lignin matrix in lignocellulosic biomass is widely recognized as the primary barrier in preventing more widespread biofuel production through such means. Improving pretreatment systems to more effectively break down lignin is therefore a priority for biofuel researchers [7] [14].

#### 2.3.1 Flow through pretreatment

Flow through (FT) pretreatment refers to a physicochemical method of pretreatment in which a solvent flows over a stationary bed of biomass in order to prepare it for processing. It is an attractive alternative to batch pretreatment options. Directly compared to the batch equivalent, FT pretreatment results in increased sugar yield and lignin removal [7]. Furthermore, current research states that "passing water through a stationary bed of lignocellulosic biomass in a flow-through configuration achieves very high yields of hemicellulose sugars, removes more than 75% of the lignin for potential valorization, and improves sugar release from the pretreated solids with lower enzyme loadings" [15].

FT pretreatment liquid uses either hot water or a dilute acid as a solvent. Dilute acid is the most common, conventional method of pretreatment. Some commonly used acids include sulfuric acid, oxalic acid, and maleic acid. These acids can either be utilized for a short period at a high temperature, or for a long period at a low temperature in order to achieve the appropriate level of pretreatment for the biomass. Occasionally, acid can be used to achieve fermentable sugars from the biomass, but the sugars must be excessively washed before they are fermented. A drawback to acid pretreatment is that it has the potential to produce inhibitors that must be removed before fermentation. Furthermore, in order for this method to be economically viable, the acid must be recovered after pretreatment, adding an additional step to the process [16].

Water is another potential solvent for FT pretreatment. Using water as a solvent allows for easier product separation from the solvent when compared to using a dilute acid. Furthermore, water does not cause any inhibitors to form. Water is also far cheaper and more widely than dilute acids. The common consensus is that the challenge of designing biomass pretreatment lies in that high sugar recovery must be achieved, but sugars tend to degrade when utilizing more severe pretreatment methods. Therefore, water is certainly a viable alternative to more extreme methods of pretreatment [17] [18].

### 2.4 Pressure Drop

However, before FT pretreatment systems can be implemented on a large scale, the fluid mechanics of the system must be understood. One characteristic that has been seldom studied in FT pretreatment systems for lignocellulosic biomass is pressure drop. Pressure drop refers to the difference in pressure at two points within a system that experiences fluid flow. Pressure drop is caused by frictional forces, which hinder the fluid's ability to flow. During FT pretreatment with water as a solvent, biomass particles absorb the water and swell over time. The swelling of biomass particles results in the flowing solvent experiencing more frictional forces, which in turn increases the pressure drop across the system.

Pressure drop in packed beds can be understood using the Ergun equation. The Ergun equation is as follows:

$$\frac{\Delta P}{L} = \frac{150\mu}{D_p^2} \frac{(1-\epsilon)^2}{\epsilon^3} v_s + \frac{1.75\rho}{D_p} \frac{(1-\epsilon)}{\epsilon^3}$$

 $D_p$  is the particle diameter (m),  $\mu$  is the viscosity of the liquid (Pa.s), v is the superficial velocity (m/s),  $\epsilon$  is the bed void fraction, $\rho$  is the density of the liquid (kg/m<sup>3</sup>), and  $\frac{\Delta P}{L}$  is the pressure

drop over the length of the packed bed (kPa/m). During operation of a FT pretreatment reactor, many of these parameters can be considered to be constant. The solvent, in this case water, flowing over the reactor will maintain its density and viscosity assuming the pressure and temperature of the system can be held constant. The velocity of the solvent also can be held constant by maintaining a consistent flow rate. However, the swelling of the biomass can create conditions that are difficult to estimate and model. As particles swell, their diameter increases over time. This increase in diameter in the presence of a constant fluid flow is difficult to accurately characterize as a function of time. If the biomass is confined to a fixed volume, the packing height can be considered to be constant since the particles cannot expand beyond the initial height. In this case, the swelling of particles can impact the void fraction. Since the swelling cannot expand outwards, the particles will instead expand into the spaces between the particles. This behavior results in a lower void coefficient for swollen biomass relative to dry biomass. Similarly to particle diameter, this parameter is difficult to accurately assess and model.

Setting up a biomass reactor system to determine the actual pressure drop over time could greatly improve efforts to model FT reactor systems. At the very least, the real pressure drop data could be used to approximate the rate of change for parameters like particle diameter and void fraction. By assigning different functions to these variables, inserting them into the Ergun equation and comparing the resulting model to the collected data, researchers could gain a general idea of the behavior of the biomass swelling within the FT system. In addition, biomass samples could be taken from the reactor at repeated time intervals and examined under a microscope to gain a more direct understanding of the effect of swelling on particle diameter. This method would make the time dependent particle size much easier to fit a function to. Since the particle size term would be produced without needing to be fit to the pressure drop data, the only variable remaining whose behavior is unknown over time would be the void fraction. Thus, models for the void fraction could be tested in the Ergun equation to fit the data without having to adjust any other variables.

### **Chapter 3: Materials and Methods**

### 3.1: Equipment

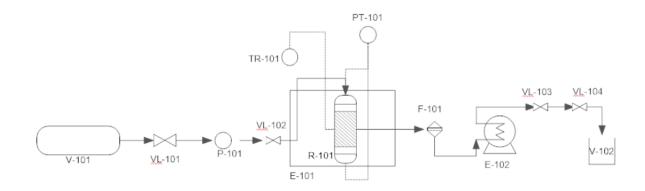


Figure 2: Process Flow Diagram of Flow-Through Packed Biomass Reactor

V-101- Vessel for deionized water VL-101- Pump Inlet Valve P-101- 1 L Syringe Pump VL102- Pump Outlet Valve E-101-Gas Chromatograph Oven R-101- Packed Bed Reactor PT-101Differential Pressure Transducer and Recorder TR-101- Temperature (Thermocouple) Recorder
F-101- 7 Micron Inline Filter E-102- Condenser VL-103- Backpressure Regulator (Diaphragm Valve) VL-104- Needle Valve V-102- Outlet Collection Beaker

The apparatus, shown in Figure 2, was built in order to study the pressure drop in a flowthrough pretreatment of a packed bed of biomass. This system was used to pump DI water over a biomass reactor at controllable flow rates and temperatures. A pressure transducer measured the pressure differential from the inlet of the reactor to the outlet. Thermocouples at each end of the reactor provided temperature data. The flow of water through the system was entirely controlled by the syringe pump controller, while the pressure of the system was adjusted using the backpressure regulator in conjunction with the pressure displays on the pump controller. The oven that we used to control the temperature of the system was a modified gas chromatograph, and could be programmed to increase temperature over time at a set rate. All of the temperature control for our system was handled by the oven. A small, 7 micron inline filter was also installed after the reactor to protect sensitive equipment such as the pressure regulator. A condenser was used as the cooling system for the outlet stream. This condenser used cold water to bring the temperature of the outlet stream down to roughly 17°C. The reactor itself, shown in Figure A2, was installed at roughly a 40 degree angle due to space limitations within the oven. It was also easily removable from the oven to enable the system to be efficiently cleaned and prepared between runs. The reactor was composed of a 6 inch long stainless steel tube with removable fittings at either end. The biomass was contained between a pair of 2 micron frits, held in place by a stainless steel spacing ring at the outlet end of the reactor. A set of stainless steel tubes arranged above the biomass and frits provided some degree of flow straightening. All of these components could be removed from the reactor for cleaning.

Both pressure and temperature data were recorded. Temperature was recorded by a thermocouple located at the exit stream of the reactor, while a differential pressure meter measured the pressure drop from the inlet to the outlet of the reactor. Data recording was handled by Dataq and Windaq software.

Two types of biomass were utilized in the reactor setup in order to compare their performance. In particular, we wanted to study how both the pressure drop and the sugar production changed between pine sawdust and corn stover. To make the comparison easier, most variables were held constant when switching between types of biomass. The packing height, water flow rate, pressure, and temperature of the system were consistent between runs. The major factor that was altered when changing from pine to corn stover was the mass of the biomass sample.

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Using the stock biomass available in the lab at consistent particle sizes, corn stover proved to be denser than pine. Due to the importance of bed height in modeling the behavior of the system, we decided that it was appropriate that packing height be held constant rather than mass.

Before running any tests with pine or corn stover, our team ran the reactor with 1 mm glass beads in place of the biomass. This test was done to establish a baseline for the FT system in terms of pressure drop and chemical output. Glass beads do not swell over time or produce sugars. As such, the system should have behaved close to ideally based on models such as the Ergun equation. The data gathered from this run, compared to the results of the Ergun equation, could therefore be used to evaluate the reliability or ideality of the pressure drop and chemistry data. In addition, the oven temperature, water flow rate, pressure, and packing height used in the glass bead test were identical to the conditions in our first set of tests with actual biomass. Comparing these results could isolate the impact of swelling on the pressure drop within the reactor, as the biomass would be expected to swell while the glass beads would not. We would expect the glass bead pressure drop to be relatively stable, so fluctuation could be due system limitations or unexpected phenomena. The notable difference between the runs would be the packing density, as the glass beads are a uniform size but the biomass particles are not. However, the data from the glass beads is still useful and valid for the purposes of comparing general trends.

Due to the limited capacity of the pump in our system, extended runs over the course of multiple hours were not possible. To account for this issue, in addition to using dry biomass, we planned to test the performance of the reactor with pre-soaked pine and corn stover. Small samples of biomass we sealed in a vial filled with water for 2-3 days before being inserted into the reactor. This soaking was done to simulate extended use of a single batch of biomass in a flow through system. Knowing the pressure drop behavior and the amount of sugars in the exit stream relative

to the dry biomass could be useful in modeling the system over extended runs. Testing with preswollen biomass could also aid in characterizing the impact of swelling on the pressure drop. Comparing the performance of the reactor with equivalent packing heights of swollen and dry biomass would allow our team to directly isolate the difference in pressure drop caused by swelling. Unfortunately, we were not able to run tests with this method due to time limitations, but this would be a step for future research.

To prepare the biomass for use in our reactor setup, we had to ensure that the particles of biomass were roughly even in size. While it would have been convenient to simply pack the stock biomass into our reactor, this would have resulted in significant complications later on. Keeping the particles at known and consistent sizes presented several advantages. For modeling equations, knowing the diameter of the particles was crucial. Without treating the biomass, we would have had to crudely estimate the average particle size. This would have introduced significant uncertainty into our results. Parameters such as void volume were also able to be estimated more accurately with consistent, known particle sizes. In addition to easier calculations, preparing the biomass for consistent size -particles also made the system easier and safer to operate. For the purposes of our experiments, the biomass had to stay within the reactor for the duration of the test. The sieve plate filters keeping the biomass contained within the reactor had pore sizes of 2 microns. If any of the biomass particles were smaller than these pores, they could end up exiting the reactor during testing. This event could potentially clog the system and create potentially dangerous levels of back pressure. Due to these factors, it was necessary to resize and sort the biomass particles before placing them in the reactor. To achieve this, our team used a coffee grinder to reduce the size of the biomass stock. Once the particles were sufficiently ground, they were passed through a

sieve to sift out any particles that did not meet our size requirements. Once the biomass particles reached the desired size, they were packed into the reactor bed for use in testing.

#### 3.2: Procedure

### 3.2.1: Pre-Startup

A series of steps were undertaken to prepare the reactor for installation in the test oven. First, the reactor, the frits, the spacing ring, and the spacing tubes were thoroughly cleaned and dried to remove any contamination from previous runs. The lower frit and the spacing ring were then returned to their place in the reactor. A sample of the desired biomass was weighed out and poured into the reactor, resting on the lower frit. The height of this sample was measured. The upper frit was then inserted on top of the biomass, followed by the spacing tubes. Once these steps were taken, the top and bottom caps were attached and tightened. The reactor could then be installed in the test oven.

### 3.2.2: Startup and Operation

Once the biomass was installed in the reactor, the reactor itself was attached to the inlet and outlet streams located inside the oven. To prepare the pump for the experiment, 800-1000 ml of water was drawn into the pump cylinder. After this was completed, the valve connecting the pump to the test oven was opened and the valve connecting the pump to its reservoir was closed. In addition, the filter between the reactor and the back-pressure regulator was cleaned or replaced between runs. Once the system was watertight, water was pumped through the reactor at a desired flow rate, set using the pump controller. For most of our runs, the flow rate was set to 20 ml/min. After a few minutes of pumping water into the system, a steady water flow would begin in the outlet stream. Once the outlet stream was devoid of air bubbles, the backpressure was set to a desired pressure. For the purpose of our experiments, this pressure was 220 psi. The pressure of the system was monitored on the display of the pump controller. Our team monitored the operating pressure of the system for the duration of the run and adjusted the back-pressure regulator if it deviated from the desired pressure by more than 10 psi. After the system was pressurized, it was heated to the desired temperature using the oven that the reactor is housed in. The program that we ran in our oven increased the temperature at a constant, set rate until reaching a desired temperature. This desired temperature was set to either 150 C or 180 C, although other values could have been tested. The temperature that the oven was set to did not necessarily correspond directly to the temperature inside the reactor, due to the presence of water entering the reactor at room temperature. Along with starting the oven, the cooling system for the outlet stream was also turned on. All of the pressure monitoring systems were also switched on before any data was recorded. As soon as the oven was started, the data collection software was turned on. Set a time? The pressure drop and temperature of the reactor were continuously measured throughout the experiment. Over the course of the experiment, the contents of the outlet stream were collected in a beaker. This beaker was periodically emptied to avoid overflow. In addition, samples were taken directly from the exit stream in 5 minute intervals. These samples were subsequently refrigerated for preservation.

#### 3.2.3: Shutdown

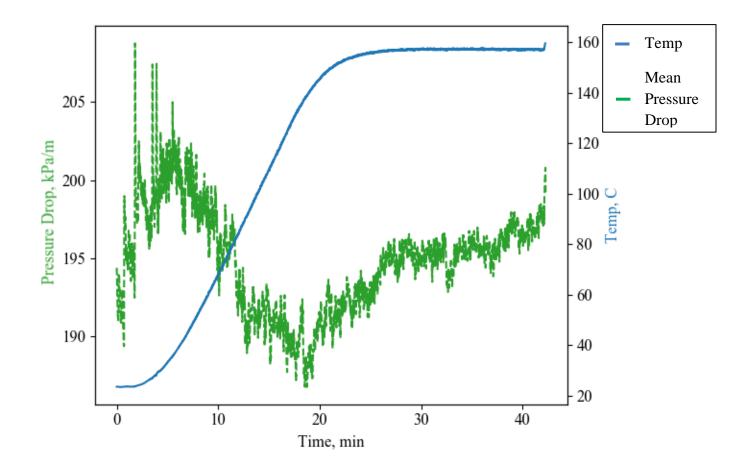
To initiate shutdown, the flow was shut off by either stopping the pump or allowing it to run until it was empty. At the same time, the oven was shut off. Due to the high temperatures experienced by the reactor, our team waited at least 10 minutes after shutdown to open the oven door. While the oven was cooling, the back pressure regulator was fully opened and any liquid left in the system was allowed to flow out the exit stream. Also, the cooling system and the data recorders for both the thermocouples and the pressure transducer were shut down during this time. Once the system had cooled to under 40 C, the oven door was opened and the reactor itself was removed. The caps were subsequently taken out of the reactor and the reactor cylinder was emptied, with a small sample of the biomass preserved for analysis. Before leaving the system, the pump and pump controller were turned off, and the valves on the pump were set to be open to the reservoir and closed to the rest of the system.

### **Chapter 4: Results**

#### **4.1 Pressure-Drop Results**

Before testing the system, expected certain behaviors in terms of pressure drop based on the assumptions we had made about biomass swelling. We assumed that the biomass would swell over time in the presence of a constant water flow, and that this swelling would cause the pressure drop to increase over time. We also assumed that this behavior would be absent when the system was tested with glass beads. Due to increasing temperature, we also knew that the viscosity of the water would decrease. We predicted that this change in viscosity would have the opposite effect on the pressure drop, lowering the pressure drop over time until the reactor reached its final reaction temperature. This behavior was expected to occur in both the glass bead tests and the biomass tests. Since we assumed that no other changes over time would occur, we thought that the impact of changing viscosity would be isolated in the glass bead test. Due to a myriad of factors that are currently only partially understood, the actual data often contradicted our expectations.

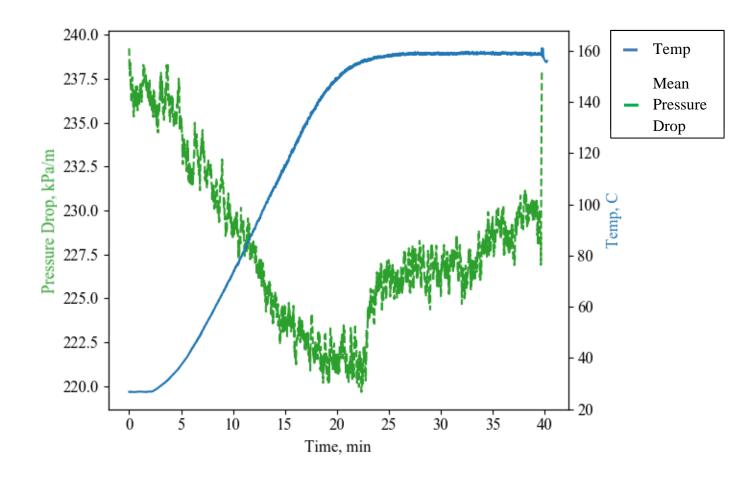
In order to develop an understanding of the mechanics within our system, we developed pressure drop graphs for multiple runs with increasing temperature. Two of these graphs can be seen in figure 2 and figure 3 (the rest in appendix B). The system had very similar curves for both types of biomass with the pressure dropping until around halfway through the run. As the temperature increased and the viscosity and density of water decreased, the pressure drop within the system decreased. After the temperature hit its maximum, the pressure drop increased slightly and seemed to be relatively stable after the increase. This aspect of the curve needs further exploration to determine the possible causes of the increase.



**Figure 3: Corn Stover Results** 

Mean pressure drop within reactor with ~3.4 g of corn stover in relation to increasing oven

temperature

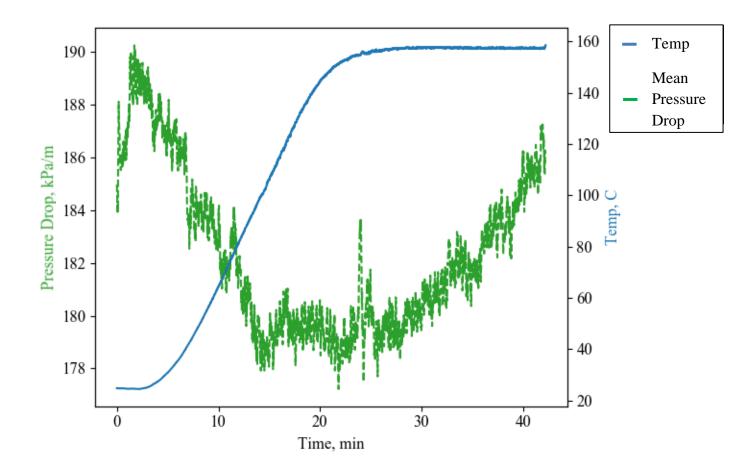


**Figure 4: Pine Sawdust Results** 

Mean pressure drop within reactor with  $\sim 2$  g of pine sawdust in relation to increasing oven

#### temperature

One possible cause of this increase in pressure drop is the presence of biomass swelling. As the water flows over the packed biomass, the biomass begins to absorb the water and swell. If this swelling is present in our system over the time of our runs, it could explain the increase in pressure drop. We also compared our biomass runs to a run of 1mm borosilicate glass beads. The glass beads run can be seen below in figure 4. The glass bead run seems to exhibit the same pattern that is present in our biomass runs. It is possible that this pressure increase is an inherent factor within our system. Unfortunately, we only have one glass bead run to compare to our biomass run.



**Figure 5: Glass Beads Results** 

Mean pressure drop within reactor with 1mm glass beads in relation to increasing oven temperature

### **4.2 Ergun Equation**

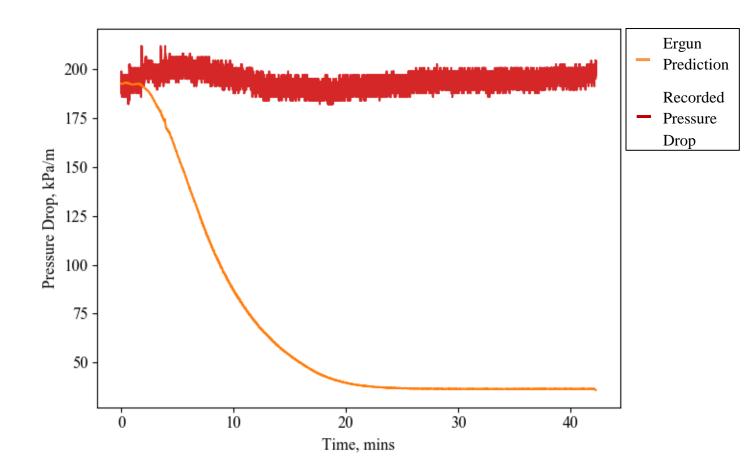
Our results were also compared to the theoretical pressure drops from the Ergun equation. This would allow us to see differences in what should be happening within our system and could provide possible evidence for biomass swelling. The form of the Ergun equation used in our calculations can be seen below. Our superficial velocity term,  $v_s$ , was held constant because our flowrate was a constant 20mL/min, so our  $v_s$ =0.000657 m/s. The particle diameter,  $D_p$ , was held

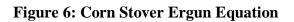
constant at 1mm for both glass beads and each biomass. Though the biomass was a large mix of particle sizes, 0.86mm-1.6mm, we simplified it to an average of around 1mm. In order to determine the density of water,  $\rho$ , with changing temperature, we used Dortmund Data Bank's DIPPR105 equation and parameters. In order to determine the viscosity,  $\mu$ , of the water with changing temperature, we used the Vogel equation to make viscosity a function of temperature. The void fraction,  $\epsilon$ , we assumed for glass beads was 0.3. In order to fit the Ergun equation for the values of the corn stover we recorded, our estimated void fraction was 0.074. We believe that this low value that is required to fit the equation to our results could be caused by pressure drop from other parts of our system.

$$\frac{\Delta P}{L} = \frac{150\mu}{D_p^2} \frac{(1-\epsilon)^2}{\epsilon^3} v_s + \frac{1.75\rho}{D_p} \frac{(1-\epsilon)}{\epsilon^3}$$

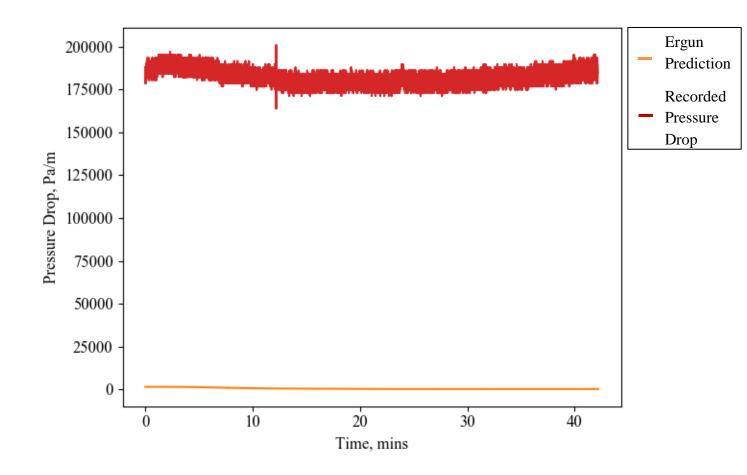
We created Ergun equation graphs for corn stover and glass bead runs. The corn stover graph in figure 5 illustrates a significant difference between our measured values and the Ergun equation. This could be a possible indication of swelling of the biomass. However, the glass bead values are very different as compared to the theoretical Ergun values, as seen in figure 6. The recorded pressure drop values were around 180 kPa/m and the starting theoretical value for glass beads were around 660 Pa/m. Unfortunately, we only have one run of glass beads for our conditions, so we cannot compare to other runs in order to determine the cause of this difference.

One possible reason is the other materials within the system affect the pressure drop much greater than we originally expected. In order to better determine what is happening within our system, significant and comprehensive pressure drop characterization experiments should be run to ensure the integrity of data. Similarly, runs with biomass should be reexamined to determine the pressure drop of biomass separate from other factors, such as the pipe straighteners or frits. The orientation of the reactor at a 40 degree angle may have also contributed to differences between the theoretical model and the data we collected. Most packed bed reactors are mounted vertically, and models describing their behavior assume that the reactor is mounted vertically as well. Because of these factors, models like the Ergun equation may not be perfectly suited to describe our reactor system. However, it is unknown how or to what extent the angle of the reactor affected the pressure drop data. Also, if there is a significant amount of pressure drop from the other parts of the system, that would explain why we need a void fraction of 0.074 in order to match the Ergun equation with the corn stover. If we were to measure pressure drop of the biomass itself, we would expect a higher void fraction value consistent with similar materials. With this great difference present in the glass beads, we also cannot determine whether any biomass swelling is present or whether something within our system or runs caused this difference.





Recorded corn stover pressure drop versus theoretical Ergun equation with void fraction of 0.074



**Figure 7: Glass Beads Ergun Equation** 

Recorded glass bead pressure drop versus theoretical Ergun equation with void fraction of 0.3

### **Chapter 5: Conclusions and Recommendations**

Although no significant statistical conclusions can be drawn from our data, flow through pretreatment of biomass for biofuel production remains a promising field of study. More work must be done to accurately model the pressure drop and chemistry of FT biomass systems. The reactor system that our team constructed should be able to provide future researchers a testbed to run additional tests.

There were major discrepancies between the data we collected and the theoretical predictions from the Ergun equation. Of course, including terms to account for the swelling of biomass over time could help, but that does not explain the behavior of the glass beads in the reactor system. Since the glass beads do not swell over time, the void fraction and particle diameter should not have changed and the experimental data should have matched the theoretical model. Instead, the pressure drop is orders of magnitude greater than the Ergun equation predicts, and the pressure drop follows a very similar pattern to the biomass tests. A major part of this issue is likely that the internal components of the system contributed to increasing the pressure drop. We do not have usable pressure drop data on frits and tubes alone within the reactor, however, so their effect on the pressure drop could not be accounted for in the data generated. Finding the amount of pressure drop that these components contribute is a crucial next step in generating more useful and accurate data out of the existing reactor setup.

Outside of the issues connecting models to the data we produced, there are several steps that future researchers should undertake to improve the accuracy and utility of the reactor setup. The data sets that we were able to produce with the FT system were fairly limited in scope. Our team only tested two types of biomass and two oven temperatures, while keeping the pressure of the system, the packing height within the bed and the water flow rate constant. In addition, we were unable to test the samples taken from the reactor for sugar content, so we do not have an accurate picture of the effectiveness of the FT pretreatment. This gap in our data, combined with the limited number of runs that we were able to execute, has severely limited the conclusions we can draw. Significant work must be undertaken to fully understand the pressure drop and chemistry that occurs in the system that we set up. We recommend that future researchers retest the system at the conditions described in this report to verify our findings before engaging in tests at new conditions. This will also allow for the sugar content to be properly analyzed, giving a more complete understanding of the chemistry of the system at the lower end of reaction temperatures. Experiments should also be expanded to include pre-swollen biomass as packing material, as we described in our methods, to develop a complete understanding of the pressure drop in this system. We also recommend that future groups prioritize increasing the oven temperature. Higher temperatures will likely lead to more reactions and more sugar produced from the biomass.

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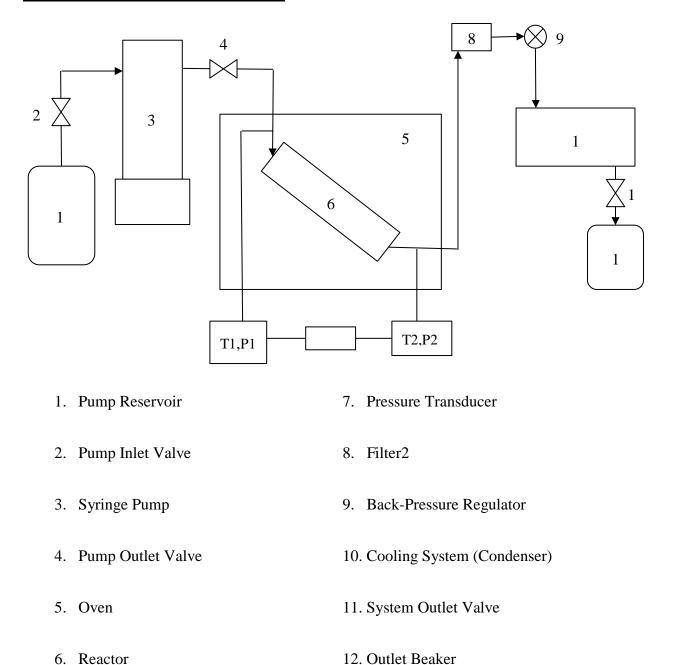
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# Appendices



### **Appendix A: Experimental Apparatus**

Figure A1: Overall reactor system and associated parts

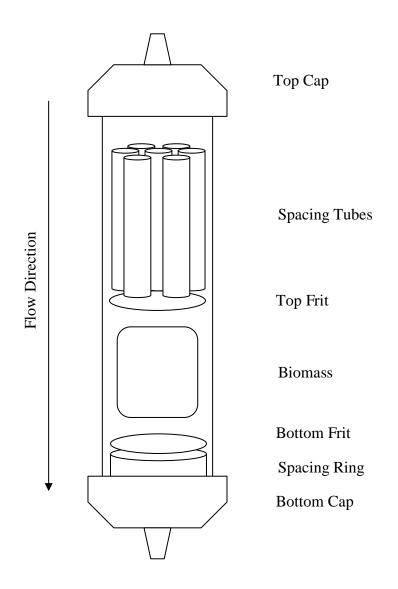


Figure A2: Reactor vessel and internal parts

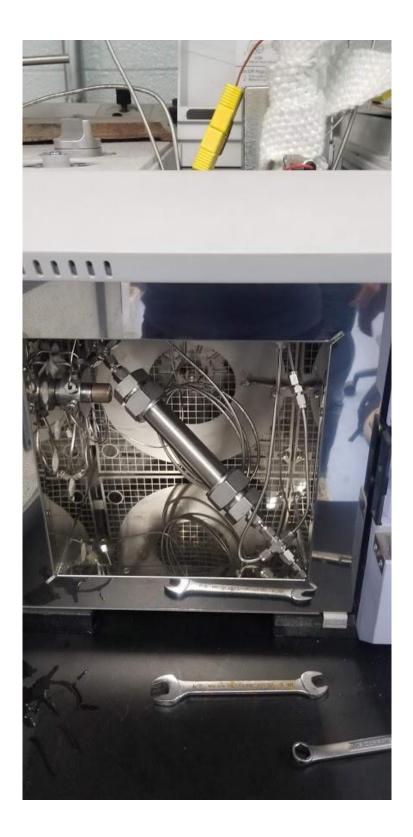


Figure A3: Reactor installed within reaction oven



Figure A4: Pump controller and pressure transducer displays. Note system PSI indicator on

pump controller.

### **Appendix B: Result Graphs**

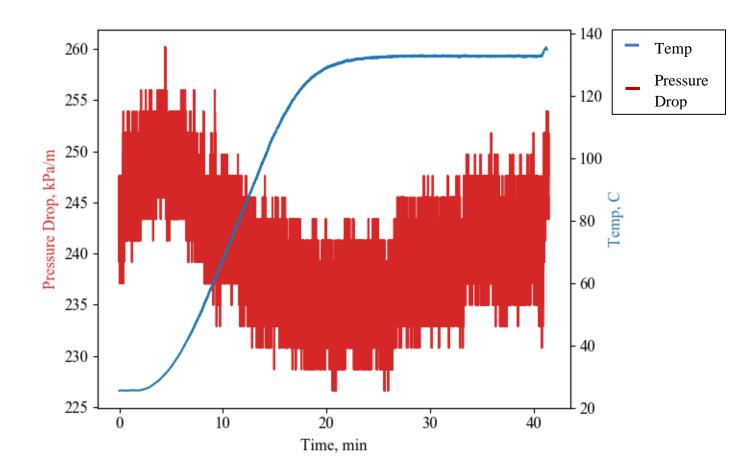
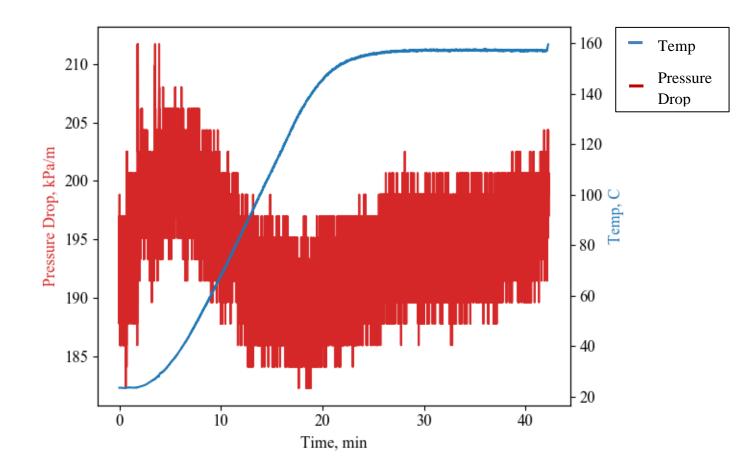
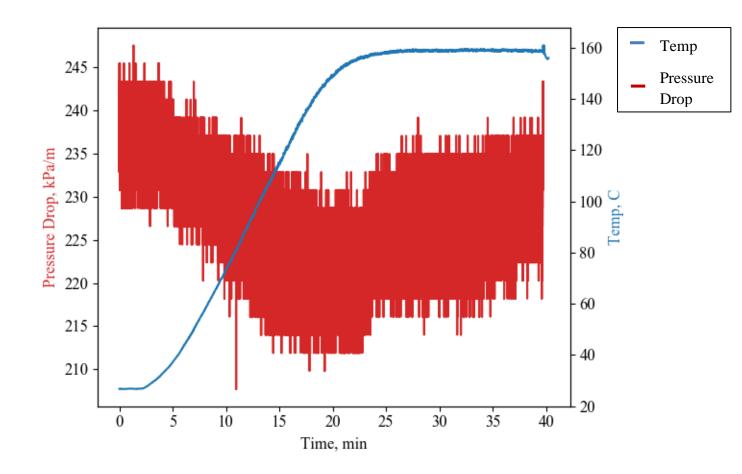


Figure B1: Pressure Drop-Temperature-Time graph for 1<sup>st</sup> run with dry corn stover, 20 ml/min flow rate, (~100 samples/sec)



**Figure B2:** Pressure Drop-Temperature-Time graph for 2<sup>nd</sup> run with dry corn stover, 20 ml/min flow rate, (~100 samples/sec)



**Figure B3:** Pressure Drop-Temperature-Time graph for run with 2<sup>nd</sup> dry pine sawdust, 20 ml/min flow rate, (~100 samples/sec)

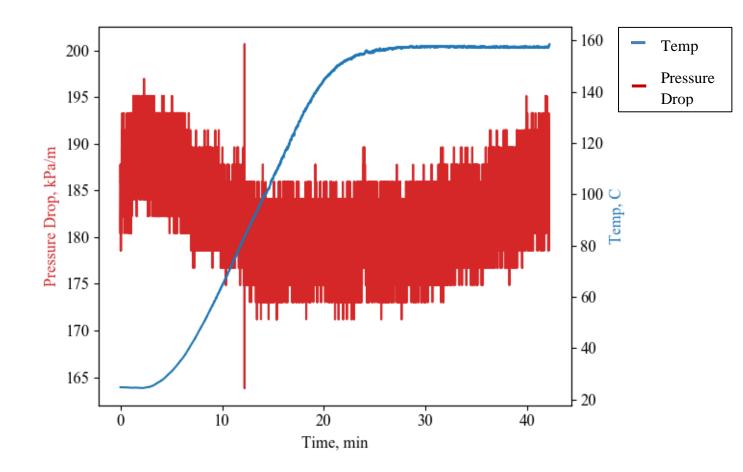
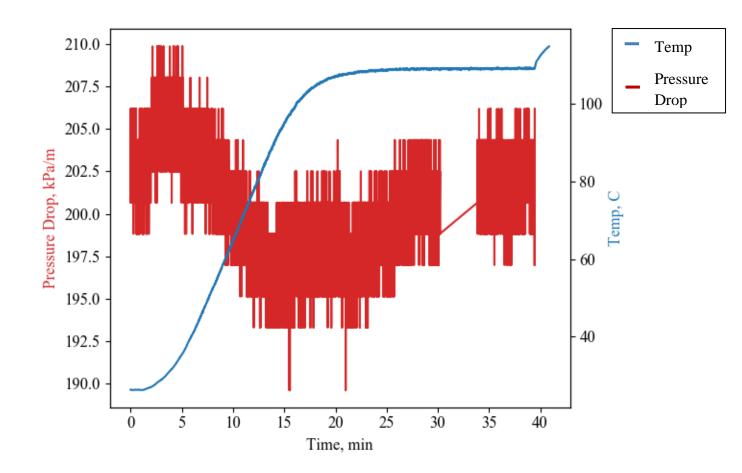
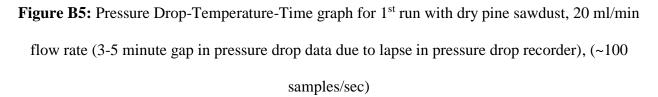
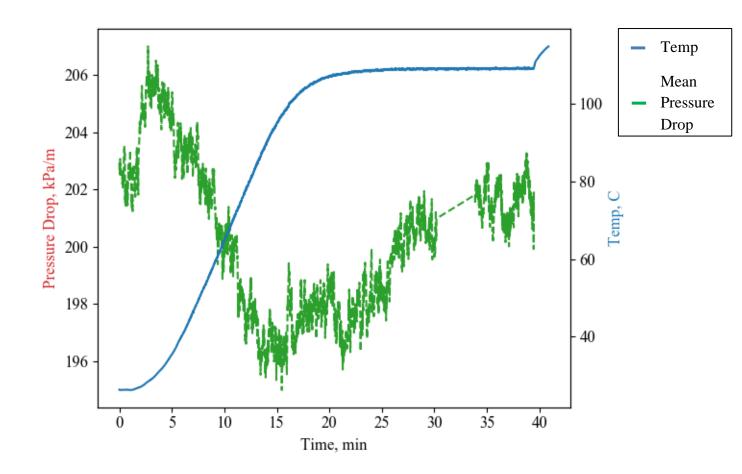


Figure B4: Pressure Drop-Temperature-Time graph for run with 1mm borosilicate glass beads,

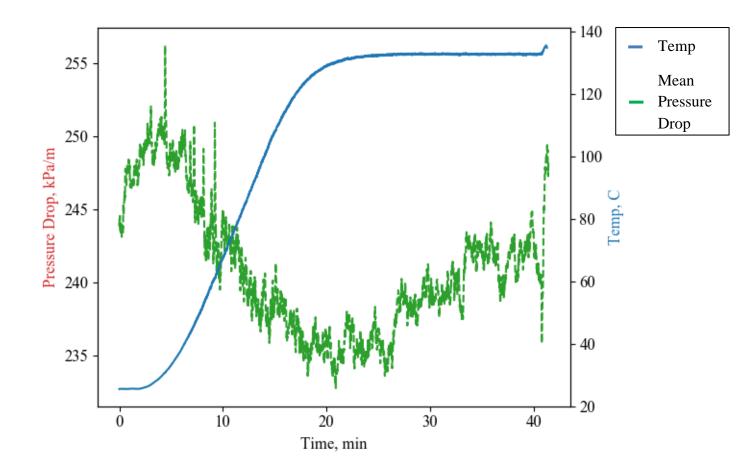
20 ml/min flow rate, (~100 samples/sec)







**Figure B6:** Mean Pressure Drop-Temperature-Time graph for 1st run with dry pine sawdust, 20 ml/min flow rate (3-5 minute gap in pressure drop data due to lapse in pressure drop recorder)



**Figure B7:** Mean Pressure Drop-Temperature-Time graph for 1<sup>st</sup> run with dry corn stover, 20 ml/min flow rate, (~100 samples/sec)