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Development of self selection torrefaction system

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Development of self selection torrefaction system

by

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A thesis submitted to the graduate faculty

in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Major: Agricultural Engineering

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Iowa State University

Ames, Iowa

2012

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Acknowledgements

First and foremost I thank my Lord and Savior Jesus Christ for his unconditional love. I thank the Lord for providing me with the ability and support to complete this research project. Soli Deo Gloria!

I thank all of my family and friends for their support through this project. I especially thank my mom, Barbara, and brother, Jonathan, for their continued support. This project would not have been the same without your constant encouragement.

I would like to thank ConocoPhillips for providing funding and support for this research project. I also would like to thank my advisor Dr. Matt Darr for his support and valuable guidance throughout the project. I could not have wished for a better fit for an advisor or research project. Thanks is given to Dr. D. Raj Raman and Dr. Kurt Rosentrater for serving on my program of study committee. I also thank Dorde Medic for his assistance and willingness to help on this project.

I dedicate this work to the memory of my father, David Brue and my grandfather, Donald Teig. Dad, you taught me all that I know about working with my hands and you encouraged me to always follow my dreams. Grandpa, you were the definition of a positive attitude and living life to the fullest. I am grateful for the memories I have of both of you and thank you for your never ending support.

Chapter 1. Introduction

In 2005, the Renewable Fuel Standard (RFS) passed by Congress mandated renewable fuel production in the United States. The RFS sought to reduce greenhouse gas emissions, reduce petroleum imports, and encourage development of renewable fuels. Revisions were made and the RFS2 was passed, requiring 36 billion gallons of renewable fuel are required to be blended into transportation fuel by 2022. Furthermore, the RFS specifies that by 2016, the majority of biofuels produced must come from cellulosic feedstocks, as opposed to food crops. These cellulosic feedstocks (hereafter referred to as biomass) include a variety of sources such as dedicated energy crops and agricultural residues, which are available in large quantities.

However, the use of biomass as an energy source presents a number of challenges such as low bulk density, particle size irregularity, and high moisture content. The low bulk density, variability in moisture content, and susceptibility to microbial degradation of biomass hinder the ability to efficiently transport, store, and process material. Cellulosic processing plants require a large area of land to supply the necessary feedstock. The economies of scale would suggest that as plant sizes become larger, unit costs will decrease. However, the larger collection radius surrounding the plant will increase transportation costs for material. Baling of biomass, such as corn stover, is one commonly used method to increase the bulk density of the material and improve the handling characteristics. Additionally, research has been conducted on thermochemical treatment of biomass to increase material density and address issues of moisture and material irregularity.

Mild pyrolysis, or torrefaction, is a thermochemical pretreatment for biomass used to address these issues. During the torrefaction process, biomass is heated to 200-300°C in an inert environment. Hemicellulose material is broken down and volatile gases are driven off. The resulting material is valuable due to the increase energy density, Higher Heating Values (HHV) and brittleness. The increase in material brittleness is attractive due to the ability to reduce energy requirements for particle size reduction. Research has been conducted to investigate the benefits of torrefaction when used as a pretreatment for pyrolysis and gasification applications. These applications often have strict particle size requirements that

create the need for particle size reduction. Torrefaction can be used as a pretreatment to optimize these processes.

A challenge with torrefaction is achieving a uniformly heated particle. Large biomass particles have limited heat transfer to internal layers and experience a varying degree of torrefaction. The focus of this project was to develop a self selection torrefaction system. The system integrated a lab scale torrefaction system with a ball mill reactor system. By incorporating the two stages of torrefaction and grinding into a single process, higher efficiencies are possible than if each stage were run separately. The central hypothesis is that as large particles experience torrefaction, the outer layers will be removed by the ball mill action and expose the inner layers. The process would eliminate the need for further grinding in downstream applications and would create a more uniform product.

Preliminary testing was conducted to investigate the impact of torrefaction on the particle size distribution of corn stover when ground in a ball mill reactor. Torrefied corn stover was found to be more brittle than untreated corn stover. The particle distribution was used to quantify the grindability of torrefied corn stover and untreated corn stover.

1.1. Thesis organization

This thesis is composed of two primary chapters on testing of the self selection process. The chapter concerning the particle size distribution of corn stover was written as a research article for publication. The article discussed the impact of torrefaction on the grindability of corn stover in a ball mill reactor. Data obtained in this study was used as a foundation for the development of the self selection torrefaction reactor system. The chapter discussing the development of the self selection torrefaction system was written as a final project summary for ConocoPhillips. This chapter describes the design and fabrication of the self selection torrefaction reactor and the testing associated with the reactor.

Chapter 2. Literature Review

2.1. Bio Energy Policy

The increase in energy usage over the past century has raised concern over the sustainability of current fossil fuel consumption rates. According to the U.S. Energy Information Administration, the United States has seen annual consumption rates of energy reach just over 98 Quadrillion BTU's. Of that total, 27.51 Quadrillion BTU's (28.07%) was designated for transportation purposes. Of that 98 Quadrillion BTU's, 83% of the energy was derived from fossil fuels compared to 8% for renewable energy. By comparison, in 1950 the rate of consumption was just under 35 Quadrillion BTU's (U.S. Energy Information Administration, 2010). Estimates on the remaining fossil fuel reserves vary greatly but as availability becomes more scarce, costs will continue to increase. Recently more focus has been placed on the development of renewable fuel sources to offset the demand for fossil fuels. Non-fossil fuel sources such as wind, solar, and geothermal may significantly reduce dependence on fossil fuels. However, these sources do not appear to be ready for industrial scale up in the near future. Wind energy is limited by intermittent supply and presents the challenge of storing produced energy. Solar technology remains an expensive option and does not appear to be cost competitive in the near future.

A driving factor behind much of the recent interest in biomass based fuel production is the Renewable Fuel Standard (RFS) passed in 2005. The RFS seeks to increase development of renewable fuels in order to reduce dependence on imported petroleum and reduce greenhouse gas emissions (United States EPA, 2010). In 2007 the RFS was revised as part of the Energy Independence and Security Act of 2007 and became the RFS2. By 2022, 36 billion gallons of biofuel must be blended into gasoline, with the majority of these biofuels coming from cellulosic feedstocks, rather than food crops. In order to meet this demand it is estimated that 180 million dry tons of biomass will be needed on an annual basis (Biomass and Research Development Board, 2011). Cellulosic ethanol production has the potential to replace a portion of gasoline consumption but will not be able to realistically offset all demand. A limiting factor to the increase of ethanol use is the maximum amount of ethanol that can be added to conventional gasoline, often referred to as the "blend wall." The current

limit on ethanol blends in the United States is a 10% ethanol to 90% gasoline mixture, referred to as E10. A 10% blend of ethanol in all gasoline would account for only 14 billion of the 36 billion gallons required by the RFS2.

Agricultural residues have gained interest as potential fuel sources for cellulosic based fuel production. These residues are the remaining material after completion of crop harvest. In the Midwest portion of the United States, corn stover is one of the most abundant residues available. It is estimated that 60-80 million dry tons can be sustainably harvested on an annual basis in the United States (Kadam & McMillan, 2002). This figure varies as availability is dependent upon crop yields, tillage practices, and competing markets. In Iowa alone, 57% of corn stover could potentially be removed (National Renewable Energy Laboratory, 2001). However, the low bulk density of biomass creates challenges for transportation and storage, especially when considering distribution on national and international levels. The need exists to increase density of material while keeping costs low.

2.2. Ethanol Production

The primary focus within the area of biofuels has been on ethanol production. Debate has escalated over the past decade over its use but ethanol has been in existence for over a century in the United States. As early as 1906 the *New York Times* ran an article entitled "Auto Club Aroused over Ethanol Bill" in which the scarcity of gasoline was discussed (Songstad et al., 2009) Henry Ford was noted for being a supporter of ethanol use as the Model T was able to run on either gasoline or pure alcohol (DiPardo, 2001). Following World War II the demand for ethanol tapered off as interest diminished. In the 1970's the ethanol industry experienced renewed interest as crude oil became an issue of national security due to the Arab Oil Embargo. It was also around this time that the petroleum industry began to eliminate the use of lead in gasoline due to environmental health concerns. In 1990 Congress passed the Clean Air Act Amendment which helped to spur on development of biofuels. Mandates were placed on the use of oxygenated fuels, with a minimum of 2.7% oxygen by volume. Since then the typical ethanol blend with gasoline has been a 10% ethanol blend with discussion of increasing to a 15% blend. Higher blends do exist, such as E85 (85% ethanol, 15% gasoline) but require additional vehicle modifications.

Flex Fuel Vehicles (FFV) have the ability to run on any blend of ethanol without problem. Ethanol production has continued to increase and today, global production has reached nearly 16 billion gallons (Songstad et al., 2009).

While use of ethanol has replaced lead usage in gasoline, there are noticeable drawbacks. The energy content of ethanol is lower than gasoline. Approximately 1.5 gallons of ethanol are required to equal the energy content of one gallon of conventional gasoline. Ethanol blended with conventional gasoline cannot be transported in multi-fuel pipelines due to separation issues. Moisture within pipelines can be absorbed by ethanol, causing it to separate from gasoline. The fuels must be shipped separately and then blended before reaching fueling stations. The chemical composition of ethanol is different enough from conventional gasoline that modifications would be required for transportation equipment, fuel station pumps, and vehicle engines.

Recently, the push has been made toward the production of cellulosic ethanol. In this type of production, ethanol is converted from cellulose instead of starch. Traditional ethanol production from corn converts the starch content to sugars and is then fermented. With cellulosic based fuels, corn is replaced by agricultural residues, such as corn stover. Hydrolysis of the biomass converts cellulose into sugars. Two stages of hydrolysis are performed: one at low temperatures to target the hemicellulose material and a second at higher temperatures to convert cellulose. Figure 2.1 shows the processing stages for ethanol production from corn and from biomass, respectively.

Ethanol from Corn

Corn — Amylase → **Glucose** — Yeast → **Ethanol**

Ethanol from Cellulose

Cellulose — Cellulase or Sulfuric Acid → **Five & Six Carbon Sugars** — Genetically Engineered Bacteria → **Ethanol**
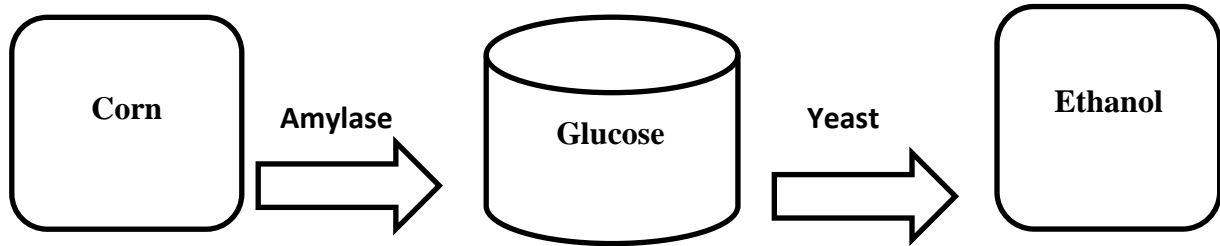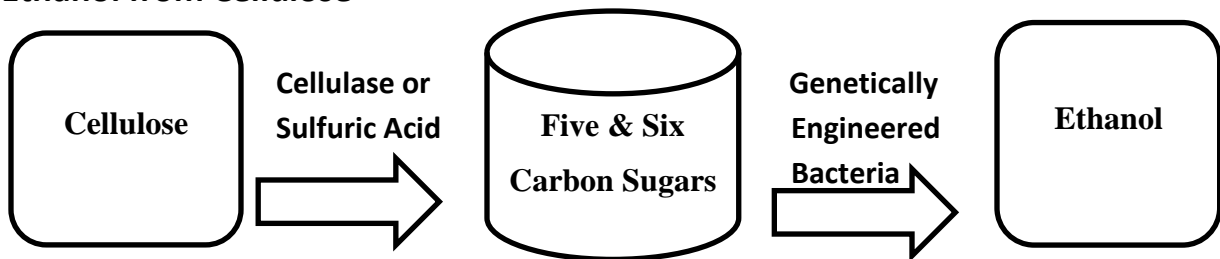
Figure 2.1: Ethanol Production from Corn and Biomass

One of the primary arguments for cellulosic fuel production is that food crops will no longer compete for the production of fuel. Estimates show that conversion rates of 60-100 gallons of ethanol per dry ton of corn stover are possible (DiPardo, 2001). However, cellulosic ethanol production is still in the early stages and will require more time before significant increases in production are achieved.

2.2.1. Biorenewable Feedstock Economics

One of the major factors limiting the advancement of biofuels is the high cost of production. Each stage of the biomass supply chain presents an additional cost to the final product: harvest of material (20-65% of total delivery cost), transportation (20-40% of total cost), and pretreatment (20-25% of total cost). The cost of the biomass feedstock is highly variable as collection techniques depend on the local infrastructure. Potential feedstocks can be categorized as milling residues, agricultural residues, or dedicated energy crops. Milling residues remain after the processing of materials such as woody biomass. Agricultural residues, such as corn stover, remain after the corn crop has been harvested. While corn

stover is necessary for nutrient replacement and ground cover for the soil, sustainable harvest of the material is possible. The production of dedicated energy crops are those produced specifically as feedstock for biofuels. These types of materials include switchgrass and woody biomass. Certain types of materials once viewed as strictly waste products, such as municipal solid wastes, can be used as potential feedstocks.

Table 2.1: Availability and cost of various feedstocks (Data from Brown, 2003)

Feedstock	Production (10^6 Mg/yr)	Price (1994 $/Mg)
Corn	191	90
Sorghum	16	90
Sugar cane	25	30
Agricultural residues		
Low cost	4	12.9
Mid cost	36	38.8
High cost	50	47.4
Forest residues from logging		
Low cost	3	12.9
Mid cost	3	25.9
High cost	3	43.1
Municipal solid waste		
Mixed paper	26	0-19
Packaging	14	0-5.2
Urban wood	3.5	12.9-25.9
Yard Waste	11	0-12.9

2.3. Torrefaction Process

The biorenewable market has seen significant interest in two main sectors: biochemical processing, which includes the previously mentioned cellulosic ethanol production, and thermochemical processing, which includes the areas of gasification and pyrolysis. Biochemical conversion uses organisms such as yeast or bacteria to convert biomass into fuels. Thermochemical conversion uses heat as the primary means of converting biomass into a fuel or other bio based products.

Torrefaction is often referred to as mild pyrolysis where biomass is heated to temperatures of 200-300°C in an inert environment. The word torrefaction is derived from the French word meaning 'roasting' (Ciolkosz & Wallace, 2011). Figure 2.2 shows a comparison of the torrefaction from raw state to 200°C, 250°C, and 300°C. The earliest use of torrefaction occurred in France during the 1930s. Much like gasification, the purpose was to produce synthetic gas, or syngas. Syngas can be used for heating and combustion as a substitute for natural gas. It was not until the 1980s that the commercial process of torrefaction was first investigated in the metallurgical industry. Over the last decade interest has continued to increase in torrefaction as a potential pretreatment option for thermochemical processes. (International Energy Agency, 2009), (Bridgeman et al., 2010)
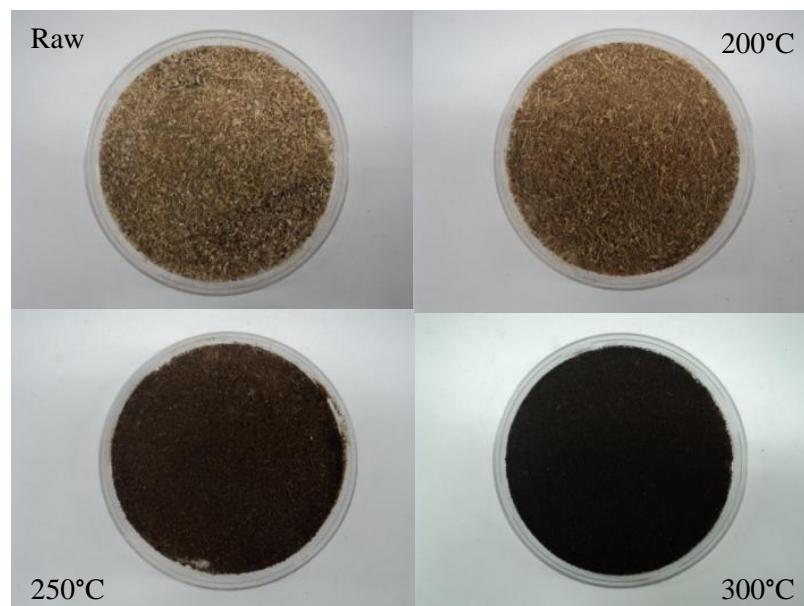
Figure 2.2: Torrefaction of Corn Stover

During this process, hemicellulosic material begins to degrade and the final product is composed primarily of cellulose and lignin. Volatile gases such as acetic acids, carbon dioxide, and carbon monoxide are driven off from the solid material. Figure 2.3 shows the decomposition of hemicellulose material during the torrefaction process. The final product of torrefied material is attractive as a biofuels feedstock due to its increased brittleness, resulting in lower energy requirements for grinding. The material also has

increased bulk density, resistance to microbial degradation, and hydrophobicity. Studies have shown that torrefaction will increase the carbon content of wood and improve flow characteristics for wood powder (Repellin et al., 2010). By pretreating the biomass, cost reductions can be achieved in storage and transportation of the material (Shah et al., 2012). Collection, processing, storage, and transportation can account for 25 – 65% of total production costs for cellulosic ethanol (Biomass and Research Development Board, 2011).
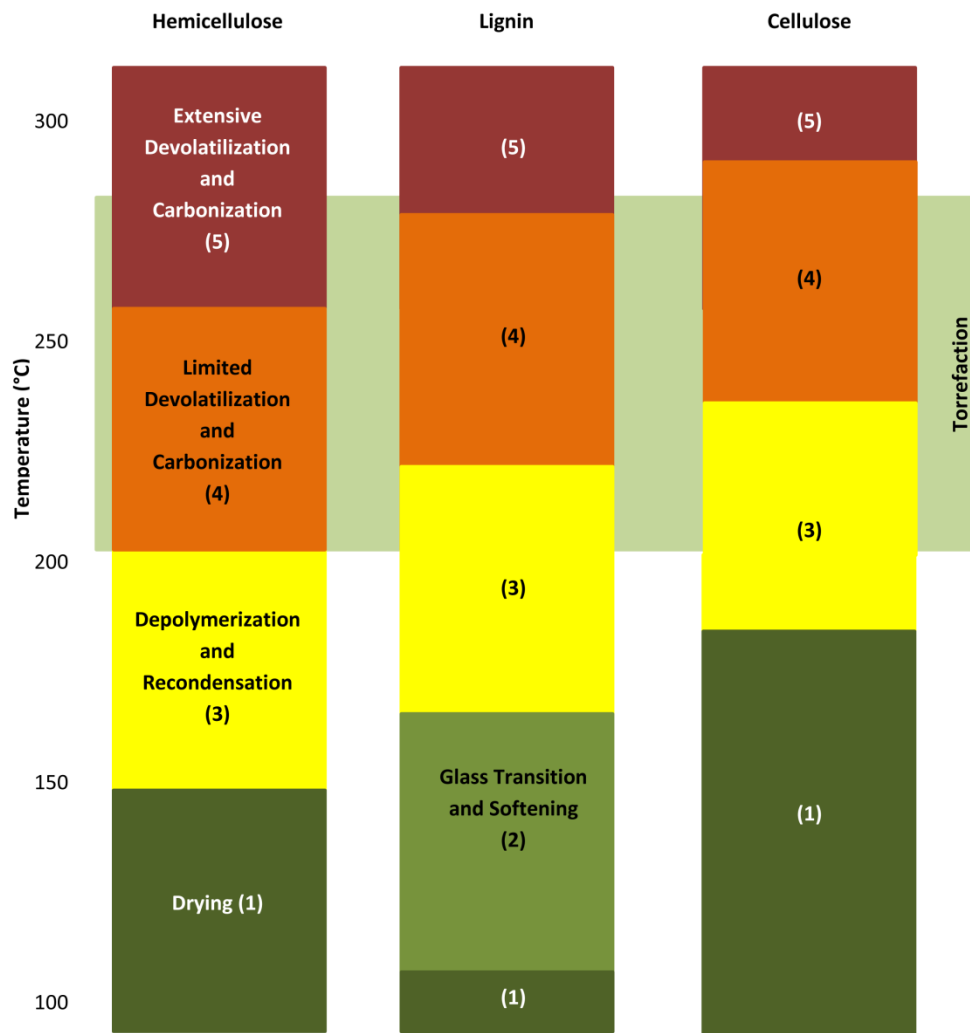
Figure 2.3: Lignocellulosic Decomposition during Torrefaction

Medic et al. have reported that increases in energy density as high as 19% are possible for corn stover (Medic et al., 2012). However, to achieve such increases in energy density, overall mass loss of material is increased significantly as processing temperatures are elevated. Increases in the Higher Heating Value (HHV) have also been observed with values being 21.6 MJ/kg and 26.41 MJ/kg for corn stover and pine residue, respectively (Medic et al., 2012), (Phanphanich & Mani, 2010). By comparison, bituminous coal and untreated corn stover have HHV figures of 27.72 MJ/kg and 18.2 MJ/kg, respectively. Figure 2.4 shows the relationship between mass and energy loss for typical torrefaction reactions. As the torrefaction process occurs, the rate of mass loss is higher than that of energy loss. The final product is found to have to have a higher energy density than the initial material. The exact values for energy and mass loss will vary, depending upon feedstock type and processing temperatures.

Gas

Mass = 0.3
Energy = 0.1

Biomass

Torrefaction

Torrefied
Biomass

Mass = 1
Energy = 1

Mass = 0.7
Energy = 0.9

Energy Densification (E/kg): 0.9/0.7 = 1.3
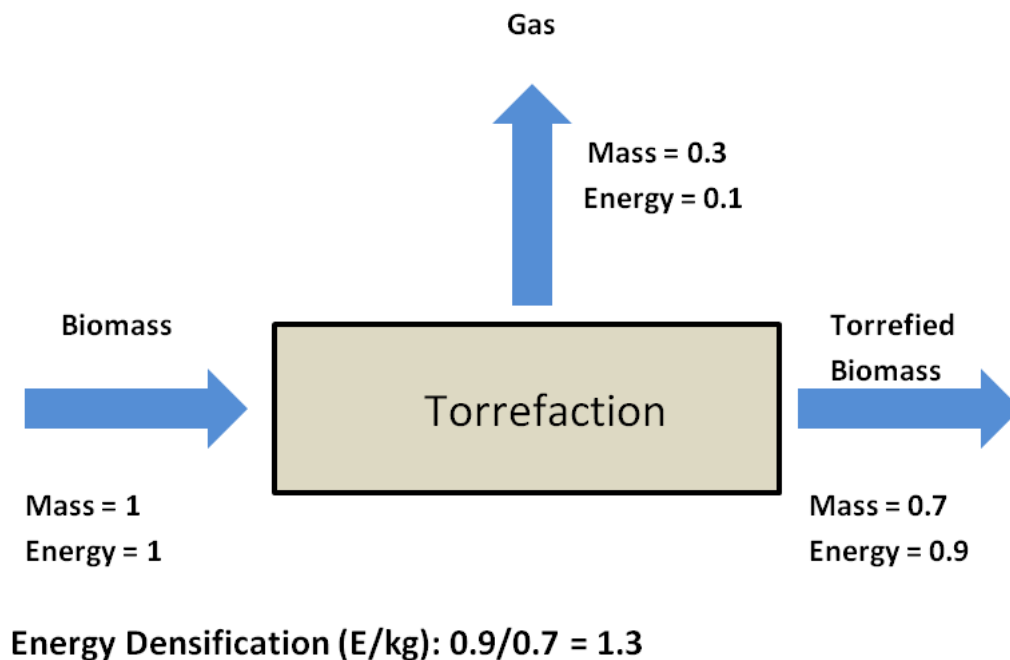
Figure 2.4: Energy and Mass Balance for Torrefaction Process

The low bulk and energy densities of lignocellulose create challenges for storage and transportation of biomass feedstocks. Typical bulk densities range from 60 -100 kg/m^3 while energy densities range from 8 – 14 MJ/kg (Phanphanich & Mani, 2010).The efficiency of

these processes is reduced by variations in moisture content, particle size, and overall quality of the material. The fibrous structure of biomass also makes particle size reduction a difficult and energy intensive process.

Cellulose, hemicellulose, and lignin are the three main structural components of biomass. This combination of material is often described by the term lignocellulose. Of these three components, cellulose is the most abundantly available. Cellulose is a structural polysaccharide, comprised of a chain of glucose molecules connected by glycosidic bonds (Brown, 2003). When these bonds are broken, glucose is released, allowing for the production of food or fuel. Rarely is cellulose found without some combination of hemicellulose and lignin content. In thermochemical processing hemicellulose begins to devolatilize at temperatures of about 200°C, while lignin begins devolatilization around 240°C (van der Stelt et al., 2011). Cellulose devolatilization begins around 260°C. The degree of torrefaction will determine the extent of devolatilization and material decomposition. Much of the gases and condensables with high oxygen content that are given off during the torrefaction process are a result of the hemicellulose decomposition (Medic et al., 2012).

Table 2.2: Hemicellulose Content for Various Feedstocks (Data from Brown, 2003)

Feedstock	Cellulose (wt %)	Hemicellulose (wt %)	Lignin (wt %)	Other (wt %)
Bagasse	35	25	20	20
Corn stover	53	15	16	16
Corncobs	32	44	13	11
Wheat straw	38	36	16	10
Wheat chaff	38	36	16	11
Short rotation woody crops	50	23	22	5
Herbaceous energy crops	45	30	15	10
Waste paper	76	13	11	0

Studies have shown mass loss for corn stover is much higher at temperatures between 250-300°C than at temperatures of 200-250°C (Medic et al., 2012). This is due to the higher degree of devolatilization and decarbonization of hemicellulose that occurs. Initial moisture

content and residence times play a factor in the final product produced but temperature is the most significant factor. Medic et al. presents the following data in Figure 2.5, showing the effect of untreated biomass moisture content on mass loss of torrefied corn stover samples. The plot shows how mass loss varies little as residence time and moisture content change. However, increases in temperature will lead to increases in mass loss.
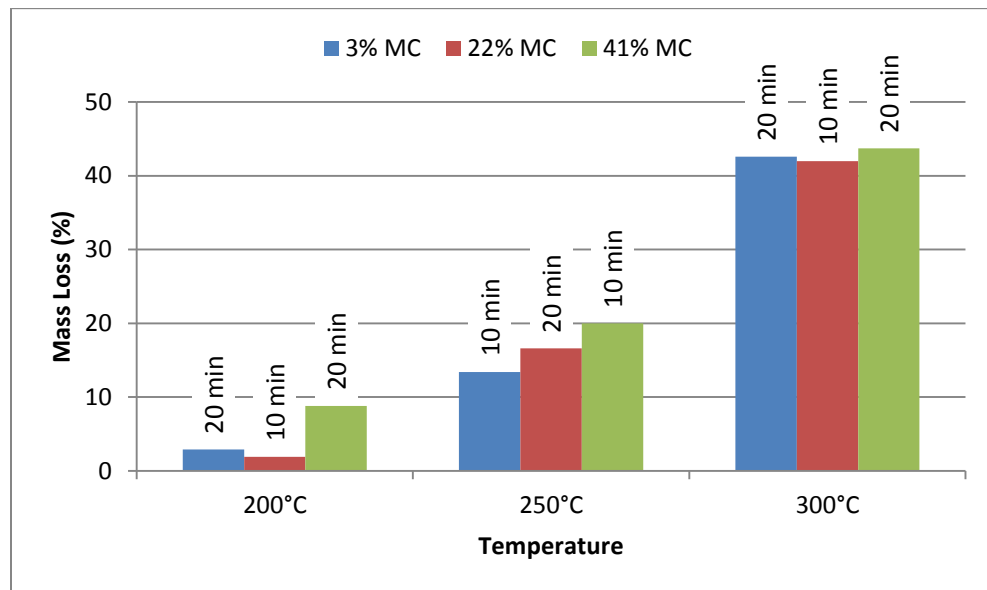
Figure 2.5: Mass Loss during Torrefaction (data from Medic et al., 2012)

Medic et al. have also studied the composition of condensables released during the torrefaction process. The largest percentage of material released is water, along with acetic acid, furfural, methanol, and hydroxyacetone (Medic et al., 2012). As processing temperatures are increased, the release of CO increases. Additionally, the ultimate analysis of torrefied material shows that carbon content increases (wt%) with increases in temperature (Medic et al., 2012). Decomposition of hemicellulose accounts for the majority of these products but lignin and cellulose experience some decomposition above temperatures of 250°C (Repellin et al., 2010).

2.4. Grinding of Biomass

The central objective of this thesis is based upon the concept of integrating the torrefaction and grinding stages into a single step. There have been several reports on the

grindability of biomass and the benefits of torrefaction as a pretreatment option prior to grinding. Grinding of biomass is energy intensive, with natural wood requiring one-sixth of its Low Heating Value (LHV) (Repellin et al., 2010). Much of the testing on biomass grinding has focused on woody biomass or perennial grasses. For beech and spruce woods, energy requirements to grind material with an ultra centrifugal mill to a 500 μm particle size require 850 kWh/t and 750 kWh/t, respectively (Repellin et al., 2010). Torrefaction of the wood to temperatures as low as 160°C can reduce energy requirements by as much as 40% for spruce. Increase of temperature will continue to reduce energy requirements. At temperatures of 280°C, energy requirements for beech can be reduced by 90% (Repellin et al., 2010).

One application for torrefied material is as a fuel source for co-fired coal plants where biomass and coal are simultaneously combusted. When investigating the grindability of coal, the Hardgrove Grindability Index (HGI) is often used. This test is used to determine the relative difficulty of grinding coal to an acceptable particle size for combustion. As the HGI value increases, the material is found to be easier to reduce in particle size. Grinding of miscanthus has been investigated as a potential fuel source for co-firing applications. Torrefaction has been found to improve the grindability of the biomass and create a material that is similar to coal, based on HGI (Bridgeman et al., 2010). Miscanthus that was torrefied to 290°C was found to be similar to coal for grinding tests. However, the HGI test has limitations concerning material that is heterogeneous. Calculating energy requirements for grinding has also been used to observe that as material brittleness increases, material strength decreases, thus reducing energy requirements. Many tests have also focused on particle size distributions to compare the grinding between feedstocks.

There have been comparably fewer tests conducted on torrefaction and particle size reduction of agricultural residues such as corn stover. Medic et al. investigated the influence of torrefaction on corn stover and studied the effects of temperature, moisture content, and reaction time on the final product. While an increase in material brittleness was observed, no specific testing was conducted on the particle size reduction characteristics of torrefied corn stover. Mani et al. examined the grinding requirements for various biomass feedstocks. In the study on corn stover, it was found that using a hammer mill to reduce corn stover to

particle sizes of 0.8, 1.6, and 3.2mm required 7.0, 14.8, and 22.0 kWh/t, respectively (Phanphanich & Mani, 2010). From this data it was shown that fine grinding of corn stover requires higher energy input. Compared to the other tested biomass feedstocks of wheat straw, barley straw, and switchgrass, corn stover was found to be the easiest to grind. With corn stover having favorable grinding characteristics, it is believed that the torrefaction process will enhance the grindability and further reduce energy requirements.

Much of the available literature on biomass grinding is focused on the use of hammer mills. They are effective for use with a variety of feedstocks and can produce specific particle sizes. However, there have been fewer studies on the application of ball mill grinding. Yu and Wu reported that that ball milling of cellulose will result in a reduction of particle size distribution (Yu & Wu, 2011). Additionally, the degree of polymerization of cellulose is reduced, thus reducing the mechanical strength of the material.

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Chapter 3. Objectives

3.1. Concept of self selection process

A significant portion of research in the area of torrefaction has been devoted to particle size reduction of biomass. The information in Chapter 2.4 shows how the introduction of torrefaction into the biomass supply chain will lead to reductions in energy requirements for grinding of biomass. However, the process of torrefaction is limited by material diameter and will experience temperature gradients within the material. Particle thickness and density will limit the degree of torrefaction that is possible, resulting in a non-uniform final product.

One solution is to grind the material prior to the torrefaction stage. However, this will increase the processing time and production costs for the biomass conversion process. Torrefaction will reduce grinding energy requirements but often requires a pre-torrefaction grinding stage. Biomass is typically densified and transported in large masses, such as bales, and can have volumes of nearly 3 m^3 and a mass of over 500 kg. The increased size and density of the bale makes for more efficient storage and transportation of material but creates challenges for processing. The tradeoff exists between the ease of material transportation and the ease of particle size reduction.

Torrefaction of biomass has been investigated on scale lab tests but few efforts have been placed on an industrial scale. Grinding of biomass prior to torrefaction can be used for lab scale testing but on an industrial level this limits efficiency and reduces economic gains. The feasibility of torrefaction requires an investigation of the entire biomass supply chain. For corn stover, the material is typically transported and stored in bale form. The design of a torrefaction system must be designed to efficiently process material without requiring significant modifications to the existing supply chain.

This research project recognizes this gap in the supply chain for incorporating a torrefaction stage. There has been some focus on the grindability of torrefied material but more research is required concerning the heat transfer issues associated with torrefaction of large particles. The focus of this research project is to not only incorporate the torrefaction process to the supply chain for biomass but to integrate the torrefaction process with the grinding process.

3.2. Objectives

Objective 1: Quantify acceptable operating parameters for a self selection torrefaction reactor.

The self-selection process reduces material particle size in a ball mill reactor as it is being torrefied. As the degree of torrefaction increases, the material will reduce in particle size more easily. With larger particles, the outer layers will become torrefied while the inner layers remain untreated. The increased brittleness of the torrefied material will allow the fully torrefied layers of the material to be removed while leaving the non-torrefied layers intact until torrefied. Impact within the ball mill will reduce the more brittle torrefied material. The non-torrefied material will remain in the ball mill reactor until fully torrefied. By integrating the system, further grinding of torrefied material would no longer be needed after the torrefaction process. The factors being studied will include torrefaction pretreatment, duration of ball milling, and speed of drum rotation. This objective will be discussed in Chapter 4.

For objective one, the P-value testing H_0: $p > 0.05$ versus H_a: $p < 0.05$ was used. The null hypothesis H_0 stated that no statistical difference exists between the particle size of torrefied and non-torrefied material when ball milled. The alternative hypothesis H_a would be true if $p < 0.05$ and would state that that the particle sizes differ between torrefied and non-torrefied material.

Objective 2: Validate operation of a self-selection torrefaction reactor in a lab scale continuous flow torrefier.

The objective is to integrate a ball milling system into the torrefaction process to allow for self-selection of torrefied material. A continuous flow torrefaction system was developed with a self-selection ball mill reactor. Scale up to the continuous flow torrefier will allow for a greater output of material. A continuous flow system would be more efficient as samples would not have to be loaded and unloaded before and after each test run. Different feed rates will be possible depending upon the type and quality of feedstock. Particle size distributions will be determined to validate the operation of the self-selection torrefaction reactor. This objective will be discussed in Chapter 5.

Chapter 4. Impact of torrefaction on particle size distribution of corn stover

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4.1. Abstract

Mild pyrolysis, or torrefaction, is a thermochemical pretreatment for biomass used to address issues of low bulk density, high moisture content, and particle size irregularity. The torrefaction process involves heating biomass to 200-300°C in an inert environment. Torrefied biomass is characterized by increases in material brittleness, hydrophobicity, and resistance to microbial degradation.

This study seeks to examine the impact of combined torrefaction and particle size reduction on corn stover biomass. Corn stover was torrefied to 275°C and compared to non-torrefied corn stover when ball milled. A ball mill reactor was used to break down the corn stover. The ground stover samples were then passed through a series of sieves to characterize the particle size distribution of torrefied corn stover versus non-torrefied corn stover. Particle size distributions were used to quantify the grindability of torrefied corn stover. Increasing the grindability of biomass will reduce costs for biomass processing and increase the value of these materials.

Torrefied material was found to increase the grindability of corn stover when compared to non-torrefied material and resulted in smaller particle sizes. When sieved through a 1.4mm screen, less than 3% of non-torrefied material passed through compared to 92% of torrefied material. The torrefied material was found to be much more brittle than the non-torrefied material and allowed for a greater reduction in particle size when ball milled under identical conditions. Results found from the testing support the concept of self selection, where biomass is simultaneously torrefied and ball milled. The distinctly different particle

size distributions allow for the torrefied material to be separated from the non-torrefied material.

4.2. Introduction

Biomass fuels are essential to the transition away from fossil fuels to a renewable based system. In 2009, the United States imported an average of 9,000 barrels of crude oil per day (International Energy Agency). Increasing oil prices and concern over global climate change have led to a renewed interest in renewable energy sources. Non-fossil fuel sources such as wind, solar, and geothermal may significantly reduce dependence on fossil fuels. However, these sources do not appear to be ready for industrial scale up in the near future. Renewable energy sources are projected to be the fastest growing energy source according to a report by the International Energy Agency (Bridgeman et al., 2010).

In 2005, the Renewable Fuel Standard (RFS) was passed in order to increase the production of biofuels within the United States. This act requires 7.5 billion gallons of renewable fuel to be blended into gasoline by 2012. In 2007 the RFS was revised as part of the Energy Independence and Security Act of 2007 and became the RFS2. By 2022, 36 billion gallons of fuel are required to be blended into gasoline. After 2016, the majority of this biofuel must come from cellulosic feedstocks, as opposed to food crops (United States EPA, 2010). In order to produce the necessary 36 billion gallons of fuel it is estimated that 180 million dry tons of biomass will be needed annually (Biomass and Research Development Board, 2011).

Biomass is composed of three primary components: cellulose, hemicellulose, and lignin. For the testing in this study, corn stover was be the primary focus. Corn stover is composed roughly of 53% cellulose, 15% hemicellulose, 16% lignin, and 16% other material (including oils and proteins) (Brown, 2003). Cellulose is the primary source of sugars used for converting biomass into ethanol. Cellulosic ethanol production is a biochemical conversion that converts cellulose and hemicellulose into sugars which are then converted into ethanol by means of fermentation. Other methods of biofuel production include gasification and pyrolysis, two thermochemical conversions that yield syngas and bio-oil, respectively. In

terms of gasoline production, gasification yields a cost of $4.27-$4.83/gallon while fast pyrolysis totals $1.80-$2.12/gallon (Swanson et al., 2010) (Wright et al., 2010).

In raw form, biomass has low energy densities, typically 8-14 MJ/kg (Phanphanich & Mani, 2010). The bulk density of biomass is also low, with lignocellulosic biomass having densities of 60-100 kg/m^3 (Phanphanich & Mani, 2010). These challenges make storage and transportation inefficient due to the low material density. Variations in moisture content, particle size and overall quality will further reduce the efficiency of biomass processing and increase costs. To overcome these challenges, several pretreatment options have been developed. One such pretreatment is torrefaction, a thermochemical process used to increase energy density and particle uniformity.

Torrefaction, or mild pyrolysis, occurs when biomass is heated to 200-300°C in an inert environment. During this process, hemicellulosic material begins to breakdown and volatile gases, such as acetic acids, carbon dioxide, and carbon monoxide are given off. The final material is composed primarily of cellulose and lignin. Torrefied material is appealing due to increases in brittleness, hydrophobicity, resistance to microbial degradation, and energy density. These factors contribute to cost reductions in transportation and storage. Downstream cleaning and processing costs are also reduced by removal of volatile compounds (Phanphanich & Mani, 2010). Costs for biomass collection, processing, storage, and transportation can account for 25-65% of the total production cost for cellulosic ethanol (Biomass and Research Development Board, 2011).

After torrefaction, the energy density of biomass is increased to 18-22 MJ/kg^3 for corn stover (Medic et al., 2012). Mass loss has been observed between 3-45%, depending on initial moisture content, residence time, and temperature. Above 250°C, mass loss is increased significantly due to increased devolatilization of hemicellulosic material (Medic et al., 2012). The torrefaction process will break down the fibrous structure of the biomass and increase the material brittleness (van der Stelt et al., 2011). The overall oxygen content of the biomass is also reduced during the process, further improving energy density.

With the abundance of available agricultural residues, there is potential for corn stover to be used as a fuel source for torrefaction. It has been found that corn stover can undergo

torrefaction immediately following harvest without requiring additional drying and particle size reduction (Medic et al., 2012). By introducing the torrefaction process early on in the supply chain, overall costs are reduced and material properties are improved. For example, laboratory tests have shown that grinding of torrefied biomass requires 10-20% of the energy necessary for grinding of unprocessed biomass (Ciolkosz & Wallace, 2011). Lab scale tests have been conducted but it remains to be seen what the economic impact of torrefaction will be on a commercial scale. The volatile gases given off can be combusted and used to heat the torrefaction process, further reducing heating costs. It is believed that the improved physical and chemical properties of torrefied material will reduce costs for pyrolysis and gasification. When used for gasification, torrefied material will produce higher levels of H_2 and CO in syngas, compared to non-torrefied material (Ciolkosz & Wallace, 2011).

Introduction of the torrefaction process early on in the supply chain will preserve the quality of the fuel while reducing costs for transportation and storage. Estimated costs for operation of a torrefaction processing plant have been found to be $17.5/Ton with an initial material moisture content of 30%$_{wb}$ and a processing temperature of 240°C (Shah et al., 2012). These costs have the potential to be further reduced as optimal operating conditions are determined. Integrating process such as drying, grinding, and pelletization with the torrefaction process can also reduce operating costs.

Tests show that the particle size of ground material will decrease as torrefaction temperatures increase (Phanphanich & Mani, 2010). Torrefied biomass will also produce a more uniform particle size range when compared to non-torrefied biomass. Some studies show that particle sizes below 200 μm allow for improved control over kinetic reactions for gasification and pyrolysis (Repellin et al., 2010). More uniform particle sizes improve combustion of co-fired biomass and coal.

An investigation into the particle size distribution of corn stover is recommended. The availability of agricultural residues makes for a suitable feedstock for torrefaction. A better understanding of particle size distribution will lead to more efficient upgrades of feedstocks in the areas of pyrolysis, gasification, and pelletization.

4.3. Experimental Design

4.3.1. Objective

The objective of this research is to develop a torrefaction reactor that is capable of self-selection. With the process of torrefaction, heat transfer is a significant factor limiting the efficiency of the process. Large biomass particles make uniform torrefaction difficult to achieve. To overcome this limitation, grinding of material is required prior to the torrefaction process. The self-selection process combines the torrefaction and grinding steps into a single process. The grinding will be achieved by use of a ball mill reactor placed inside of a torrefaction chamber. A ball mill reactor has been chosen for this application due to the characteristics of the material being ground. Corn stover has non-uniform density and particle sizes. Also, as the material becomes torrefied, there will be varying degrees of material brittleness. A ball mill reactor allows for a high level of impact between the grinding media and the non-uniform biomass material. The brittle nature of the torrefied material will result in a breakdown of material structure in the ball mill reactor.

The central hypothesis is that as large particles are torrefied, the outer layer will become more brittle and will be removed by the ball mill action. The inner, untreated layers will then be exposed and allowed to become fully torrefied. Removal of the outer, fully torrefied layers will prevent material from being exposed to excess heat. Once the material has reached the maximum temperature of the surrounding air, residence time will have very little influence on the outer layers of the material. Continued exposure to heat will fail to reach the inner layers of the material. By removing the outer layers, heat transfer through the particles will be improved, resulting in a more uniformly treated material. Once the torrefied material has been removed from the untreated biomass, it can be screened and removed from the reactor.

The specific hypothesis is that the grinding of torrefied and untreated biomass will result in distinctly different particle size distributions. Untreated biomass will be more difficult to grind than torrefied biomass due to the increased brittleness of the torrefied material. The resulting particle size distributions should show that ground torrefied corn stover will result

in smaller particle sizes than that of ground, untreated corn stover. This difference in particle size will allow for torrefied material to be separated from the untreated corn stover.

4.3.2. Corn Stover Samples

Corn stover samples, harvested during the fall 2010, were collected from the Department of Agriculture and Biosystems Engineering at Iowa State University, Ames, Iowa. The samples were taken from baled corn stover and were divided into two treatment levels: untreated and torrefied. The moisture content of the corn stover at the time of collection was 20%. An Ohaus Moisture Analyzer was used for moisture sampling. The torrefied and untreated corn stover samples were stored in a cooling chamber, which is kept at 3-5°C to reduce feedstock degradation and moisture loss. Selected corn stover was dried to moisture levels of 13-15% before being tested.

The ash content of the material was found to be between 6-7%. Corn stover samples were placed in a muffle furnace and heated to a temperature of 570°C for 8 hours. The samples were cooled to a temperature of 100°C and then removed from the furnace and weighed to determine final ash content.

4.3.3. Torrefaction Reactor

Corn stover samples treated by means of torrefaction were processed in a Thermo Scientific Tabletop Muffle Furnace. The furnace had an overall capacity of 45 L with the ability to hold two 125g samples of corn stover. The furnace was modified to include a nitrogen inlet line in order to create the necessary inert environment. Nitrogen was purged through the reactor at a rate of 15 L/min. PID controllers regulated the temperature within the furnace. The controller was set to ramp from room temperature to 160°C at a rate of 20°/min. Once at 160°C, the temperature was held constant for 15 minutes. The reason for holding the temperature at 160°C for 15 minutes was to allow adequate time for internal layers of material to reach the desired temperature. The temperature then increased to 275°C at a rate of 20°/min. The temperature was held for 17 minutes and then the system was allowed to cool. Material was cooled to 200°C before being removed from the furnace and placed into a cooling chamber, which is kept at a temperature of 3-5°C. Figure 4.1 shows the furnace used for torrefaction of the corn stover.

A maximum temperature of 275°C was selected based upon the physical characteristics of the material. Increasing the torrefaction temperature would increase the brittleness of material but would also increase total mass loss. This temperature was chosen to provide material with sufficient brittleness without inducing significant mass loss. For all samples of torrefied material, the mean mass loss was 40%.

Figure 4.1: Torrefaction Reaction Chamber

Torrefaction Reactor Temperature
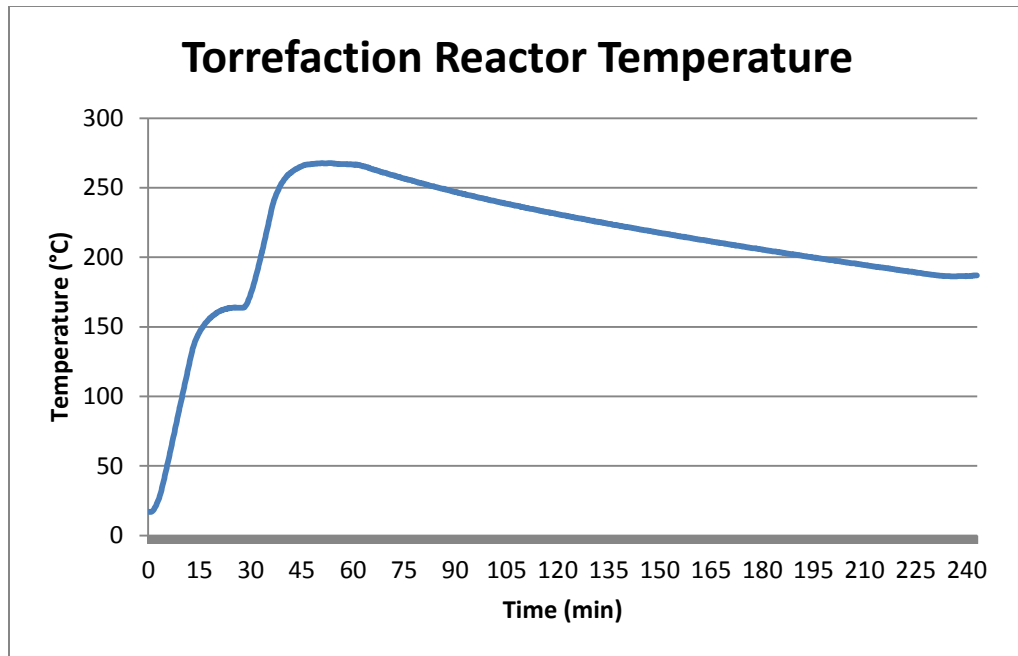
Figure 4.2: Torrefaction Reaction Chamber Temperature Profile

4.3.4. Ball Mill Reactor

Ball mill testing was conducted in a 19 L (5 gallon) steel container with a 0.3 m diameter and 0.34 m height. Steel ball bearings with a diameter of 1.9 cm (0.75 in) were used as the grinding media inside of the drum. In total, 27 steel bearings with a total mass of 778.5 g were used. The steel container was placed on a Morse Single Stationary Drum Roller. The drum roller consisted of two parallel shafts, each with a series of rollers for the drum to rest upon. Variable frequency drive controls were installed for controlling the motor speed. Corn stover samples were placed in the ball mill reactor and ground for a period of 5, 10, and 20 minutes. The drum was sealed to reduce mass loss of material while being ground. Following each test, the material was removed and analyzed.

Figure 4.3: Ball Mill Drum for Grinding Tests

4.3.5. Design of Experiment

During testing, the torrefaction and grinding steps were conducted separately. The sample size for each test condition was 30 g. This size was selected for sampling due to the size of the steel drum. Placing too large of a sample inside of the drum would reduce the collisions of the steel bearings on the corn stover and reduce the grinding capacity. The sample size was also based on the volume of the sieve shaker used for separating the material. A larger sample size would have been difficult to fit in the sieve shaker and would have required further separation of material. This additional handling of material would increase the chance of unwanted material loss and impact the data results.

Corn stover samples were torrefied prior to the ball mill grinding. Untreated corn stover and torrefied corn stover were ball milled separately to find the particle size distribution of each treatment type. The two steps were conducted separately in order to remove the interaction of the torrefaction and grinding stages and focus on the grinding stage. The hypothesis is based upon the combination of these two steps but before that interaction can be investigated, an understanding on the material grindability must be reached first. From

previous studies, an acceptable range of parameters regarding torrefaction temperature and residence time have been determined (Medic et al., 2012). Looking specifically at the particle size distribution of the torrefied material will determine the acceptable parameters for grinding of material. The study looked at the particle size distribution of untreated corn stover and torrefied corn stover to determine if a definitive difference exists between the two types of material. If it can be concluded that the particle distributions of each material type are significantly different, then evidence exists to support the design of a self-selection torrefaction system.

The set of torrefaction experiments conducted for this project were based on the following parameters: treatment of corn stover, speed of ball mill reactor rotation, and duration of ball mill grinding. The two treatment factors used for the corn stover were untreated and torrefied. For the ball mill reactor, rotation speeds of 45 rpm and 60 rpm were selected. For the duration of grinding, times of 5, 10, and 20 minutes were selected. Based on previous tests, complete torrefaction of corn stover can be achieved in 20 minutes (Medic et al., 2012). The duration of grinding times were selected to coincide with the time required for torrefaction.

A full factorial design was set up to test the parameters of treatment, duration of grinding, and speed of ball mill reactor rotation. Three replicates were made at each test condition, giving a total of 36 samples. Table 4.1 shows the design of the experiment.

Table 4.1: Design of Experiment for Ball Mill Grinding Tests

Factor	#	Levels
Duration	3	5, 10, 20 min
Speed of Rotation	2	45, 60 rpm
Material	2	Raw, Torrefied
Material mass	1	30 g
Replicates	3	
Total # of Runs	36	

Six screen sizes were selected for the material sieving: 1.4mm, 1.18mm, 0.85mm, 0.71mm, 0.5mm, and 0.3mm. The ground corn stover samples were sieved using a Ro-Tap Testing Sieve Shaker. The untreated samples were sieved for 15 minutes; the torrefied samples for 10 minutes. Sieving times varied due to the increased brittleness and reduced particle size of the torrefied material. These samples were found to pass through the sieves with more ease than the non-torrefied material. The samples were then weighed and a mass percentage was found for each screen size. With the grinding of the material there was some mass loss associated with each sample. Untreated corn stover samples had an average mass loss of 3% while the torrefied samples had an average loss of 4%.

4.3.6. Statistical Analysis

To analyze the data, Minitab software will be used to run the General Linear Model (GLM). For the statistical analysis, p-value testing will be used with an α-value of 0.05. The null hypothesis H_0: $p > 0.05$ will be accepted if $p > 0.05$ and will state that the particle size of torrefied and raw material are not statistically different. The alternative hypothesis H_a will be accepted if $p < 0.05$ and will state that the particle size of torrefied and raw material are statistically different.

4.4. Results and Discussion

Figures 4.4 and 4.5 show the results for both the untreated corn stover and the torrefied corn stover at 60 rpm. Figure 4.4 shows the untreated corn stover results. The plot shows the cumulative distribution of material. The distribution of material changes very little between the different screen sizes. Less than 5% of the material was reduced below the 1.4mm screen. A 95% confidence interval is included. The plot shows that the change in grinding time does not produce a statistical difference. The data from the ball milling showed little variation between replicates of the same test conditions.
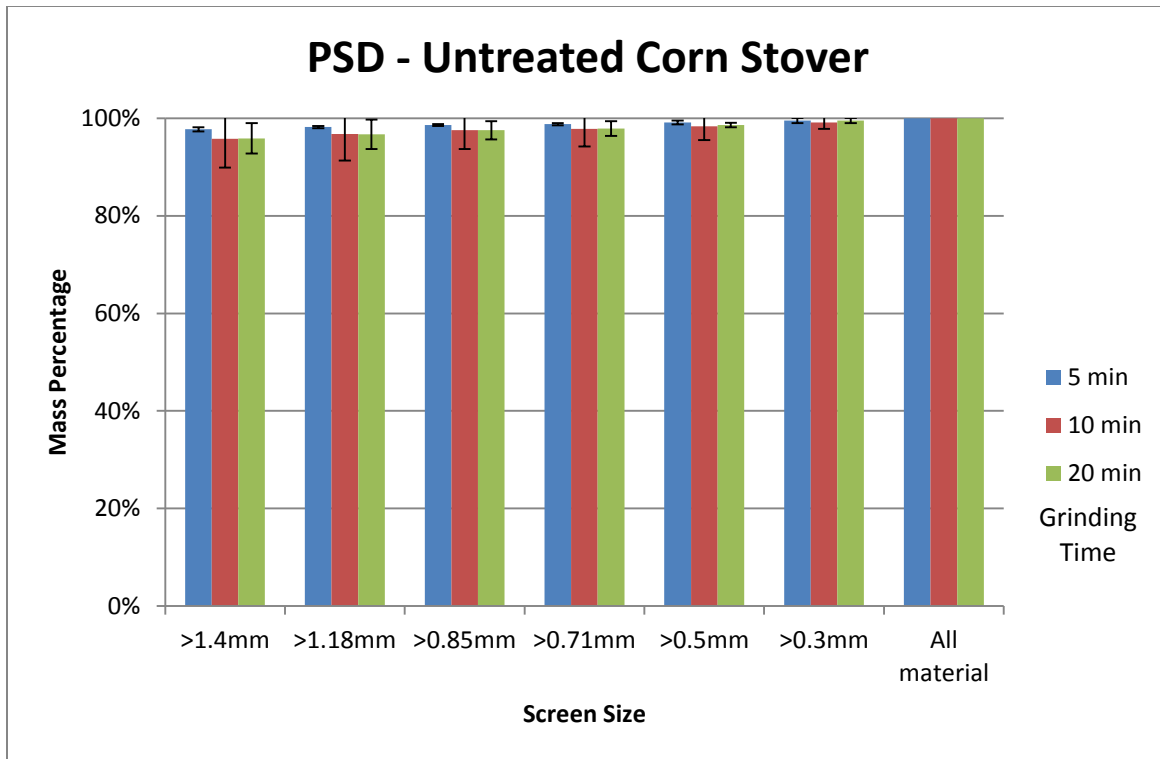
Figure 4.4: Particle Size Distribution for Untreated Corn Stover

Table 4.2: Untreated Corn Stover Analysis of Variance

Source	DF	Seq SS	Adj SS	Adj Mean Square	F ratio	Prob. >F
Screen Size	6	0.0081447	0.0081447	0.0013575	15.55	0.000
Time	2	0.0011017	0.0011017	0.0005509	6.31	0.003
Error	54	0.0047136	0.0047136	0.0000873		
Total	62	0.01396				

S=0.00934282 R^2 =66.24% R^2 (adj)=61.23%

Figure 4.5 shows the cumulative distribution of material for the torrefied corn stover. The plot shows that for grinding times of 5, 10, and 20 minutes, 29%, 13%, and 4% of the material remained larger than the screen size of 1.4mm. The plot shows the increasing trend in the amount of material that passes through as the screen sizes decrease in size. As the time of grinding increases, the average particle size continues to decrease. The trend shows that over time, very little of the original material will remain intact. The results give support to the idea of a self-selection torrefaction system. The torrefied material will break off from the

inner layers and will expose the non-torrefied material. The error bars from this plot show a higher level of variation. The smaller particle size also increased the mass loss during handling and measurement which accounted for some of the error.

For the 20 minute ball milling tests, one of the three samples was removed from the analysis. The data was inconsistent with the other two samples and created a significant amount of error in the data. Inspection of the material showed that a large corn cob remained unground in the sample. The samples were randomly selections of corn stover, thus having a corn cob with the material sample was acceptable. However, the cob was found to be lighter in color than the rest of the material sample and was not torrefied to the same extent as the rest of the sample. The brittleness of the cob was not consistent with the material used for ball milling and was concluded to be an inaccurate representation. Additionally, the cumulative particle size distribution was affected at each of the screen sizes. By increasing the mass percentage of the largest screen size, the other screen sizes experienced a decrease in mass percentage and also experienced an inaccurate level of uncertainty. The sample was concluded to be an outlier and removed from the data analysis.

Figure 4.5: Particle Size Distribution for Torrefied Corn Stover
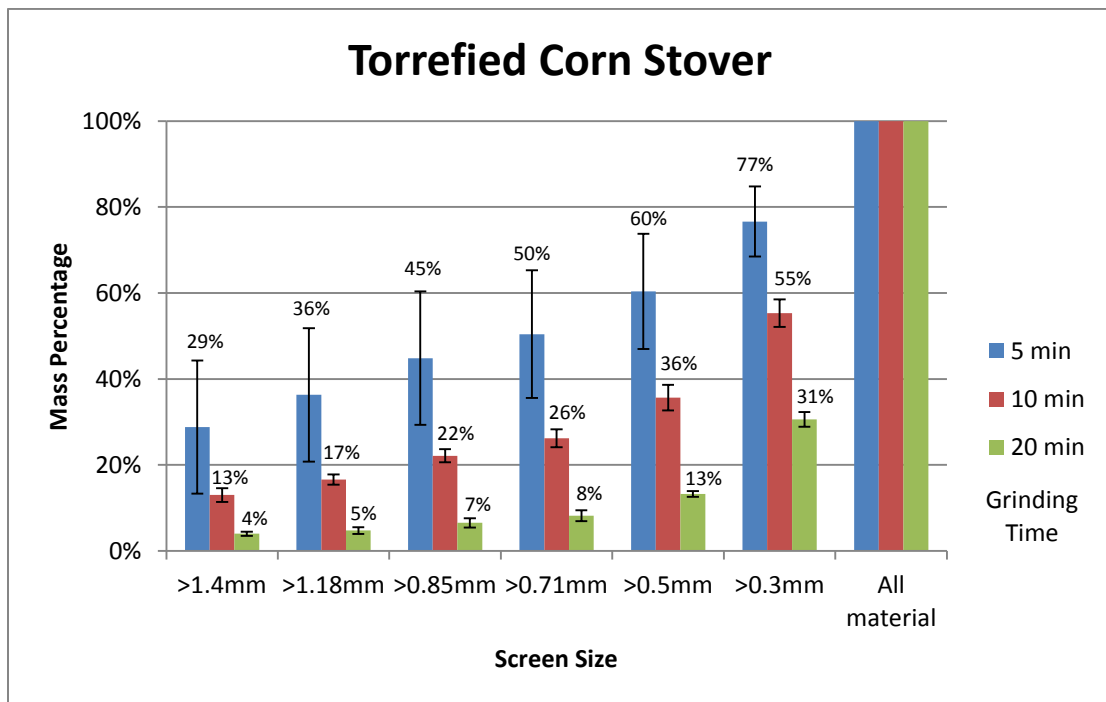
Table 4.3: Torrefied Corn Stover Analysis of Variance

Source	DF	Seq SS	Adj SS	Adj Mean Square	F ratio	Prob. >F
Screen Size	5	4.03046	4.03046	0.67174	128.9	0.000
Time	2	0.943	0.943	0.4715	90.48	0.000
Error	47	0.24492	0.24492	0.00521		
Total	55	5.21839				
S=0.0721884 R^2=95.31% R^2(adj)=94.51%						

The torrefied corn stover was found to be more brittle than the untreated corn stover. When the corn stover samples were run through the ball mill reactor the torrefied material resulted in a more finely ground product than the untreated material. The speed of the ball mill reactor rotation and the duration of the ball mill testing were found to have a greater effect on the torrefied corn stover than on the untreated corn stover. The main factor influencing the grindability of the material was the torrefaction treatment of the corn stover.

Previous experiments have used energy consumption measurements as a means of quantifying grindability of biomass (Repellin et al., 2010). Other studies have used the Hardgrove Grindability Index (HGI) test to compare treatments of biomass (Bridgeman et al., 2010). For this study, the Particle Size Distribution of the ground corn stover was used to compare the effectiveness of torrefaction treatment. For the ball mill reactor, measuring the power consumption would not have yielded significant differences for the different treatments. With each sample having an identical mass, the drum roller would not have experienced a difference in load conditions. Sieving of the ground corn stover allowed for analysis of the particle size distribution of the material.

For the untreated corn stover there was very little difference between the grinding times of 5, 10, and 20 minutes at 60 rpm (Figure 4.4). The majority of the material remained unground and above the 1.4mm screen size. The fibrous nature of the corn stover prevented the ball mill reactor from reducing particle size. The torrefied corn stover resulted in a greater percentage of material being ground below the largest screen size of 1.4mm. The trend in the data showed that the longer the grinding period, the smaller the final particle size of the material. For the 60 rpm tests, the percentage of material above the 1.4mm screen size

was reduced. For times of 5, 10, and 20 minutes, 28.8%, 13%, and 3.99% of the material remained above 1.4mm, respectively. With over 95% of the untreated corn stover remaining unground, there was very little material collected by the 1.18mm and smaller screen sizes. The trend for the torrefied corn stover showed an increase in mass percentage as the screen size decreased.

4.4.1. Material Purity Calculations

The results from the ball milling of the material showed that non-torrefied material will experience less reduction in particle size than torrefied material. A comparison of non-torrefied to torrefied material was calculated as material purity. For the ball milling tests conducted, the torrefied material and non-torrefied material were ground separately. However, in the self selection process, there will be a mixture of torrefied and non-torrefied material as the material is being processed. One objective of this research is to develop a method for separating torrefied material from non-torrefied material in the self selection torrefaction reactor. The torrefied material will be removed by a screen in the reactor and the non-torrefied material will remain in the reactor until torrefied.

In order to select the proper screen size, a separation point needed to be found that would allow for the torrefied material to be separated while minimizing the contamination of non-torrefied material. Using the data from the ball mill tests, a material purity level was found for each sieve screen size. This calculation shows the fraction of non-torrefied material compared to the sum of torrefied and non-torrefied material for each screen size. This contamination level will identify the optimal screen size for separating torrefied material. The contamination level data will also be used for quantifying results found in the self selection torrefaction reactor described in Chapter 5.

$$\text{Contamination level} = \frac{\text{Raw material}}{\text{Raw material} + \text{Torrefied material}}$$

Table 4.4: Contamination level of non-torrefied material

	>1.4mm	>1.18mm	>0.85mm	>0.71mm	>0.5mm	>0.3mm
5 min	1.4%	1.4%	1.0%	0.9%	0.6%	0.3%
10 min	2.3%	3.0%	2.2%	1.8%	1.3%	0.6%
20 min	4.1%	3.3%	2.4%	2.0%	1.2%	0.4%
Mean	2.6%	2.6%	1.9%	1.6%	1.0%	0.4%

Table 4.4 shows the percentage of non-torrefied material at each screen size of separation. The trend among the data shows that as the screen size decreases, the contamination level decreases. Likewise, as the time decreases, the level of contamination for each screen size decreases. This is due to the fact that the non-torrefied material experiences greater particle size reduction as time increases. As the time increases, the torrefied material will breakdown to smaller particle sizes than the non-torrefied material. Because of this greater rate of particle size reduction, the larger screen sizes will see a higher level of non-torrefied material and a higher level of contamination. The 10 min and 20 min periods produce similar levels of contamination while the 5 min period remains lower. This follows the hypothesis of torrefied material being more brittle than non-torrefied material. The torrefied material will reduce in size more rapidly than the non-torrefied material. As the time of ball milling increases, the percentage of non-torrefied material that has reduced in size increases. Figure 4.6 shows the contamination levels for each screen size at the three time periods. Even at the highest level of contamination at the 1.4mm screen size, the level is still below 5% contamination and shows that separation of material is possible.
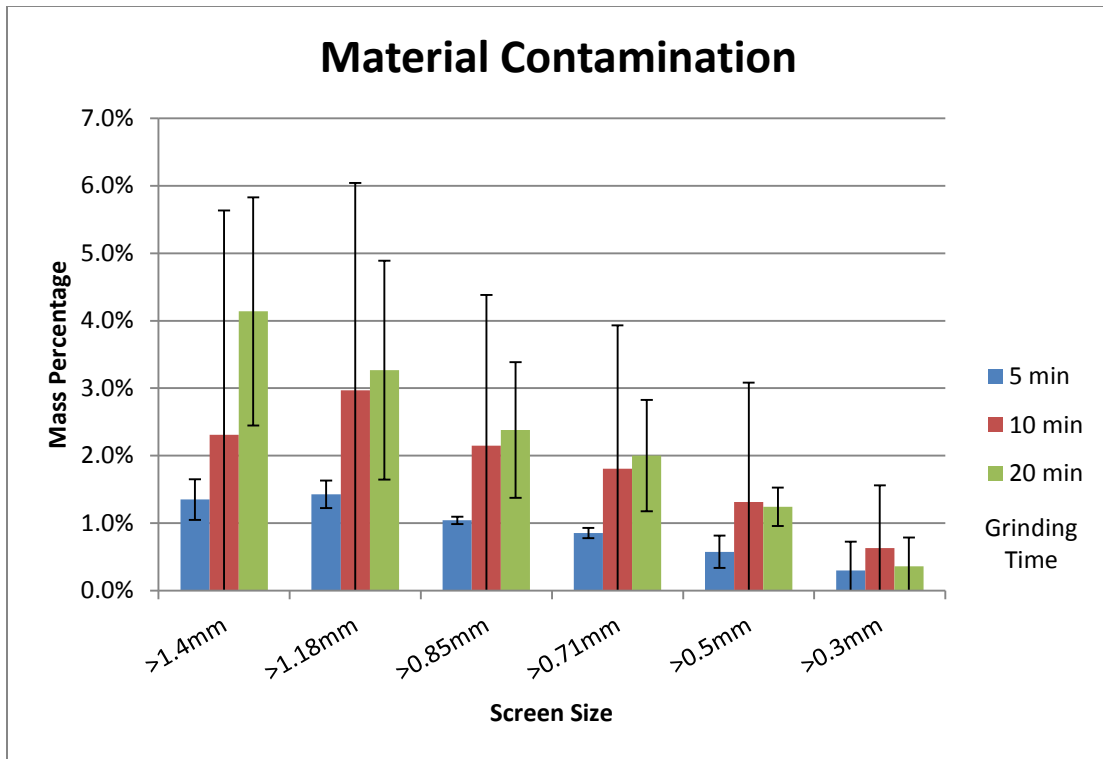
Figure 4.6: Material Contamination

Table 4.5: Contamination level Analysis of Variance

Source	DF	Seq SS	Adj SS	Adj Mean Square	F ratio	Prob. >F
Screen Size	5	0.0013198	0.0013198	0.000264	8.41	0.000
Time	2	0.0002596	0.0002596	0.0001298	4.13	0.022
Error	46	0.0014439	0.0014439	0.0000314		
Total	53	0.0030233				

S=0.00560257 R^2=52.24% R^2 (adj)=44.97%

4.4.2. General Linear Model

From the General Linear Model (GLM), the significant terms for all tests were material treatment and duration. Of the significant terms, the material treatment was found to have a statistically significant impact (at an α-value of 0.05). This shows that torrefaction of corn stover will result in a different particle size distribution compared to untreated corn stover.

Table 4.3 shows that the factors of screen size and time are significant terms for the grinding of the torrefied material. From this table it is shown that there is strong evidence the mean values of the torrefied and non-torrefied particle sizes are not equal. The ANOVA table includes 7 screen sizes, 3 time factors, and 3 replicates. As previously mentioned, one of the torrefied samples from the 20 minute test was removed. This created an unbalanced ANOVA model; a general linear model was used to find the adjusted sum of squares and mean squares. With the p-value for the factor of screen size being less than 0.05, the null hypothesis H_0 was rejected and the alternative hypothesis H_a was selected. The torrefied material was found to create particle size that is statistically different from raw, non-torrefied material.

4.5. Conclusion

Torrefaction increases the brittleness of the material by breaking down of hemicellulose and lignin content. Treatment of biomass by means of torrefaction will improve material grindability. In this work, torrefied corn stover, when ground in a ball mill reactor, resulted in smaller particle sizes compared to untreated corn stover. The factors of speed of rotation and duration of grinding also impact the distribution of particle sizes. As the duration of grinding increased, the particle size of the torrefied corn stover became smaller. The greater the speed of rotation, the smaller the particle sizes will become. However, this factor had less impact than the material treatment and duration of grinding.

The untreated corn stover was found to be mostly unaffected by the duration of grinding or speed of rotation. This data shows that the grinding of untreated and torrefied corn stover will result in distinctly different particle size distributions. This gives support to the feasibility of a self-selection torrefaction system. It is possible to distinguish torrefied particles from untreated particles on the basis of particle size. This will allow for material to be separated during the self-selection process by use of a screen system. The breakdown of the torrefied material will be dictated by the degree of torrefaction and the physical properties which it exhibits.

4.6. Acknowledgements

4.7. References

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Chapter 5. Self Selection Torrefaction Reactor System

A year-end report prepared for ConocoPhillips

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5.1. Introduction

The design of the torrefaction reactor was based upon the concept of developing a self selection torrefaction system. Two main components made up the self selection torrefaction system: the torrefaction system and the ball mill reactor system. The torrefaction system would be responsible for the heating of biomass in an inert environment while the ball mill reactor would reduce the particle size of biomass. Together these systems would address the central hypothesis that as large particles become torrefied, the outer layers will become brittle and be removed by a ball mill reactor. The inner layers will be exposed and allowed to torrefy more easily.

The torrefaction chamber was constructed out of 12 gauge steel. This would allow for the system to sustain the high heating temperatures and positive pressure created by purging the system with nitrogen. The dimensions of the main chamber were 0.9m length by 0.9m width by 0.8m high. The chamber has a removable front panel, allowing for modifications to be made to the internal components of the system. Additional components within the reaction chamber include the ball mill reactor, heating elements, purge gas system, fan system, and thermocouples.

5.2. Objectives

The objective of this research was to design a self selection torrefaction system. The design of the system was based upon the results observed for the properties of material grinding. In that testing, untreated and torrefied material were separately placed in a ball mill reactor. The parameters tested included duration of ball milling, speed of rotation, and

material type. These parameters were examined to identify the operating limits that would allow for material to be reduced in size. The primary focus was to observe the impact of torrefaction on the particle size reduction.

The focus of the testing in Chapter 5 was to take the information obtained in the previous testing and design a self selection torrefaction system that would integrate the torrefaction and ball milling stages into one process. Initial testing will be conducted in the same manner as previous tests. Material will be torrefied prior to ball milling. This will be done to confirm that the ball milling action of the self selection torrefaction system operates similarly to the previous ball mill testing apparatus.

Once the operation of the ball mill has been confirmed, the system will be tested with simultaneous torrefaction and ball milling stages. The particle size distribution of the material will be used to compare the self selection process to the previous testing of separate torrefaction and ball milling.

5.3. Materials and Methods

5.3.1. Process Design

The initial design of the torrefaction reactor focused on the design and interaction of the components in the system. It was known that electric heaters would be used as the heating source for the system. The laboratory where the torrefaction reactor was housed was equipped with sufficient electrical power to run the reactor. A nitrogen injection system would be used for purging the system. A vacuum pump system would be installed for removal of air within the system prior to the purging stage. A fan system would also be installed to improve heat transfer within the chamber and to ensure the ball mill reactor would reach acceptable temperatures for torrefaction. A data collection system would utilize thermocouples for temperature collection. Figure 5.1 shows the initial process flow diagram for the system.
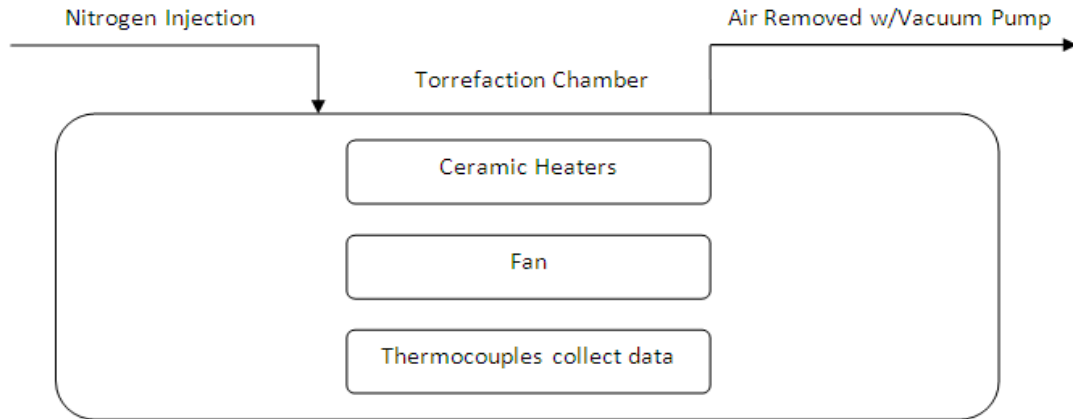
Figure 5.1: Torrefaction Reactor Process Flow Diagram

5.3.2. Purge Gas System

A vacuum pump was installed in order to remove the oxygen from the torrefaction chamber prior to heating of material. Once a vacuum was created within the chamber, nitrogen was purged into the system to create an inert environment. An oxygen sensor was installed to monitor levels within the chamber. During the torrefaction process, volatile gases are driven off from the biomass, creating a potential pressure buildup within the chamber. A pressure transducer monitored the pressure levels and regulated an exhaust valve. The exhaust valve removed the buildup of volatile gases and allowed for the collection of liquid phase volatiles.

Shakedown testing of the chamber pressure regulation system was conduction prior to running torrefaction tests. Two separate tests were conducted in which the chamber was exposed to positive pressure values of up to 1 psi and a vacuum of -0.2 psi. For the initial shakedown testing, an air compressor with pressure regulator was connected to the chamber. For vacuum testing an electric high vacuum pump was installed. This vacuum pump would also serve as the pump used during torrefaction tests. A response curve was plotted for both tests and is shown in Figures 5.2 and 5.3.
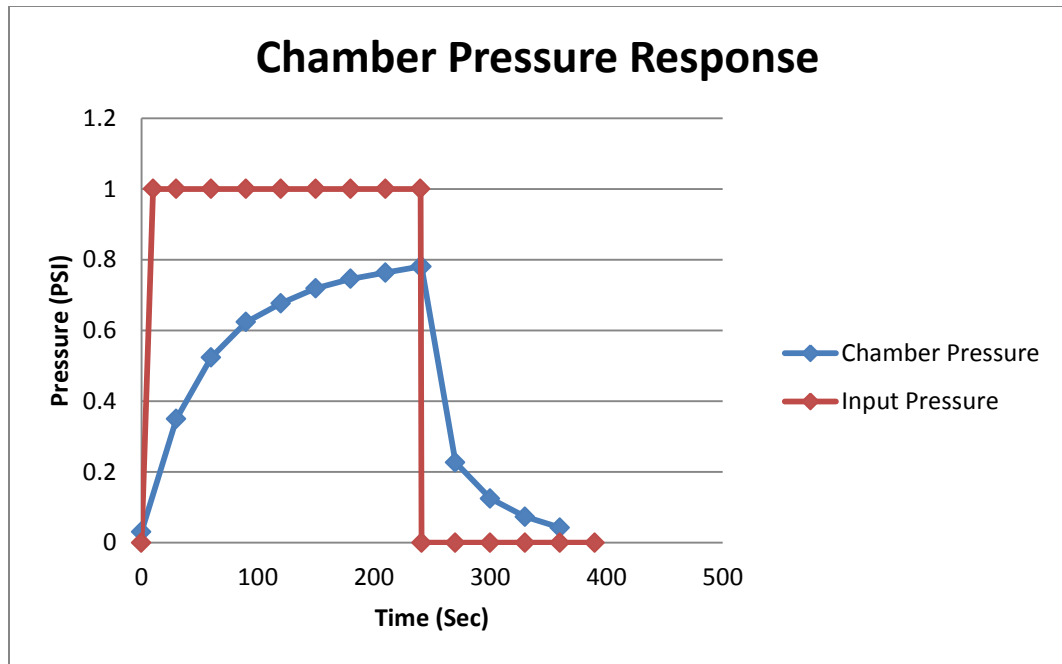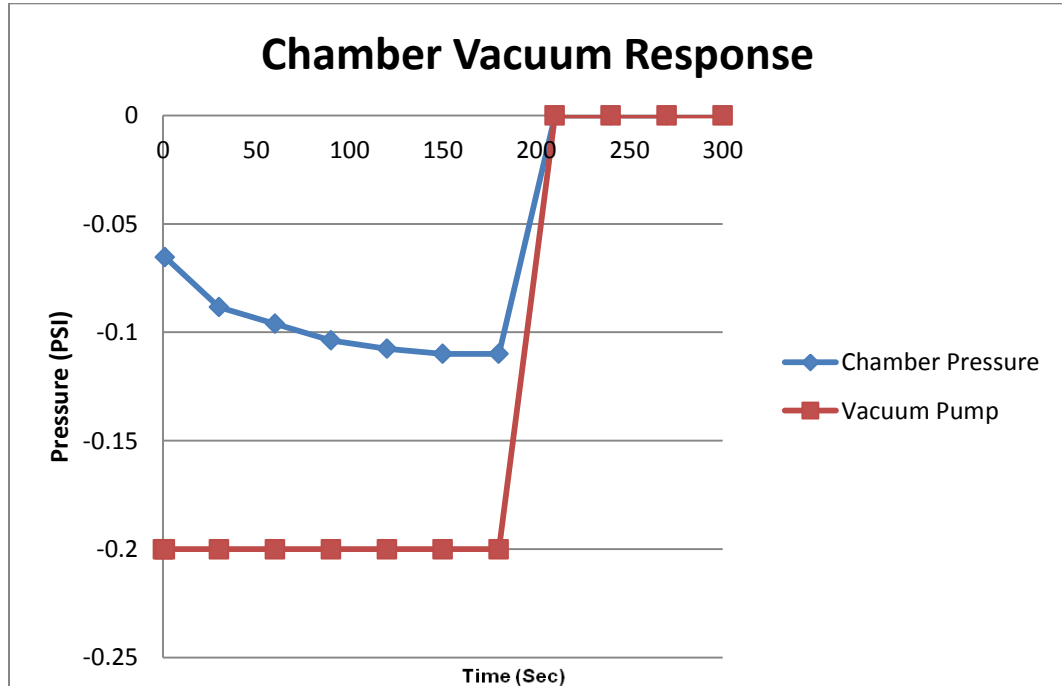
Figure 5.2: Chamber Pressure Response Curve

Figure 5.3: Chamber Vacuum Response Curve

The response curve shows the chamber's ability to hold a positive pressure while being supplied with nitrogen. There is some delay in the response of the chamber pressure level, however, this delay is only a few minutes and will not be a concern during testing. Likewise, the vacuum response experienced a delay of only a few minutes and will not be a concern. Based upon these results and an inspection of the chamber it has been concluded that the system is not perfectly sealed and will experience some leakage of gas during testing. Gaskets have been installed to minimize this but have been unable to eliminate all leakage. However, the chamber will be held to a positive pressure during the torrefaction process and will be able to maintain an inert environment.

In order to control nitrogen levels during the torrefaction process, a purge gas system was installed. Nitrogen tanks were connected to pressure regulators and a series of solenoid valves. The solenoid valves were integrated into the control system to allow for automated control of the system. The monitoring system would read oxygen and pressure levels within the chamber and control nitrogen flow as needed. Nitrogen lines were installed inside of the chamber to direct nitrogen flow directly into the ball mill reactor.

To account for pressure buildup within the chamber an exhaust system was installed. The system was expected to maintain a positive pressure due to the nitrogen purge gas; however, pressure was monitored to ensure excessive pressure buildup was prevented. A solenoid valve would be opened when pressure levels went above a desired set point.

5.3.3. Heating System

Heating of the torrefaction chamber was achieved by use of electric heaters. Based upon the volume of the reaction chamber and the temperature requirement of 260-280°C, a series of six, 2800 Watt strip heaters were installed. The heaters had a length of 0.78m and had a series of fins attached to improve heat transfer to the surrounding environment. Each heater ran off of 240V power supply.

To improve airflow and heat transfer throughout the reactor, a fan system was installed in the reactor. A centrifugal fan system was used to move air within the reactor. A series of six 0.13m diameter fans were installed in the upper corner of the reaction chamber. Three of the electric strip heaters were placed directly in front of the fans. This would allow maximum

airflow across the heaters and increase the heat transfer throughout the rest of the chamber. The remaining heaters were placed in each of the other corners of the chamber. By placing the heating elements in each of the corners of the chamber, the ball mill reactor would be in the center of all heating elements.

With the torrefaction chamber having such a large area, testing was conducted to record the temperature profile. The chamber was equipped with a series of 30 thermocouples. 27 of the thermocouples were positioned within the chamber in a 3 x 3 x 3 grid structure. The remaining 3 thermocouples were placed in the biomass collection region below the reaction chamber. By collecting a temperature profile within the chamber it would be possible to identify 'cold regions' where the temperature would not reach sufficient torrefaction temperatures. The primary area of focus was the center of the chamber where the ball mill reactor would be placed. The design requirement for the system was to heat material to a temperature range of 260-280°C. If the central region of the chamber could sustain these temperatures the design requirements would be met. Figure 5.6 shows the profile of the chamber temperature collection system.

5.3.4. Chamber Insulation

In order to maintain the required temperatures within the chamber, insulation was added to the exterior walls of the reaction chamber. Insulation of the system would be necessary as the chamber had an outer surface area of just over 5 m^2. The thermocouple probes, nitrogen purge gas lines, motor drives, and electrical connections for heating elements attached to the exterior walls made insulating more challenging. Additionally, the insulation would have to be removed every time the front panel of the chamber was removed.

Two types of insulation material were selected for the chamber: calcium silicate insulation sheets and ceramic insulation rolls. The calcium silicate had a maximum temperature rating of 650°C and had a K factor of 0.73 W/m °C (rated at 426°C). These sheets were rigid and were used on the outer walls of the chamber where the insulation would rarely be removed. The sheets were cut to size to mold around the thermocouple and nitrogen line ports. The ceramic insulation was a blanket that could easily be removed from the chamber and could conform to irregular shapes. The ceramic insulation had a maximum

temperature rating of 1100°C and had a K factor of 0.65 W/m °C (rated at 426°C). This material was used on the removable front panel of the chamber and on the sections where the calcium silicate sheets could not fit.

5.4. Control System and Data Acquisition

5.4.1. Control Panel Box

An initial layout of the control box was sketched to show the available space for all the design components. A large control box was selected to not only hold necessary components for the system but also to allow for future expansion of the system. The control box contained the motor controls, relay switches, data acquisition system, and connections for heating, purge gas and exhaust systems.

To control the electrical devices such as heaters, vacuum pump, and solenoid valves, twelve solid state relay switches (Automation Direct, AD-SSR225-DC) were installed. The relays were selected based upon their voltage capacity (120/240 VAC) and their 25 Amp output current rating. The relays were switched from a signal received from the DAQ board.

A custom circuit board was developed to act as in interface between the DAQ board, manual user controls, and relay switches. The circuit board was designed by Dr. Matthew Darr and produced by Advanced Circuits. The circuit board utilized MOSFET chips (Metal Oxide Semiconductor Field Effect Transistors) to read in a signal from either the DAQ board or the manual switches and provide a signal to the relay switch. The MOSFET would serve as a signal amplifier to provide sufficient current to the relay switches (output current from DAQ board would not be sufficient). The circuit board was able to simplify the wiring of the control system into a single compact unit.

The largest items would be the VFD controls for the electric motors. Four VFD boxes were installed. The fan system and ball mill reactor would each have an electric motor and would require VFD control. The two additional VFD controls were installed for potential future expansion of the torrefier reactor such as the addition of a feed system, allowing for continuous flow as material is added and removed from the system during operation. The VFD controls were selected from Automation Direct and operated under 230V AC power.
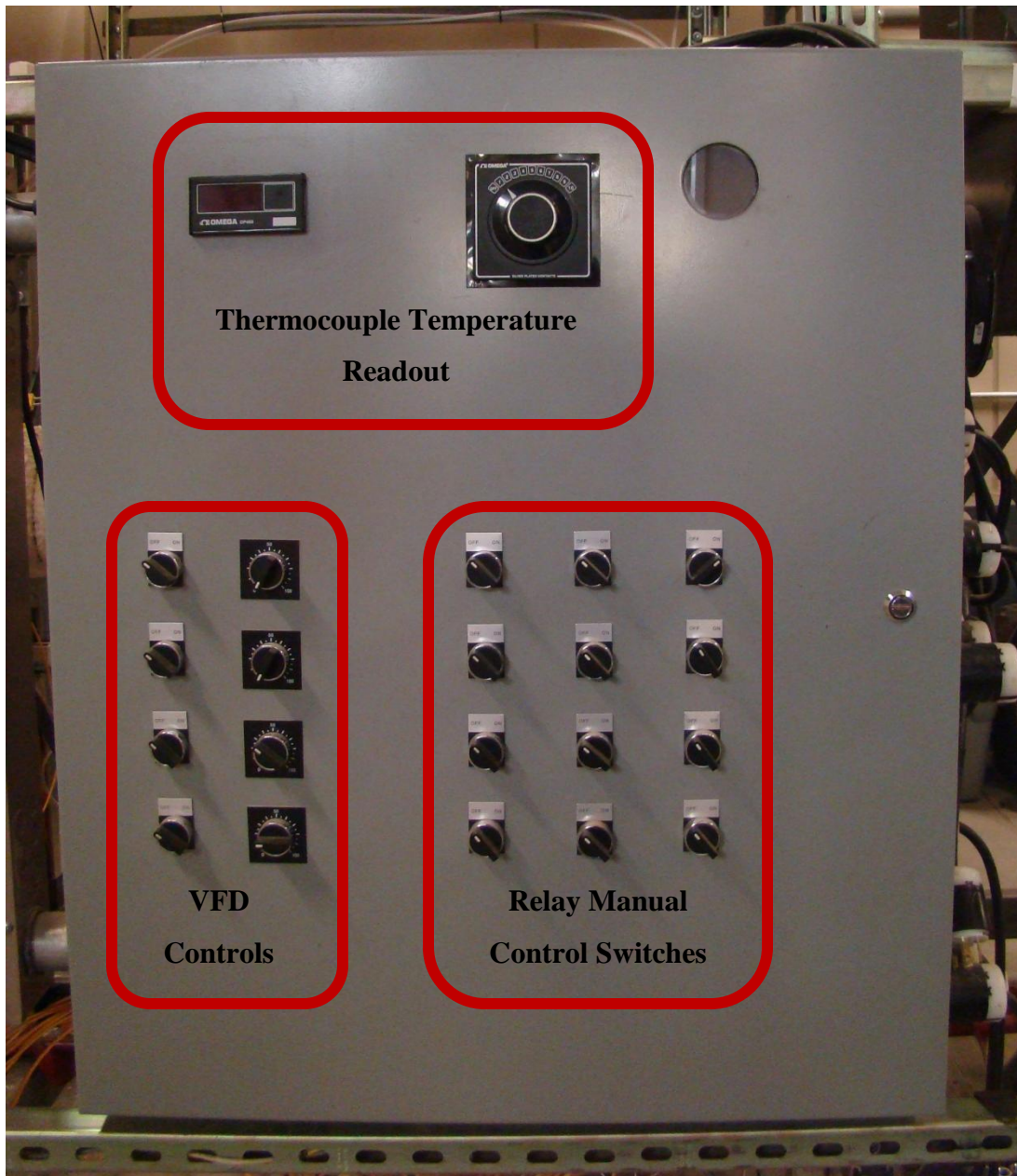
Figure 5.4: Control Panel Exterior Controls
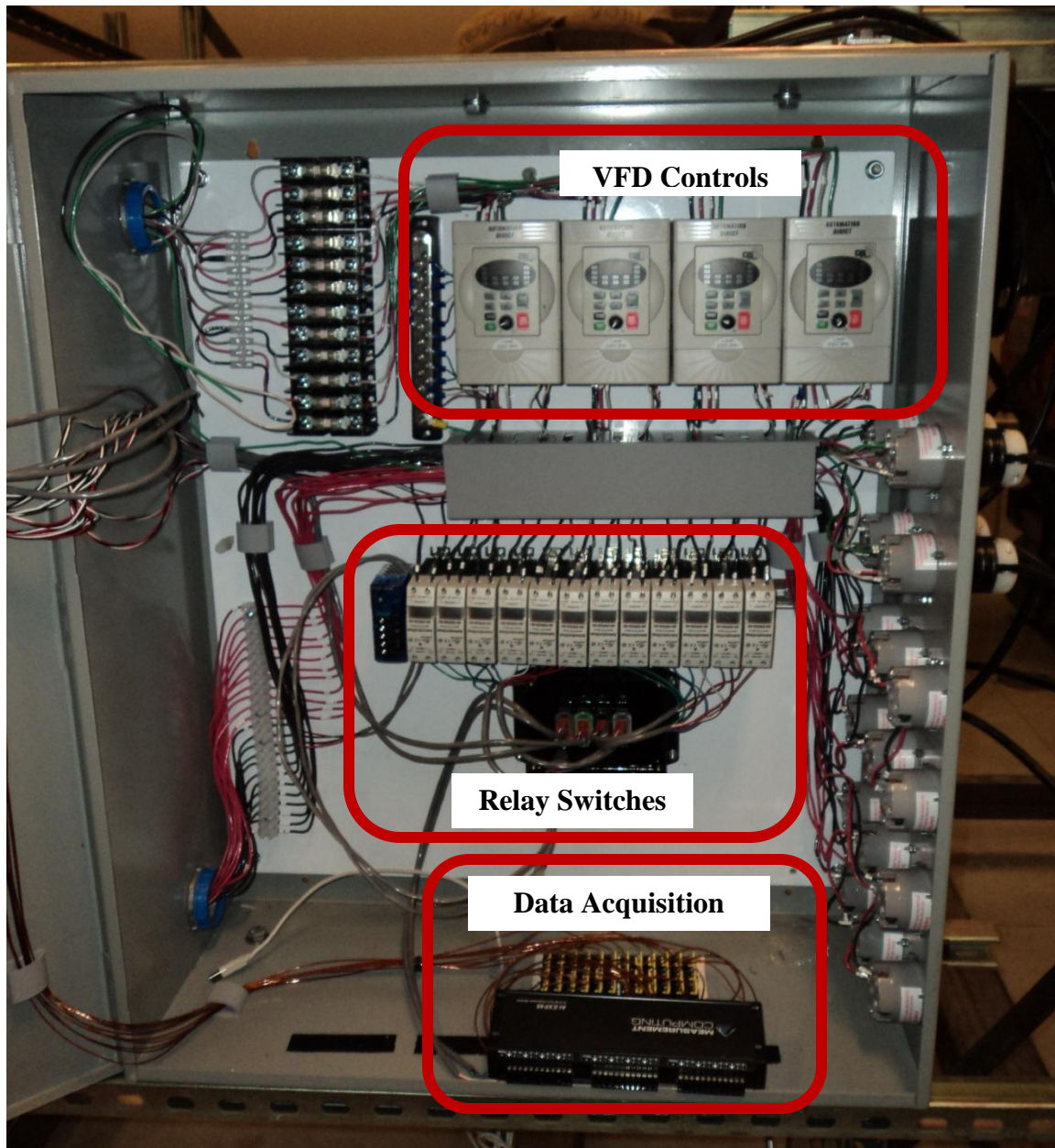
Figure 5.5: Control Panel Interior Controls

5.4.2. Thermocouple Probes

K type thermocouples were selected for the temperature readings in the torrefier. These thermocouples observed temperature and sent an analog signal to the DAQ board, which would then be displayed as a temperature. K type thermocouples probes were selected based upon their ability to handle a maximum temperature of 1300°C. The probes also provided accurate readings with very little temperature drift (under 2.8°C). The stainless

steel probes had a diameter of 6.35mm (0.25in) and lengths of 15cm, 30cm, and 60cm (6in, 12in, and 24in respectively) were selected. The probes were bendable to fit inside of the reaction chamber as needed but sturdy enough to maintain their shape.

5.4.3. Oxygen Sensor

To monitor oxygen levels within the chamber an oxygen sensor was installed. Figaro Sensor offered two oxygen sensors, the KE-25 and KE-50. The KE-25 was found to have a shorter response time than the KE-50 (14 seconds vs 60 seconds for 90% response) but offered a lower output voltage (0-60mV vs 0-250mV). The response time was not a critical factor but the higher output voltage would provide simplified data acquisition. The KE-50 was selected based upon this criterion. The sensor did not come with a calibration equation but a calibration curve was provided to match output voltage to oxygen concentrations. From the provided calibration curve a linear relationship was calculated and used with the Labview control system to report oxygen concentrations based upon input voltages. The input analog signal (Y_{analog}) was converted to a digital signal.

$$X_{digital} = 10 * \frac{Y_{analog}}{2^{16}} - 5$$

The $X_{digital}$ signal was converted into an oxygen concentration value by the following calibration equation.

$$Oxygen\ Concentration = (X_{digital} * 277.77) + 4$$

5.4.4. Labview Control Program

Data acquisition and control of the system were regulated through Labview. The Labview control program would simultaneously monitor conditions within the chamber, such as temperature, pressure levels, and oxygen levels, while regulating heaters, nitrogen flow,

and exhaust flow. The control system was designed with the ability to be run either in an automated mode controlled via Labview or in a manual mode, controlled by a series of switches located on the torrefier control panel. The system was designed with a manual control mode to allow the user the ability to change control parameters as necessary during operation without having to alter the software.

A Data Acquisition (DAQ) board was selected to interface between the Labview control program and the torrefier. The large number of thermocouples and components being controlled by the system required a DAQ board that was supplied with sufficient input/output channels. The USB-1616HS-4 board (Measurement Computing) was selected. The AI-EXP48 board was used to expand the number of input channels available. In total, the DAQ boards had the ability to handle 32 differential ended analog inputs and had 24 digital Input/Output channels.

The control program was designed in Labview to provide a Graphical User Interface (GUI) that allowed monitoring of the system performance and allowed user inputs for control parameters. The GUI showed the temperature at selected locations within the reaction chamber as well as the pressure level and oxygen concentration. The user has the ability to enter in a desired value for temperature set point, maximum pressure level, and Oxygen concentration. The GUI indicates which heaters are turned on, which solenoid valves are open, and if the exhaust system is engaged. The system also will plot the temperature for the chamber and record data for analysis.

5.4.5. Heating Shakedown Testing

For shakedown testing the chamber was sealed shut and heated with no biomass material present. The data plot was broken down into three regions: upper temperature, center temperature, and lower temperature. The electric heaters were turned on until the system either reached the required temperature range of 260-280°C or reached an equilibrium temperature below the desired range. The plot in figure 5.7 shows the recorded data for the heating test. After a period of 2 hours the system was found to successfully reach torrefaction temperatures. There was a large temperature gradient within the chamber as the bottom of the system only reached 100°C while the top level reached 350°C. This was due

to the fact that the top thermocouples were placed within close proximity (10cm) of the heater and fan system. The lower thermocouples were placed in the collection region where no heating elements were present. Figure 5.6 shows the profile of the torrefaction chamber with the three regions (top, middle, lower) where the thermocouples were placed.
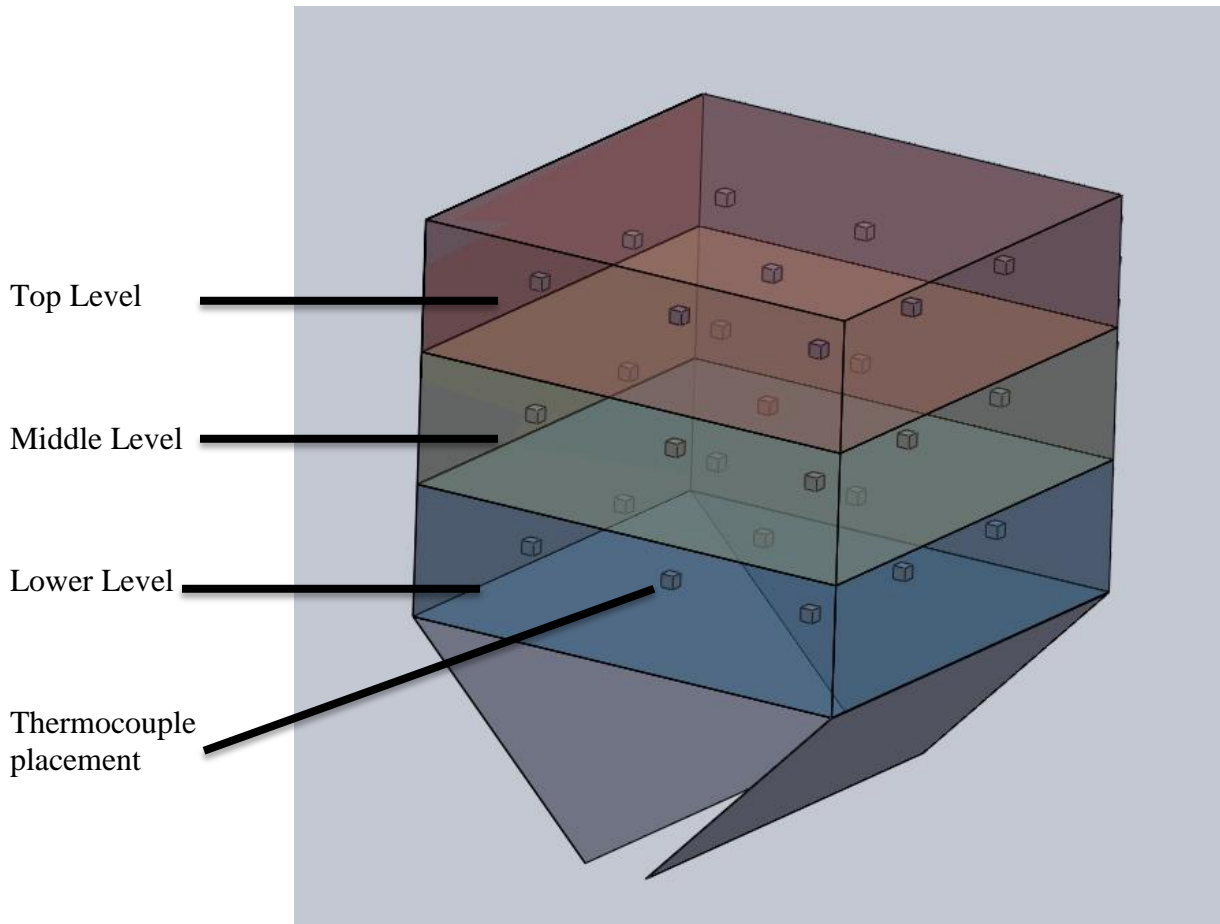
Figure 5.6: Chamber thermocouple placement

However, the temperature gradient was found to be advantageous for the torrefaction process. With the ball mill reactor installed, biomass will be torrefied and will then pass through a screen, allowing the material to collect in the lower region of the chamber. At this point the material will no longer require additional heating and lower temperatures will initiate the cooling process for the material. Testing has shown that torrefied material above 200°C has the potential to ignite when exposed to oxygen in atmospheric air conditions. The

lower exit temperature will ensure that material will be adequately cooled before removal from the reaction chamber. Figure 5.7 shows the upper region of the chamber reached 260°C in about 30 minutes. The center region, where the biomass material will be located, required about 90 minutes to reach 260°C. The design requirement for reaching torrefaction temperatures of 260°C-280°C were met for this system.
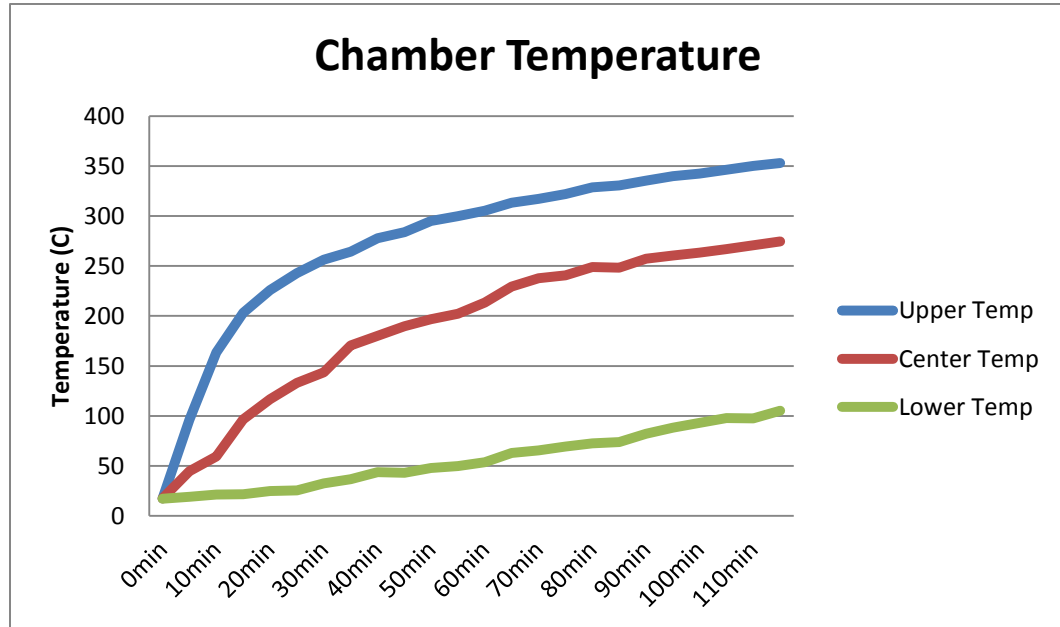
Figure 5.7: Torrefaction Chamber Temperature Response

5.5. Design of Self Selection Torrefaction Reactor

The objective of the self selection torrefaction reactor testing was to validate the operation of the system. Design of the self selection reactor was based upon results from the grinding tests previously conducted. The testing of the system was focused primarily on conceptual objectives. The particle size distribution data and purity calculations obtained in Chapter 4 provided information for analytical comparison of material quality. However, this does not give a complete analysis on the degree of torrefaction. Visual comparisons of material color were used as a metric for determining torrefaction levels. This can be used to compare between samples of biomass torrefied at different temperatures or to evaluate the degree of torrefaction within a single particle. Dense biomass particles, such as corn cobs, have a noticeable color gradient from the outer layers to the core. This gradient gives

evidence for the benefits of a self selection torrefaction system. Integration of a ball mill reactor will produce a final product that is uniform in the degree of torrefaction.

Figure 5.8: Cross section of torrefied corn cob

5.5.1. Ball Mill Reactor

Testing was conducted to investigate the impact of torrefaction on the grindability of corn stover. The specific hypothesis of the experimentation was that the grinding of torrefied and untreated biomass will result in distinctly different particle size distributions. Untreated biomass will be more resistant to grinding than the torrefied material, due to its increased brittleness. The resulting particle size distribution should show that ball milled torrefied corn stover will produce smaller particle sizes than ball milled, untreated corn stover. By creating a separation of particle sizes, torrefied material will have the ability to be separated from non-torrefied material by a use of a screen. During the torrefaction process inside of the ball mill reactor, torrefied material will be mixed with material that is yet to be torrefied. It was necessary to find the particle size distributions of each material type when ground. The

testing conducted in Chapter 4 showed that this distinct difference in particle size distributions exists when torrefied and non-torrefied material are ball milled separately.

Based upon these grinding tests, the ball mill reactor was designed for the self selection reactor. A 75.5 liter (20 gallon) steel drum with a diameter of 0.475m and depth of 0.54m was selected. The size of the container was based upon the dimensions of the reaction chamber. The drum would be large enough to test large samples of corn stover material but would fit between the heating elements within the reaction chamber. The drum had a lid that would seal shut and minimize material loss during testing. During the grinding of torrefied material there is some disbursement of finely ground particles into the air; a sealed container minimized this loss of material.

5.5.2. Test Design

. Material was placed in the reactor, ball milled for a set period of time, and the material was sieved to determine particle size distribution. For this testing the time period was fixed at 20 minutes. The 20 minute period coincides with typical residence times for the torrefaction process. However, for these tests the material will be torrefied prior to ball milling to study only the effect of the grinding process. Prior testing in Chapter 4 examined the parameters of speed of rotation, duration of grinding, and material type. This testing will eliminate the variability in the speed of rotation and duration of grinding. Three types of material were testing: raw, torrefied, and mixed. The mixed material was not previously tested but was added to observe if raw material affected the ball milling of torrefied material. The total mass of the mixed material was 100 grams, with 50 grams of raw material and 50 grams of torrefied material.

The testing was conducted to confirm that ball milling in the self selection torrefaction reactor would operate in a similar manner as the ball mill testing in Chapter 4. The particle size distributions of the samples were used to compare the results. An additional metric for comparing the results of the ball milling was a visual inspection of material. This was used to verify that the mixed material samples would separate large, non-torrefied particles from the smaller, torrefied particles. These results are further discussed in Section 5.5.5.

Table 5.1: Design of Experiment for Self Selection Reactor

Factor	#	Levels
Duration	1	20 min
Speed of Rotation	1	20 rpm
Material	3	Raw, Mixed, Torrefied
Material mass	1	100 g
Replicates	3	
Total # of Runs	9	

Corn stover was collected from the Iowa State University Bio-Century Research Farm. The baled corn stover was divided into 3 groups. The first group was for the raw, non-torrefied tests; it was divided into 100 gram samples and randomly assigned a test number. The second group was torrefied to a temperature of 275°C and then divided into 100 gram samples and randomly assigned a test number. The third group of material was a blend of 50 grams of torrefied material and 50 g of raw, non-torrefied material.
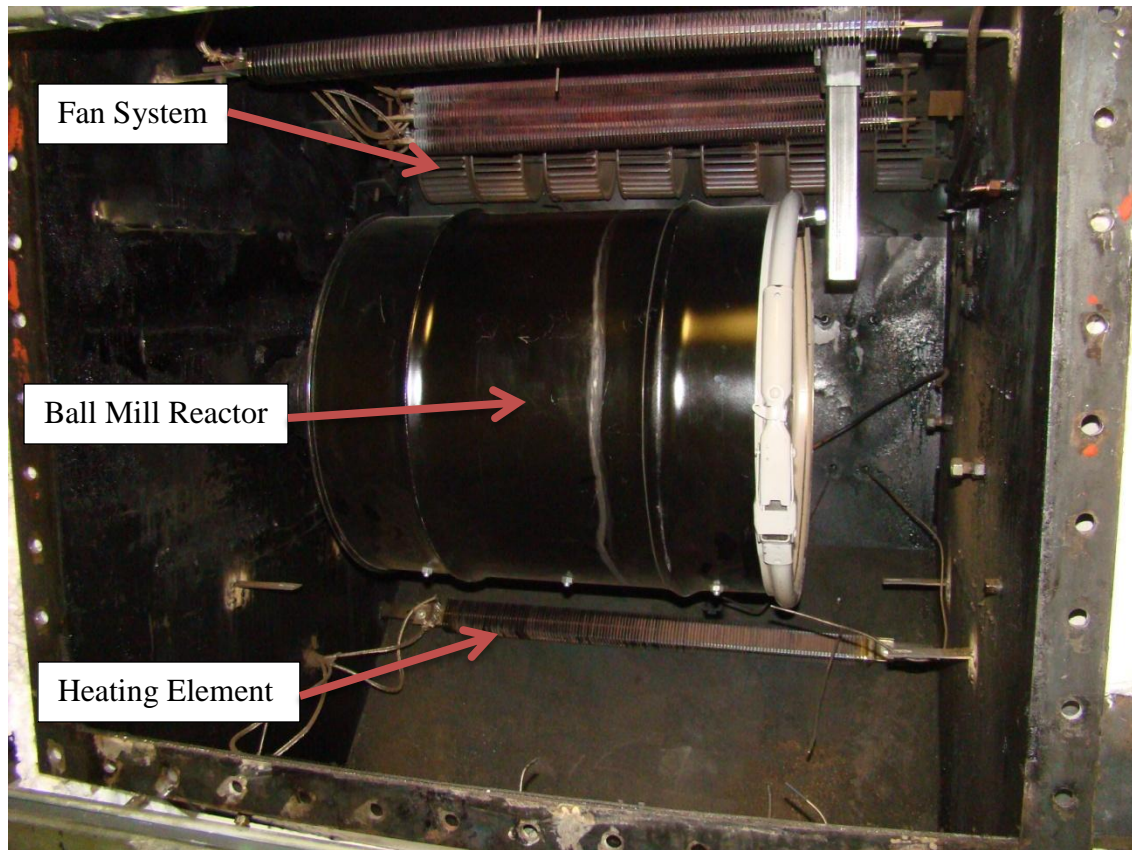
Figure 5.9: Self selection torrefaction reactor

5.5.3. Ball Milling Results

Table 5.2 shows the data for the raw, untreated corn stover. The results from the grinding tests are similar to that of the previous results for untreated corn stover. Once ground, the material was sieved for 15 minutes through a series six screens on a Ro-Tap sieve shaker (Tyler Industrial Products, OH). The size of the screens, descending from largest to smallest were 1.4mm, 1.18mm, 0.85mm, 0.71mm, 0.5mm, and 0.3mm. From the three replicates for untreated corn stover, the average mass percentage above the largest 1.4mm screen size was 94.4%. This is slightly less than the previous grinding results of 96.45% but still shows the lab scale ball mill system will be capable of producing similar results.

Table 5.2: Untreated Corn Stover Particle Size Distribution

Untreated Corn Stover PSD							
	>1.4mm	>1.18mm	>0.85mm	>0.71mm	>0.5mm	>0.3mm	All Material
R-100-1	96.08%	96.70%	97.29%	97.32%	97.94%	98.65%	100.00%
R-100-2	97.18%	97.91%	98.12%	98.35%	98.83%	99.32%	100.00%
R-100-3	97.41%	97.96%	98.43%	98.64%	99.05%	99.46%	100.00%
Mean	96.89%	97.53%	97.95%	98.10%	98.61%	99.14%	100.00%

Table 5.3 shows the results for the blended raw and torrefied samples. This tests represents the intermediate stage between the raw state and the fully torrefied state of the material. This test shows the interaction between the two material types when mixed. The table shows the particle size distribution being between the results for fully torrefied material and raw material. A visual inspection of the material was used to quantify the degree of interaction between the material types. As expected, the smaller the particle size, the higher the percentage of torrefied material in the sample. The smaller particles had a darker color and produced results comparable to that of the fully torrefied material samples. Figures 5.12-5.18 show the visual comparison of the blended material to the fully torrefied material.

Table 5.3: Blended Corn Stover (50% raw, 50% torrefied) Particle Size Distribution

Blended Corn Stover PSD							
	>1.4mm	>1.18mm	>0.85mm	>0.71mm	>0.5mm	>0.3mm	All Material
B-100-1	67.31%	73.00%	78.01%	80.71%	85.18%	91.42%	100.00%
B-100-2	62.09%	67.07%	72.49%	75.72%	81.25%	89.34%	100.00%
B-100-3	58.65%	63.20%	68.37%	71.83%	77.63%	85.97%	100.00%
Mean	62.63%	67.70%	72.90%	76.04%	81.31%	88.88%	100.00%

Table 5.4 shows the results for the torrefied samples. The test represents the material as it has reached full degree of torrefaction. The results from this test were similar to the results obtained for the torrefied material grinding tests conducted in Chapter 4. The torrefied material had 70% of the material grind below the largest screen size of 1.4mm. The grinding times required for achieving similar results did vary. The lab scale ball mill required 20 minutes of grinding time to replicate the previous grinding results for a grinding time of 5 minutes. However, the volume of material has been increased from 30g to 100g which

requires more time to breakdown in particle size. The mass of the steel ball bearings used for grinding can be increased in order to decrease the time required for grinding.

Table 5.4: Torrefied Corn Stover Particle Size Distribution

Torrefied Corn Stover PSD							
	>1.4mm	>1.18mm	>0.85mm	>0.71mm	>0.5mm	>0.3mm	All Material
T-100-1	31.03%	39.39%	48.80%	54.54%	64.14%	77.43%	100.00%
T-100-2	33.40%	41.55%	50.60%	56.15%	65.02%	77.74%	100.00%
T-100-3	32.31%	39.84%	49.35%	55.36%	65.30%	78.87%	100.00%
Mean	32.25%	40.27%	49.59%	55.35%	64.82%	78.02%	100.00%

Figure 5.10 shows the comparison between the raw, torrefied, and blended grinding tests. The plot shows the cumulative particle size distribution. As expected, the blended material results are between the raw and fully torrefied material results. The blended material plot line approaches the fully torrefied plot line as the material size decreases. This confirms the idea that even with blended material the smaller particles will contain a very high percentage of torrefied material with very little non-torrefied material.
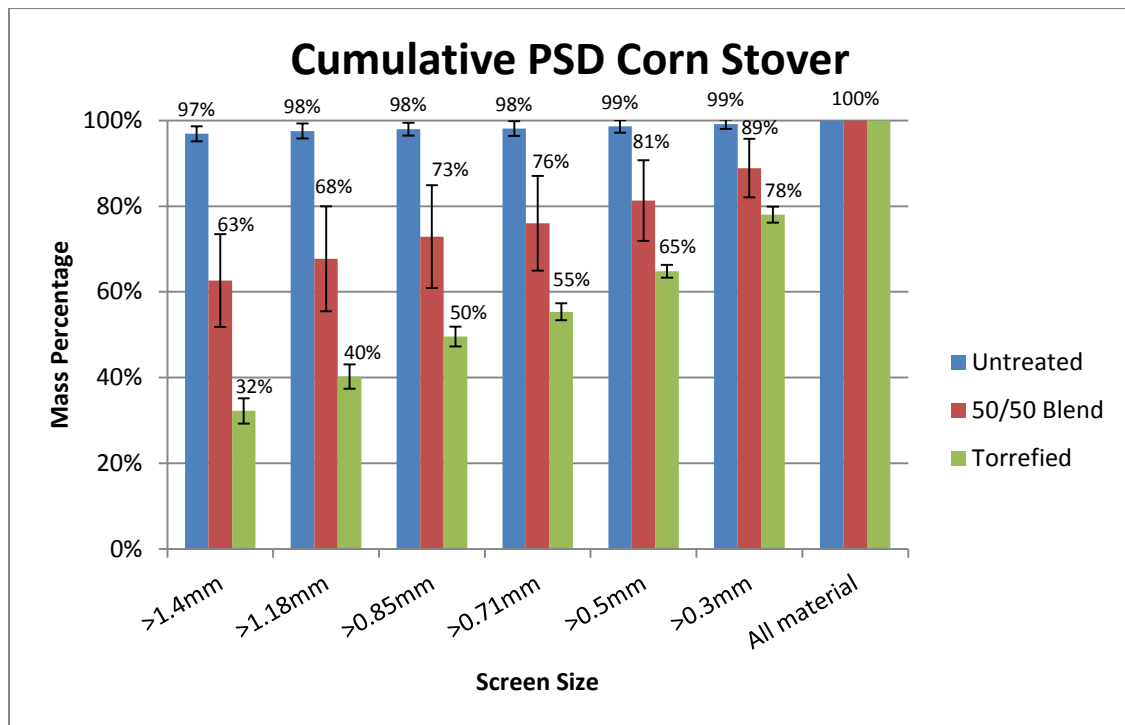
**Figure 5.10: Cumulative Particle Size Distribution Comparison**

Table 5.5: Cumulative PSD Analysis of Variance

Source	DF	Seq SS	Adj SS	Adj Mean Square	F ratio	Prob. >F
Material	2	1.79552	1.79552	0.89776	195.85	0.000
Screen Size	5	0.35872	0.35872	0.07174	15.65	0.000
Error	46	0.21086	0.21086	0.00458		
Total	53	2.36510				
S=0.0677049 R^2 =91.08% R^2 (adj)=89.73%						

5.5.4. Material Purity Calculations

The material purity was calculated for the untreated and torrefied material that was ball milled. The procedure for this calculation was discussed in Chapter 4. This calculation shows the level of non-torrefied material compared to the sum of torrefied and non-torrefied material at each screen size. The contamination level for the self selection torrefaction
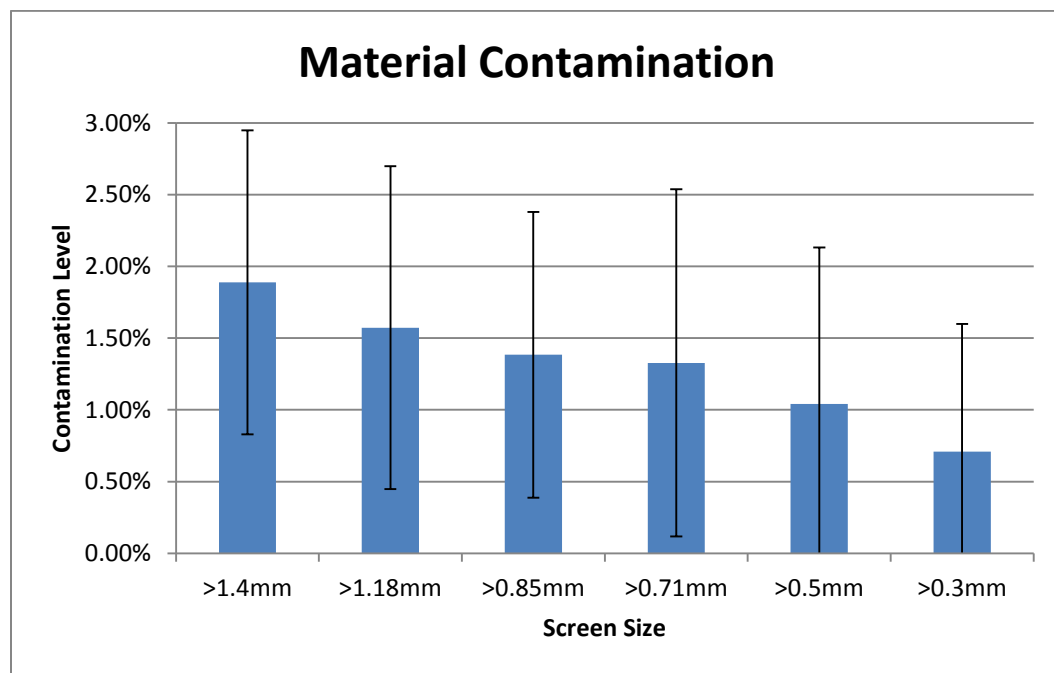
Figure 5.11: Material Contamination

5.5.5. Blended Material Grinding Results

The following photos show the comparison for blended material (50% torrefied, 50% raw stover) and fully torrefied material grinding tests. The visual inspection shows that as the

material size decreases, the color of the material becomes darker. This confirms that when the blended material is being ground during the self selection process, only the fully torrefied material will be ground. Figures 5.12-5.18 show that at large particle sizes, there is a mixture of torrefied and non-torrefied material in the blended material sample, with the majority being non-torrefied material. This supports the hypothesis that non-torrefied material will remain unground, or retain at least a larger particle size than torrefied material when ground in a ball mill reactor. As the particle size is reduced, there is a smaller level of contamination of non-torrefied material in the torrefied material.

Blended material   **Torrefied material**

Figure 5.12: Material Comparison (>1.4mm)

Blended material   **Torrefied material**

Figure 5.13: Material Comparison (1.4mm-1.18mm)

Blended material   **Torrefied material**

Figure 5.14: Material Comparison (1.18mm-0.85mm)

Blended material   **Torrefied material**

Figure 5.15: Material Comparison (0.85mm-0.71mm)

Blended material   **Torrefied material**

Figure 5.16: Material Comparison (0.71mm-0.5mm

Blended material   **Torrefied material**

Figure 5.17: Material Comparison (0.5mm-0.3mm)

Blended material   **Torrefied material**

Figure 5.18: Material Comparison (<0.3mm)

5.6. Self Selection System Testing

Upon completion of the ball mill testing previously discussed the self selection testing was conducted. The testing conditions used were similar to those in prior tests. For the self selection application the heating and grinding tests were run simultaneously. 100 grams of untreated corn stover were placed in the ball mill reactor and heated to a temperature of 275°C. The system was heated for 1 hour until the temperature reached 260°C. Once the temperature reached 260°C, the ball mill reactor was engaged and the self selection process began. The duration of grinding was 25 minutes. During the grinding stage, the temperature was held between 270°C - 276°C.

The material was allowed to cool for a period of 2 hours until temperatures were below 200°C before removing from the reactor. The visual inspection of the material showed that the material was uniformly torrefied. The particle size had been decreased but not to the same extent as previous grinding tests. This was due to the interaction between the torrefaction and grinding stages being run simultaneously. Previous tests allowed material to become fully torrefied before being ground. A review of the test results shows that a longer period of time may be required for grinding of material. The outer layers of the material may not have reached a full degree of torrefaction before the ball mill reactor was engaged.

Table 5.5 shows the data from the self selection torrefaction test. The data shows a large percentage of material that has not been decreased below the largest screen size. Visual inspection shows that the material has been reduced in size but not to an extent to allow the material to pass through the screen. Increasing the grinding time will allow for a greater percentage of material to be reduced in size.
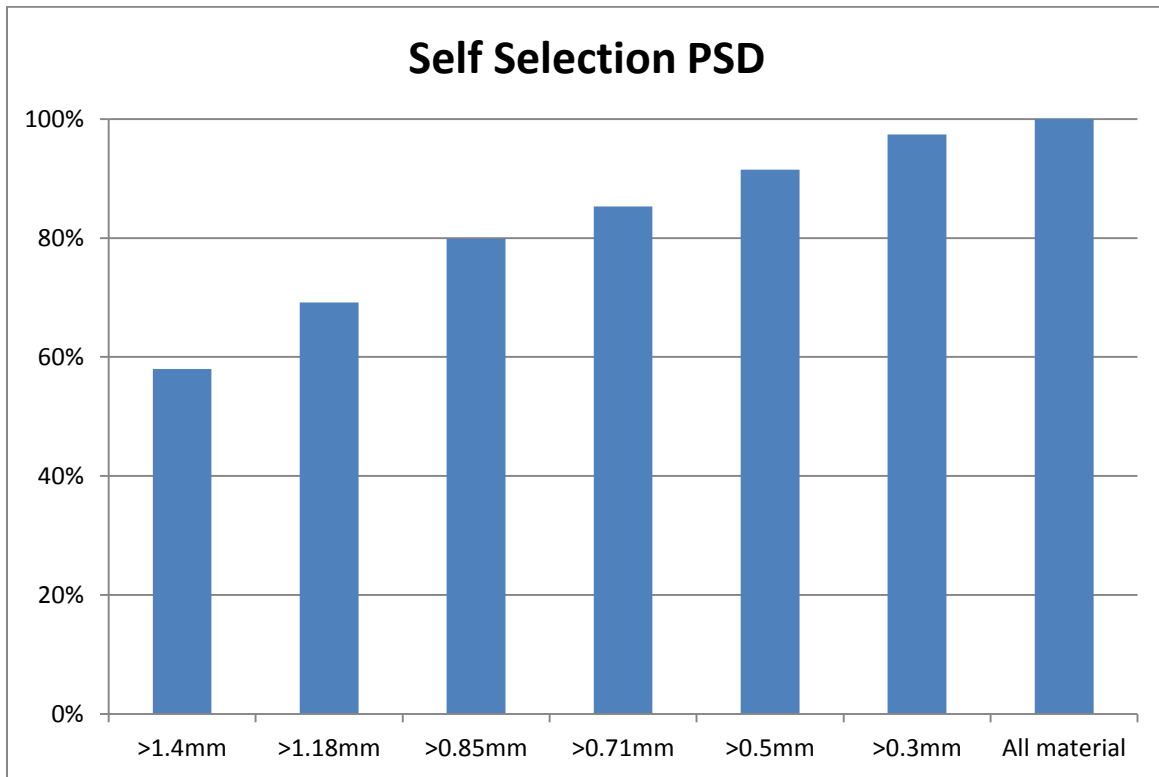
Table 5.6: Self Selection Particle Size Distribution

Self Selection Torrefaction PSD							
	>1.4mm	>1.18mm	>0.85mm	>0.71mm	>0.5mm	>0.3mm	All material
Test 1	58.0%	69.2%	79.9%	85.3%	91.5%	97.4%	100%

However, the time factor will not be the driving force in future testing when the separation screen is added to the ball mill reactor. By removing the smaller particles that have been fully torrefied, the non-torrefied particles will be more fully exposed for torrefaction and grinding. Also, the smaller particles may act as a cushion between the non-torrefied material and the steel bearings, thus reducing the impact force between the grinding media and the corn stover.

Visual inspection of the material showed that the degree of torrefaction is uniform for the entire batch. The larger, denser corn cobs did not break down entirely and examination of the cross section shows that the material has nearly been torrefied to the core. As previously mentioned an increase in grinding time will further break down the dense cobs and allow for uniform torrefaction and particle size. Figure 5.20 shows the image of the self selected torrefied material. The plot shows 58% of the material remained above the largest

screen size of 1.4mm. While this shows a greater percentage of material remaining above the screen size of 1.4mm than the previous tests where torrefaction and grinding were conducted separately, it is still a larger percentage than the non-torrefied material. The self selection torrefaction process will create a particle size that is smaller than non-torrefied material.

Figure 5.19: Self Selection Particle Size Distribution

>1.4mm

1.4-1.18mm

1.18-0.85mm

0.85-0.71mm

0.5-0.3mm

<0.3mm

Figure 5.20: Self Selection Torrefied Material

5.7. Conclusion

The self selection torrefaction process appears to be a feasible option for integrated torrefaction and particle size reduction for biomass. The results show that torrefied material will produce a smaller particle size than non-torrefied material when placed in a ball mill reactor. Additionally, when the blended material (50% torrefied, 50% raw) was placed in the ball mill reactor, the results were between the torrefied and non-torrefied material. The visual inspection of the mixed material shows that ball milling of the material will reduce the particle size of the torrefied material. The non-torrefied material retains its original structure without experiencing a significant level of reduction. The material purity calculations show confirm that raw material will reduce in particle size at a lower rate than the torrefied material. Material purity will remain high with a low level of contamination (less than 2%).

One of the objectives of the testing was to find an adequate screen size for separation of material during the self selection process. Several factors have to be considered for this decision. The mass percentage of torrefied will heavily influence the screen size selection. A very small screen size (such as <0.3mm) will require a longer period of time for particle size reduction. A larger screen size (1.4mm or 1.18mm) will have a higher quantity of raw material mixed with the torrefied material. Additionally, the application of the torrefied material being produced will influence the necessary screen size. Torrefied material can be used as a feedstock for application such as pyrolysis. The material can also be pelletized to form a more dense product that is easy to store and transport. Based upon these considerations, a screen size between 0.85mm-0.5mm would meet these criteria. This screen size could larger or smaller that this range but will alter the self selection process. A large screen size will allow non-torrefied or under-torrefied material to pass out of the reactor system. A smaller screen size will create very small fines that are more difficult to transport.

Chapter 6. Conclusions

Testing of the torrefaction and grinding processes have shown that a self selection torrefaction system is feasible. The first stage of testing involving the ball milling of torrefied and non-torrefied material showed that a distinct separation exists between the particle size distribution of each material. Over the same period of time, torrefied material will breakdown to smaller particles while non-torrefied material will experience very little particle size reduction. For grinding times of 5, 10, and 20 minutes, 71%, 87%, and 91% of the torrefied material was ground below the largest selected screen size of 1.4mm, respectively. For non-torrefied material, 2%, 4%, and 4% of the material was ground below the screen size of 1.4mm for times of 5, 10, and 20 minutes, respectively.   From this data collected, it is confirmed that torrefaction will increase the brittleness of corn stover and reduce particle size when reacted in a ball mill. Non-torrefied material will retain its structural integrity, even during grinding, and will not reduce in size to the same extent as torrefied material.

The particle size distribution of material showed that there will be some contamination of non-torrefied material with torrefied material when ball milled. The highest level of contamination found was just over 3%. This low level of contamination supports the concept of self selection and that the torrefied material will be removed from the non-torrefied material.

When the ball mill reactor was integrated into the lab scale torrefaction reactor, the results showed that the self selection process can be successful. The initial tests investigated the stages of torrefaction and ball milling separately. The self selection torrefaction system was found to operate successfully and produce a uniformly torrefied material. The ball milling tests confirmed the functionality of the reactor. The particle size distribution of the material showed that torrefied material will achieve smaller particle sizes than non-torrefied material. Additionally, the ball milling of mixed torrefied and non-torrefied material will reduce the torrefied material's size without reducing the non-torrefied material size.

6.1. Future Work

Future testing will allow for improvements on the efficiency of the system. The operating parameters of the system were based upon results found during the separate torrefaction and grinding tests. However, these parameters could be changed to improve the heating and grinding characteristics. The scale up of the system may cause unforeseen changes in performance.

The speed of drum rotation has been set at 20 rpm (0.11 g force) for testing. Based upon previous testing, as this speed is increased, the kinetic impact between the grinding media and biomass is increased. The VFD motor control will allow for rotation as high as 60 rpm. The physical limitations of the drum have not been tested for rotations of this magnitude. While it is likely that rotation of the drum can be increased, the speed will remain well below the motor limit of 60 rpm. Increasing the drum rotation speed will increase the rate of grinding and result in particle size reduction in a shorter period of time. This will further enhance the heat transfer through particles as torrefied layers are removed and could potentially decrease processing times. Testing of the system will need to be conducted to find an acceptable operating speed that will maximize system performance without approaching the physical limitations of the system.

A longer residence time for torrefaction and ball milling of material will further reduce the particle size of large particles, such as corn cobs. The large ball mill reactor has been found to require additional time for ball milling of material. During self selection testing, the ball mill reactor was not engaged until temperatures in the torrefier reached 250°C. It is possible that the ball milling could begin at a lower temperature. However, this alteration will need to be studied more to observe the influence upon final material quality. If the material begins to reduce in particle size prematurely the material may exit the reactor having been torrefied at a lower temperature than expected.

Chapter 7. Bibliography

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