BIOCHAR PRODUCTION FOR CARBON SEQUESTRATION

A Major Qualifying Project
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ABSTRACT

The primary objective of this project was to produce biochar from different biomass materials with a variation of temperature conditions. Once the biochar sample was made, numerous tests were used to determine the carbon content and the characteristics for each sample. Production and analysis were used for theoretical application of biochar for atmospheric carbon dioxide sequestration. Through research, daily production, and experimentation a methodology was formed that set a basis to find the yield of biochar produced, percent of carbon in biochar and the surface areas of ideal temperature samples. Many conclusions and recommendations were drawn to understand the application of and further study of biochar as a beneficial sorbent.

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ACRONYMS AND ABBREVIATIONS

MQP	Major Qualifying Project
SJTU	Shanghai Jiao Tong University
WPI	Worcester Polytechnic Institute
CO_2	Carbon Dioxide
N_2	Nitrogen Gas
KBr	Potassium Bromide

EXECUTIVE SUMMARY

Carbon dioxide (CO_2) emissions have been and will continue to increase over time, many countries are looking for ways to reduce or alter the amount of CO_2 harming our environment. By 2050 the goal set by President Obama and his administration is to reduce U.S. greenhouse gas emissions by about 83 percent from 2005 levels as seen in Figure 1 below.

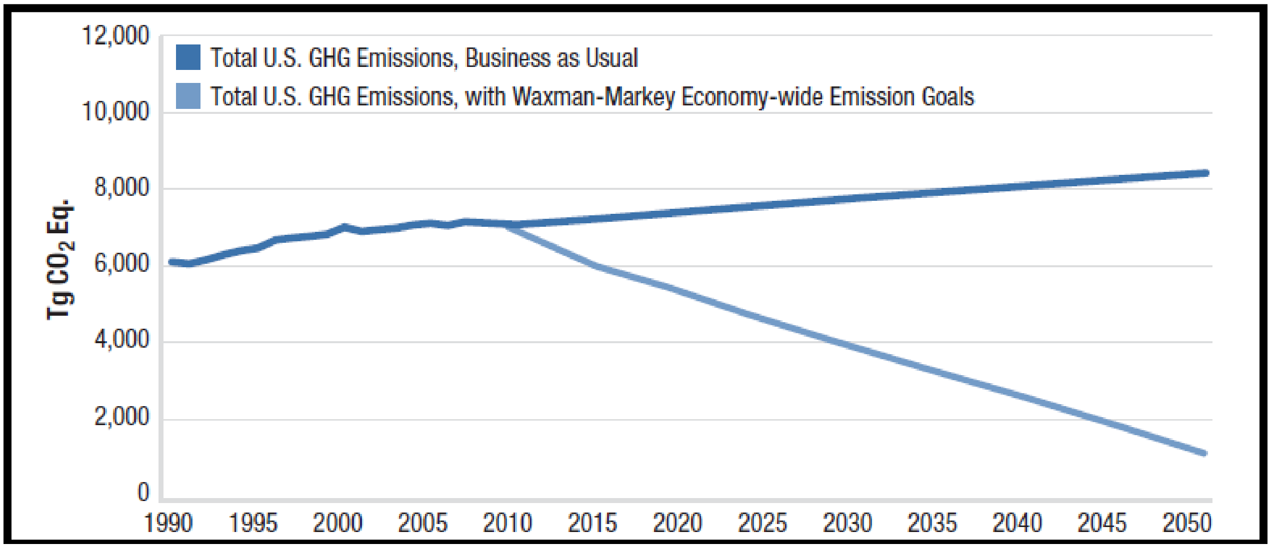


FIGURE 1: PROJECTED U.S. GHG EMISSIONS MEETING RECENTLY PROPOSED GOALS **(1)**

Many other industrial countries are following the actions of the United States. China is at the center of discussion, high industrialization yields a high output of harmful emissions. If there is no action toward implementing change by 2035 China alone will be responsible for 31% of the world carbon dioxide emissions (2). Government officials, researchers and the general public want to be able to find means to reduce output and decrease the current parts per million present in the atmosphere, one viable option is the use of underground biochar sinks (3) and (4).

FIGURE 2: A LAYER OF RICH, BLACK *TERRA PRETA* LIES ON TOP OF A LAYER OF LIGHT BROWN, NUTRIENT-POOR RAINFOREST SOIL IN BRAZIL **(35)**

Biochar is an organic charcoal material that is the final product of pyrolysis, or high temperature burning of agricultural biomass without the presence of oxygen. The limitation of oxygen in the system prevents the complete burning, instead producing the charcoal that captures much more of the natural carbon from the biomaterial. Such a form of carbon will not only be able to capture additional carbon, but also store carbon dioxide in sinks and out of the atmosphere for thousands of years. The ability to hold carbon underground has been studied based off of the *terra preta* soils found in the Amazon Basin where the ancestors put the ash onto the ground for hundreds of years compiling multi-colored, thick but fertile layers of soil seen in Figure 2. Scientists have studied this carbon rich soil phenomenon, and been able to show that applying biochar to soil can have multiple benefits, such as the carbon sinks and soil additives to increase plant productivity.

The purpose of this project was to help the Environmental Science department at Shanghai Jiao Tong University initiate a biochar research sector and begin the first stages in research toward biochar for carbon sequestration. Over the course of two months research in the field of biochar was completed, in order to fulfill the overall purpose of attending the project site the following objectives were achieved:

- Complete and understand background information on the conversion of biomass to biochar via pyrolysis.

- Determine an experimental methodology for production using the new reactor and furnace provided by Professor Xinde Cao.

- Upon post production of the biochar perform characterization tests to determine the best production conditions and to calculate the amount of potential carbon dioxide that can be sequestered.

Background research was conducted prior to the trip to Shanghai to have knowledge on the area of interest, this included looking into how biochar is made, pyrolysis systems and the uses of the charcoal. Each area was pertinent to the laboratory research completed at Shanghai Jiao Tong University. During the first three weeks at the university, while waiting for materials, a methodology was formed in addition to understanding how the reactor and furnace were used for pyrolysis. The methodology included determining the amounts of biomass needed, the temperatures that would be ideal for production, how to prepare the biomass for charring, flushing oxygen from the reactor and the rate of heating of the furnace.

With methods in place and biomaterials obtained production of the charcoal began after a series of practice runs. Each sample took four hours to complete, plus cooling time. Due to time constraints three biomaterials were used and charred at the three temperatures of 200, 350 and 500 Celsius, corncob, dairy manure, and rice hulls. Each sample was characterized based on the elements present post production, the functional groups, and the surface area. Two additional samples were made at 500 Celsius from peanut shells and corn straw to make additional carbon sequestration calculations to find a theoretical value of the amount of carbon that could potentially be captured if all agricultural waste was converted into a charcoal to form underground sinks.

After research was completed a number of conclusions were drawn from the experimental procedure. With an increase in temperature, there is a decrease in the yield of biochar. At 500°C corncobs yield the greatest percentage of carbon potentially due to biomass composition or initial material size. The surface area of the sample increases with temperature. FTIR yielded the presence of water, some carbon dioxide, oxygen, carbon, and nitrogen. Finally, corncob samples have the highest % C present in biochar and subsequently the highest potential CO_2 sequestration due to mass production. Each of the conclusions are significant for Professor Cao and his students in later work, testing and production exemplifies the ideal temperature of pyrolysis and the evidence supporting the notion that biochar has carbon capturing and remediation abilities.

CHAPTER 1: INTRODUCTION

Every year the world wide carbon dioxide (CO_2) emissions from energy needs increases, and by the year 2020 the world will produce 33.8 billion metric tons up from 29.7 billion metric tons in 2007. (5) The emission of carbon dioxide is not solely derived from energy sources, but also from fires, the natural carbon cycle and deforestation each adding to the amount produced on a yearly basis. Over time the emissions have contributed to overall green house gases and the effects of global warming. In 1990, China and India accounted for 13% of the world carbon dioxide emissions, by 2007 the combined for 26% of the emitted green house gas. (5) This was largely in part to economic and technological growth which has been seen in countries around the world. At the current rates the two countries will start to increase the rate of emission from fossil fuels while drastically impacting the levels of carbon dioxide in the atmosphere. Alternative energy sources such as wind or solar are able to replace such demands for high CO_2 emitting sources but do not aid in reversing the climate change process (6).

With a large emission of carbon dioxide there is an increase in the threat to the natural environment and its inhabitants. Scientists and scholars have predicted impacts on health, agriculture and food supply, ecosystems, coastal zones, water resources, energy production and usage, land usage, deforestation, in addition to extreme or rapid changes in the climate (7). Action is required that is far more ambitious than only decreasing worldly emissions generated by humanity. Due to the current trajectory in conjunction with the continuous need for energy, a mitigation program will decrease the current concentration of CO_2 in order to prevent humanity from reaching a level of cumulative emissions no longer deemed safe. Such an initiative would produce a usage energy source and decrease CO_2 while still maintaining the natural carbon cycle by photosynthesis (8).

Carbon dioxide is removed from the atmosphere when it is absorbed by plants as part of the biological carbon cycle but due to the current and estimated amounts of carbon dioxide emitted, biological carbon cycles are not adequate enough to handle the billions of metric tons emitted. In the carbon cycle photosynthesis intakes carbon for sugar production then uses respiration or waste removal to re-release CO_2 into the atmosphere.

Photosynthesis: $6CO_2 + 6H_2O \text{ (+ light energy) } \rightarrow C_6H_{12}O_6 + 6O_2$
Respiration: $C_6H_{12}O_6 + 6O_2 \rightarrow 6CO_2 + 6H_2O$

Thus, the carbon cycle has a net carbon withdrawal from the atmosphere of 0 percent; or carbon neutrality as seen in Figure 1 (9). In a basic cycle eventually the plants decay, and this dead biomass begins to release captured carbon dioxide into the atmosphere yielding an ineffective natural cycle (10). Organic biomass from decaying plant species or remnants of agriculture can be converted into a charcoal or biochar that can aid the in preventing "...global climate change by displacing fossil fuel use, by sequestering carbon into soil carbon pools and by dramatically reducing emissions of nitrous oxides, a more potent greenhouse gas than carbon dioxide." (3) Biochar slows down the decaying and mineralization of the biological carbon cycle to establish a carbon sink and a net carbon withdrawal from the atmosphere of 20 percent, as seen in Figure 3. Additionally calculations have shown that putting this biochar back into the soil can reduce emissions by 12-84 percent of current values; a positive form of sequestration that "...offers the chance to turn bioenergy into a carbon negative industry." (4)

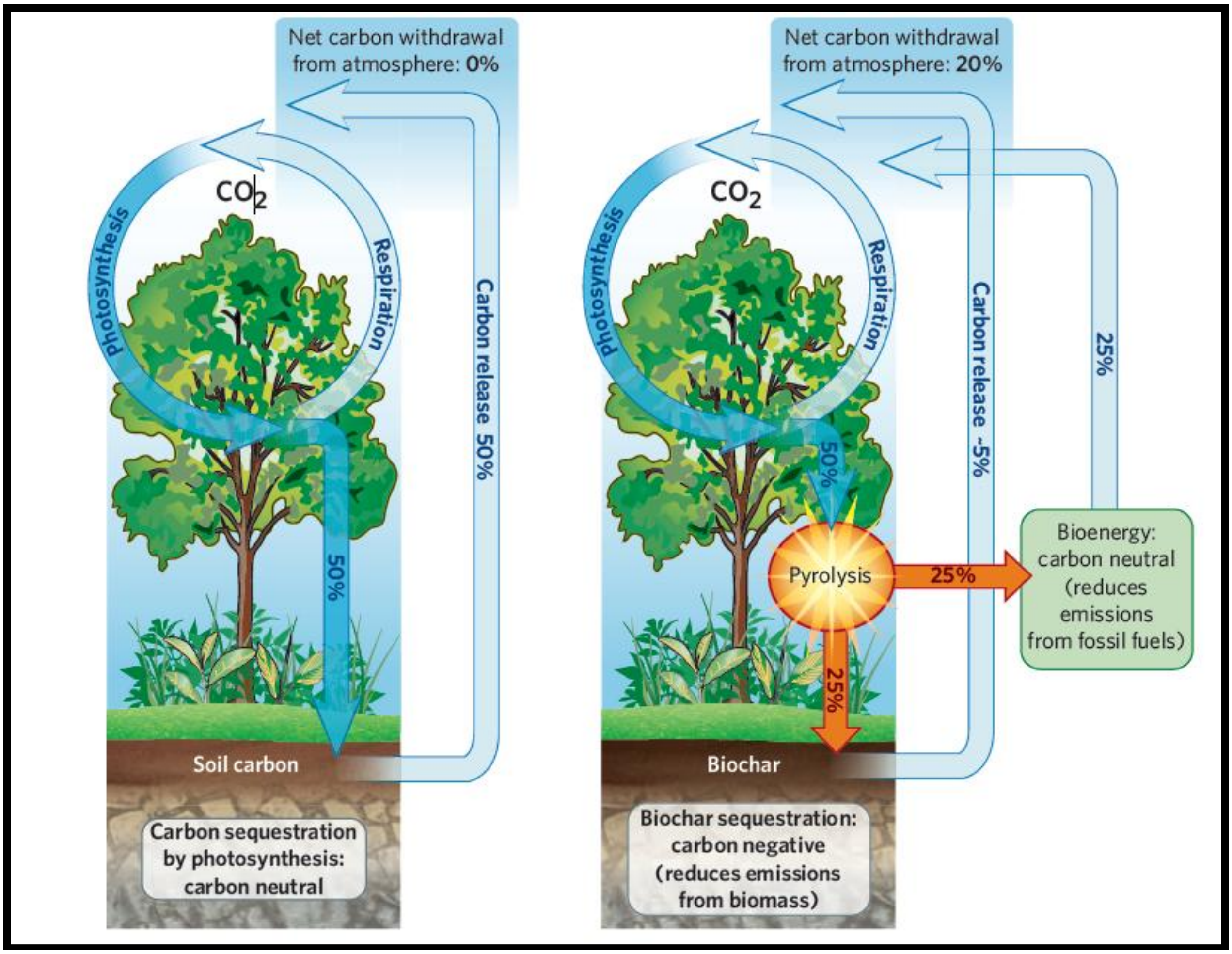


FIGURE 3: COMPARISON OF NORMAL AND BIOCHAR CARBON CYCLES **(4)**

In recent years the use of surplus organic matter or biomass to create biochar has yielded promising results in the reduction of CO_2. Biochar is a carbon rich charcoal that is formed by the pyrolysis (thermal decomposition) of organic biomass.

Biomass$_{(solid)}$ $\xrightarrow{\text{PYROLYSIS}}$ biochar + liquid or oil(tars, water etc) + volatile gas (CO_2, CO, H_2)

The reaction also produces bio-oil and gases such as hydrogen that can be used for energy sources to power homes or automobiles, not produced at certain high temperature choices with small initial biomass quantities. Additionally, biochar is a soil amender that is a viable option for underground storage of the carbon that will remain in soil for centuries. As discussed, each store exceeds the potential sequestration than that of biological carbon cycles, with the ability to increase soil nutrition (11). Biochar has the capability of improving the conditions of contaminated soils to provide additional benefits to the environment. Recently studies regarding production and deposition of biochar on a larger scale have been brought to the forefront of combating global climate change (6).

Research into understanding biochar has become common across the globe because of the positive potential in green initiatives. Studying and understanding previous work on biochar will be beneficial for formation of a methodology and testing procedure for Shanghai Jiao Tong University. Professor Xinde Cao and his team of students are in the early stages of biochar production from different sources of biomasses for further application in soil remediation and plant fertilization. This project specifically focuses on the production and characterization of biochar samples from dairy manure, corncobs, rice hulls, peanut shells and corn straw biomasses at varying pyrolysis temperatures for future application.

CHAPTER 2: BACKGROUND

The focus of this project was to study the production of biochar at different conditions from a variety of biomasses and then apply to soil for contaminant immobilization and CO_2 sequestration. Prior to experimental procedures the project requires a familiarity with biochar properties, production, and application. This section of the report presents known information about biochar necessary to complete studies and experiments at Shanghai Jiao Tong University.

Biochar is obtained from the pyrolysis or thermal decomposition of surplus and readily accessible biomass which has been proved to be an effective means of carbon sequestration and immobilization of organic contaminants in soil (12). The use of biochar for remediation is not a new concept, it has been found in areas of the fertile Amazon Basin. The *terra preta* soils of the region received large amounts of charred organic debris from the former inhabitants. Thus carbon stocks have remained in the soil present day, thousands of years after abandonment providing a historical basis for the benefits of biochar for sequestration and fertility (11) and (13).

Biomass in China is in the form of agricultural residue, firewood, forestry residue, organic waste (such as manure, municipal domestic waste), and industrial waste (such as grain factories, paper mills, sugar refineries, etc). Of the latter, residuals from agriculture and animals are the largest forms of biomass energy resource in China, more than 50 percent derived from rice, wheat, corn, beans, cotton and sugarcane and 22 percent from animal excreta or manure seen in Figure 4 (14).

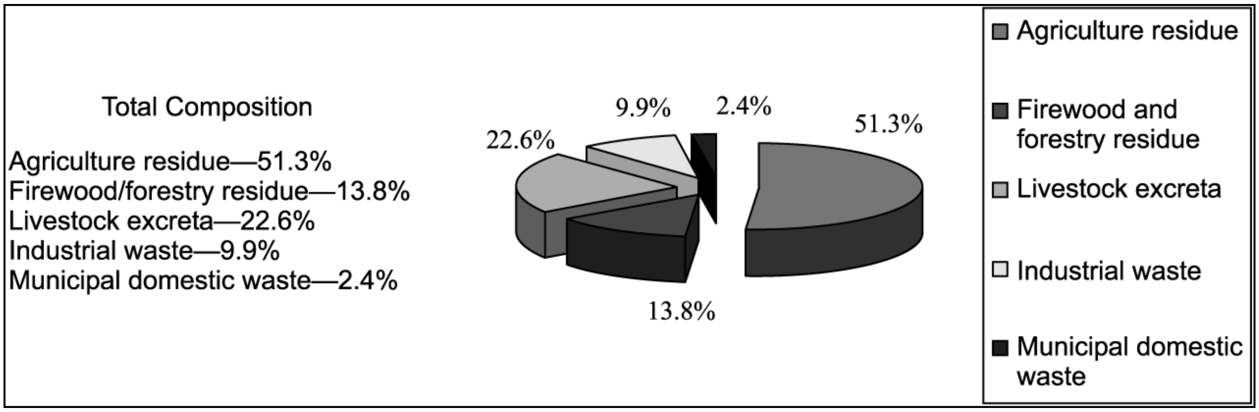


FIGURE 4: THE STRUCTURE OF BIOMASS ENERGY RESOURCES IN CHINA (%) (TOTAL 487 MILLION TONNES OF OIL EQUIVALENT/YEAR) **(14)**

From pyrolysis of biomass is yields biochar and additional products of incomplete combustion such as H_2 and CH_4 through heating biomass at varying temperatures typically between 300 and 500°C with a restriction of oxygen (15). According to Johannes Lehmann pyrolysis of biomass results in, "...a highly aromatic organic material with carbon concentrations of about 70-80 percent." Generally the carbon concentrated charcoal is stored and used by plants and organisms in a carbon pool as well as carbon used for energy and fuel, hence carbon negative energy production (10). After production, biochars result as a fine and porous charcoal like substance applicable in soil amendment for reduction of carbon dioxide emissions. When applied to soils the biochar is reluctant to decompose therefore it remains in the soil for centuries and potentially longer, like in the Amazon Basin. The carbon sinks alter the carbon cycle and immobilization of soil contaminant (3). To fully understand the benefits of biochar, it is important to understand production, the charcoal properties, and environmental implications and applications.

2.1 BIOCHAR PRODUCTION

2.1.1 Forming Biochar from Biomass

Biochar is most commonly produced by pyrolysis of the biomass. Pyrolysis allows the production of biochar by heating with essentially little to no oxygen present. In addition to the biochar, the process also results in bio-oils and synthesis gas or syngas that are used for further combustion and renewable fuels, the process can be seen in Figure 5 (11).

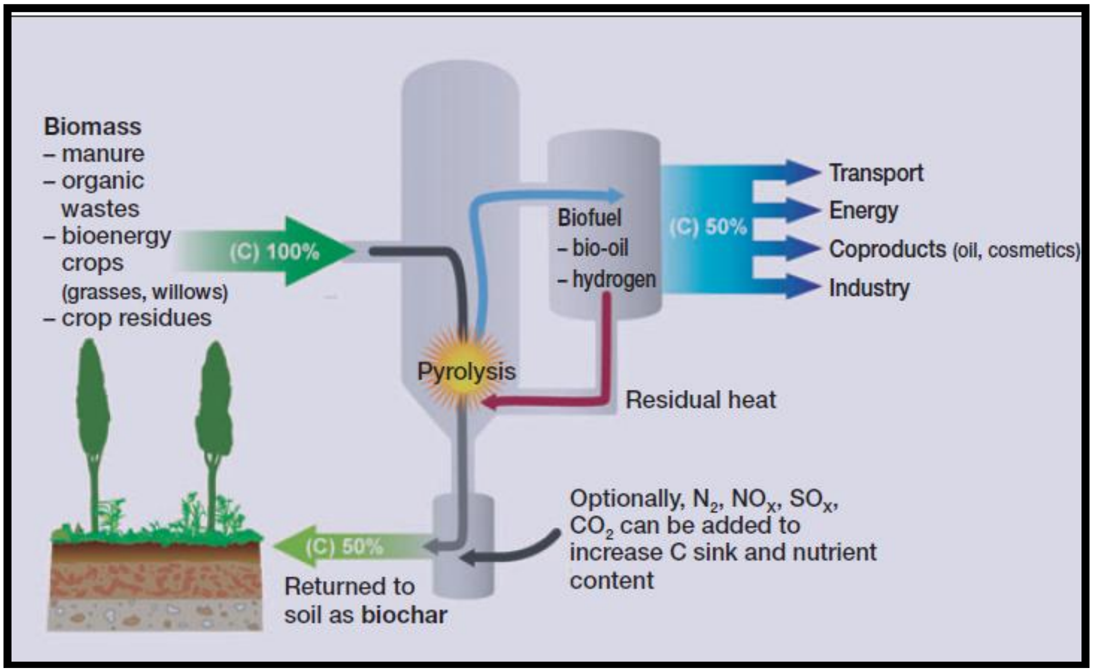


FIGURE 5: PYROLYSIS OF BIOMASS, TYPICALLY, ABOUT 50% OF THE PYROLYZED BIOMASS IS CONVERTED INTO BIOCHAR AND CAN BE RETURNED TO SOIL **(6)**

As seen above, pyrolysis requires the use of kilns and furnaces to heat the biomass in a three stage reaction process noted in the equations below. In the initial stage of production the biomass loses water and other residue. Then the residue goes through further pyrolysis and biochar begins to form. Finally the biochar produced begins to decompose forming the carbon rich charcoal used for application (16).

$$\text{Biomass} \xrightarrow{yields} \text{Water + Unreacted residue}$$
$$\text{Unreacted residue} \xrightarrow{yields} (\text{Volatile + Gases})_1 + (\text{Char})_1$$
$$(\text{Char})_1 \xrightarrow{yields} (\text{Volatile + Gases})_2 + (\text{Char})_2$$

In the laboratory experiments conducted at Shanghai Jiao Tong University slow pyrolysis was used to convert the different biomasses into biochar at varying conditions.

2.1.2 Slow Pyrolysis

Slow pyrolysis has been used as a means to produce alcohol, tar, and charcoal such as biochar by feeding large batches of dried pellets or chips into a furnace or kilns. According to the International Biochar Initiative characteristics of slow pyrolysis include (17):

- Long solids and volatiles residence times, typically greater than 5 seconds for volatiles; solids residence times can be minutes, hours or days.
- Relatively low reactor temperatures normally, but not necessarily less than 400°C.
- Atmospheric pressure.
- Very low heating rates ranging from 0.01°C/s to up to 2°C/s.

Ideally low temperatures and low heating rates are used for the production of biochar for maximization purposes since there is less production of bio-oil and syngas and the priority is the production of the charcoal (11). Upon production through pyrolysis the biochar is either characterized or applied to soil.

2.3 APPLICATION OF BIOCHAR

Biochar has a number of specific functions in the natural environment that will be beneficial to prevent global warming and also to increase the functionality of soils. Biochar, as previously mentioned is use for the sequestration of carbon dioxide becoming "...mechanism to enhance photosynthesis but decelerate the decay of the products of photosynthesis (release of CO2). Thus the production of biochar is a way to manipulate the carbon cycle." (10) Greenhouse gases will be removed from the atmosphere, with every unit of biochar that is in production. Also biochar can be applied to soils, similar to that of the *terra preta* soils to enrich lands for agricultural purposes and also for reduction of contaminants.

2.2.1 Carbon Sequestration

Recent green movements have brought environmentally friendly initiatives and reducing carbon emissions to the forefront of political, economic, and social activity. This provides a need for an inexpensive means of capturing or removing carbon from the atmosphere. Biochar, produced from biomass which is in abundance, fills this void while being a form of waste disposal and recycling (18). By an adaptation of the current biological carbon cycle, the biochar is produced from biomass and half is returned to the soil as charcoal and the other half is return to commercialization for an organic fuel.

The percent that is being used as a biofuel, is the off syngas and oil which can be applied as transportation, energy, and industry to reduce the amount of petroleum used, thus reducing the amount of carbon dioxide emitted. Biochar is returned to the soil where carbon sinks are formed for sequestration. Biochar, like plants, captures carbon dioxide and has the ability to store the greenhouse gas in a sink. A carbon sink in this case is an artificial reservoir that accumulates and stores some carbon dioxide for a biomass dependant time period through sequestration.

2.2.3 Reduction of Contaminants in Soil

While biochar is being applied to soils for the conditioning and fertilization purposes, this application can also be beneficial in reduction of toxic components. Studies have shown that biochar is also capable of absorbing metals such as lead, and organics that contaminate soils which harm people, plants and animals (12). This is because biochar as an additive to a soil can be expected to improve its overall absorption capacity impacting toxicity because there is a decrease in transportability and depletion of the presence of metal or organic compounds. Biochar comes with the appeal of being a low cost and low-environmental-impact strategy for remediation of common and health concerning environmental pollutants. Studies have shown that biochar in combination with activated carbon sinks have comparable absorption abilities, which helps in removal of contaminants such as the herbicide atrazine (12).

Through experimental methods, results have shown that metal ions are strongly adsorbed onto specific active sites containing acidic carboxyl groups at the surface of the charcoal (19). According to the Scientific Review of Biochar Application to Soils, contaminant metal intake by charcoals involves replacing already existent ions contained in the charcoal with the metal ion in the soil, suggesting a potential correlation between content of the biochar and its remediation potential for metals (19). With the studying, monitoring, and understanding of biochar, real world application to benefit environmental concerns will be in the near future the simplicity of production alludes to the capability for integration into less advanced nations.

2.4 BACKGROUND CONCLUSIONS

The concept of biochar has been around for centuries, but in recent years application for environmental benefits has prompted more in-depth research. Studies have shown that the charcoal has the potential to be a major contributor to global carbon dioxide capturing and soil decontamination. When producing biochar for use, there are a number of considerations and testing variations of importance. The research done incorporates how to produce biochar from biomass via slow pyrolysis and characterization which will be essential for future production. In the future the background knowledge will be useful to test the charcoal outside of controlled laboratory experience to pursue environmental changes. All the information in the background section is a foundation for laboratory research conducted in Shanghai, China regarding biochar, the following chapter provides an experimental methodology for the production and characterizing completed.

CHAPTER THREE: EXPERIMENTAL METHODOLOGY

This section of the report outlines the procedure to collect and analyze data obtained during the experimental process. My research took place in the Biochar and Soil Contaminant Laboratory at the School of Environmental Science and Engineering, Shanghai Jiao Tong University (SJTU), from October 25th to December 15th of 2010. The initial laboratory work included the production of the different biochars from varying biomass and conditions for further evaluation. Once the biochar was produced on each sample I performed a carbon balance, elemental analysis, solid phase determination, functional group detection, and finally found the different surface area of each sample. The final objective of the project was to determine the amount of carbon that would be sequestered from the atmosphere if each type of biomass was pyrolysized into biomass. The research focused on these three areas which are categorized into production, analyzing, and application of biochar outlined as follows.

3.1 PRODUCTION OF BIOCHAR

The production process was one that included trial runs and error before experimental production began, this was because the groups of students and Professor Cao had yet to produce their own biochar using the equipment and facilities at SJTU. Thus the methodology of production will have future importance when the biochar is produced for other experiments in their laboratory.

3.1.1 Materials

The process consisted of producing biochar from three different biomasses in order to obtain comparable data and analysis. Corncobs, dairy manure, and rice hulls were obtained from Bengbu City in Anhui Province of China and can be seen in Figure 6. All materials were received in an already dried condition. The corncob were hammered into fourths then cut into pieces approximately 1 to 3 cm in size, as seen in Figure 7 to ensure that the charring was evenly distributed and occurred to the fullest potential. Similarly the dairy manure was hammered into pieces that ranged from small particulates to pieces about 1 to 3 cm in size, seen in Figure 7. Finally, the rice hulls were maintained at the same size of approximately 1 cm, also seen in Figure 7.

FIGURE 6: BIOMASS IN ORIGINAL FORM

FIGURE 7: SIZING OF BIOMASS (L TO R) CORNCOB, DAIRY MANURE, RICE HULL

3.1.2 Experimental Set-Up

The experimental design used in the laboratory pyrolysis production can be seen in Figure 8. Pyrolysis of each biomass took place in a stainless steel reactor that was 45 cm long and had a diameter of 11. The reactor had an internal tray to hold and sieve bio-oils throughout the heating process; also there were two holes present in the reactor which are use for flushing the packed reactor with nitrogen gas (N_2) for oxygen gas (O_2) removal. Both can be seen in Figure 8. After the reactor is filled with the appropriate amount of dried biomass it is placed in an electric furnace, seen in Figure 9 for the start of the charring process.

FIGURE 8: REACTOR (TOP) AND SIEVE TRAY FILLED WITH CORNCOB BIOMASS (BOTTOM)

For each biomass tested, a sample of approximately 100 grams was initially placed on the sieve tray. As previously mentioned this was flushed with N_2 gas for about 10 minutes at a pressure of 5 kPa to remove the air. One end of the reactor was use for gas was input and the other hole was covered with a finger and occasionally uncovered in the time span to allow the release of the unwanted oxygen. The flushed reactor was then placed in the furnace at room temperature, rising at an average rate of 20°C min^{-1} until the specific final temperature was reached and held constant. The rate was calculated by timing and measuring the temperature difference (See Appendix A, Tables 1 and 2). Once at a constant specific temperature the biomass remained in the furnace that that temperature for four hours. After the four hours of heating and charring the power supply was turned off and the system was cooled over night to reach room temperature in order to remove the reactor. The following day the reactor was removed and the biochar

FIGURE 9: FURNACE USED FOR PRODUCTION

was bagged, weighed, labeled and the reactor and furnace were prepped for additional production. The temperature was set on the furnace (in green) and above this goal temperature reading was the actual temperature (in red), which was used to calculate the rate of heating as a seen in the furnace Figure 9. Each biomass was produced at 200, 350, and 500°C to obtain nine samples for analyzing purposes; the temperatures were suggested by Professor Cao from previous biochar work performed at the University of Florida. The most significant recordings were when the biomass was weighed before pyrolysis and the biochar was weighed after production. This was done to calculate the percent of biochar produced in the system. Tables in Appendix A show the calculation completed by the equation:

$$Biochar\ \% = \frac{Biochar\ Mass}{Biomass\ Mass} \cdot 100$$

The recording was done to ensure the proper replication in the future; other information included the beginning and ending times and the rate of heating, also seen in Appendix A.

3.2 ANALYZING BIOCHAR

As previously mentioned, the biomass was weighed before pyrolysis and the biochar was then weighed after production for calculation purposes, and to better understand how the conditions affect the experimental methodology. Following the production the final week in the laboratory was dedicated to analyzing the characteristics of the different biochars. In order to do so, a fourth of each sample was hand ground with a mortar and pestle and sifted to approximately 1 mm in size to complete a Fourier Transform Infrared Spectroscopy (FTIR) and then also a size of 0.23 mm for Elemental analysis and Brunauer, Emmett, Teller (BET) testing. The series of tests and calculations helped to analyze the elemental composition, functional groups, and particle surface area. It became a tedious process completed in a number of departments throughout the SJTU campus but beneficial to understanding the application of biochar and for comparison purposes.

3.2.1 Elemental Analysis

Elemental analysis was performed on the samples in the seventh week of the project. This was completed in the Analysis Center at SJTU by the CHNS/O Analyzer Perkin Elmer 2400 II seen in Figure 10. Elemental analysis is a means of determining the elemental composition of the biochar sample produced. The main focus of the testing was to determine the amount of carbon present for sequestration purposes in addition to the ratio of carbon to other elements such as hydrogen and sulfur. In this case the elemental analysis sent to the analysis center where the procedure was conducted by a professional. This was because there was a shortage of time. Also for this analysis only the samples of 500°C were used for the reason of time and also because Professor

FIGURE 10: PERKIN ELMER 2400 II ELEMENTAL ANALYSIS (PHOTO COURTESY OF XIANYUN XU)

Cao suggested this, mainly because only 500°C will be used for further calculation. Additionally this included peanut shell and corn straw biochar produced only for the same carbon sequestration calculations.

3.2.2 Fourier Transform Infrared Spectroscopy (FTIR): Functional Groups

FTIR is used for the identification or fingerprint of a sample or solution to determine the functional groups. In infrared spectroscopy, IR radiation is passed through a sample and some of the infrared radiation is absorbed by the sample while the remainder is transmitted through. This laser radiation is a spectrum that represents absorbance and transmittance that is created and unique to the sample. In particular the analysis identifies unknowns, determines the quality or consistency of the sample, and determines the component. In this analysis of biochar the FTIR was used specifically to determine the functional groups present for each temperature and biomass, especially carbons (20).

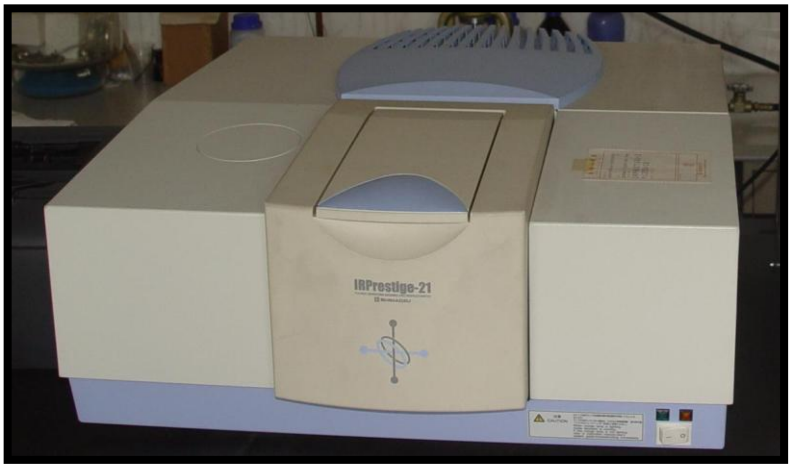


FIGURE 11: IR PRESTIGE-21

At SJTU the FTIR Machine was found in the Environmental Engineering building in the analysis center this model, seen in Figure 12 is the IR Prestige-21. The procedure to prepare and conduct the analysis required multiple steps to create the potassium bromide, KBr tablets. The KBr tables were used to complete the testing of the solid sample. Figure 11 shows both the tablet dies and the tablet punching device that we used in series with one another. First a small amount of the sample with a small amount of the KBr were ground together in an agete mortar, this was not an exact measurement. After being ground for 5 minutes the powder was transferred to the sample base ③, and the concave tablet frame ⑤ with a spatula. The other sample base ③ is pressed downward on the tablet frame ⑤ to create a

Figure 12: VACUUM PUMP

flat powder surface that is free of white areas which would indicate KBr. This is then inserted into the base ① and then the spring system ⑧ is placed on the top and pressed downward. This system is then placed in to a vacuum pump, seen in Figures 11 and 12. It is pressurized to 60 KN, where it remains for one minute. After one minute the pressure is released and the system is separated. The sample base and tablet frame ③ and ⑤ remain together holding the sample. It is then placed into the punching base ⑩ where the spring system ⑧ is placed on top again. This is then put into the vacuum pump, where the pressure goes to about 5 KN or when there is a clicking noise. It is then immediately removed from the pump and the tablet is removed. The tablet is then used for the laser scanning of the FTIR reading. After the blank was completed, this process occurred 9 times for each sample for later comparison and analysis through the peaks present in each graph.

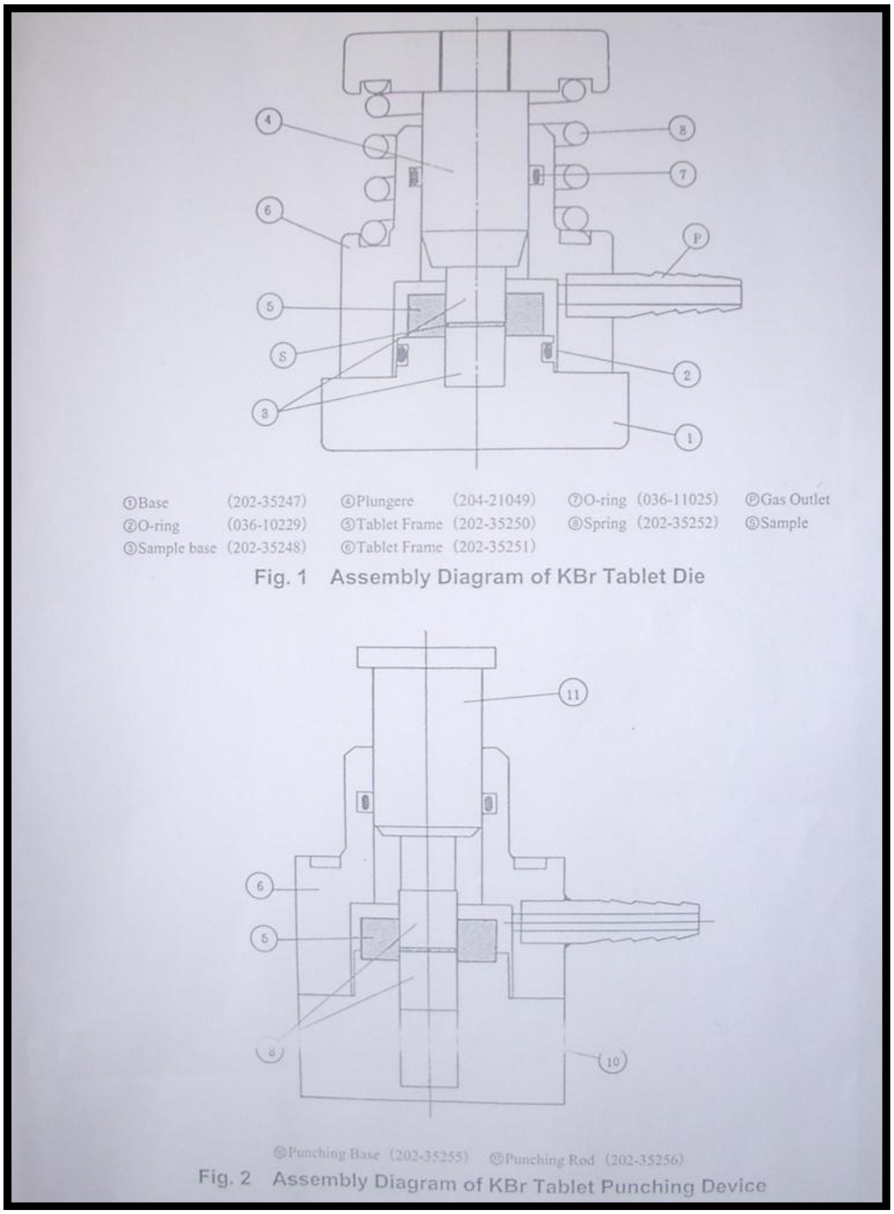


FIGURE 13: TABLET ASSEMBLY DIAGRAM

3.2.2 Brunauer, Emmett, Teller (BET): Surface Area

BET was the method used to determine the surface area of each sample of biochar. This method is and explanation of the physical adsorption of gas molecules on a solid surface which is important in the specified surface area. The experimentation process is derived from multiple theories that deal with how gas molecules physically adsorbed on a surface in layer and the lack of interactions between each layer. The general equation for BET is:

$$\frac{p}{v(p_0 - p)} = \frac{1}{v_m c} + \frac{c-1}{v_m c}\frac{p}{p_0}$$

p: are pressures
c: BET constant based on adsorption
v_m: monolayer adsorbed gas quantities

For this procedure, the total surface area, derived from the previous equation was used. This can be denoted by:

$$S_{BET,total} = \frac{(v_m N s)}{V}$$

And also,

$$S_{BET} = \frac{S_{total}}{a}$$

N: Avogadro's Number
V: molar volume
A: mass of adsorbent in grams
s: cross section
(21)

The BET analysis was completed by an analysis center at SJTU.

3.3 CARBON BALANCE FOR UNDERSTANDING CARBON SEQUESTRATION

The final stage in the characterization after conversion of the biomass to biochar was calculating the amount of carbon that would be captured by each biomaterial if the experimental conversion was applied. This required outside research on the amount of biomass wasted produced in China each year. With Food and Agriculture Organization estimated production values for dairy manure, rice hulls, corncobs, peanuts, and corn straw in 2009 the amount of biochar that can be produced is calculated:

$$Millon\ Tonnes\ of\ Biomass\ Produced\ in\ 2009\ \cdot \%\ Conversion$$
$$= Biochar\ in\ Million\ Tonnes$$

With the previously calculated amount of biochar created in an ideal situation the amount of potential carbon was found based on the percent of carbon detected during the elemental analysis.

$$Biochar\ in\ Million\ Tonnes\ \cdot \%\ Carbon = Total\ Potential\ Carbon$$

Lastly, using total potential carbon for sequestration the amount of carbon dioxide reduced in the atmosphere in one year was calculated. This is based off the new carbon cycle seen in Figure 1, 20 percent of carbon is withdrawn therefore 80 percent is remained in a stable state which was then converted to potential carbon dioxide.

$$Total\ Potential\ Carbon\ \cdot Carbon\ Stability\ of\ 80\%\ \cdot \frac{44\ g}{12\ g} = Carbon\ Dioxide\ Reduction$$

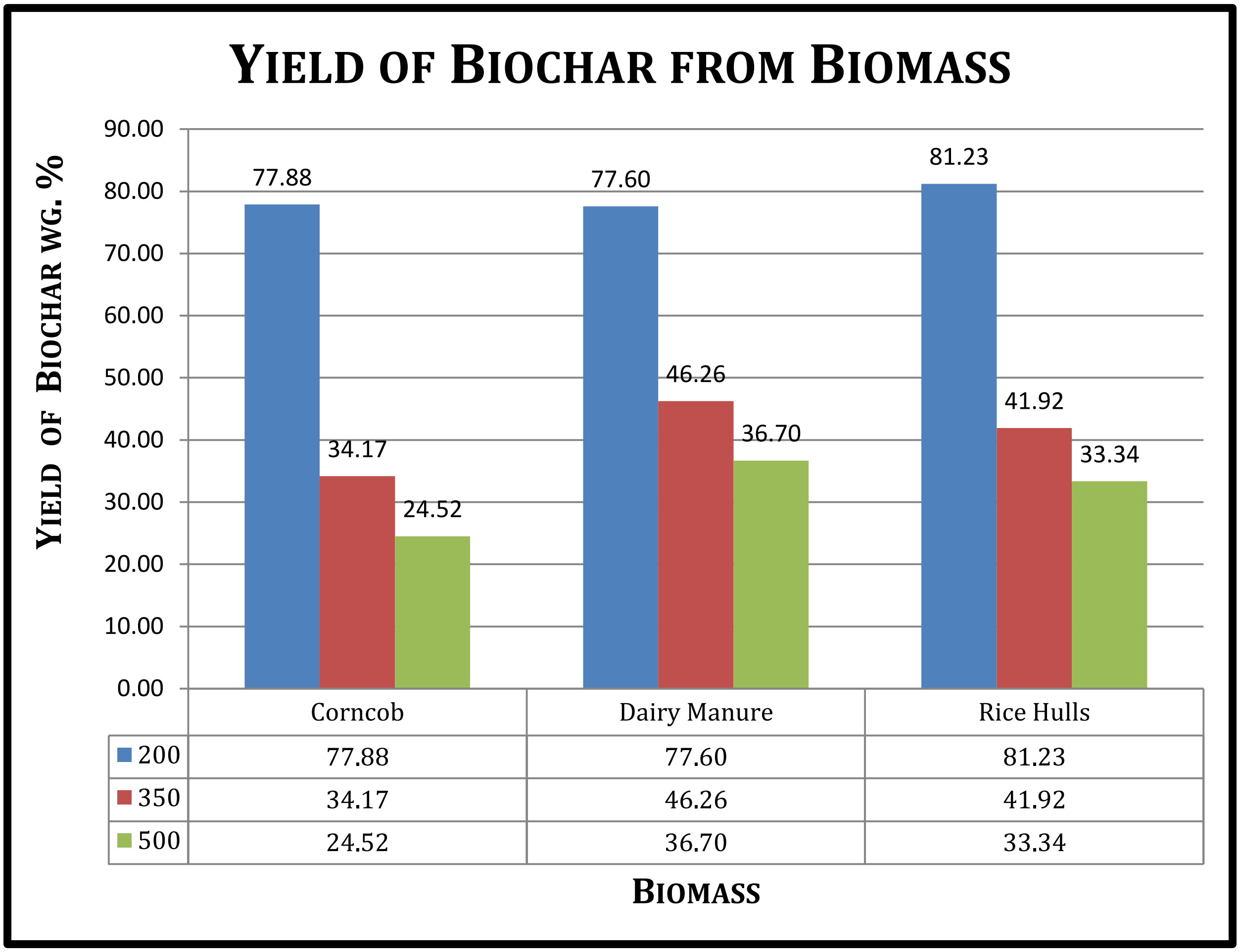
The calculations help to understand the significance and the role that biochar has the ability to play in the atmosphere.

CHAPTER FOUR: RESULTS AND DISCUSSION

In this section production of biochar and the data collected from the testing will be discussed in detail for future application as a soil additive for CO_2 remediation. Each stage of the experimental methodology will be covered.

4.1 PRODUCTION OF BIOCHAR

Producing biochar from corncobs, dairy manure and rice hulls resulted in not only a first time methodology for Professor Cao and his team of students, but also data to determine the best temperature and biomass for yield percent of biochar. In Appendix A the full data tables can be found with the pre and post pyrolysis masses for the biomass and the biochar samples at each temperature. The bar graph below shows the percentage of biochar that was produced from each set of conditions.



	Corncob	Dairy Manure	Rice Hulls
🟦 200	77.88	77.60	81.23
🟥 350	34.17	46.26	41.92
🟩 500	24.52	36.70	33.34

FIGURE 14: BAR GRAPH YIELD OF BIOCHAR

From the graph evident correlations can be made due to the temperatures and the initial states of the biomass. Temperature played a major role in the in the yield. As the temperature was increased for each sample, the yield decreased. This is the due to the obvious correlation that with exposure to extreme temperature conditions the biomass will be diminished faster than normal. At 200°C rice hulls had the highest remaining yield; this may correlate with the initial conditions/form of the biomass. Rice hulls were added to the reactor in the normal form, whereas the dairy manure and the corncobs had to be hammered. The hammering produced fine particles in addition to the ones of one to three centimeters in size. Such small particles would burn faster and become non-existent. Additionally the rice hulls, due to the biomass surface area, had a larger density of packing in the reactor. Therefore the rice hulls were thoroughly packed into the sieve tray whereas the other materials had sufficient space for even burning. This packing may have had an influence at the lower temperature.

At 350°C dairy manure had the highest yield. It would be expected that the same trend would occur and rice hulls would have the largest yield. The reason in which the dairy manure was the higher yield at this temperature is something that was questioned and there is not an evident reason behind the high yield. The biomass composition may be the main driving force in the post production yield. Dairy manure in the natural form is composed of not only the plant vegetation consumed by the animal, but also digesters and other excretions that could have potential impact on the yield. Corncobs have the smallest yield at 350 as well which may have to do with the initial size or the composition of the biomass in an original state. The outer layer of the cob is dry and brittle and can be charred much easier than the compacted dirt remnants of the manure or the hard protective shell of the rice grain.

Corn also was the lowest yield at 500 Celsius formulating a trend in the temperature relationship between corncobs and charring. Similar to 350 degrees the dairy manure had the highest yield of 36.70 percent, which would prove that there was an error or problem that took place in the charring at 200 Celsius, or that the temperature is too low for the ideal charring conditions. This can be supported by the visual representation of the samples seen in Figure 15; all of the samples are still a red and brown color which does not represent the ideal coloring for a charcoal according to previous research and background information on the *terra preta* soils.

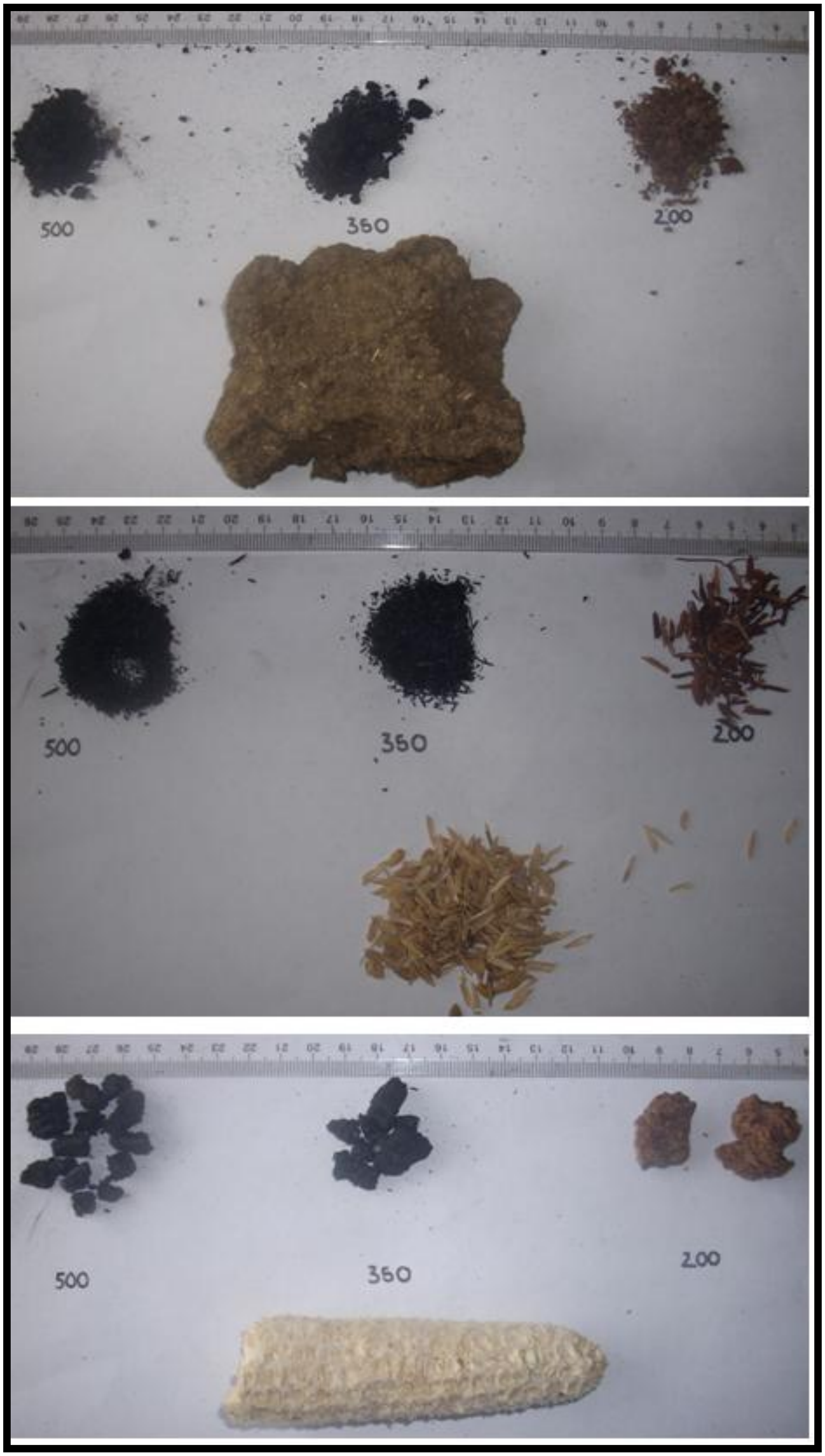


FIGURE 15: DAIRY MANURE, RICE HULLS AND CORNCOB BIOCHAR

4.2 ELEMENTAL ANALYSIS AT 500°C

Determining the elemental composition of biochar will help to determine the biomass that would be ideal for application as a biochar for sequestration. In the analysis, due to time and money constraints each biochar was analyzed at only 500 degrees as asked by Professor Cao because of his prior production experience this was the temperature he felt would be most important to look into more in-depth. The results can be seen in Figure 16 as well as in Appendix B. The laboratory technician who completed the analysis took four reading of the samples of the biochar; from this the mean of each percentage was taken to obtain the values that are found in the bar graph. It can be noted that the percentage is not equal to 100; this is because the machine is only able to detect carbon, nitrogen and sulfur; hydrogen, oxygen and other elements may be present but this is not known nor is the quantity.

The graph indicates that corncob, which had the lowest yield had the highest presence of carbon. Having a high percent of carbon means that the charcoal can absorb more of the atmospheric carbon, the main research focus of the project. The second was peanut shells, which were not analyzed in any other manner because of the multiple constraints, but if there was further studies it may be an ideal source of charcoal since the yield was 42 percent at 500 degrees as seen in Appendix A. Corn straw, rice hulls and dairy manure had smaller percentages of carbon present. Dairy manure, which is an abundant source of agricultural waste, does not seem to be an ideal source for charring based on the elemental analysis. Even though this material does have a high yield more of it would be require to attain the same level of carbon capturing compared to other biomasses. The dairy manure was the only biomass that was not in original organic form which may be a reason for the results obtained.

Nitrogen and sulfur were also detected in the analysis. The results indicate the amounts of each component are trace and not significant for determining what type of biomass should be used for biochar production. Nitrogen was used in flushing oxygen out of the system, a potential cause for the slight indication in the elemental determination. Corncobs have the least amount of nitrogen, with this small amount and further elements detected it might be found that there is a higher presence of oxygen in the sample which would hinder the pyrolysis and be a form of error in the report.

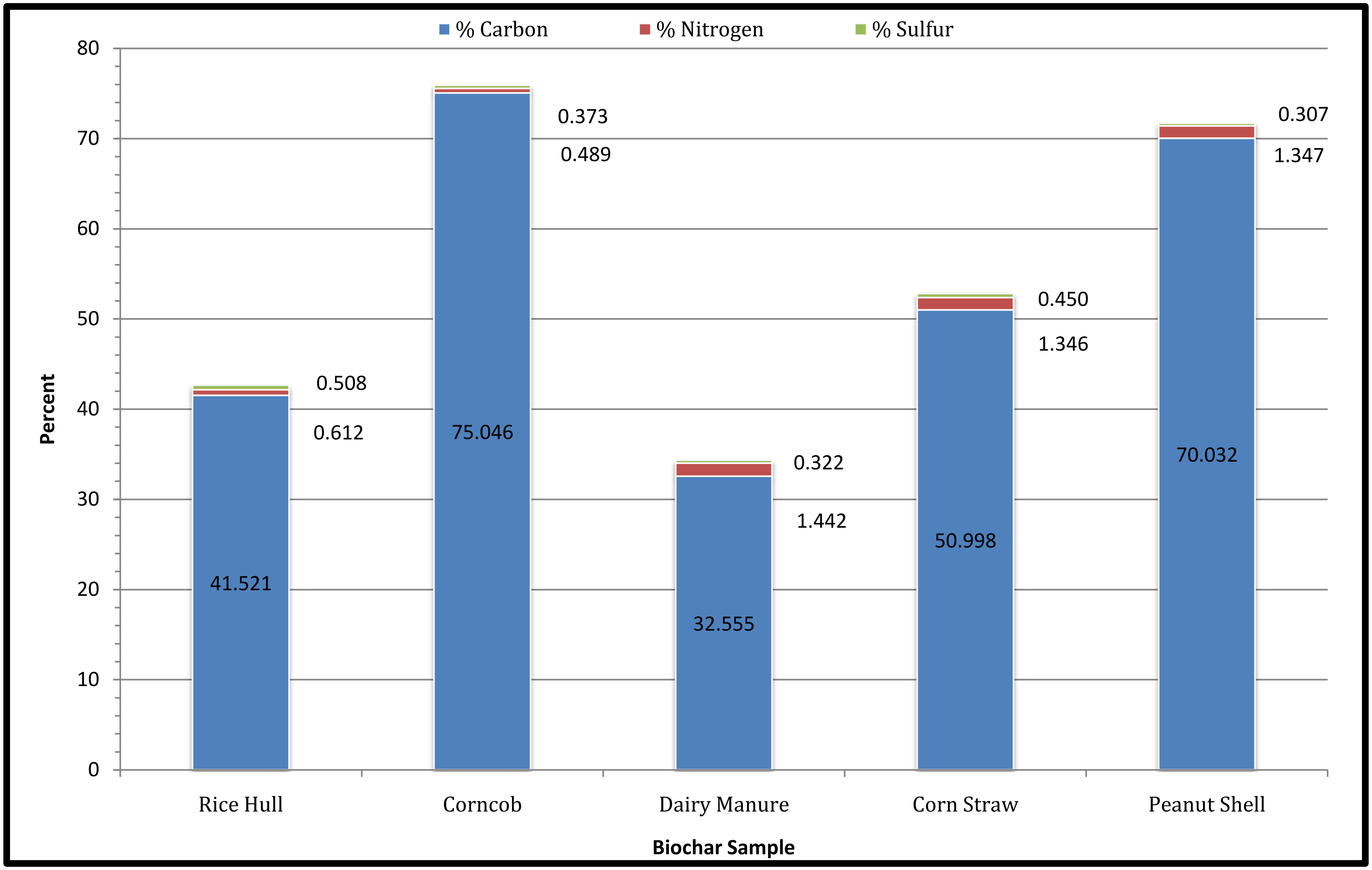


FIGURE 16: ELEMENTAL ANALYSIS OF FIVE BIOCHAR SAMPLES AT 500 CELSIUS

4.3 FOURIER TRANSFORM INFRARED SPECTROMETRY (FTIR)

FTIR is used to characterize the samples of biochar because carbon functional groups, impurities, and water can be detected that may alter the sequestration ability. Some of the differences in the spectra are a result of organic matter combustion and the concentration of the mineral components that was changed when heated (18). Appendix B has the FTIR data graphed and shows a number of similarities amongst the functional groups present for the three different biochars at the same temperature. The only error can be seen at 500 ˚C with the dairy manure sample, this could be a human error or have to do with the fact it was tested on a different day than all of the other samples. The sample should follow the same trend as rice hulls and corncobs at the same temperature but with different intensity. Table 1 below is a detection of the functional groups for each sample (22).

TABLE 1: FUNCTIONAL GROUPS IN EACH SAMPLE

Wave numbers (cm^{-1})	Functional Groups
200 ˚C	
3000	C-H
2802-1820 (stretch)	Large amounts of C-H present
2355	C≡N (could be CO_2 impurity)
1554	C-C
788	C-H
3459 (CORNCOB)	O-H
350 ˚C	
2355	C≡N(could be CO_2 impurity)
1853	C-O
1473	C-C
850	C-H
646	C-Br or C-X
500 ˚C	
(CORN AND RICE)	
3446	O-H
2357	C≡N(could be CO_2 impurity)
1635	C-C or N-H or C=N
844	C-H
(MANURE)	
2335	C≡N(could be CO_2 impurity)
1803	C-O
650	C-Br or C-X

It was difficult to determine the actual groups, but it was done to the fullest extent with the assistance of the graduate students as well as Professor Cao. At 200 Celsius, there is carbon and hydrogen bonding (2802-1820) that with the addition of heat dissipates and is just remaining in the 800 region with the other two higher temperatures because the sample has yet to reach a charring form. Around 1500s there is C-C in all of the samples with varying intensities and widths, carbon interaction is expected in the organic material as well as the carbon rich charcoal more prevalent in lower temperatures and becomes a trace at 500 Celsius. All of the samples also have a C≡N bonding, expected due to the result of nitrogen present in the biochar, the prior nitrogen flushing, and the initial biomass composition. At 350 the samples have a main peak at about 1850, this was determined to be carbon oxygen interaction, that that is slightly present at 500 temperature sample but this trace of oxygen as the temperature increases does not seem to be an accurate analysis, and may be unwanted trace organics or even carbon dioxide. At 500 Celsius the significant peak is around 3500, which was –OH stretching that could indicate water, but because the sample is at high heating this would not be the case but could be a syngas contributor. This was a common result in previous studies completed by Professor Cao.

Through the FTIR analysis more information about the contents of the charcoal is discovered. Oxygen and hydrogen are present, important for contaminant interaction in soil. Also the results show that no matter what the biochar, the temperature is a determination of what functional groups will be in any sample. The percent transmittance of the samples can also be noted for each run.

4.4 BET

BET was used to find the surface areas of the charcoals. Having a high surface area is important to the placement of the biochar underground. With larger surface area per gram of a sample there is less erosion and more ability to capture any particulates that may pass through the sink or into the biochar fertilized soil. Therefore the longevity is increased and carbon capturing can take place over a longer period of time. Table 2 below includes the data from the BET detection. From the table, the higher temperatures have the larger surface area and rice has the larger square meter per gram. Similar to previous characterization, this can be connection to the initial sample of biomass that was used and also the grinding method that was used for preparation prior to the testing.

TABLE 2: SURFACE AREA

Biochar	**Surface Area (m^2/g)**
Corn 200	1.91
Corn 350	2.44
Corn 500	31.7
Rice 200	1.92
Rice 350	27.8
Rice 500	198
Manure 200	2.09
Manure 350	5.82
Manure 500	24

4.5 CARBON SEQUESTRATION

TABLE 3: CARBON SEQUESTRATION CALCULATIONS

Biomass	Million Tonnes/Year 2009	Total Biochar Million Tonnes	Total Potential Carbon in Biochar Million Tonnes	CO_2 Reduction Million Tonnes
Rice	678	226.04	93.85	275.3
Peanuts	29	12.249	8.574	25.15
Manure	900	330.21	108.9	319.64
Cob	817	200.32	150.4	441.31
Husks	250	77.225	39.38	115.53

Table 3 above shows the amount of carbon dioxide that has the potential to be captured if all the agricultural waste from biomass was converted to biochar to form carbon sinks. Corn is mainly produced by the United States and China, and based on the experimental data, yields the best conversion of total potential carbon thus the largest amount of carbon dioxide capturing. Table 4 below shows that each year more than 9 billion metric tonnes of carbon are emitted, forming over 34 billion metric tonnes of carbon dioxide. Biochar will be a small, but significant contributor to reducing the emitted amounts.

TABLE 4: YEARLY EMITTED CARBON DIOXIDE **(23)**

Year	Carbon Emissions (billion metric tonnes)	Carbon Dioxide Emissions (billion metric tonnes)
2009	9.28	34.0576
2008	9.45	34.6815
2007	9.31	34.1677
2006	9.22	33.8374

CHAPTER FIVE: CONCLUSIONS AND RECCOMENDATIONS

Over the two month project period, all of the objectives set by Professor Cao were brought to completion. Understanding the production of biochar and determining a methodology for his students was fulfilled. Also the characterization of the charcoal samples provided information to draw a number of conclusions that with further production and analysis may determine the best biochar for soil nutrition, immobilization and sequestration. The time constraints did have a slight impact on the amount of data collected, also resources and language barriers proved to hinder experimental procedures and communication of ideas and theory. Despite all of the minor issues and setbacks the project was completed with the following conclusions drawn:

- With an increase in temperature, there is a decrease in the yield of biochar and at 500°C dairy manure has the largest % yield.
- At 500°C corncobs yield the greatest percentage of carbon potentially due to biomass composition or initial material size.
- Surface area increases with temperature and rice proves to have the largest surface area, which is at 500°C.
- FTIR yielded the presence of hydroxide ions, some possible carbon dioxide, oxygen, carbon, and nitrogen.
- Corncobs have the highest % C present in biochar and subsequently the highest potential CO_2 sequestration due to mass production.
- Corncobs have the highest sequestration capabilities based on the world production rates.

In order to aid the team at SJTU with research and production later, the following recommendations have been made:

- Altering the reactor tray design to include removable end walls to prevent biochar from falling out during the weighing process.
- Stoppers for the reactor to ensure that after flushing more oxygen does not enter.
- Seal the door of the furnace, flush the furnace with nitrogen gas before pyrolysis, keep the door closed during cooling and chose what is considered a cooled temperature.
- Set/Weight Ratio of KBr to Sample in the FTIR
- Grinding
  - Before production

    - Use a machine instead of hand grinding.
    - Grind a sample once; select the best size of the biochar first before characterization.

- Elemental/BET
    - Select and maintain a sample weight.

With additional testing and production, the application of biochar as a significant means of carbon dioxide sequestration can be highly beneficial to China and the rest of the world. This is a simplistic low cost means of adding nutrients to soil and helping agriculture flourish, therefore it can be useful in third world countries. With carbon capturing, there is very little impact on people or other organisms and the effects of global warming could be reduced. Environmental protection and human health will be the leading benefactors in large scale biochar production.

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APPENDICES

APPENDIX A: PRODUCTION DATA TABLES

Table A1: Furnace Rate of Heating (1)

RATE OF HEATING		
Temperature 1	**Temperature 2**	**Difference in 1 Min**
47	51	4
51	60	9
60	73	13
73	97	24
97	111	14
111	133	22
133	157	24
157	178	21
178	203	25
203	225	22
225	246	21
246	269	23
269	290	21
290	309	19
309	334	25
334	347	13
347	370	23
370	382	12
382	404	22
404	416	12
416	431	15
431	447	16
447	462	15
462	479	17
479	491	12
	AVERAGE	**18.5 °C min^{-1}**

Table A2: Furnace Rate of Heating (2)

RATE OF HEATING		
Temperature 1	**Temperature 2**	**Difference in 1 Min**
65	70	5
70	80	10
80	95	15
95	113	18
113	134	21
134	158	24
158	180	22
180	204	24
204	229	25
229	251	22
251	273	22
273	295	22
295	320	25
320	336	16
	AVERAGE	**19.36 °C min^{-1}**

Table A3: Corncob Biochar Production Data

CORNCOB BIOCHAR PRODUCTION		
DATE	BEGIN TIME	END TIME
18-Nov-10	**10:35**	**2:35**
500		
MASS (GRAMS)	**Before Pyrolysis**	**After Pyrolysis**
Beaker Mass	**316.55**	**319.17**
With Corn	**414.51**	**343.19**
	97.96	**24.02**
	BIOCHAR PERCENT	**24.52**
DATE	BEGIN TIME	END TIME
19-Nov-10	11:35	3:35
350		
MASS (GRAMS)	**Before Pyrolysis**	**After Pyrolysis**
Beaker Mass	316.56	319.2
With Corn	416.66	353.4
	100.1	34.2
	BIOCHAR PERCENT	**34.17**
DATE	BEGIN TIME	END TIME
22-Nov	11:01	3:01
200		
MASS (GRAMS)	**Before Pyrolysis**	**After Pyrolysis**
Beaker Mass	316.51	319.15
With Corn	416.12	396.73
	99.61	77.58
	BIOCHAR PERCENT	**77.88**

Table A4: Dairy Manure Biochar Data

DAIRY MANURE BIOCHAR PRODUCTION		
DATE	BEGIN TIME	END TIME
2-Dec	9:15	1:15
500		
MASS (GRAMS)	**Before Pyrolysis**	**After Pyrolysis**
Beaker Mass	316.56	319.2
With Manure	418.96	356.77
	102.4	37.57
	BIOCHAR PERCENT	**36.69**
DATE	BEGIN TIME	END TIME
24-Nov	2:00	6:00
350		
MASS (GRAMS)	**Before Pyrolysis**	**After Pyrolysis**
Beaker Mass	316.54	319.21
With Manure	416.15	365.29
	99.61	46.08
	BIOCHAR PERCENT	**46.26**
DATE	BEGIN TIME	END TIME
25-Nov	11:36	3:36
200		
MASS (GRAMS)	**Before Pyrolysis**	**After Pyrolysis**
Beaker Mass	**316.53**	**319.24**
With Manure	**417.16**	**397.33**
	100.63	**78.09**
	BIOCHAR PERCENT	**77.60**

Table A5: Rice Hull Biochar Production Data

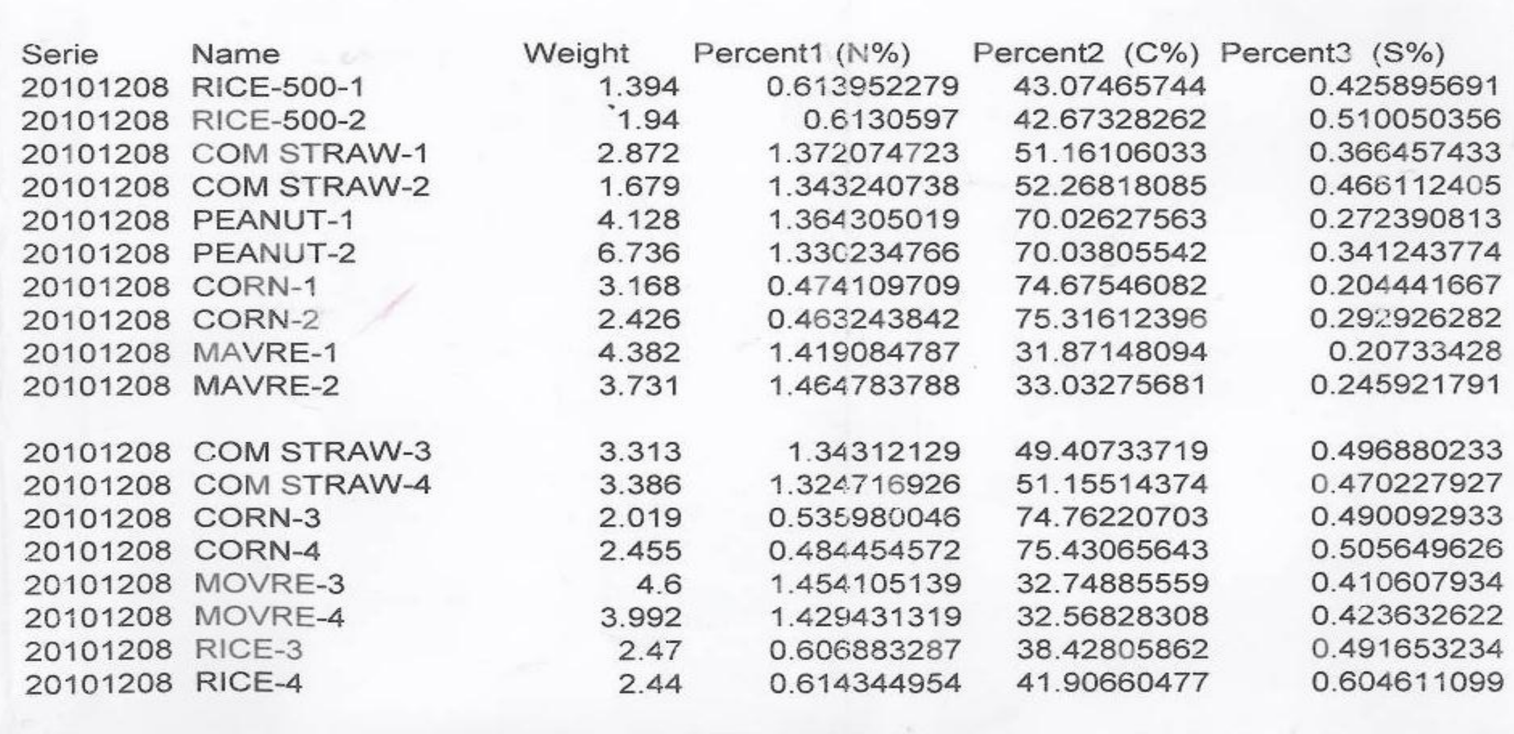
RICE HULL BIOCHAR PRODUCTION		
DATE	BEGIN TIME	END TIME
29-Nov	10:40	2:40
500		
MASS (GRAMS)	**Before Pyrolysis**	**After Pyrolysis**
Beaker Mass	316.55	319.88
With Rice	416.83	353.31
	100.28	33.43
	BIOCHAR PERCENT	**33.34**
DATE	BEGIN TIME	END TIME
30-Nov	9:15	1:15
350		
MASS (GRAMS)	**Before Pyrolysis**	**After Pyrolysis**
Beaker Mass	316.58	319.21
With Rice	416.78	361.21
	100.2	42
	BIOCHAR PERCENT	**41.92**
DATE	BEGIN TIME	END TIME
1-Dec	9:24	1:24
200		
MASS (GRAMS)	**Before Pyrolysis**	**After Pyrolysis**
Beaker Mass	316.54	319.24
With Rice	416.24	400.23
	99.7	80.99
	BIOCHAR PERCENT	**81.23**

Table A6: Other Biochar Produced at 500˚C

OTHER PRODUCED BIOCHAR		
3-Dec	11:30	3:30
Corn Straw 500		
MASS (GRAMS)	**Before Pyrolysis**	**After Pyrolysis**
Beaker Mass	316.55	319.23
With CS	389.95	341.9
	73.4	22.67
	BIOCHAR PERCENT	**30.89**
DATE	BEGIN TIME	END TIME
6-Dec	10:00	2:00
Peanut Shells 500		
MASS (GRAMS)	**Before Pyrolysis**	**After Pyrolysis**
Beaker Mass	316.58	319.21
With PS	390.01	350.23
	73.43	31.02
	BIOCHAR PERCENT	**42.24**

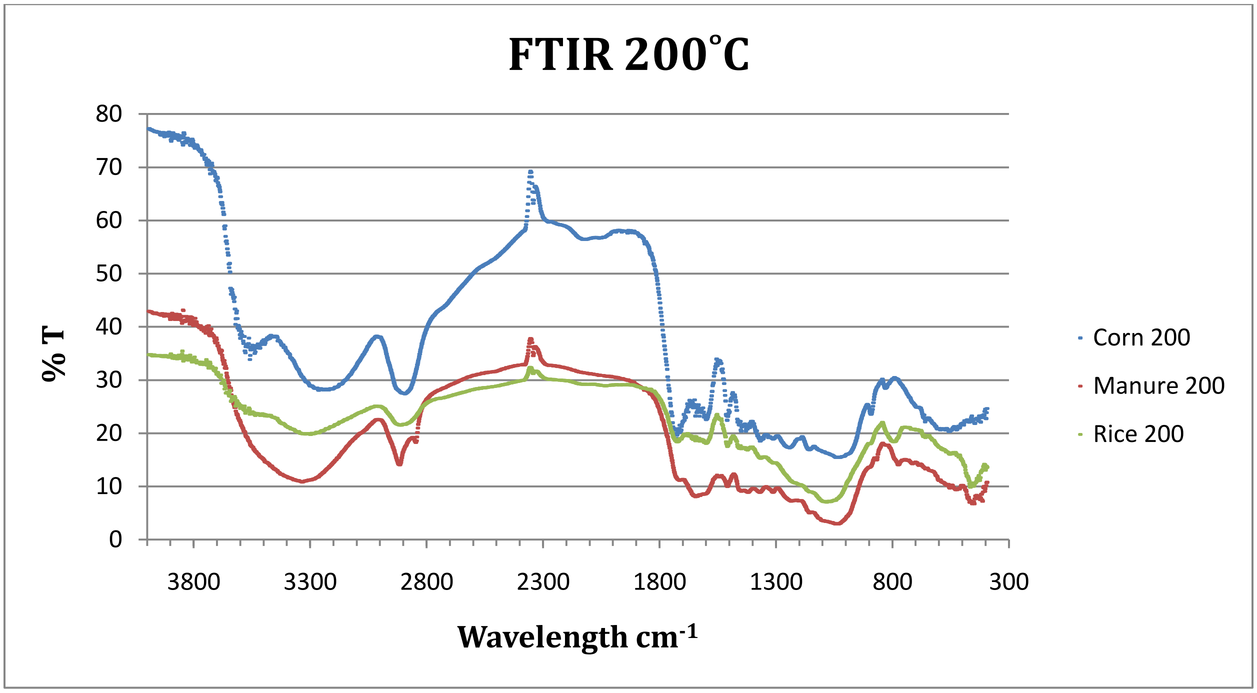
APPENDIX B: TESTING RESULTS

Table B1: Elemental Analysis

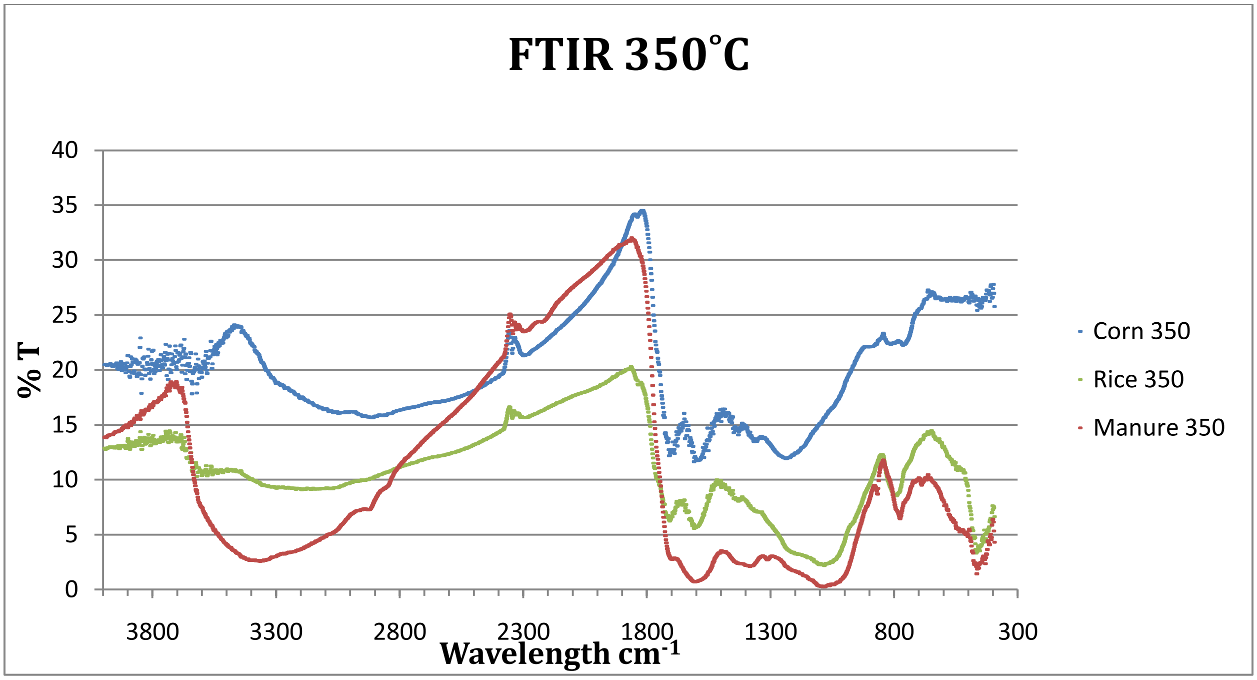


Serie	Name	Weight	Percent1 (N%)	Percent2 (C%)	Percent3 (S%)
20101208	RICE-500-1	1.394	0.613952279	43.07465744	0.425895691
20101208	RICE-500-2	1.94	0.6130597	42.67328262	0.510050356
20101208	COM STRAW-1	2.872	1.372074723	51.16106033	0.366457433
20101208	COM STRAW-2	1.679	1.343240738	52.26818085	0.466112405
20101208	PEANUT-1	4.128	1.364305019	70.02627563	0.272390813
20101208	PEANUT-2	6.736	1.330234766	70.03805542	0.341243774
20101208	CORN-1	3.168	0.474109709	74.67546082	0.204441667
20101208	CORN-2	2.426	0.463243842	75.31612396	0.292926282
20101208	MAVRE-1	4.382	1.419084787	31.87148094	0.20733428
20101208	MAVRE-2	3.731	1.464783788	33.03275681	0.245921791
20101208	COM STRAW-3	3.313	1.34312129	49.40733719	0.496880233
20101208	COM STRAW-4	3.386	1.324716926	51.15514374	0.470227927
20101208	CORN-3	2.019	0.535980046	74.76220703	0.490092933
20101208	CORN-4	2.455	0.484454572	75.43065643	0.505649626
20101208	MOVRE-3	4.6	1.454105139	32.74885559	0.410607934
20101208	MOVRE-4	3.992	1.429431319	32.56828308	0.423632622
20101208	RICE-3	2.47	0.606883287	38.42805862	0.491653234
20101208	RICE-4	2.44	0.614344954	41.90660477	0.604611099

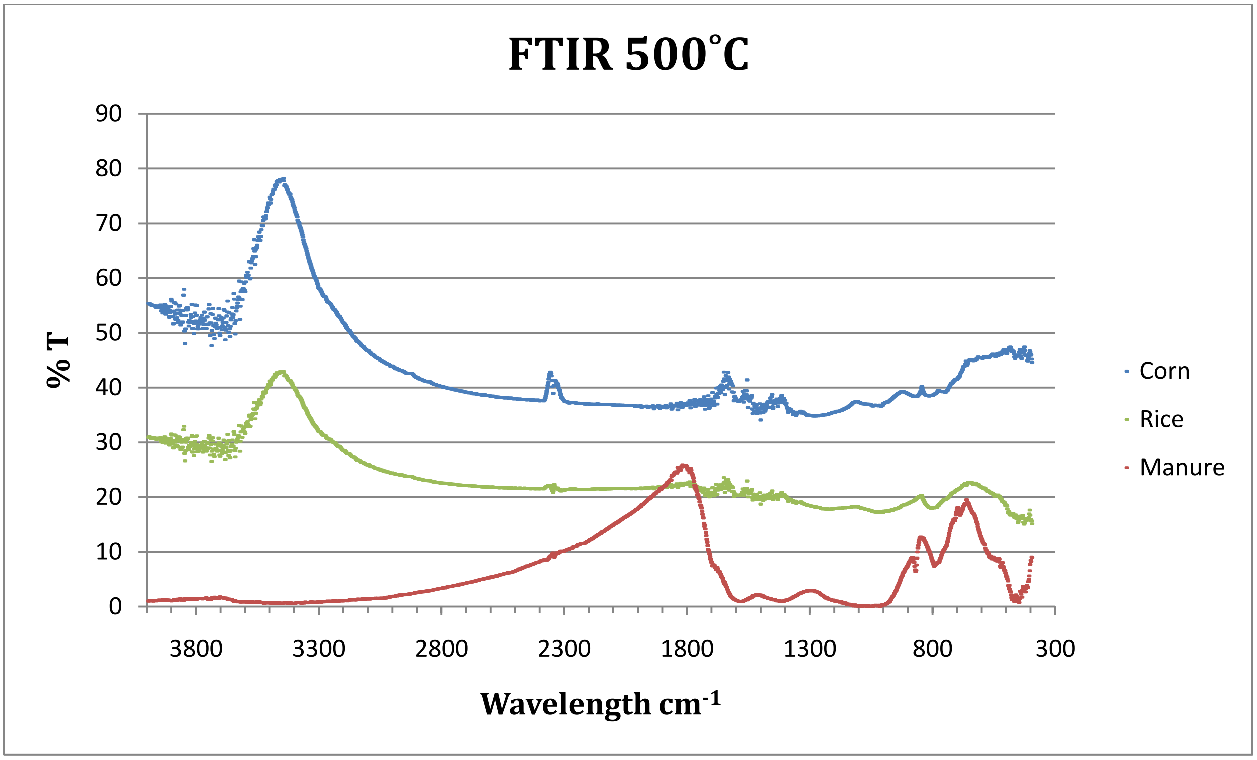
Graph B2: FTIR Analysis at 200 °C



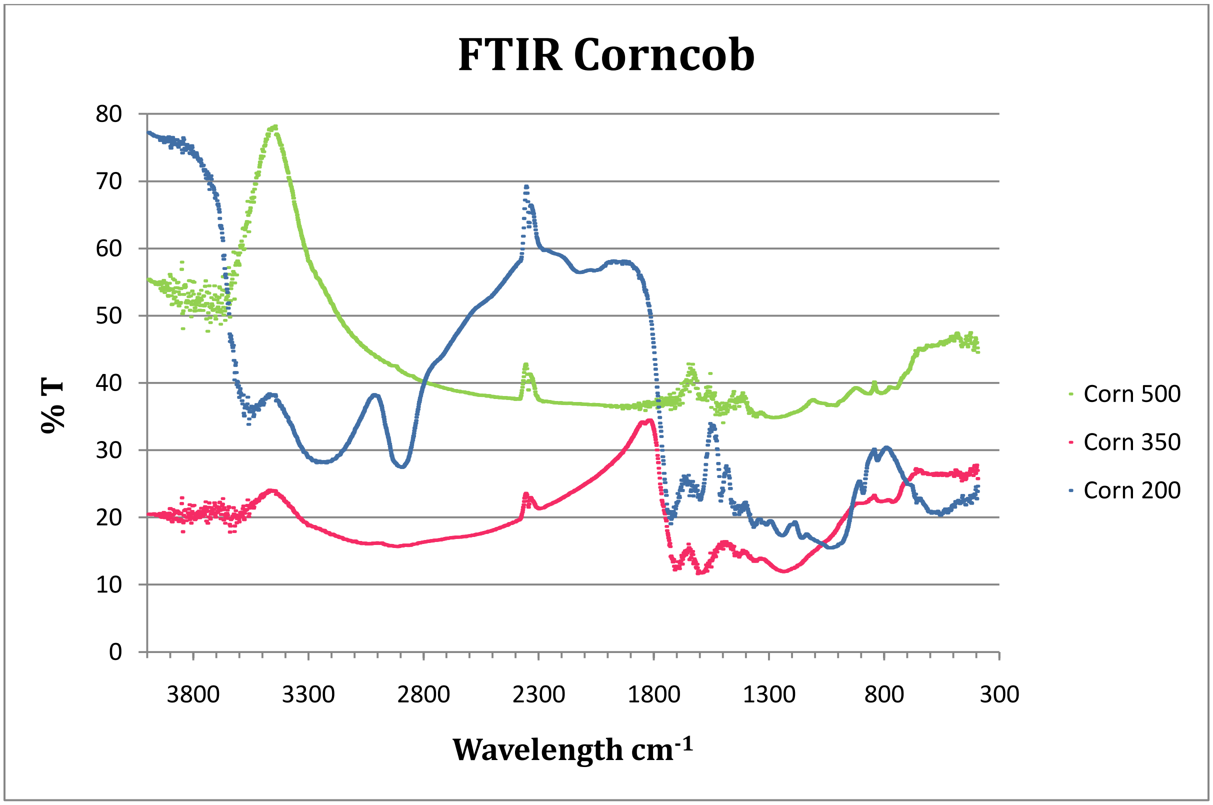
Graph B3: FTIR Analysis at 350 °C



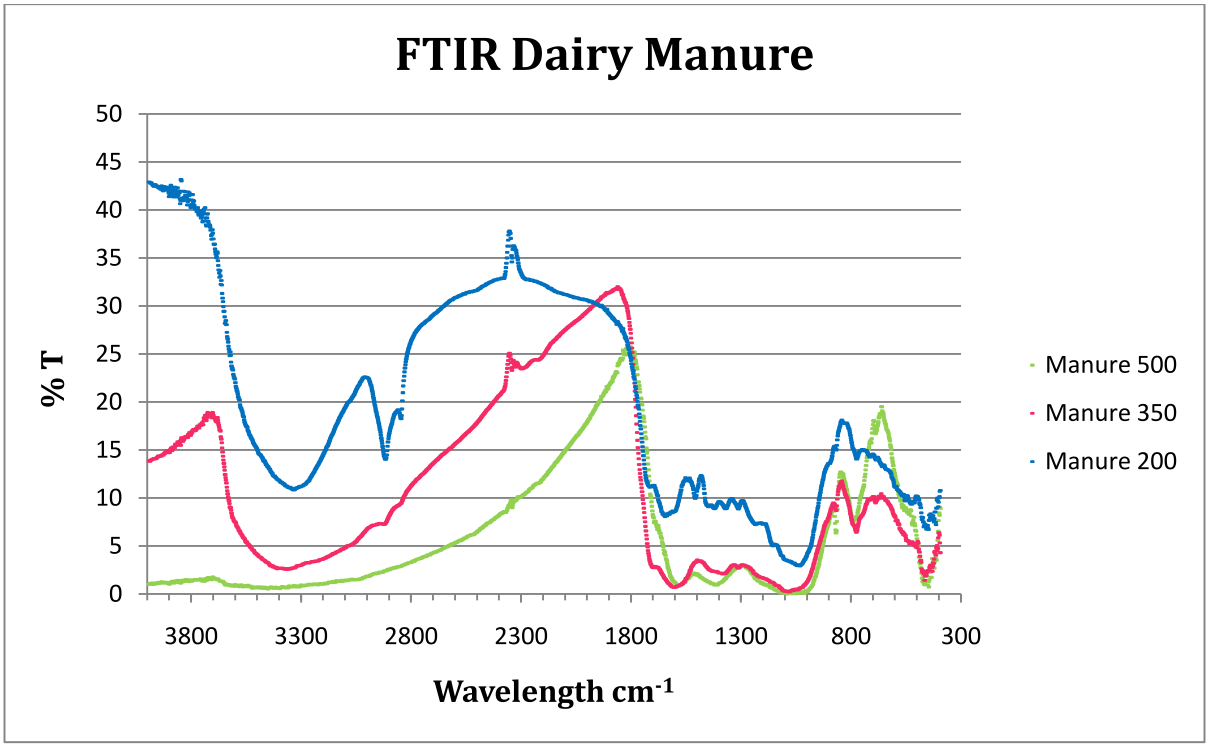
Graph B4: FTIR Analysis at 500 ˚C



Graph B5: FTIR Analysis Corncob



Graph B6: FTIR Analysis Dairy Manure



Graph B7: FTIR Analysis Rice Hulls