BIOMASS THERMAL PRE-TREATMENT FOR CO-GASIFICATION: CO_2 TORREFACTION

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BIOMASS THERMAL PRE-TREATMENT FOR CO-GASIFICATION: CO_2 TORREFACTION

MOHAMMAD HAFIZUDDIN BIN MOHD YUSOFF

Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering

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JUNE 2015

SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering.

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Date	: JUNE 2015

STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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Dedication

Special dedication to my supervisor, Dr. Ruwaida binti Abdul Rasid for your Time, Guidance and Support.

And,

To my beloved parent (Mohd Yusoff Abdullah & Naliha bt Abd Ghani), my siblings and friends, that encouraged and fully supports me throughout completing this thesis.

ACKNOWLEDGEMENT

First and foremost, I would like to express my deepest appreciation to my dear supervisor, Dr. Ruwaida Binti Abdul Rasid for her valuable suggestion and endless support. She has provided me with an excellent atmosphere for doing this research. Special gratitude again goes to her guidance in all the time of research and writing of this thesis. I could not have imagined having a better supervisor for my bachelor study. Also, special thanks to our FYP coordinator, Dr Sumaiya and to all our FKKSA lecturer's especially to the panels who in during presentation give tips, advice and endless cooperation. Apart from that, I would also like to thank the lab technician for providing their services during my experimental work in the FKKSA laboratory. Not forgetting my friends for being helpful and gives information on URP throughout the semester. Last but not least, I would like to express my heartiest appreciation to my family for their unfathomable encouragement. They provided me with transportation and financial support whenever I need them. My family's love and support were crucial for me to pass through all difficult stages in the completion of this research.

ABSTRACT

Biomass has been a very reliable alternative for renewable fuel to reduce the use of conventional fuel due to environmental concern and sustainability issue. In order to reduce the emission of the harmful greenhouse gases and to promote sustainable environment, the renewable energy is the perfect solution to achieve the targets. But there are some problems that needs to be overcome before the biomass can be utilised as biochar or biofuel through gasification by using the torrefaction method. Torrefaction of biomass is a pre-treatment process that aims to improve the fuel properties by lowering the moisture content and increasing the energy density at temperature between 200°C to 300°C in either inert or non-inert environment. The objective of this work is to study the effect of CO_2 concentration present during torrefaction at temperature 240°C and 280°C. In addition, the impact of torrefaction time was also evaluated at 15 and 30 minutes. In this study, the sample used is empty fruit bunches (EFB) from palm oil mills in Felda Lepar. The torrefaction reactor consists of a vertical stainless-steel tubular reactor equipped with an electrically heated furnace. The experimental apparatus is setup consist of gas tanks, N_2 and CO_2 that are controlled by regulators and furnace. The gas will be mixed at 0%, 10%, 15%, and 21% value of CO_2 while the rest is N_2. It was determined that the mass yield is decreased with increased in the CO_2 concentration. This suggests the occurrence of oxidative reaction, for example the Boudouard reaction and an enhancement of volatile matter release. The heating value of the torrefied biomass shows an increasing trend, with temperature increased. This is consistent with the findings in the literature, which primarily indicates improved fuel properties. The comparable mass loss at lower temperatures improved fuel properties, makes utilization of carbon dioxide as a torrefaction medium for pre-treating biomass as an attractive technology.

ABSTRAK

Biojisim adalah alternatif yang sangat berguna untuk bahan api yang boleh diperbaharui bagi mengurangkan penggunaan bahan api konvensional berikutan kebimbangan keatas isu kelestarian alam sekitar. Dalam usaha untuk mengurangkan pelepasan gas rumah hijau yang berbahaya dan untuk menggalakkan persekitaran yang sihat, sumber tenaga boleh diperbaharui adalah penyelesaian yang sempurna untuk mencapai sasaran tersebut. Tetapi terdapat beberapa masalah yang perlu diatasi sebelum biojisim boleh ditukar ke arang atau bahan api bio dengan menggunakan kaedah gasifikasi. Kaedah 'torrefaction' untuk biomass adalah satu proses pra-rawatan yang bertujuan untuk meningkatkan sifat-sifat bahan api dengan mengurangkan kandungan kelembapan dan meningkatkan ketumpatan tenaga pada suhu antara 200 ° C hingga 300 °C dalam persekitaran lengai atau tidak lengai. Objektif kajian ini adalah untuk mengkaji kesan kepekatan CO_2 yang hadir dalam kaedah "torrefaction" pada suhu 240 ° C dan 280 ° C. Di samping itu, kesan masa torrefaction juga telah dinilai pada 15 dan 30 minit. Di dalam kajian ini, sampel yang digunakan ialah Buah Tandan kosong (EFB) yang diambil dari kilang kelapa sawit di Felda Lepar. Reaktor torrefaction terdiri daripada reaktor tiub menegak yang diperbuat dari keluli tahan karat dilengkapi dengan perapian elektrik yang dipanaskan. Antara peralatan eksperimen yang digunakan terdiri daripada tong gas yang mengandungi N_2 dan CO_2 dikawal bersama dengan pengawal selia dan relau. Gas tersebut akan bercampur pada 0%, 10%, 15%, dan 21% kepekatan gas CO_2 yang mana selebihnya adalah gas N_2. Ia telah didapati bahawa kadar hasil jisim telah berkurang dengan peningkatan dalam kepekatan CO_2. Ini menunjukkan berlakunya reaksi oksidatif, sebagai contoh reaksi Boudouard dan penambahan pada ruapan bahan. Nilai kalori pada biojisim yang telah dipanaskan menunjukkan peningkatan pada aliran, dengan suhu meningkat. Ini selaras dengan keputusan daripada lain-lain artikel, terutamanya yang menujukkan ciri-ciri bahan api yang lebih baik. Perbandingan kehilangan jisim pada suhu yang lebih rendah yang memperbaiki sifat bahan api pada biojisim menjadikan penggunaan karbon dioksida sebagai medium torrefaction untuk kaedah pra-rawatan biojisim yang menarik.

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LIST OF SYMBOL

%	-	Percentage
°C	-	Degree Celsius
g	-	Gram
min	-	Minute

LIST OF ABBREVIATION

EFB	-	Empty Fruit Bunch
O/C	-	Oxygen-Carbon Ratio
y_M	-	Mass yield
CO_2	-	Carbon Dioxide
CV	-	Calorific value (MJ/kg)
y_E	-	Energy yield
TGA	-	Thermogravimetric analysis
HHV	-	Higher heating value

CHAPTER 1

1 INTRODUCTION

1.1 Background study

Biomass has been known as a renewable fuel that can be used to produce heat and power. The increasing of world population and economic growth leads to increase in world energy demand and consumption especially in developing countries. Many of the renewable energies sources such as solar, wind, biomass, tide, wave and geothermal energy are being developed nowadays (Lund, 2007). Moreover, the depletion of conventional fuel over the years has urged scientists all over the world to think about the solution for this problem. Therefore, a reliable affordable and clean energy supply such as biomass as a substitute to fossil fuels has become more promising.

Renewable energy especially biomass seems to be most promising option for Malaysia. Malaysia was the second largest producer of palm oil, producing 18.9 million tonnes, or 38% of the total world supply in 2011 (PalmOilWorld.org 2011). There are many of waste products from the palm oil industry that can be used as a biomass. They include empty fruit bunches (EFB), mesocarp fiber, kernel shells, fronds and trunks (Uemura et al., 2013). Normally, biomass is burned to release the chemical energy in the material and convert it into heat energy and carbon dioxide (CO_2) produced is released into the atmosphere. It is considered a carbon-neutral fuel that is based on the cycle between carbon and the atmosphere (Chen and Wu, 2009). Even though there are some problems need to be overcome such as lower calorific value, high moisture content (Chen et al., 2011), low energy density and high ash content (Uemura et al., 2013), its potentials are limitless.

CO_2 is one of the greenhouse gases that is released into the environment during combustion of fossil and renewable sources. Hot gases produced from boiler exhaust that have higher percentage of CO_2 in it that easy to obtain makes it an alternative option to be used as the biomass pre-treatment medium.

Among technologies developed to improve these problems is the torrefaction process, in which the biomass are thermally treated in temperature between 200 to 300°C under atmospheric conditions without the presence of any types of oxidizer (Peng et al., 2012). Recently, it was found that the presence of a small quantity of oxidizer at the torrefaction condition has the potential to improve the properties of torrefied biomass (Thanapal et al., 2014, Uemura et al., 2013).

As for this research, the study will be concerning on the investigation of the optimum temperature and the effectiveness of carbon dioxide in the torrefaction process that will help to increase the fuel properties of biomass using empty fruit bunches. Therefore, the study will be the seed of hope for the solution to our global energy problem besides promoting the potential of local agricultural waste.

1.2 Motivation and statement of problem

There are many reports about global warming and changing of earth's climate that is a big threat and challenge in our era. The main factor that causes this kind of disaster is the release of greenhouse gases to the environment. So, to reduce the emission of these greenhouse gases and to promote sustainable environment, the renewable energy is the perfect solution to achieve the targets. On the other hand, Malaysia also one of the biggest producer of palm oil in the world. There are a lot of biomass feedstock that can be used to produce many type of products such as bio-based value added products, bio-fuel and direct fuel for power generation.

Basically for this study, the aim is to improve the fuel properties for the biomass so that the gasification process that takes place after torrefaction will be more efficient. Mainly the biomass has low energy density in combination with its high moisture content result that uses more energy in the gasification process. This is because higher gasification efficiencies can be achieved for fuels with low O/C ratios, such as coal, than for fuels with high O/C ratios such as biomass (Prins et al., 2003).

Using raw biomass for the gasification process will results in poor syngas production and will results in high tar concentration. Thermal pre-treatment or torrefaction of biomass is a promising method to convert biomass to high energy density hence a superior biomass in the form of bio char. So, under certain torrefaction conditions, the torrefied biomass can have properties comparable to low rank coals that may be used for heat and power generation (Chen and Kuo, 2010).

In this study, torrefaction of empty fruit bunches was carried out in a fixed tubular reactor in the presence of carbon dioxide ranging from 0% to 21%. The effects of carbon dioxide, temperature and torrefaction time on the mass and energy yields were investigated. The EFB was dried, ground and sieved before torrefaction.

1.3 Research objectives

This research project aims to investigate the effectiveness of CO_2 in enhancing the torrefaction process in producing bio-char with properties compatible to coal. Moreover, this study aims to improve the torrefaction process by using different concentration of carbon dioxide, CO_2 as an enhancer. The optimum temperature to be used in this process will also be determined. This is then followed by the characterization of the properties of biomass based on the analysis that will be conducted.

1.4 Scope of this research

The following are the scope of this research:

a) To study the effect of temperature at 240°C and 280°C on the biomass samples in the torrefaction process.
b) To study the effect of concentration of CO_2 at 10%, 15% and 21% in enhancing the torrefaction process.
c) To investigate the effect of torrefaction time in the torrefaction process.

1.5 Organisation of this thesis

The structure of the reminder of the thesis is outlined as follows:

Chapter 2 provides a description of the torrefaction process that is the important theory in this study. Also, there are general description on the condition of the torrefaction process and why this process is suitable to change the properties of biomass that will be used in the gasification process. This chapter also provides a brief discussion of previous study conducted in the torrefaction process.

Chapter 3 gives a review of the procedure be conducted for the experiment. First, the preparation of samples was explained before proceed to the experiment. In this chapter, we explained about how to prepare the samples and the initial drying before the EFB can be torrefied. Also, there are explanation about the procedure to conduct the torrefaction experiment whether for inert torrefaction gas or the mixture between nitrogen and oxygen.

Chapter 4 gives the results for preparation of samples and the expected result obtained from the journal. In this chapter, some of graph was plotted to shown the trend of mass yield over the temperature as well as the concentration of CO_2. Some of discussion also presented to support the result from the previous works.

Chapter 5 draws together a summary of the full thesis and outlines the future work which might be derived from this work.

2 LITERATURE REVIEW

2.1 Overview

This chapter presents the basic principles of torrefaction process that will be conducted in this study. It also contains the general knowledge about the torrefaction process and the way to conduct the experiment regarding this process. Moreover, the extensive review from selected journals with the citation is also included in this chapter. Some of the findings in the review and method of previous works also included.

2.2 Biomass

Biomass is organic material from recently living things, including plant matter from trees, grasses, and agricultural crops. The chemical composition of biomass varies among species, but basically consists of high, but variable moisture content, a fibrous structure consisting of lignin, carbohydrates or sugars, and ash.

Common sources of biomass are included agricultural which varies from food grain, bagasse, corn stalk, straw, seed hulls, nutshells and manure from cattle. In Malaysia, the biomass from oil palm wastes such as mesocarp fibre, kernel shell, empty fruit bunch, fronds and trunk are mostly available. Some disadvantages of biomass are it cannot be stored or transport for a long shipping because of the hygroscopic nature (Chen and Kuo 2010).

The hydrophilic nature of biomass is related to the presence of OH groups in biomass. Hemicellulose was found to have the highest potential to adsorb water, followed by cellulose and lignin. Those structure are contain in the biomass that will increase the moisture content of it. Hence, the reason of the hydrophobic nature of torrefied biomass can be recognized to reduce amount of hemicellulose and OH groups in the biomass (Thanapal, et al. 2014).

2.3 Torrefaction Process

Torrefaction is one of the thermal pre-treatment techniques used to improve the properties of biomass (Deng et al., 2009). In this process, the raw biomass is heated in an inert or nitrogen atmosphere at temperatures between 200 - 300°C (Uemura et al., 2013, Deng et al., 2009). It is based on the removal of oxygen from biomass which aims to produce a fuel with increased energy density. A fraction of the volatile matter of the dry biomass is removed as well as the moisture. In the torrefaction process, the properties of biomass can be altered depending on its hydrophilic, fibrous and severity so that the product is brittle and easy to grind (Koppejan et al., 2012). There are studies that proved that characteristic of lignocellulosic can be improved into a useful biofuel.

There are many advantages of torrefaction process such as (1) reduce the moisture content and O/C ratio (Couhert et al., 2009), (2) increase the higher heating value (HHV) or energy density (Yan et al., 2009) of biomass. Also, the torrefied biomass shows more uniform properties such as moisture content compared to the raw biomass (Chen Hsu et al., 2011).

Torrefied biomass mainly used as a renewable fuel for combustion or gasification (Prins, et al., 2003). Torrefaction process is one of best pre-treatment method for biomass before the gasification process take place. Torrefaction studies involving biomass have mostly been conducted on woody (Lauan) blocks (Chen Hsu et al., 2011), pine chips and logging residue chips (Phanphanich and Mani, 2011), rice straw and rape stalk (Deng et al., 2009), empty fruit bunches (EFB) (Uemura et al., 2013), bamboo (Chen et al., 2011), mesocarp fiber, kernel shell, beech wood (Couhert et al., 2009) and willow.

Chen et al., (2011) torrefied the woody biomass (Lauan) blocks and recommended that the operation should be conducted at the temperature of 250°C and time longer than 1 hour. Deng et al. (2009) studied the yields of solid and liquid product of rice straw and rape stalk at temperature 200°C, 250°C and 300°C. The result showed that as the temperature increases, the yield of solid decreases. Couhert et al.(2009) stated that the energy density increases with torrefaction temperature and found an increase of 20% at a torrefaction temperature of 280°C.

2.4 Previous work on Torrefaction Process

One of the previous study of the torrefaction process using the CO_2 as the enhancer used woody biomass which are mesquite and juniper as their samples (Thanapal, et al. 2014). The higher mass loss at the temperature range for torrefaction was obtained by using the CO_2 compared to the inert atmosphere. Other than that, by comparing the mass and energy yield of the torrefaction of mesquite and juniper, the optimum temperature seems to be at 240°C. The use of CO_2 as the torrefaction medium was found to improve the grindability of the biomass because of the increased surface area of biomass samples.

Research done by Arias et al. (2008) on the torrefaction of eucalyptus showed that torrefaction of raw biomass increases the grindability of the biomass. The samples (eucalyptus) were torrefied between 240°C and 280°C with varying residence times between 15 minutes to 3 hours. The size distribution analysis showed an increase in the residence time at a given temperature resulted in smaller particle sizes, indicating easier grindability.

Some of the previous work is mostly discussed on the effect of temperature and residence time on different biomass but most of them are not focused about the effect of biomass in non-inert atmosphere for torrefaction. The uses of CO_2 in torrefaction process will provide an opportunity for a lower pretreatment cost of biomass.

2.5 Torrefaction Technologies in Industries

Some torrefaction technologies are proficient of handling feedstock with small particles such as sawdust and other are capable of processing large particles. Example of torrefaction technologies that can be found in industries nowadays such as rotating drum, screw reactor, torbed reactor and fixed bed. The most important reactor technologies are briefly described below according to their advantages.

The rotating drum is a continuous reactor and can be regarded as proven technology for various application (Koppejan, et al., 2012). For torrefaction applications, the biomass in the reactor can be either directly or indirectly heated using superheated steam of flue gas resulting from the combustion of volatiles.

Besides that, screw type reactor also being used in torrefaction process. It is a continuous reactor, consisting of one or multiple auger screws that transport the biomass through the reactor (Junsatien, et al., 2013). A disadvantage of screw reactor is the formation of char on the hot zones that caused limited mixing of the biomass. A screw reactor is relatively inexpensive, however the scaleability is limited because the ratio of screw surface are to reactor volume decreases for larger reactors.

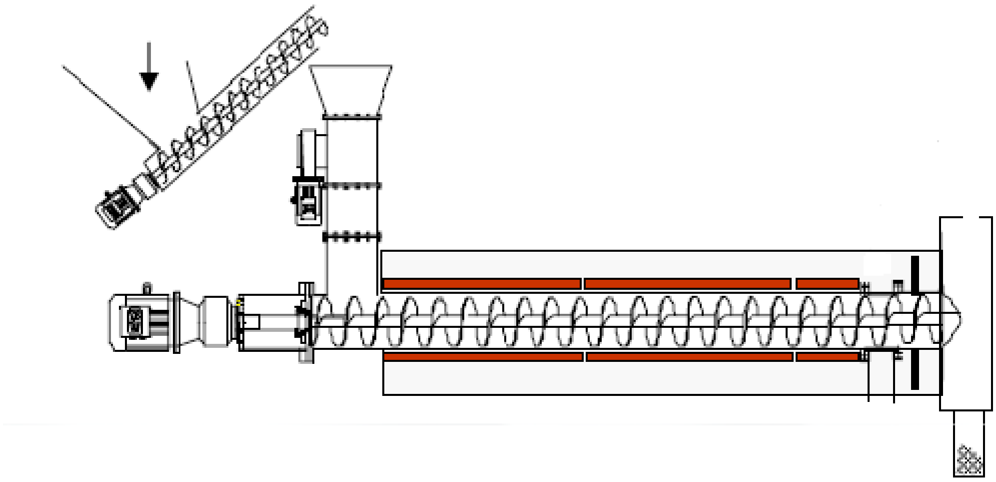


Figure 2-1-Auger screw type reactor

The torbed reactor basically are proven technology for various applications, including combustion. Recently, torrefaction process are done in torbed technology only in batchwise that is very small scale (2 kg/h). In a torbed reactor, a heat carrying medium is blown from the bottom of the bed with high velocity that gives biomass particles inside the reactor both a vertical and horizontal movement (Harrison 2014). It can heat up to 380°C that resulting in higher loss of volatiles.

2.6 *Gasification of Biomass*

Gasification is the conversion of raw materials into solid or liquid fuel gas that is valuable and convenient or chemical raw materials that can be burned to release energy or used for production of high-value added chemicals (Prabir, 2013). The low moisture content and good ratios of C/H/O in the biomass samples after torrefaction is excellent for gasification. Meanwhile, gasification that involved biomass is the conversion of an organically derived, carbonaceous feedstock by partial oxidation into a gaseous product, synthesis gas or "syngas" consisting primarily of hydrogen (H_2) and carbon monoxide (CO), with lesser amounts of carbon dioxide (CO_2), water (H_2O), methane (CH_4), higher hydrocarbons and nitrogen (N_2) (Jared P., 2002).

Sometimes pyrolysis, which takes place in higher temperatures and produces a mixture of gases with hydrogen content can be considered as biomass gasification. Gasification of biomass is generally observed to follow these reaction:

$$Biomass + Heat \xrightarrow{yields} CO, CO_2, H_2O, H_2, CH_4 \ and \ other \ CH_s, + ash + char$$

In the gasification process, the particle size and moisture contents are the crucial factor for operation. Hence, the smaller particle size gives additional surface area to react in the gasification process and there is more energy is required at the high operating temperature to break the complex long chain of lignincellulosic biomass into simplest carbon chain of syngas. Gasification using torrefied biomass could potentially benefit from improved flow properties of the feedstock, increased levels of H_2 and CO_2 in the resulting syngas, and improved overall process efficiencies (Koppejan, Sokhansanj, et al., 2012). The grindability could be considered positive aspect in the case of entrained flow gasifier. As of yet, there is hardly any practical knowledge available on the options and limitations of torrefied biomass for gasification. Gasification of low quality biomass results in poor synthetic gas quality and high tar concentration.

3 MATERIALS AND METHODS

3.1 Overview

This chapter presents a methodology to conduct the torrefaction experiment by using certain raw materials and experiment setup. Preparation of EFB samples and procedure for torrefaction experiment is included here. Several characterization tests are done to determine the characteristic of EFB and the effectiveness of torrefaction process.

3.2 Preparation of biomass samples

The raw materials that will be used in this study is the Empty Fruit Bunches (EFB) shown in Figure 3-1. EFB is the residue generated at the thresher, where fruits are removed from fresh fruit bunches. EFB were collected from an oil palm plantation in Lepar, Pahang. About 5 kg EFB samples were collected from the site. The raw EFB is dried in the sun before proper drying process. For the drying process, a bunch of EFB samples is weighed and then placed in a tray. Then, the EFB samples were dried in an oven at the temperature of 65°C for 24 hours to provide a basis for experiments. The step was repeated until 500 gram of the EFB samples was accumulated. For this, the raw EFB samples were further ground using the mechanical grinder and sieved using sieve shaker. From the sieved samples it was determined that the largest particle size that would allow for an adequate sample amount during torrefaction experiment were samples below 1 mm size. The ground samples were removed and placed in airtight plastic bags and stored until needed.

For the initial analysis, the samples will be analysed for proximate, elemental (ultimate) analyses as well as the calorific value (CV). For further preparation that is required for use in TGA experiment, the EFB will be chopped into small pieces by using a chopper and grounded using a laboratory grade blender. The Figure 3-2 shows the flow chart of preparation of samples.

Figure 3-1 Raw Empty Fruit Bunches (EFB)

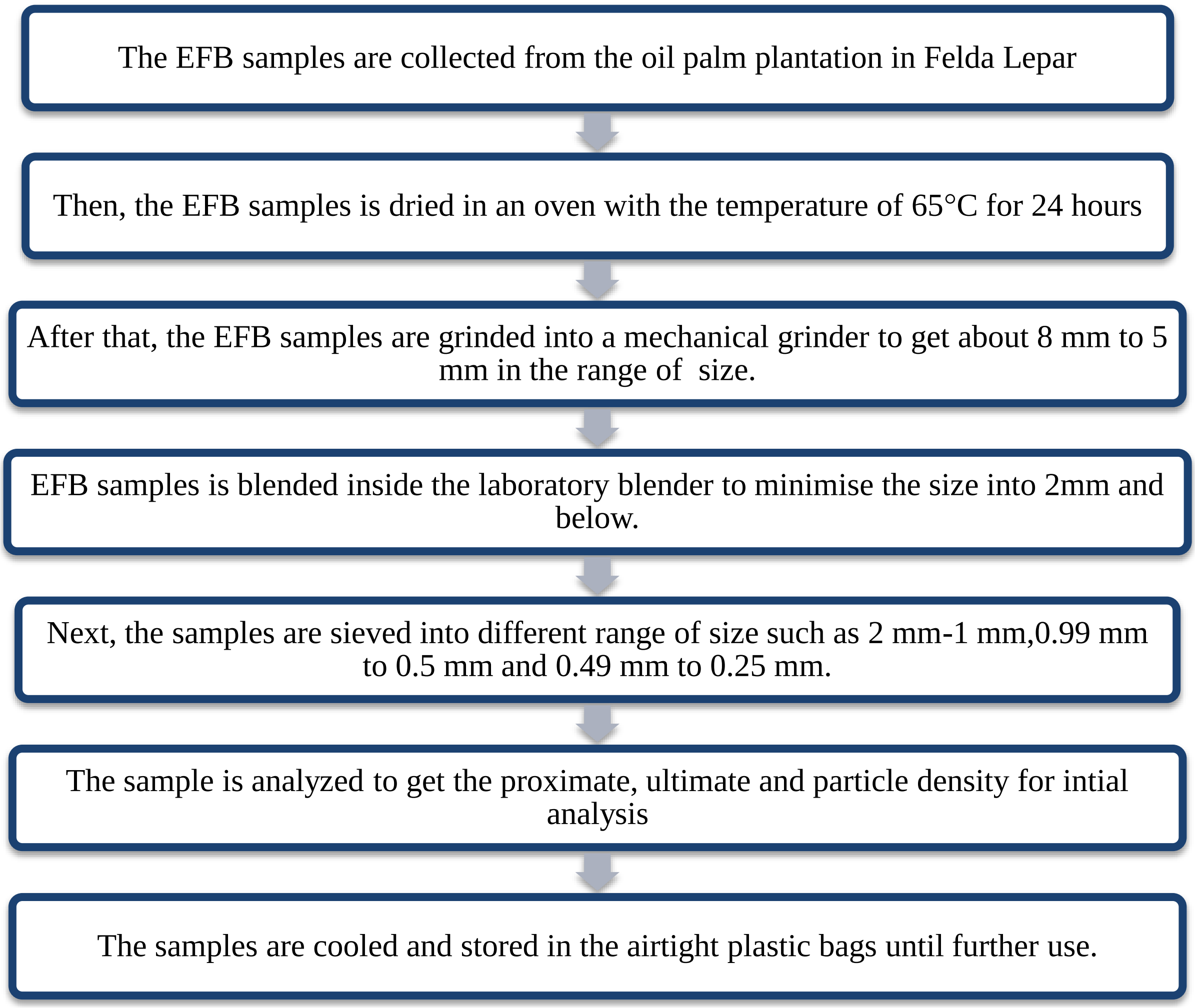


Figure 3-2 Flow Chart of Preparation of Samples

3.3 *Torrefaction experiments*

The experimental setup consists of a N_2 cylinder tank, CO_2 cylinder tank, and a vertical stainless-steel tubular reactor equipped with an electrically heated furnace and a liquid/gas collection unit. The experiment consists of two parts; the first part is inert torrefaction that only nitrogen gas flowed into the reactor. The second part is carbon dioxide and nitrogen that co-feed flowed into the reactor. The nitrogen and carbon dioxide tank was used to supply nitrogen and carbon dioxide as a carrier gas for providing an inert environment in the torrefaction experiment. The volumetric flow rate of nitrogen was controlled by an electronic mass flow controller. A schematic diagram of the experiments is shown in Figure 3-3.

A total of 3 g of EFB sample was weighed and put in a tubular reactor. The reactor was placed at the center of the furnace. When the EFB samples were in the furnace, they are heated in the furnace at a heating rate of 10 °C/min until the desired torrefaction temperature is reached at 200 - 300°C. N_2/CO_2 was used to purge the reactor depending on the medium used for torrefaction.

A constant flow of 50 mL/min of N_2/CO_2 was set using variable area mass flow controller to maintain inert/non reacting environment during the process. After flushing the reactor with torrefaction gas and different concentration of CO_2, the temperature is set at 240°C and 280°C. The samples were heated from room temperature to the desired temperature and kept constant within 30 minutes. After being torrefied for 30 minutes, the furnace is turn off and the reactor is left to cool to ambient temperature. Then the torrefied sample is recovered, weighed and kept in airtight plastic bags prior to further analysis. The gas product from the decomposition reaction of biomass is channelled to the bottom of reactor, passed through a condenser at room temperature. The concentration of carbon dioxide in the gas stream is also adjusted to 0%, 10%, 15% and 21vol% to investigate the effect of carbon dioxide concentration on torrefaction. The torrefaction gas is prepared by mixing the nitrogen with carbon dioxide under ambient temperature. Figure 3-4 summarizes the work flow of experiments after 30 minutes in the furnace.

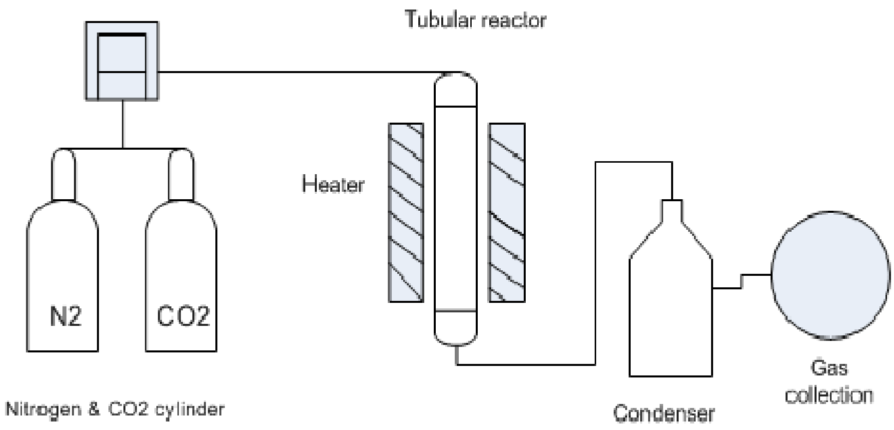


Figure 3-3 A schematic of the experimental setup

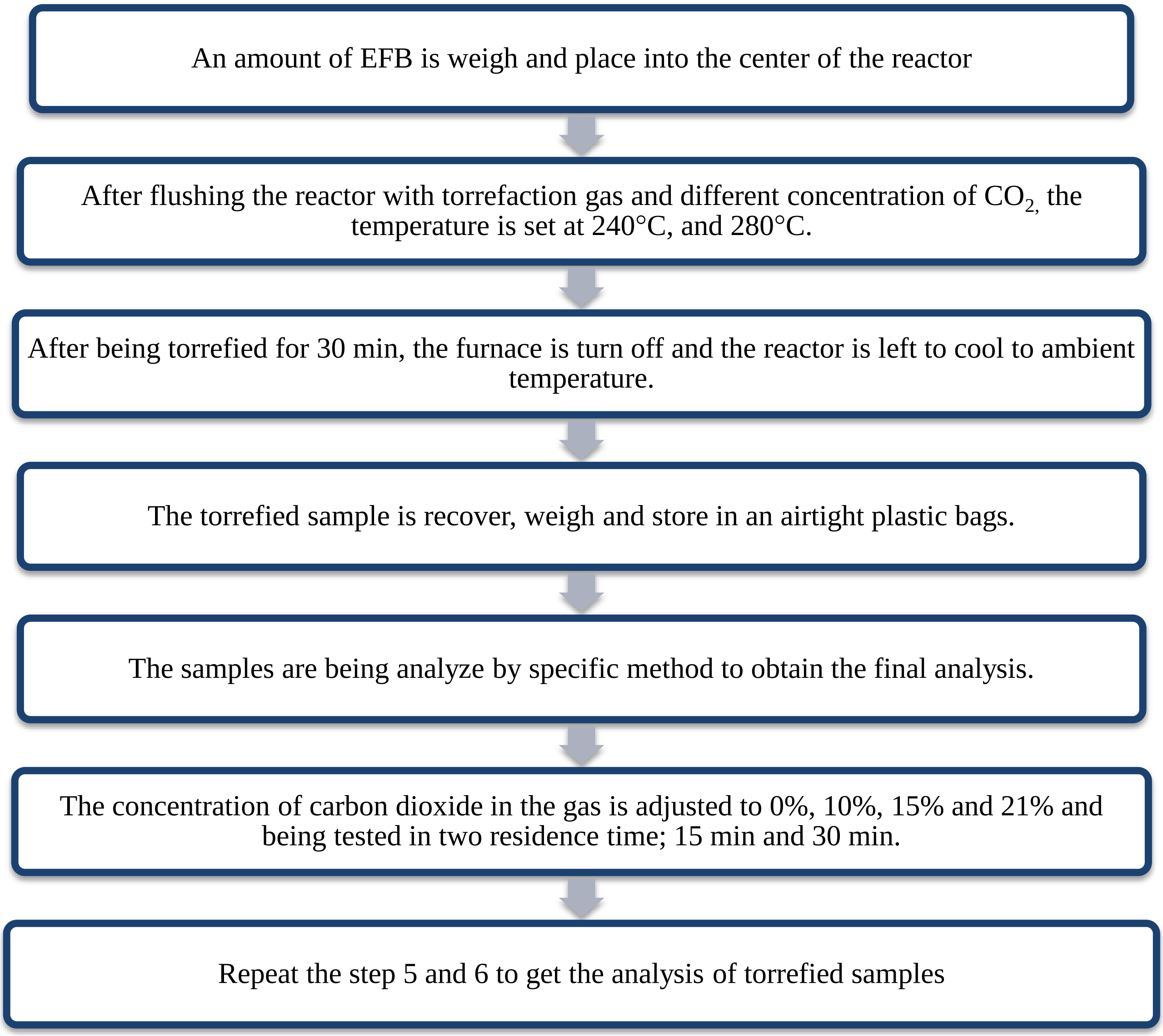


Figure 3-4 Flow Chart of torrefaction experiment

Grinder is used to shred or grind the EFB into small, about 8 to 10 mm in length is shown in Figure 3-5.

Figure 3-5 Mechanical Grinder in FKKSA Open Lab

3.4 Data Analysis

3.4.1 Gas Pycnometer

A pycnometer shown in Figure 3-6 is a device used to determine the density of a material. A pycnometer is usually made of glass, with a close-fitting ground glass stopper with a capillary tube through it, so that air bubbles may escape from the apparatus. The particle density of a powder, to which the usual method of weighing cannot be applied, can also be determined with a pycnometer.

Figure 3-6 Gas pycnometer

3.4.2 Thermogravimetric analysis (TGA)

The samples are also tested by using a thermogravimetric analyzer (TGA) to obtain the proximate analysis. TGA is a thermal analysis technique performed to determine the changes in the weight of a sample as a function of temperature. TGA of the raw samples is carried out using PerkinElmer Pyris 1 TGA. The sample (5mg) is loaded into a sample pan, and 100 mL/min of nitrogen is used to maintain inert environment. The samples are heated at a constant rate of 10^{o}C/min from room temperature to 900 ^{o}C. The results from TGA will be displayed in term of decomposition graph and used to determine moisture content, volatile matter, fixed carbon and ash content in the EFB. The increased mass loss showed when using the CO_2 as purge gas when tested with TGA (Eseltine, 2011).

The the heating rate of 10°C /min was executed by a thermogravimetry where the temperature ranged from 25 to 600°C. In TGA, 5 mg of sample was tested where the flow rate of carried gas was 40ml/min. The data collected by the TGA can be analyzed in a many ways. The most basic analysis of TGA data is in the creation of thermograms which plot the weight loss of the substance being tested versus temperature (time can also be used as the x-axis). Thermograms give a basic picture of the thermal breakdown of a substance over a given temperature range.

3.4.3 CHNS analyzer

The Ultimate analysis can be determined by using the Carbon, Hydrogen, Nitrogen and Sulphur (CHNS) analyzer to determine the elemental composition of a solid sample in wt%. In this test, samples are weighed around 3 to 5 mg in the holder before placed in a tin capsule and capsule was crimped. This capsule together with capsule serving as a blank and capsules containing standard (sulfamethazine) were placed into the auto sampler of the CHNS analyzer. Analyzer oxidation temperature was set at 1000°C. Helium, nitrogen and also nitrogen were used as carrier combustion and auto sampler injection gases. After that, the analyzer will show the properties of the carbon, hydrogen, nitrogen and sulphur that is contained inside the samples. The analyzer uses a combustion process to break down substances into simple compounds which are the quantified usually by infrared spectroscopy.

3.4.4 Scanning Electron Microscopic (SEM)

Scanning Electron Microscope is used to examine the topology at the surface of solid specimens, where it uses a focused beam of high energy electrons that scan across the surface of the specimen in a raster pattern. The increase in the number of pores or increase porosity of the EFB samples in the experiment can be determine using BET surface analysis and SEM image analysis. To study the effect using CO_2 as the torrefaction medium, the SEM images of the torrefied samples and raw biomass can be used to study the presence of pores on the samples torrefied under different conditions (Eseltine et al., 2013). The images were obtained at a magnification of 2000× to identify the pores formed on the samples when using different torrefaction mediums.

3.4.5 Characterization of EFB samples

For biomass analysis, the most important properties that need to be quantified are among others, the proximate, elemental (ultimate), as well as the higher heating value (HHV). The proximate analysis of the EFB is performed to determine the moisture, ash, volatiles and fixed carbon contents in accordance with standard procedure of American Society for Testing and Materials (ASTM E870-82) (Chen , et al., 2011). The ultimate analysis is performed for the determination of basic elemental composition of the EFB using an elemental analyzer for carbon, hydrogen, nitrogen and sulphur while oxygen is determined by the difference. The calorific values are measured using a bomb calorimeter, model C2000 series manufactured by IKA Werke (Uemura et al., 2013).

Three samples of different particle size is use in this study, and their masses and the calorific value is measure before and after torrefaction. Calorific values or HHV is measure using a bomb calorimeter. By using the results of the torrefaction process, three parameters were calculated using the following equations:

$$y_M = \frac{Mass\ of\ solid\ after\ torrefaction}{Mass\ of\ EFB\ used} \quad \text{Equation 3-1}$$

$$CV\ ratio = \frac{CV\ of\ solid\ after\ torrefaction}{CV\ of\ EFB\ used} \quad \text{Equation 3-2}$$

$$y_E = y_M \times CV\ ratio \quad \text{Equation 3-3}$$

Where,

y_M is the mass yield

CV is the calorific value (MJ/kg)

y_E is the energy yield

4 RESULTS AND DISCUSSION

4.1 Overview

This chapter presents the results obtained that includes the comparison between torrefaction experiments using CO_2 obtained from the journals. In the first semester, the preparation of samples for EFB was conducted for experiment. So, some of the result shown was actually based on the results from the preparation of samples. The TGA analysis was conducted to determine the effect of torrefaction temperature and concentration of CO_2 had upon the biomass. Some of the range of torrefaction temperatures (200- 300°C) was investigated in order to provide a thorough representation of the effect of varying torrefaction temperatures

4.2 Analysis on raw EFB

In this study, two different torrefaction temperatures of 240°C and 280°C were considered. Meanwhile, it can be found that the torrefaction time was usually controlled within 30 minutes. Consequently, two different torrefaction times of 15 and 30 minutes were tested. In the following discussion, the basic characteristic of raw EFB such as proximate analysis and calorific analysis, are shown in Table 4-1. According to the proximate analysis, moisture, VM, fixed carbon (FC) and ash content in the raw empty fruit bunch are 0.0 wt. %, *8.205* wt. %, 68.79 wt. %, and *22.99* wt. % respectively.

Table 4-1 Proximate, heating value and particle density of the raw Empty Fruit Bunch

Analysis	Value
Proximate analysis (wt. %)	
Moisture	8.411
Volatile matter (VM)	57.45
Fixed Carbon (FC)	29.9
Ash	4.24
Elemental analysis (wt. %)	
C	46.71
H	7.335
N	1.22
O	44.658
HHV (MJ/kg)	40.43
Particle density (g/cm^3)	1.3075

4.3 Effect of temperature and CO_2 concentration on mass yield of EFB

In this experiment, the parameters that need to be compared are the mass yield and temperature. The mass yield after torrefaction is defined as the ratio of the amount of mass left after pre-treatment to the original mass of the raw biomass.

Figure 4-1 presents the relationship between mass yield and carbon dioxide concentration of 10%, 15% and 21% at temperature of 240 °C and 280 °C. The graph showed that the mass yield of torrefied EFB decreased with increase in torrefaction temperature. The reason of the mass loss was due to thermal decomposition of hemi-cellulose and some short chain lignin compounds (Bergman, et al. 2005). The mass yield also decreased with increasing carbon dioxide concentration, even though the mass yield at 21% CO_2 for 280 °C was increasing. According to Chen, et al.,(2011) once the torrefaction temperature was a high as 280 °C, the weight loss of the biomass became drastic (>50%) where they concluded that temperature of 280 °C was not a proper condition for biomass torrefaction.

The effect of CO_2 presence in torrefaction was investigated to strengthen the objective of this study. The mass yield of torrefied biomass using CO_2 torrefaction shows a decrease when compare to the mass yield of torrefied biomass in inert torrefaction. The influence of CO_2 altered the reaction between volatiles evolved from the degradation of cellulose and CO_2 feed gas (Lu , et al. 2012). Saadon et al, 2013 stated that the concentration of CO_2 altered the ratio of solid formation during the torrefaction process. The presence of CO_2 suppressed the formation of liquid and boosted the generation of volatiles compound. Moreover the optimum temperature can be identified as the temperature of 240 °C showed more decrease in mass yield compare to temperature of 280 °C.

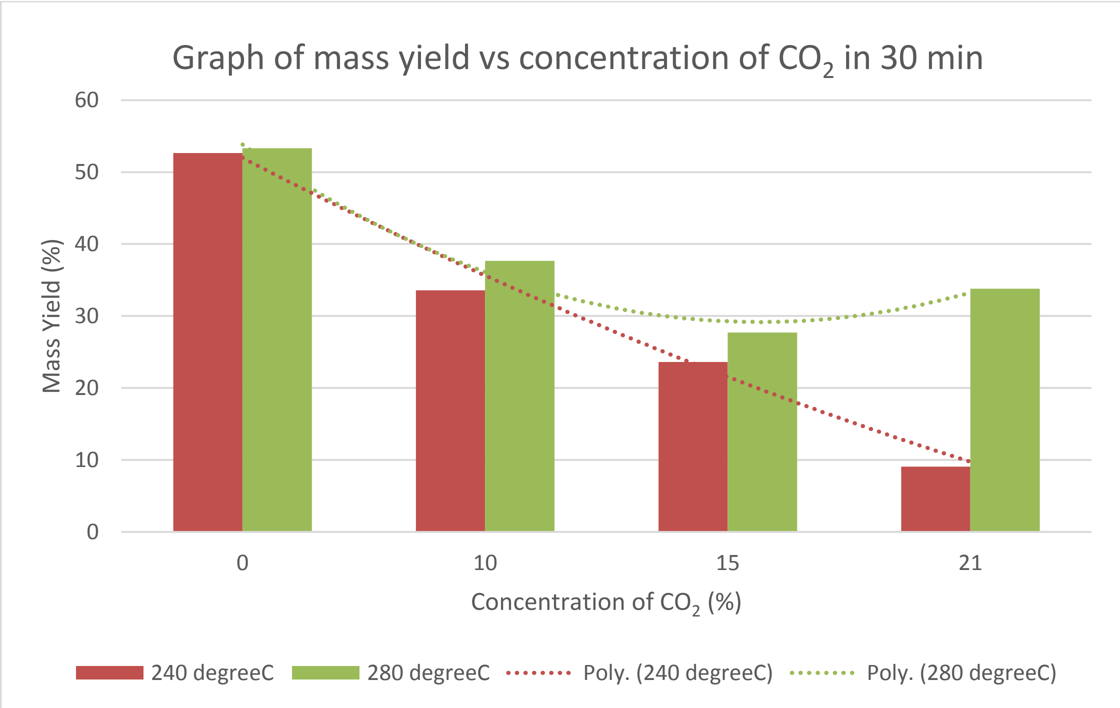


Figure 4-1 Graph of Mass yield and Temperature

When the torrefaction temperature is 240°C, the mass yield showed the uniform decrement from 52.67%, 33.57%, 23.59% and 9.06% at CO_2 concentration of 0%, 10%, 15% and 21% respectively. When the concentration of CO_2 increased, the mass yield of torrefied biomass become more lowered because of the thermal decomposition of biomass. This implies that the impact of CO_2 on the carbonization of EFB is heavier than N_2, whether the temperature is 240°C or 280°C. This reflects that CO_2 can be utilized as a carrier gas to perform EFB torrefaction. The carbonization extents of EFB at the temperature of 280°C are higher than at 240°C for all different concentration of CO_2.

Based on the different in residence time for torrefaction process, the mass yield for torrefied biomass treated in 30 minutes show slight decrease compared to the torrefied biomass treated in 15 minutes. According to Arias, et al. (2008) and Sadaka and Negi (2009), the higher residence time (more than 30 min) had minor effect on mass loss behavior.

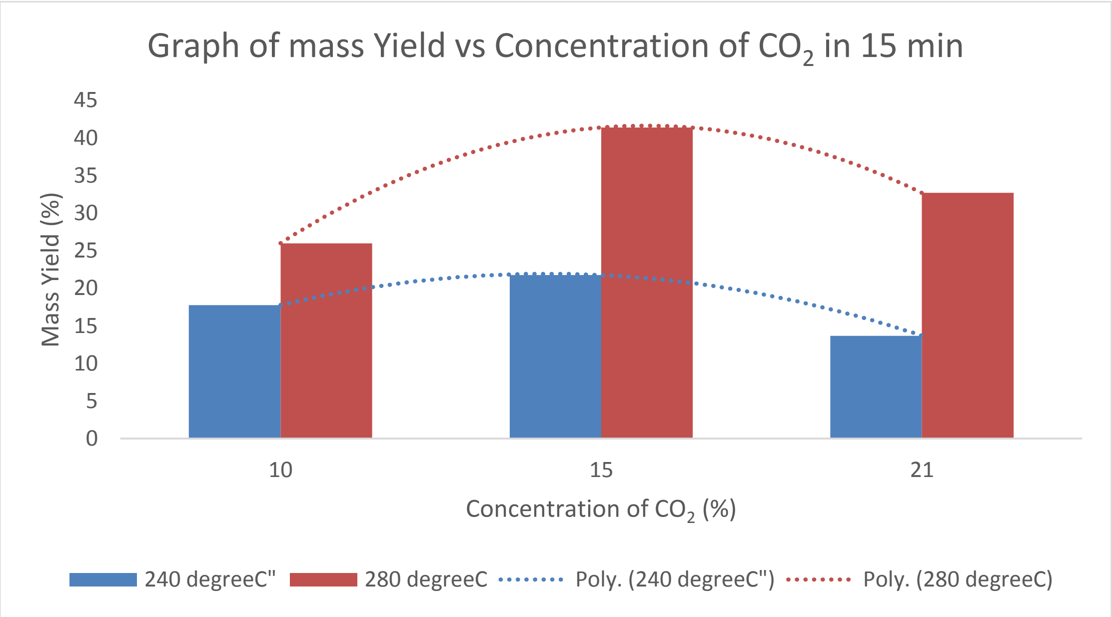


Figure 4-2 Graph Mass yield vs concentration CO_2 in 15 min

4.4 *Fuel Characteristic of torrefied biomass*

Data from proximate and ultimate analyses of torrefied empty fruit bunches (EFB) given in Table 4-2. It was observed that moisture content of torrefied biomass was decreased as the torrefaction temperature increases when stored at room temperature. It indicates that torrefied biomass cannot absorb moisture compared to untreated EFB. It may due to the decrease of hydroxyl (~OH) groups from biomass during torrefaction reaction. It can be observed from Table 4-2 that the ash percentage of the samples treated using CO_2 and N_2 are higher than the raw biomass samples. Some of the ash percentage shows a slight deviations that might be caused by distribution of ash within the samples that not much uniform.

Table 4-2- Proximate Analysis of torrefied EFB

Biomass	MC (%)	VM (%)	FC (%)	Ash (%)
Raw Empty Fruit bunch (EFB)	8.411	57.45	29.9	4.24
EFB-240°C/10% CO_2	6.453	41.64	46.01	5.801
EFB-280°C/10% CO_2	5.347	36.64	26.41	31.59
EFB-240°C/15% CO_2	5.222	46.21	20.31	28.24
EFB-280°C/15% CO_2	6.077	38.80	20.98	34.02
EFB-240°C/21% CO_2	4.915	54.94	33.40	6.724

As the torrefaction temperature increased, the elemental carbon content of torrefied biomass was increased. Moreover, hydrogen and oxygen contents of torrefied biomass decreased, resulting in decreased H/C and O/C ratios. Lower hydrogen and oxygen contents of torrefied biomass also support the fact of reduction in hydroxyl (~OH) groups during torrefaction.

4.5 *Effect of temperature and concentration CO_2 on calorific value of biochar*

Figure 4-3 shows the relationship between calorific value and temperature at CO_2 concentration of 0%, 10%, 15% and 21%. The calorific value increased with increasing temperature. The same trend had been reported previously using another type of biomass. The increasing of calorific value caused by the increasing in carbon content through torrefaction (Y. Uemura, et al. 2011). For different concentration of CO_2, the caloric value does not very depend on it but the calorific value slightly became uniformly at concentration of 21% CO_2. This result suggests that EFB that undergoes CO_2 torrefaction does not interrelate with calorific value except at 21% concentration CO_2.

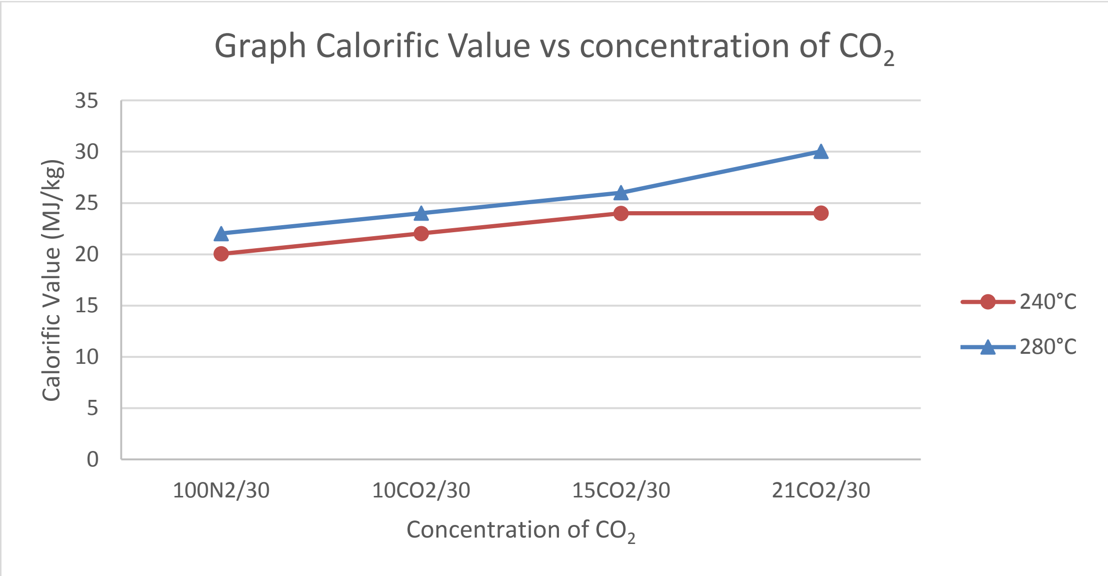


Figure 4-3 Graph Calorific Value vs concentration of CO_2

4.6 The effect of temperature and CO_2 concentration on energy yield of EFB.

Pretreated EFB with higher fixed carbon and lower moisture content will have higher heating value than the raw EFB. However, to attain the relationship between mass yield and pretreatment process, energy yield is used and defined according to equation 3. The energy yield of the torrefied samples obtained using equation 3 is presented in Figure 4-4. A rise in the energy yield value indicates an increase in heating value and lower mass loss behaviour at that particular temperature. The torrefied EFB showed an increase in energy yield at temperature around 280°C. Yet further increase in temperature shows an increase in energy yield for EFB samples that will result in higher loss in combustible volatile matter from the biomass (Thanapal, et al. 2014).

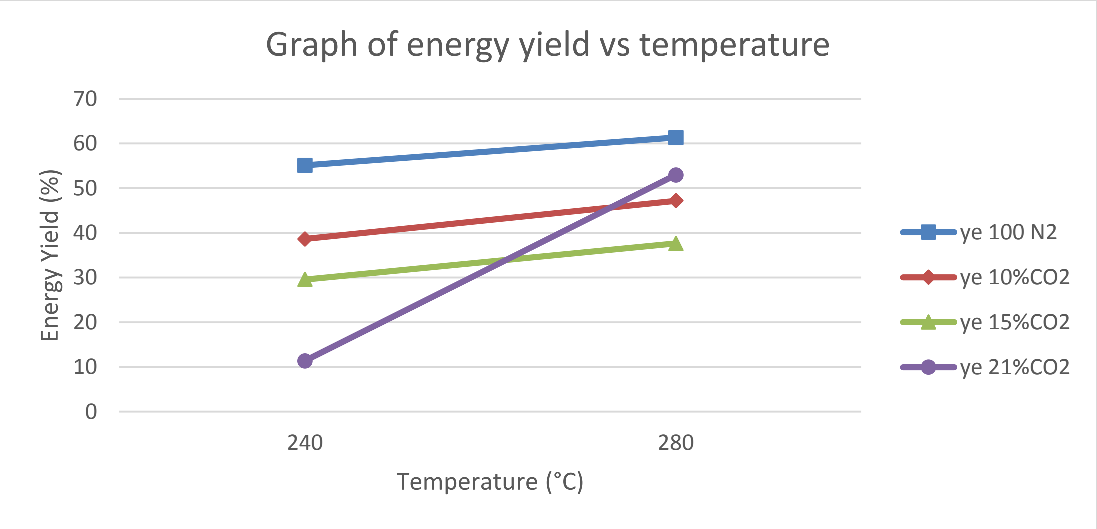
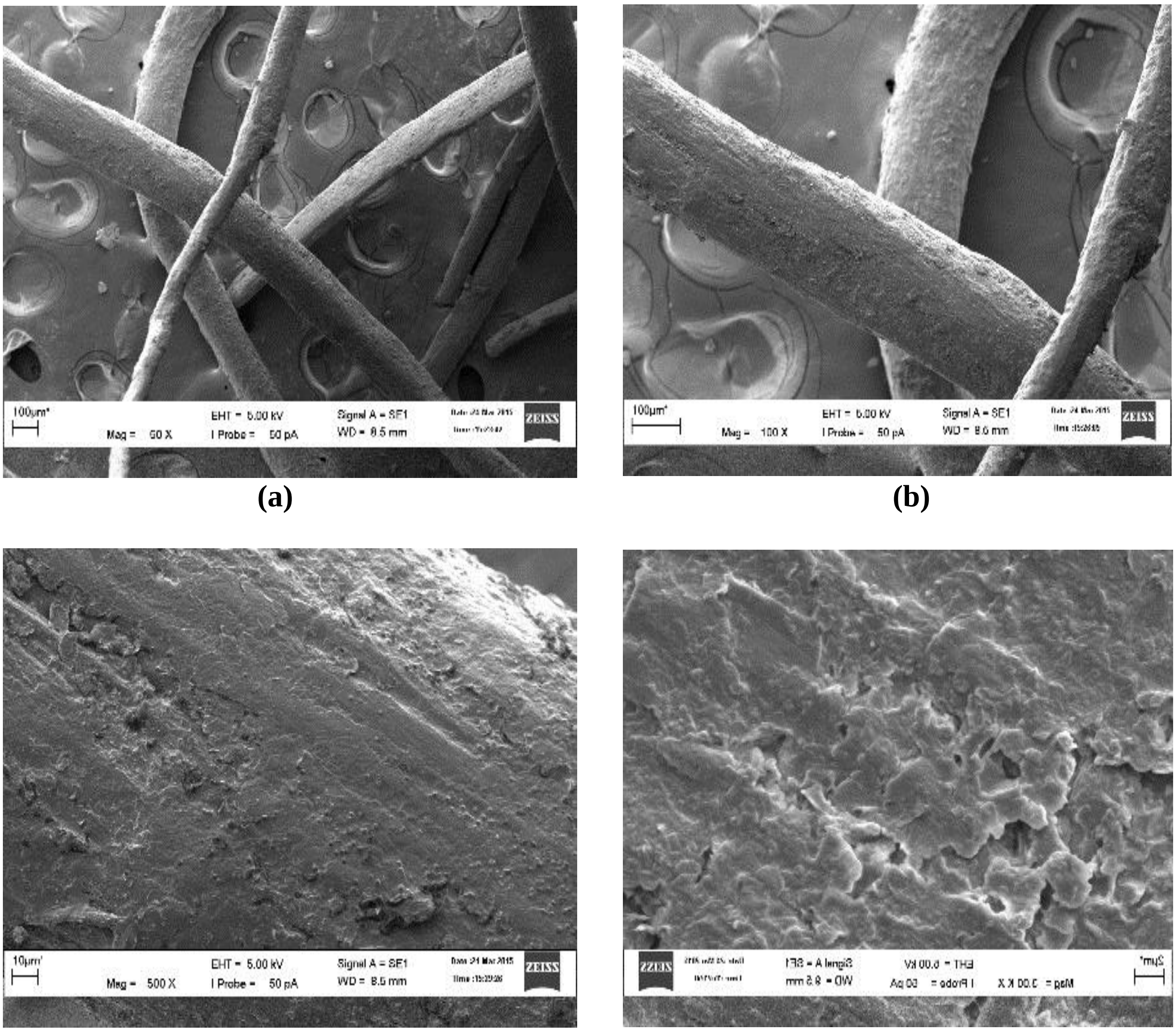


Figure 4-4 Graph of energy yield vs temperature

4.7 SEM Analysis on torrefied biomass

The difference in temperature also can affect the SEM observation for the torrefied biomass. Figure 4-5 shows indicates the Scanning Electron Microscopy (SEM) image for raw EFB at magnification 50x, 100x, 500x and 1000x. The surface of the raw wood shown is integrated but after undergoing torrefaction, it make a pronounced impact on the EFB surface. In temperatures of 240 and 280°C, the surfaces of the EFB can be characterized using the different in cell structures. In 280°C, the surfaces is further damaged and provide a tubular-shape surface (Chen, Hsu, et al. 2011).

SEM analysis were carried out on raw and torrefied EFB for a range of ash content in order to study their surface structure and elemental composition. These analyses were performed at Central laboratory of Universiti Malaysia Pahang. We found that the raw EFB might not be easily to feed in the gasification equipment as it is tended to stick to each other easily.



(a) **(b)** **(c)** **(d)**

Figure 4-5- Scanning Electron Microscopy (SEM) image for raw EFB at magnification (a)50x, (b)100x, (c)500x and (d)1000x.

Table 4-3- SEM images of torrefied EFB at 240°C and 280°C, 10% CO_2 in 30 min

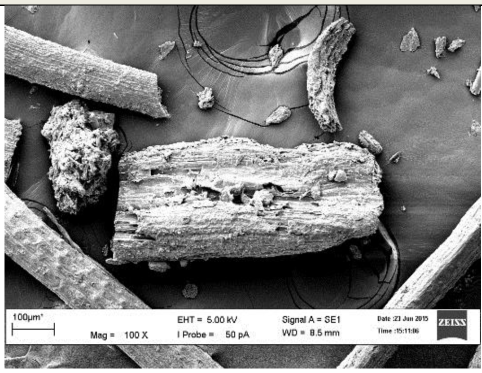
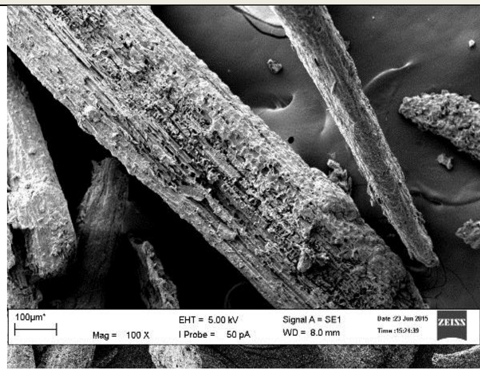
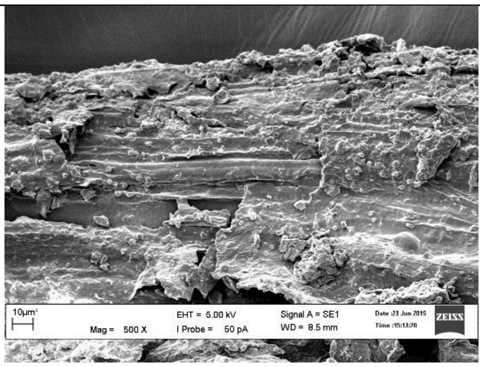
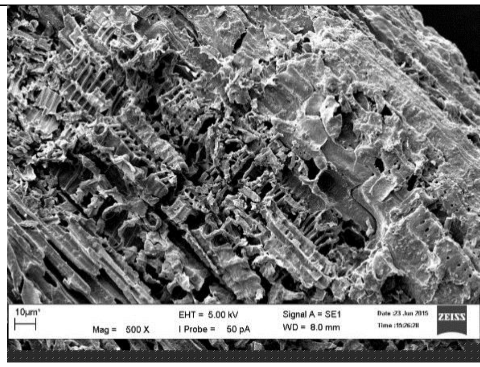
Magnification	10% CO_2, 240°C, 30 min	10% CO_2, 280°C, 30 min
100x		
500x		
1000x		
3000x		

Table 4-6- SEM images of torrefied EFB at 240°C and 280°C, 10% CO_2 in 15 min

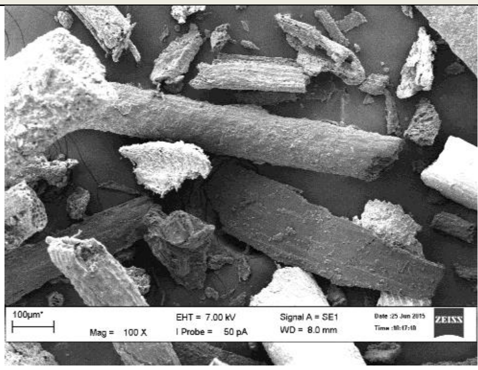
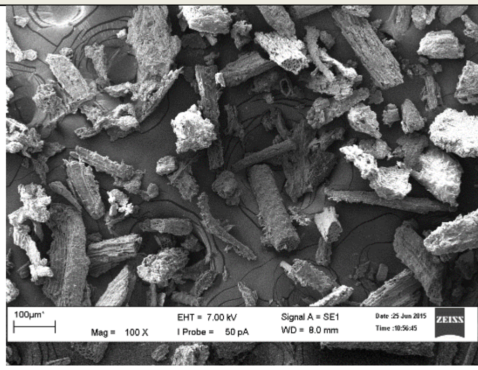
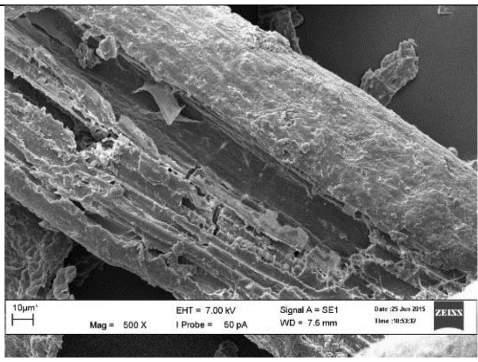
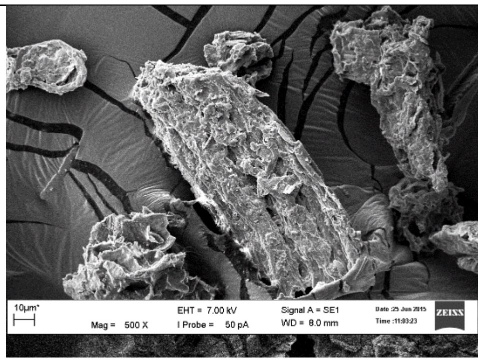
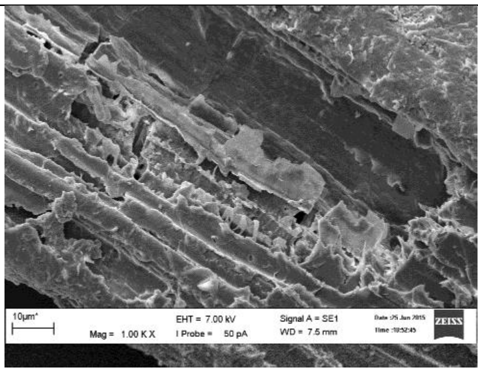
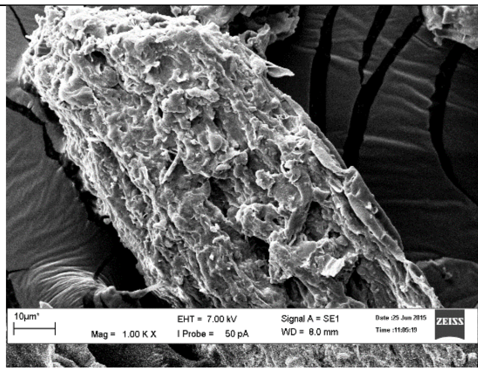
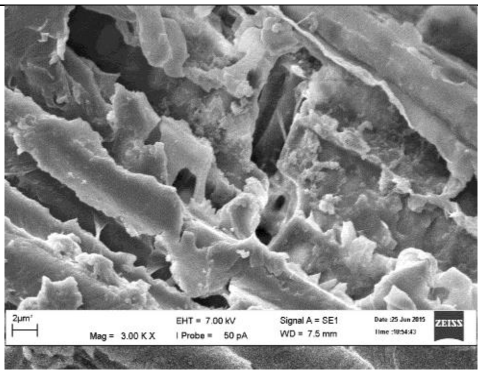
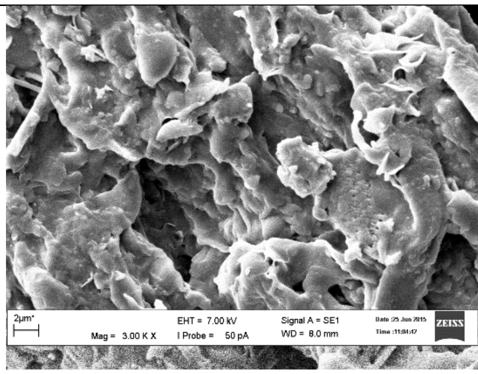
Magnification	10% CO_2, 240°C, 15 min	10% CO_2, 280°C, 15 min
100x		
500x		
1000x		
3000x		

Table 4-7 - SEM images of torrefied EFB at 240°C and 280°C, 15% CO_2 in 15 min

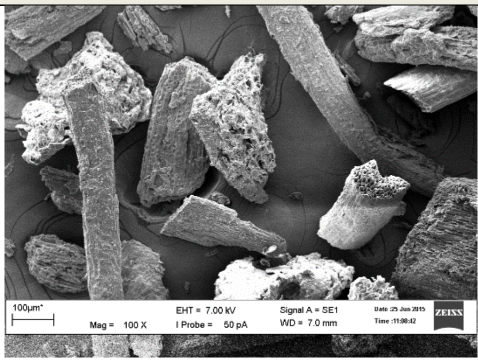
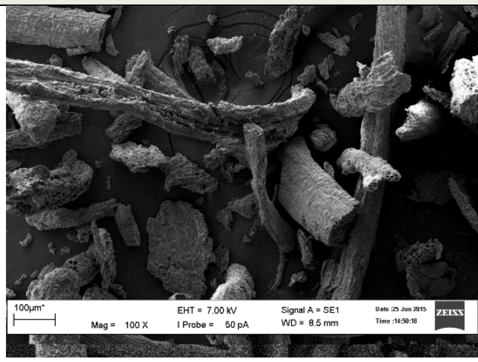
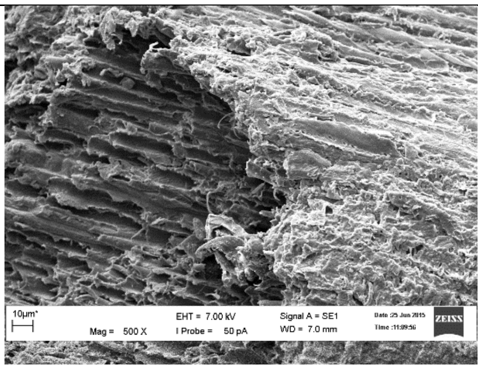
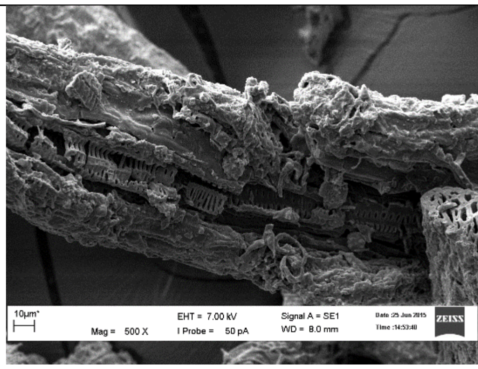
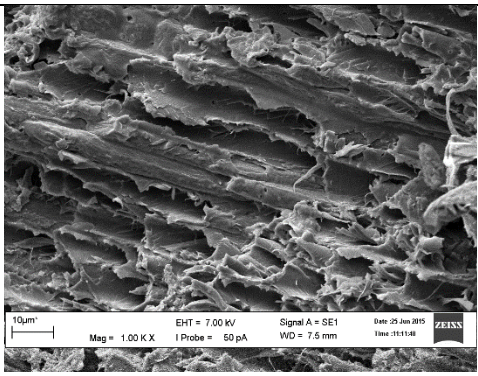
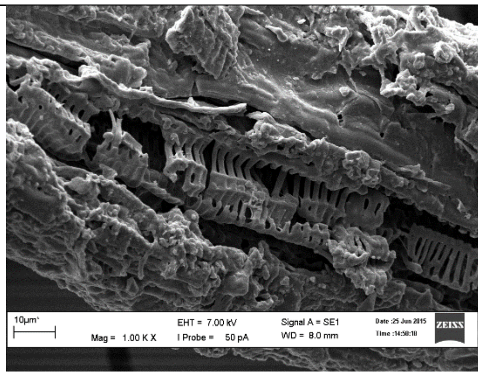
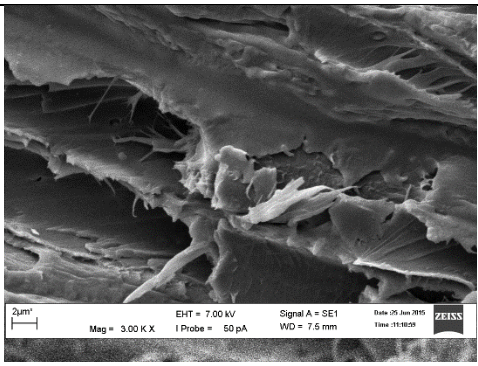
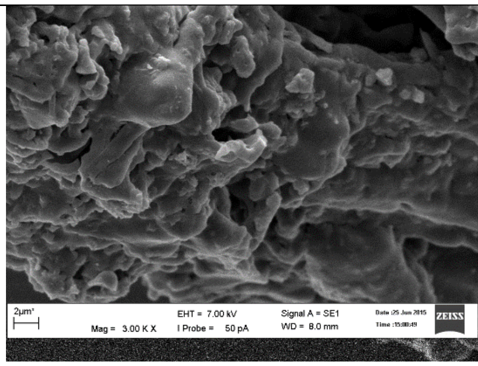
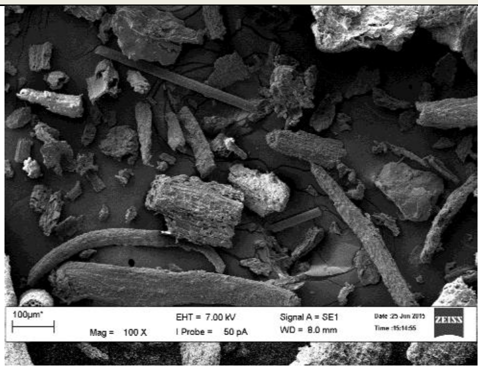
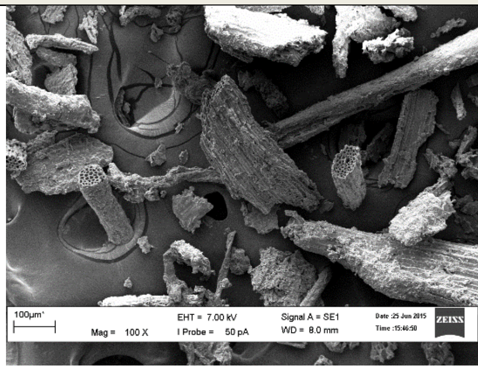
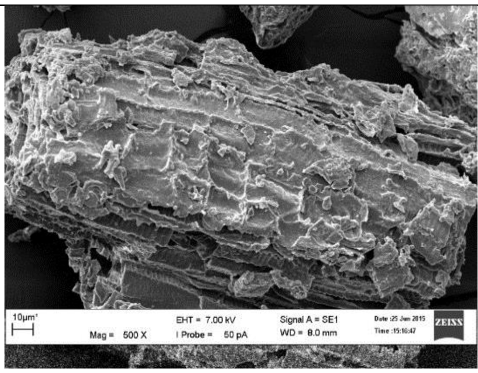
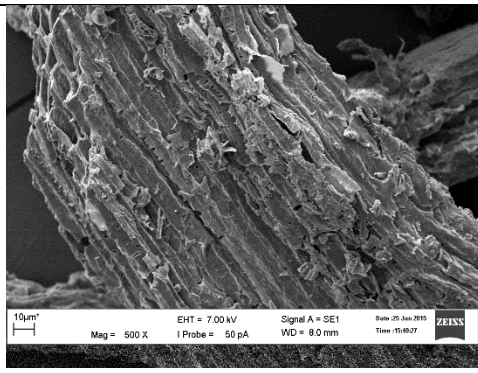
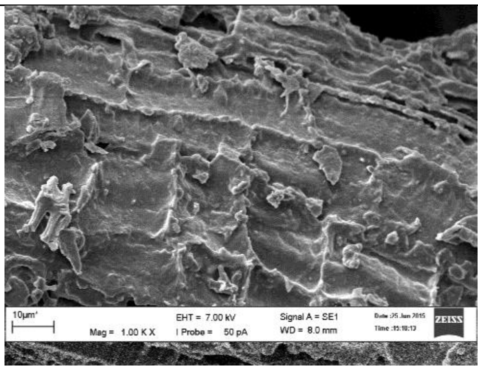
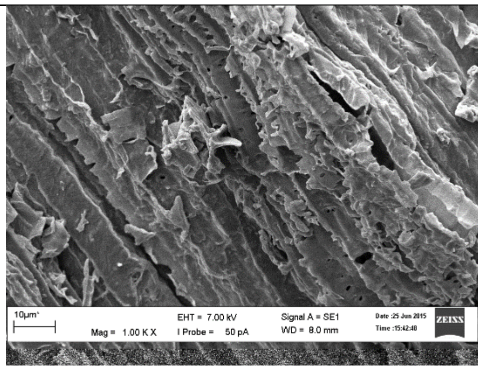
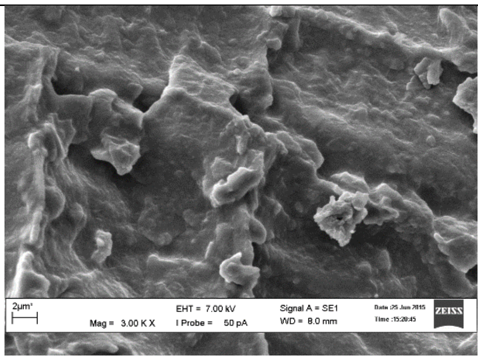
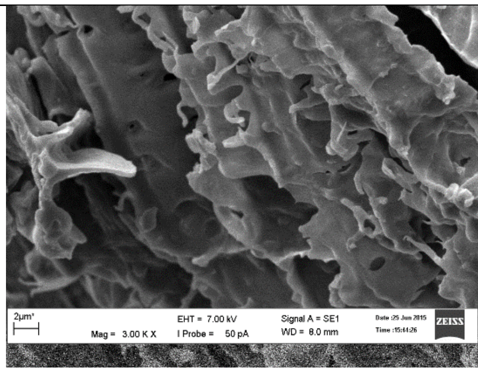
Magnification	15% CO_2, 240°C, 15 min	15% CO_2, 280°C, 15 min
100x		
500x		
1000x		
3000x		

Table 4-8 - SEM images of torrefied EFB at 240°C and 280°C, 21% CO_2 in 15 min

Magnification	21% CO_2, 240°C, 15 min	21% CO_2, 280°C, 15 min
100x		
500x		
1000x		
3000x		

4.8 Summary

This chapter presents all result from the torrefaction experiment that conducted during this semester. Some of the results were discussed according to the analysis and referred from the journal with citation to be compared with results obtained.

5 CONCLUSION

5.1 Conclusion

This work focuses on the effect of CO_2 in the torrefaction process for EFB biomass. Overall the improvements of torrefaction process can be made by using the CO_2 torrefaction. The mass yields and characteristic of torrefied empty fruit bunch at the temperatures of 240°C and 280°C using N_2 and CO_2 as the carrier gases have been investigated. The results showed that the higher torrefaction temperature makes the properties of solid products approach those of coal. In the discussion earlier stated that the mass yield decreased as the CO_2 concentration increased. The mass yield decreased resulted from the removal of moisture content in the EFB that help in gasification process. The concentration of CO_2 below 15% has been chosen as the manipulated variables by co-feeding with nitrogen to simulate the composition of torrefaction gas. The existence of CO_2 in torrefaction caused a minor decrease in mass yield of torrefied EFB. Moreover, the surfaces of biomass observed from SEM images shows improvement when increases the torrefaction temperatures. Compared to N_2, CO_2 is a better choice than conventional torrefaction to increase the ignition temperature of torrefied biomass. Accordingly, CO_2 can replace N_2 as a carrier gas for biomass torrefaction to improve the properties of the torrefied biomass and increase the safety of biomass storage and transportation. As conclusion, the torrefaction using CO_2 as enhancer gas will improve the fuel characteristic of the EFB samples compared to the N_2 (inert) torrefaction.

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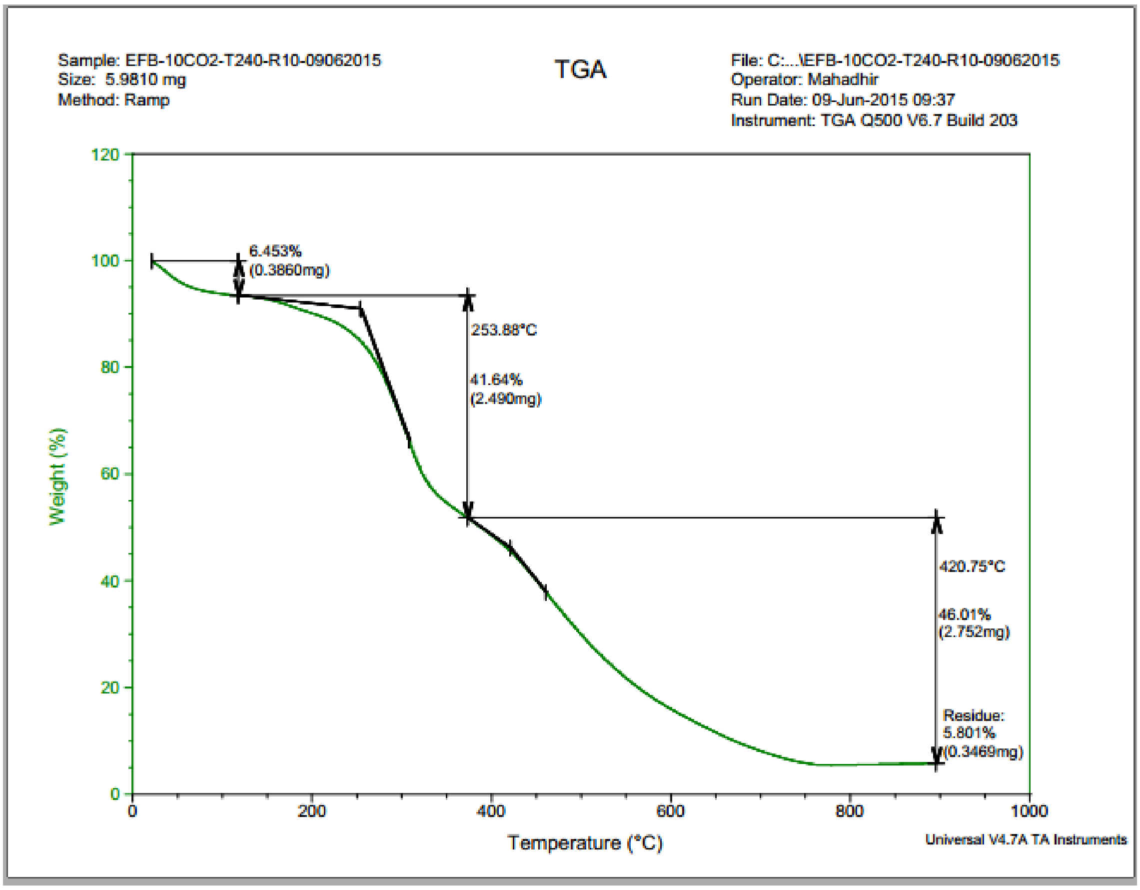
7 APPENDICES

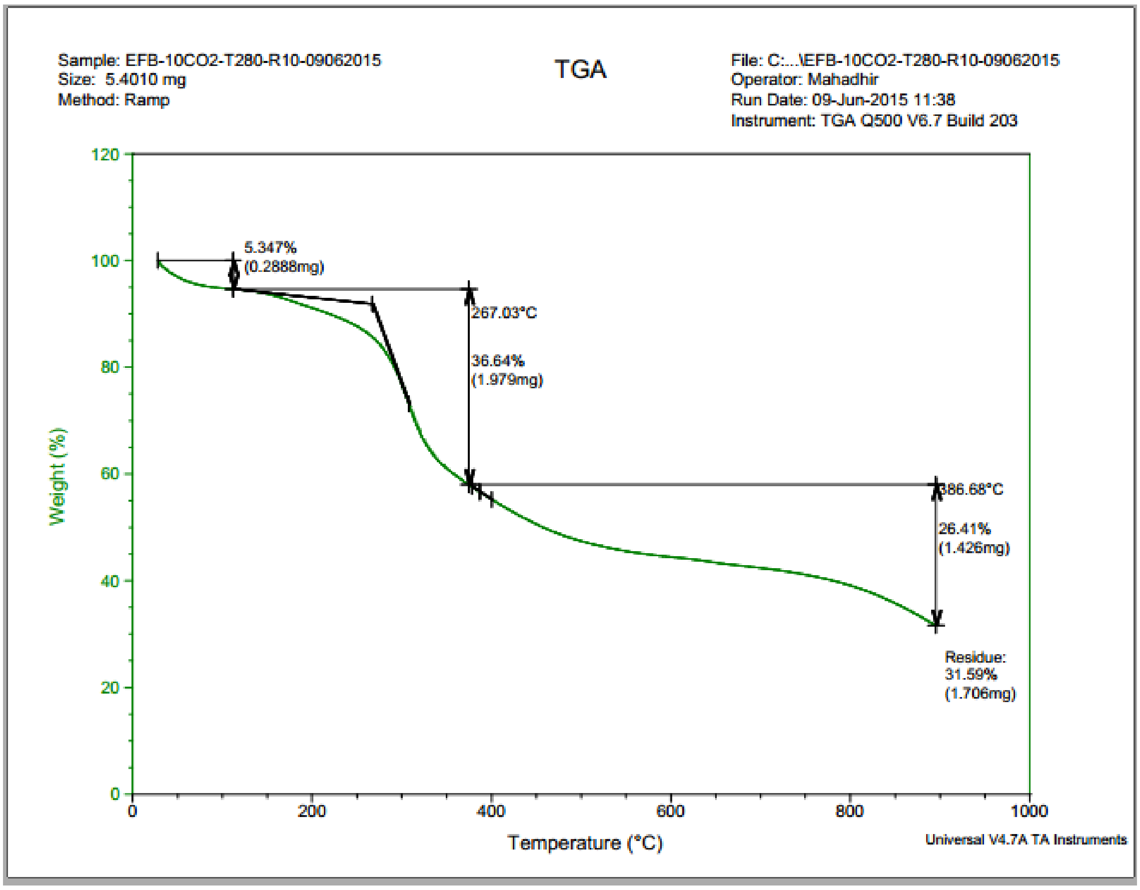
(a) TGA Analysis – EFB-10%CO_2 T=240°C, t= 30min



TGA Result Analysis

Material	Composition	Tem.Decomp
	%	ºC
1	6.453	N/A
2	41.64	253.88
3	46.01	420.75
4	N/A	N/A
Residue	5.801	at 900

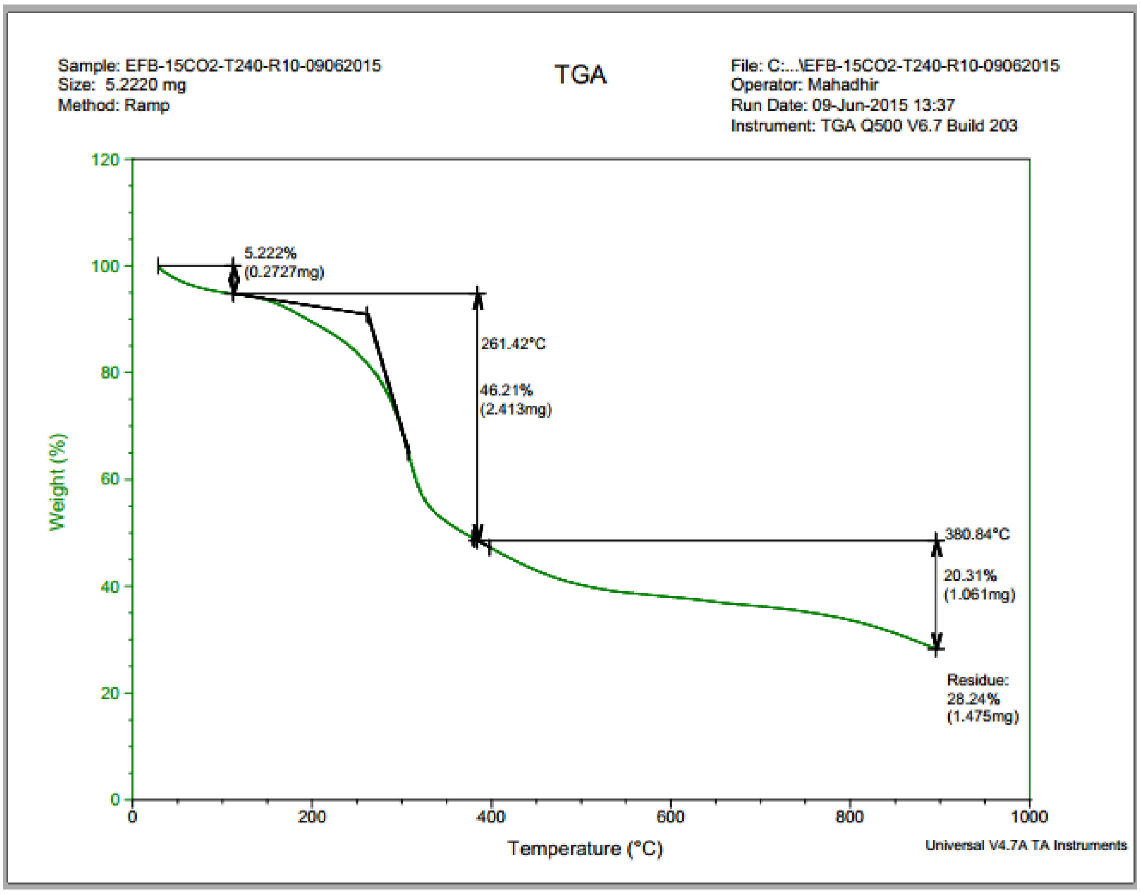
(b) TGA Analysis – EFB-10%CO_2 T=280°C, t= 30min



TGA Result Analysis

Material	Composition	Tem.Decomp
	%	°C
1	5.347	N/A
2	36.64	267.03
3	26.41	386.68
4	N/A	N/A
Residue	31.59	at 900

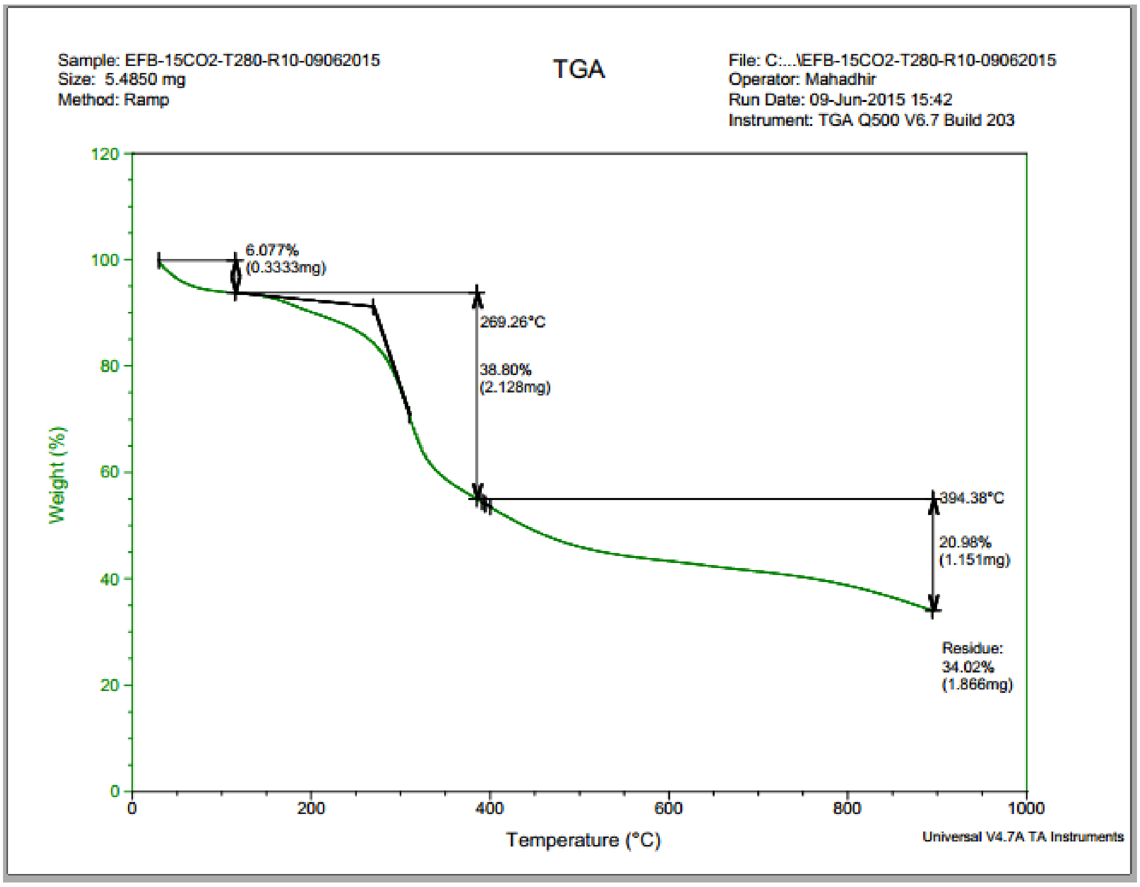
(c) TGA Analysis – EFB-15%CO_2 T=240°C, t= 30min



TGA Result Analysis

Material	Composition	Tem.Decomp
	%	°C
1	5.222	N/A
2	46.21	261.42
3	20.31	380.84
4	N/A	N/A
Residue	28.24	at 900

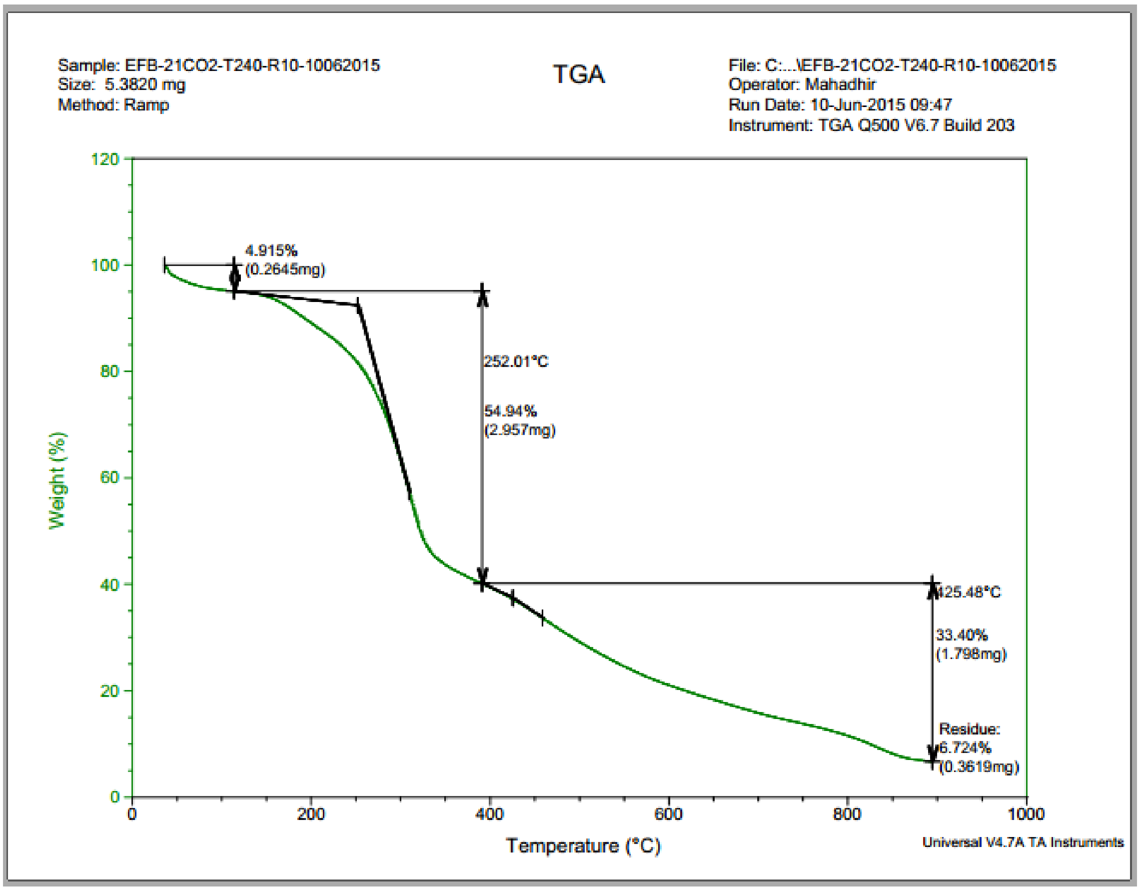
(d) TGA Analysis – EFB-15% CO_2 T=280°C, t= 30min



TGA Result Analysis

Material	Composition	Tem.Decomp
	%	°C
1	6.077	N/A
2	38.80	269.26
3	20.98	394.38
4	N/A	N/A
Residue	34.02	at 900

(a) TGA Analysis – EFB-21%CO_2 T=240°C, t= 30min



TGA Result Analysis

Material	Composition	Tem.Decomp
	%	°C
1	4.915	N/A
2	54.94	252.01
3	33.40	425.48
4	N/A	N/A
Residue	6.724	at 900