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MORPHOLOGY AND BONDING ANALYSIS OF TORREFIED MATERIAL

by

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DECLARATION

I declare that this thesis entitled "Morphology and Bonding Analysis of Torrefied Material" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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Morphology and Bonding Analysis of Torrefied Material

ABSTRACT

Torrefaction of oil palm empty fruit bunch (EFB) was carried out by using microwave oven as reactor without the presence of oxygen. The process take place under a varied temperature between 200 ℃ to 300 ℃ for about 20 – 60 minutes, respectively. Resulting from the torrefaction process of EFB causing changes in terms of its surface morphology alongside with bonding behaviour. Thus, this studied was conducted in order to investigate the impact of torrefaction process on the surface morphology as well as functional group of EFB before undergo further processing method, so that, it can be used as renewable energy. Scanning Electron Microscope (SEM) showed images of the surface structure EFB after undergo torrefaction by which it was completely decomposed internally by producing pores while the structure become flattened with almost disappeared sharp edge compared to the raw EFB. The changes of presence functional groups before and after the particular torrefaction process were observed under certain wavelength using Fourier Transformation Infrared (FTIR) which were O-H bonds around 3420 - 3435 cm^{-1}, C-H bonds around 2930 – 2950 cm^{-1}, 1430 – 1440 cm^{-1} and 850 – 890 cm^{-1}; C=O bonds at 1730 - 1750 cm^{-1} and much more. These functional groups determined the changes of functional groups as well as the wavelength whereby the degradation of hemicellulose, cellulose and lignin take place.

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Morfologi dan Analisis Ikatan untuk Bahan Terbakar

ABSTRAK

Pembakaran telah dijalankan ke atas tandan buah kosong (EFB) kelapa sawit dengan menggunakan ketuhar gelombang mikro sebagai reactor tanpa kehadiran oksigen. Proses ini berlaku dalam keadaan suhu yang bervariasi di antara 200 ℃ sehingga 300 ℃ selama 20 – 60 minit, masing-masingnya. Hasil daripada proses pembakaran EFB menyebabkan perubahan dari segi morfologi permukaan beserta tingkah laku ikatan. Oleh yang demikian, kajian ini telah dijalankan agar kesan proses pembakaran terhadap morfologi permukaan beserta kumpulan berfungsi EFB dapat disiasat sebelum proses selanjutnya dilaksanakan supaya dapat digunakan sebagai tenaga boleh diperbaharui. Mikroskop Imbasan Elektron (SEM) menunjukkan imej-imej struktur permukaan EFB selepas menjalani pembakaran yang mana struktur itu mengalami pereputan dalaman secara keseluruhan melalui penghasilan liang-liang roma sekaligus menjadi leper dengan hampir kehilangan struktur hujung yang tajam jika dibandingkan dengan EFB mentah. Perubahan yang berlaku sebelum dan selepas proses pembakaran terhadap kumpulan berfungsi yang ada dapat diperhatikan melalui kepanjangan gelombang tertentu dengan menggunakan Fourier Transformasi Inframerah (FTIR) di mana ikatan O-H dapat dikesan pada gelombang sekitar 3420 - 3435 cm^{-1}, ikatan C-H pula pada sekitar 2930 – 2950 cm^{-1}, 1430 – 1440 cm^{-1} dan 850 – 890 cm^{-1}; ikatan C=O ada disekitar 1730 - 1750 cm^{-1} dan banyak lagi. Kumpulan-kumpulan berfungsi ini menentukan perubahan ikatan yang ada serta gelombang di mana degradasi hemiselulosa, selulosa dan juga lignin berlaku.

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LIST OF ABBREVIATIONS

IEA	International Energy Agency
EFB	Empty Fruit Bunch
SEM	Scanning Electron Microscope
FTIR	Fourier transformation Infrared
OPF	Oil Palm Fronds
OPT	Oil Palm Trunks
MF	Mesocarp FIber
PKS	Palm-kernel Shells
BSE	Backscattered Electron
SE	Secondary Electron
IR	Infrared
RSM	Response Surface Method
CCRD	Central Composite Rotatable Design
HHV	Higher heating rate

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LIST OF SYMBOLS

℃	Degree Celsius
%	Percentage
℃/min	Heating rates
MHz	Frequency (Megahertz)
GHz	Frequency (Gigahertz)
K	Thousands
X	Magnification of microscope
µm	Micrometre
mL min^{-1}	Flow rate
W	Watt
cm^{-1}	Wavenumber
g	Mass in gram
℃$^{-1}$	Thermal expansion
MPa	Megapascal

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Based on the *Energy Statistics Manual* by International Energy Agency (IEA), fuel is defined as burned substances as a source of heat by which carbon and hydrogen in the fuel substance undergo combustion process with the presence of oxygen to release heat (International Energy Agency, 2005). The heat is referred as energy. Increasing risk of depletion of fuel is due to the increasing demand of energy which eventually causing an increase in price of crude oil. The increasing in energy demand is because of a rapid growth of development in population and industrialization (Saxena et al., 2009). A continuous consumption of fuels makes them unable to be used in future generation since they act as a limited resource, nowadays, by providing energy and feedstock for materials (BRE Academy, 2016).

The depletion of fuel will eventually lead to the environmental issues such as the economic activities likes in the industry, services, agricultural and transport become limited and restrained, mitigation of climate change as well as declining and the insecurity of other non-renewable resources. These negative environmental consequences had led to the exploration and study of finding alternatives to fuels which is renewable energy resources.

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The use of renewable energy resources had become a big concern to the worldwide for energy production because the resources is quite efficient ways to reduce environmental issues. Chen et al., (2012) listed that the renewable energy resources are solar, wind, biomass, geothermal, hydropower and ocean. According to Basu (2010), biomass is a promising alternative as it can provide a full range of feedstocks. Its percentage account to 10% of world’s annual energy consumption compared to natural gas, coal and petroleum oil, each with 21%, 27% and 33%, respectively (International Energy Agency, 2010).

Every biomass will undergo several pretreatment process before being a further processing as renewable energy. The pretreatment process composed of carbonization, gasification, pyrolysis and torrefaction. The more preferable process among those is torrefaction due to its production of coal substitute from biomass which caught the attention of power industries (Basu, 2013). Moreover, torrefaction is known as its ability to prepare biomass for further use instead of directly use from its raw substance.

1.2 Problem statement

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The use of biomass as fuel already being studied for a long period of time started with its production from raw materials. Unfortunately, there are serious limitation on the extent usage of biomass as a source to generate energy. It is undeniable that biomass have a low inorganic matter and impurity contents (Pohlmann et al., 2014), but the drawback made biomass difficult to be process in becoming resources energy. The irreversible negative impact is on the properties or features of biomass. A raw biomass has a high moisture and oxygen content, less brittle structure and more fibrous which make them difficult to be grind (Werther et al., 2000).

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Nevertheless, biomass also have low density, low heating value and soot pulverised combustion system (Khorshidi et al., 2014). Furthermore, its strength, toughness and abrasion resistance are reduced (Chen et al., 2012).

Many studies have been conducted in order to overcome the drawbacks of biomass. Unfortunately, the studies only restricted to its morphology and less studies have related it with bonding in the structure of biomass. Upon undergo research, only a little research focusing on morphology, physiochemical properties, composition of torrefied biomass, chemical and physical characteristics (Ibrahim et al., 2013) whereas the bonding analysis of torrefied biomass is very limited.

1.3 Objectives

The objectives of this research are:

1. To observe the surface morphology of raw and torrefied material
2. To analyse the bonding behaviour of torrefied material

1.4 Scope of work

This study is conduct to identify the morphology and analysing the bonding of torrefied material. Biomass material used in this case is empty fruit bunch (EFB) will undergo thermal pretreatment method known as torrefaction before being analyse. The process take place within the temperature of 200 – 300 ℃ in an inert gas atmosphere under a low heating rate (Bergman & Kiel, 2005). EFB is used to analyse its morphology and bonding due to the curiosity regarding its properties, since only a few study referred EFB to be used as source for renewable energy. Sabil et al. (2013) states

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that EFB, traditionally, is burnt in a simple incinerator to be disposal and its ash is used as fertilizer.

1.5 Expected outcome

In this study, the morphology variations for both raw and torrefied EFB will be assessed by using Scanning Electron Microscope (SEM). The changes in structure involve the decomposition of hemicellulose and partial polymerization of lignin and cellulose, resulting from torrefaction process (Chen et al., 2014). The changes in structure of torrefied material will also affecting its bonding. Thus, Fourier Transformation Infrared (FTIR) is used to looked on compositional bonding of torrefied material.

Extension to FTIR is the functional group of torrefied material. The functional group will be reflecting on the effect of torrefaction process as causes by the heat treatment process.

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CHAPTER 2

LITERATURE REVIEW

2.1 Renewable energy

Basically, energy can be in all forms including electricity, heat and lights. Energy also can be divided into non-renewable energy such as natural gas and fossil fuels, and renewable energy such that hydro, geothermal and biomass. Due to the depletion of fuel (Ibrahim et al., 2013) as well as mitigation of climate change (Sabil et al., 2013a), the development of alternative energy sources had been driven out by renewable energy as stated by (Svanberg et al., 2013).

Renewable energy as defined by Sathaye and Meyers (1995) is any energy sources that are derived either directly or indirectly from solar energy. Nevertheless, renewable energy had play an important role in providing energy with sustainability to the vast populations in developing countries (Painuly, 2001). There are many energy sources which are biomass, coal power, hydro power nuclear power and wind power (Vattenfall, 2011).

Most of previous studies focusing biomass as an alternative energy compared to other renewable energy (Abbasi & Abbasi, 2010; Aziz et al., 2012). The considerations of biomass as a carbon neutral fuel in the net carbon emissions from burning of biomass is zero (Chen et al., 2012), potential size of the resource is large in global scale, and the ability to be convert into thermal energy, liquid, solid or gaseous

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fuels and other chemical products (Sriram & Shahidehpour, 2005) had made biomass most preferable renewable energy source.

2.1.1 Biomass

Biomass is defined as organic materials that are derived from plants and animals according to Loppinet-Serani et al. (2008) either living or was living in the recent past that is consumed directly from the solid state. According to Twidell and Weir (2006), biomass is stated as all of materials involving plants and animals included their wastes and also residues which are organic and carbon-based material undergo combustion process by reacts with oxygen to release heat. Biomass only required a short period of time to be replace as energy resource compared to fossil fuels (Sriram & Shahidehpour, 2005).

As stated by Basu (2010), biomass is a promising alternative as it can provide a full range of feedstocks accounted to 10% of world's annual energy consumption (International Energy Agency, 2010). Its potential size of source is large in global scale and able to be convert into thermal energy, liquid, solid or gaseous fuels and other chemical products (Sriram & Shahidehpour, 2005). Biomass that are mostly used in previous research are from plant – based such as wood pellet, rice husk, pine sawdust, wood chips (Alauddin et al., 2010). These type of biomass are known as lignocellulose biomass which plays a major role in the production of sustainable energy because they are abundance, inexpensive and easier to be found as locally available (Young-Hun et al., 2015) in the country. The formation of biomass from plant take place as the conversion of carbon dioxide in the atmosphere into carbohydrate by the energy of sun in the presence of chlorophyll and water (Basu, 2010).

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2.2 Empty fruit bunches

Empty fruit bunches or better known as EFB are waste generated after removal or stripped of fruits or nuts from fruit bunches (Law et al., 2007, Jinn et al., 2015) either from the processing mills. EFB is left as waste had weighed same as for the production of oil palm (Asia Biomass Energy Cooperation, 2016). EFB used in this study is generated from oil palm industry as Malaysia is one of the main producers for oil palm in the world (Sabil et al., 2013b). Not only that, the industry also produce a huge amount of biomass product that will create wastes as the products does not being utilized properly (Sabil et al., 2013a)

Other biomass being produced by oil palm industry apart of EFB are included oil palm fronds (OPF) and oil palm trunks (OPT) which are generated in the plantations whereas mesocarp fiber (MF), palm-kernel shells (PKS) are by-products of oil extraction process (Uemura et al., 2013), respectively.

Among all of these biomass, EFB is the most often investigated by researches, since its quantity is one third of the oil palm biomass. EFB usually has fibrous texture and presence in a wet condition at its raw state. It is containing 30 - 35% lignocellulose, 1 - 3% residue oil and roughly 60% of moisture (Gunawan et al., 2009). The lignocellulose of EFB comprises of 17 - 33% of hemicellulose, 43 - 65% of cellulose and 13 - 37% of lignin on the dry weight basic (Palamae et al., 2014).

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2.3 Torrefaction

In existing literature, torrefaction is stated as a thermochemical process in an inert or limited oxygen environment. The biomass is slowly heated within a specified temperature range within 200 to 300 ℃ (Bergman & Kiel, 2005) under an atmospheric pressure and characterised by low particle heating rates (<50℃/min) (Basu, 2013) within the time less than 60 minutes. This process also known as roasting (Ciolkosz & Wallace, 2011), slow-and mild pyrolysis (Tumuluru et al., 2010), wood cooking and high temperature drying as well as pretreatment method for biomass conversion techniques such as gasification and co-firing (Van der Stelt et al., 2011).

During the torrefaction process, biomass is partly decomposing, giving off water eventually losing its moisture content and volatile matter (Koppejan et al., 2012). Biomass also loses 20 to 30% of its mass compared to energy content loss that is only 10% (Wilèn et al., 2013). The characteristics of the biomass are changed drastically compared to its original condition whereby it becomes more fragile as its mechanical strength is loses (Kongkeaw & Patumsawad, 2011). On the other hand, a torrefied biomass do have its own benefits or strength upon being torrefied such that increased stability and reduced susceptibility to microbial degradation, improved hydrophobic properties and higher carbon density than its raw biomass (Ciolkosz & Wallace, 2011).

2.4 Microwave technique

In general, microwave is an oven that cook foods very quickly by using electromagnetic waves rather than heat. In this context of research, microwave ovens or reactors are devices that involved electromagnetic wave in the range of 300 MHz to 300 GHz. Wang et al. (2012), stated that a single-mode microwave with 2.45 GHz frequency was applied.

Microwave when being compared to other directly heated reactors being used in the torrefaction process, it heated biomass particles within it whereas other reactors used conductive heating by an external source to heat the biomass by which the heat

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absorbed on the biomass surface is conducted inside. A direct coupling between microwave energy with molecules of biomass producing an efficient heating process. The heating process take place internally.

Electric component of electromagnetic microwave radiation causes by two main heating mechanisms which are dipolar polarization and ionic conduction. Microwave does not require other heat transfer medium, hot wall, particle or gas (Dhungana et al., 2012). In simple words, microwave reactor uses electromagnetic heating of water molecules in biomass as the mode of heat transfer, plug flow solids - unidirectional motion of gas and solids as the gas-solid motion and the particle heating take place directly and internally.

There are many reactors can be used in the torrefaction process for example; fluidized bed, hydrothermal reactor, entrained flow reactor, rotating drum and screw or stationery shaft. Among all of these reactors, microwave is preferable for a lab-scale research as it is less time consuming that lead to conservation of energy as the energy consumed is lowered, easy to be clean after in used and eco-friendly.

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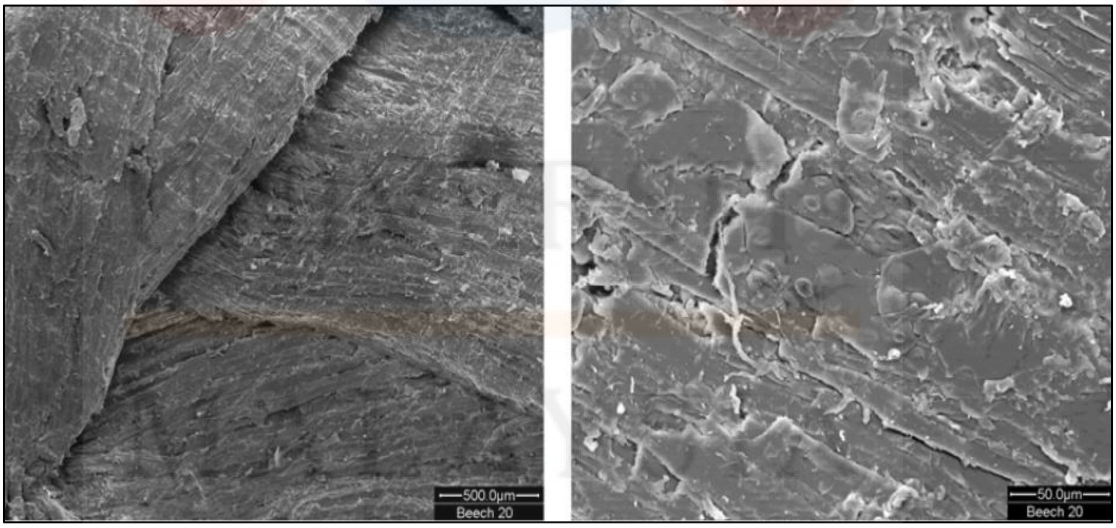
2.5 Torrefied material

Torrefied material is a material that already undergo torrefaction process or final product of torrefaction. In this context, the material is biomass. Torrefied biomass is strongly influenced by the parameters of torrefaction which are temperature and time. The properties of torrefied biomass are enhanced compared to its raw material. As an example, the lower oxygen-carbon-ratio compare to the original biomass as oxygen is removed from the material (Van der Stelt et al., 2011). The inconvenient of raw biomass such as high oxygen content, low calorific value, hydrophilic nature and high moisture content (Chew & Doshi, 2011; Pimchuai et al., 2010; Sarvaramini et al., 2013; Uemura et al., 2011).

The upgraded properties of biomass through torrefaction had to be studied further such as its chemical composition, surface morphology and many more. Many comparisons can be made between torrefied and its raw biomass likewise the previous study had obtained (Law et al., 2007; Sabil et al., 2013a).

2.6 Morphology of biomass

Surface morphology, in terms of materials, is the surface formation or structure of any materials. The torrefaction process does change the surface morphology of biomass (Cruz et al., 2012; Ibrahim et al., 2013) in terms of its particle sizes and structures look. Different biomass gives different surface morphology upon undergo torrefaction.



(a) (b)

Figure 2.1: Fracture surface of beech pellets manufactured at 20℃ at low (a) and higher magnification (b) (Stelte et al., 2011)

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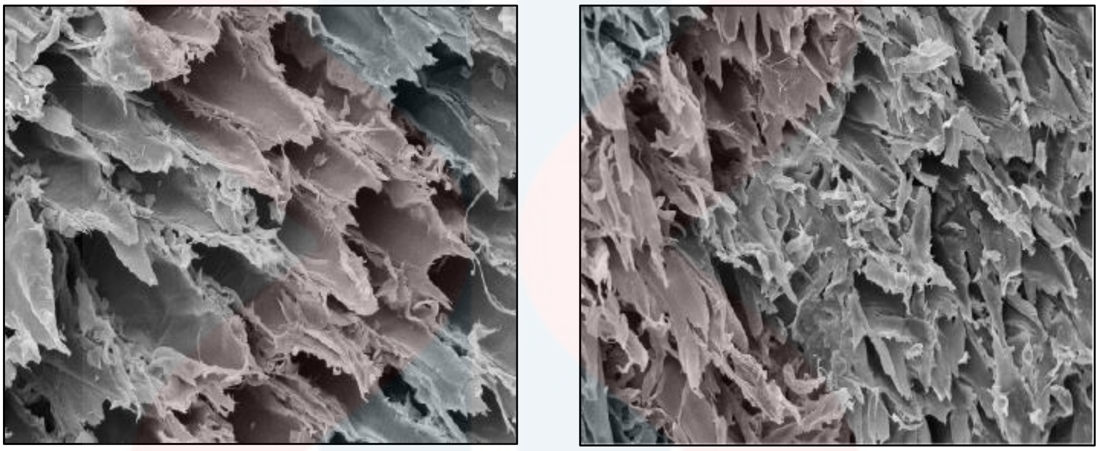
When the biomass is observed under scanning electron microscope (SEM), different magnification gives different images of surface morphology of the biomass (Figure 2.1). Stelte et al., (2011) explained that at high magnification, a brittle failure structure of beech is shown crystal clear as inter-particle gaps and voids are existing, but at low magnification, areas of a brittle and cohesive fracture are only shown.



(a) (b)

Figure 2.2: Images SEM of pine sawdust: in natural (a) and torrefied (b) at magnification of 1.00K X (Cruz et al., 2012)

The determination of minimum and maximum magnification used need to be considered as those magnifications will determine what need to be seen. For example, Cruz et al., (2012) using 1.00K X of magnification which is relatively moderate magnification to see an average particles size of pine sawdust decreased in natural by the loss in structure with tubular format as well as the presence of lamellar and pith structures and pores with average diameter of 10 μm whereas for torrefied material, the lamellar structure characteristics are maintained and average diameter underwent an expansion giving a range of 10 μm to 30 μm. Even though the magnification used is same, but the condition of the pine sawdust is varied from its raw to torrefied pine sawdust as shown above on Figure 2.2.

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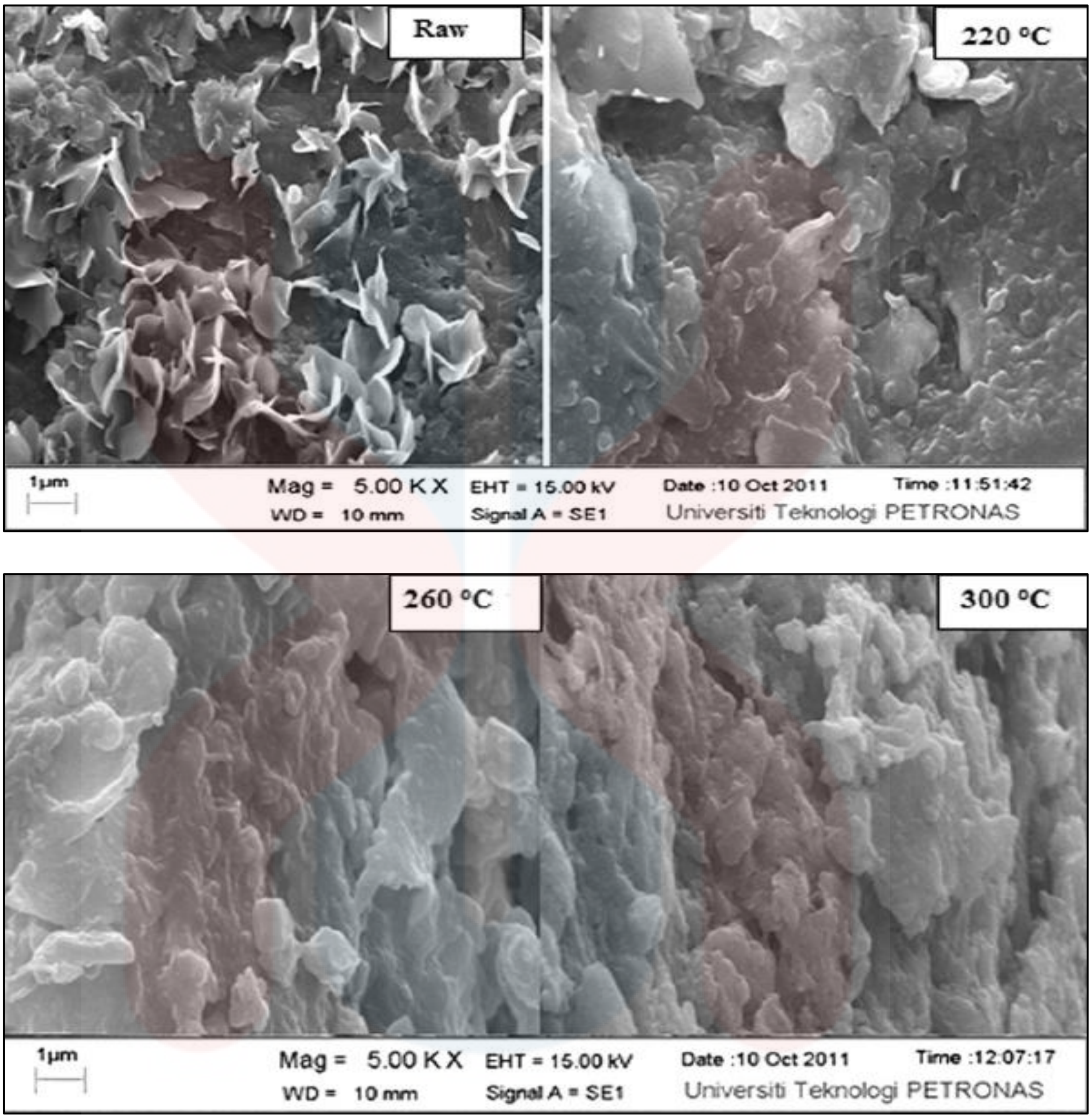

Figure 2.3: SEM images for raw and torrefied EFB (with torrefaction temperature of 220, 260 and 300) at magnification of 5.00K X (Sabil et al., 2013)

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As for EFB, the changes on surface structure upon undergo torrefaction are observed under SEM at the magnification of 5.00K X as shown in Figure 2.3. Structure of raw EFB showing a clear sharp edge but when under severe torrefaction condition, it has almost flattened and disappeared. Another observation is the internal structure of EFB is completely decomposed at the temperatures of 260 ℃ and 300 ℃ (Sabil et al., 2013a). Thus, magnifications of SEM are really important when a comparison between a raw and torrefied need to be examined.

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2.7 Bonding behaviour of biomass

In the thermal process of biomass, bonding behaviour can be determined by examined the functional groups presence in certain wavelength on fourier transformation infrared (FTIR). Generally, FTIR is a non-destructive analysis instrument which provides information about chemical bonding in a material.

Different wavelength associated with different types of functional group that are exists in EFB. Functional groups that are often detected by FTIR on oil palm empty fruit bunch (EFB) are shown in Table 2.1 (Hamzah et al. 2013):

Table 2.1: Functional groups of oil palm empty fruit bunch (EFB) on certain wavenumbers of FTIR (Hamzah et al., 2013)

Wavenumbers (cm^{-1})	Functional Group
3391.23	Phenols & alcohols; O-H
2918.74	Alkanes; C-H
2849.30	Carboxylic acid; C=O
2327.30	Nitrile; C=O (Could be CO_2 impurities)
1578.59	Nitro group; N=O

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CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

Materials that were used in this research were empty fruit bunches (EFB) which were derived from oil palm wastes of oil palm *(Elaeis guineensis sp.)* at an oil palm plantation in Felda Kemahang, Tanah Merah, Kelantan. EFB is choose due to its potential as renewable biomass since it is locally abundant in Malaysia and rich in lignocellulosic components such as cellulose, hemicellulose and lignin (Pua et al., 2012; Zakaria et al., 2013).

(a) (b)

Figure 3.1: Oil palm tree (a) and empty fruit bunch (b)

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3.2 Methods

Process involved in this research could be divided into; materials preparation, torrefaction process, characteristics testing and analysing, evaluating and comparison of obtained experimental data.

3.2.1 Materials preparation

EFB was collected from an oil palm plantation in Kelantan, Malaysia which was crunched using a mechanical grinder. Then, the ground powders were sieved into desired mesh size of 250 μm, 500 μm and 750 μm in diameter. Later on, the raw EFB was dried in an oven for a one day, 24 hours at a constant temperature of 105 ℃.

The purposes of crunching and sieving the raw EFB were to obtained a small scale of sample without any contaminations of unwanted particles such as dust. This was because the unwanted particles might affect the final results when the sample being analysed. On the other part, the drying process was used to ensure the moisture contents of raw EFB were reduced to 16 – 18%.

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3.2.2 Torrefaction process

The torrefaction process was pictured in Figure 3.2. The experimental system consists of nitrogen cylinder, a temperature controller, microwave, gas cooling system and peristaltic pump.

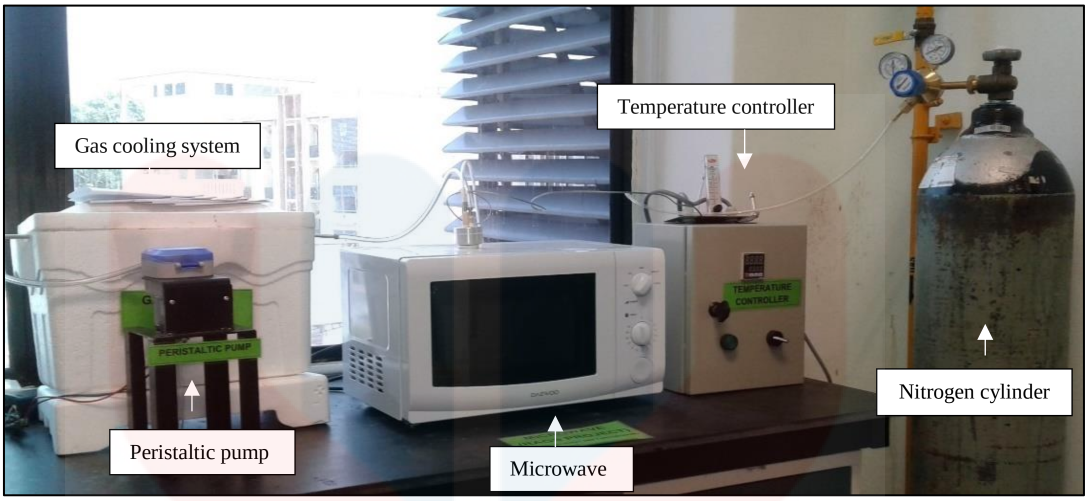

Figure 3.2: Experimental set-up in Universiti Malaysia Kelantan (UMK)

The steel cylinder was used to purge nitrogen gas to the system, so that a non-oxidizing environment was created. Flow rate of nitrogen gas is 15 mL min^{-1}. Nitrogen was used instead of oxygen because oxygen will cause oxidation and ignition in the microwave as stated by Tumuluru et al. (2010). The volumetric flow rate of nitrogen was controlled by rotameter.

Torrefaction process was carried out in an ambient pressure using a domestic microwave oven as a reactor at a maximum power range between 385 W, 540 W and 750 W. Temperature of the microwave was controlled by the temperature controller, placed at the right of the microwave as in Figure 3.2. The temperature was varied at 200 ℃, 225 ℃, 250 ℃, 275 ℃ and 300 ℃, respectively, for about 20 - 60 minutes. The residence time starts when the EFB temperature had reached the set temperature until it starts to cool down (rapidly quenched) under nitrogen flow to prevent any further reaction.

Inside the microwave, generator will convert electrical energy to the microwave energy. Later on, the microwave energy was guided to the reaction chamber. Then, in the reaction chamber, sample of EFB will react once microwave energy was in contact with it. A gas cooling system was connected to the microwave

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device to prevent overheating during the experiment (Wang et al., 2012). The product of this torrefaction process was known as torrefied EFB.

3.2.3 Characteristic testing

Torrefied EFB will undergo characteristics testing in terms of surface morphology and its bonding behaviour. The surface morphology and bonding behaviour of torrefied EFB were analysed using Scanning Electron Microscope (SEM) and Fourier Transformation Infrared (FTIR). Nevertheless, the raw EFB also conducted the same testing as the torrefied EFB, so that the comparison between raw and torrefied EFB can be made.

Scanning Electron Microscope (SEM)

Figure 3.3: Images of Scanning Electron Microscope, SEM

(Source: nanofabrication.unt.edu)

SEM analysis wit model JEOL JSM6360 LA was carried out to study the changes in surface structure of the torrefied EFB alongside with its raw material under different magnification. Basically, analysing the surface morphology of EFB using SEM takes place by placing EFB sample inside the specimen’s chamber. Then, the

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EFB is exposed to electron beam which is bombarded by electron gun. The electron beam is scanned across the surface of specimen causing emissions to be released either backscattered electron (BSE), secondary electron (SE) or x-ray. Intensity of the electrons produce are depending on the shape and composition of the irradiated EFB. Later on, these electrons are collected by a detector which generate electrical signals. An image is formed on the cathode ray screen. The image produce is large in depth of focus. Thus, surface structure of EFB is able to be analysed (Pujari, 2014).

Fourier Transformation Infrared (FTIR)

FTIR is conducted to study the changes in bonding behaviour of torrefied material in terms of the presences of its functional groups (Ibrahim et al., 2013).

Figure 3.4: Images of Fourier Transformation Infrared, FTIR

(Sources: gatewayanalytical.com)

The main purpose of FTIR is to provide information about chemical bonding in a material. FTIR instrument with model Nicolet iN10 FTIR Microscope relies on the fact of molecules have specific frequency absorb light in the infrared (IR) region of electromagnetic spectrum. This absorption is corresponding to the bonds present in the molecule. The range of IR region that is measured in terms of wave numbers are between 4000 cm^{-1} – 600 cm^{-1}. Background emission spectrum of IR source is

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recorded first, followed by emission spectrum of IR source with EFB sample. Ratio of sample spectrum to background spectrum is directly related to the absorption spectrum of sample. The presence of various chemical bonds and functional groups present in the sample are indicated by absorption spectrum from the natural vibration frequencies (Steven, n.d.)

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3.2.4 Analysis, evaluation and comparison of obtained data

Last but not least, raw and torrefied EFB are being analyse, evaluate as well as compare between the surface morphology and bonding behaviour based on data obtained.

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CHAPTER 4

RESULTS AND DISCUSSION

4.1 Overview results

Fundamentally, torrefaction process was studied for observing microstructure in empty fruit bunch (EFB) as well as the alteration of its functional groups. EFB had to undergo several steps of torrefaction process to achieve desirable outcome. As stated in previous chapter, this process was carried out under four considerable parameters, namely; size of EFB, generated power, temperature and residence time. A suitable power generated had been achieved through the effect of heating rate, temperature profile and study of magnetron which were further discussed on the next subtopic. Next, size of EFB and its initial mass were chosen using an analytic software known as Response Surface Method (RSM) based on Central Composite Rotatable Design (CCRD). Generally, RSM was functioned as design of experiment by minimizing the number of possible experiments with a lots of detailed information (Jamaluddin et al., 2013). As the results, EFB with 500 µm in size with the mass of 10 g were chosen. Later on, the exact torrefaction process took place after choosing EFB at the power generated of 385 W under a varied temperature and time, respectively. Then, the analysis of characteristic testing took over whereby the surface structures of fibrous

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EFB were observed to see the effect of torrefaction as well as changes of bonding behaviour that causing the degradation of lignocellulose.

4.1.1 Effects of heating rate towards microwave power selection

Heating rate of torrefied EFB under selected power generated was corresponding with energy yield of the samples. Higher heating rate (HHV) would facilitated down the energy yield as temperature and time increased. Huang et al. (2016) stated that increasing of heating rate is directly proportional to the increasing of power generated from the microwave.

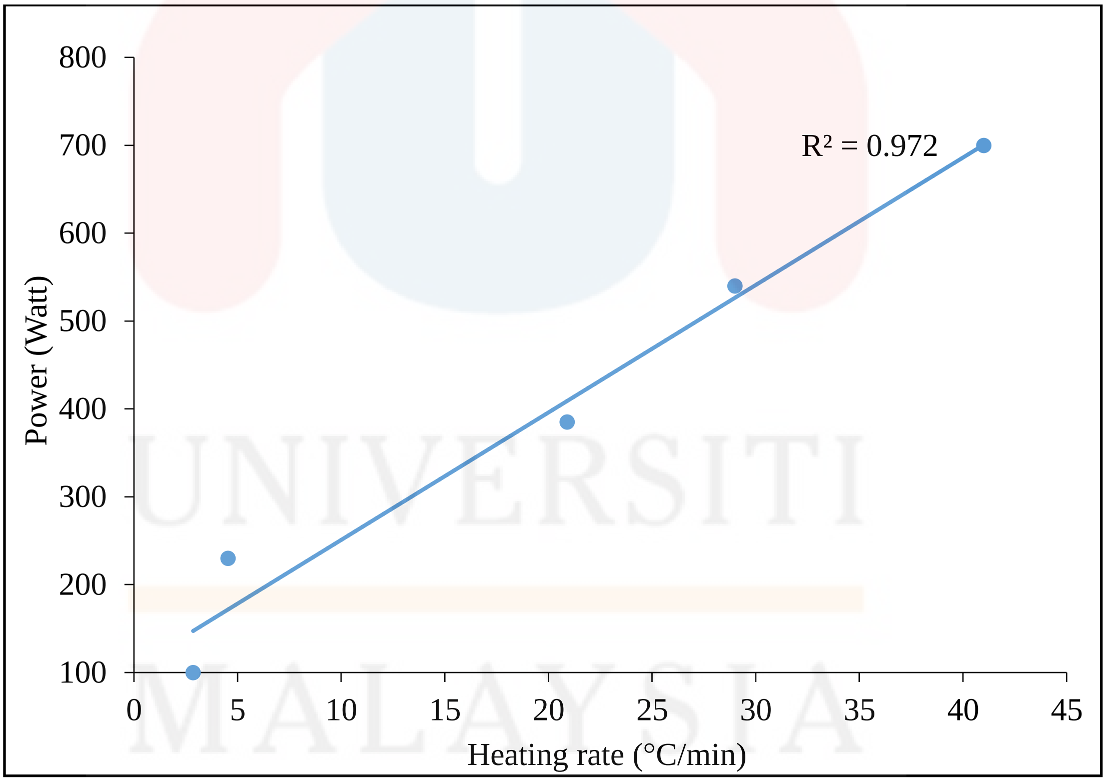

Figure 4.1: Effects of heating rate (℃/min) towards Power (Watt)

Based on the Figure 4.1, it could be clearly seen that low power generated showed a low heating rate which was not ideal. The heating rate produced at 385 W was 20.90 ℃/min which was suitable to reached the desired temperature. As the power

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generated increasing, high thermal expansion was produced. Thermal expansion inside the alumina crucible did affecting the value of heating rate. At a very high temperature, the tendency for alumina to crack was huge because the inability of alumina to withstand high thermal expansion causing thermal shock to be occurred. Mertke and Aneziris (2014) claimed that alumina crucible able to retain strength when thermal shock was 14.51 MPa at the maximum temperature of 500 ℃. However, when high temperature used for relatively high power generated, the alumina unable to retain strength. This case was presented by the power generated of 540 W and 700 W. The values of heating rate (℃/min) and thermal expansion (℃$^{-1}$) for each power generated were stated in Table 4.1.

Table 4.1: Heating rate (℃/min) and thermal expansion (℃$^{-1}$) of alumina crucible under different power generated

Power (Watt)	**Heating rate (℃/min)**	**Thermal Expansion (℃$^{-1}$)**	**Condition of alumina crucible**
100	2.86	6.94×10^{-6}	Not crack
230	4.54	7.34×10^{-6}	Not crack
385	20.90	7.74×10^{-6}	Not crack
540	29.00	27.8×10^{-6}	Crack
700	41.60	37.7×10^{-6}	Crack

Based on Y. F. Huang et al. (2012), as the microwave generating power kept on increasing, it will have caused the heating rate value to become higher. As heating rate increased, the mass yield decreased alongside with low energy yield (Wen et al., 2014). For the biomass undergo torrefaction, the total mass maintained would be

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lowered as it tends to loss in the form of gas released and liberated of liquid (van der Stelt et al., 2011). These conditions were presented in Figure 4.2.

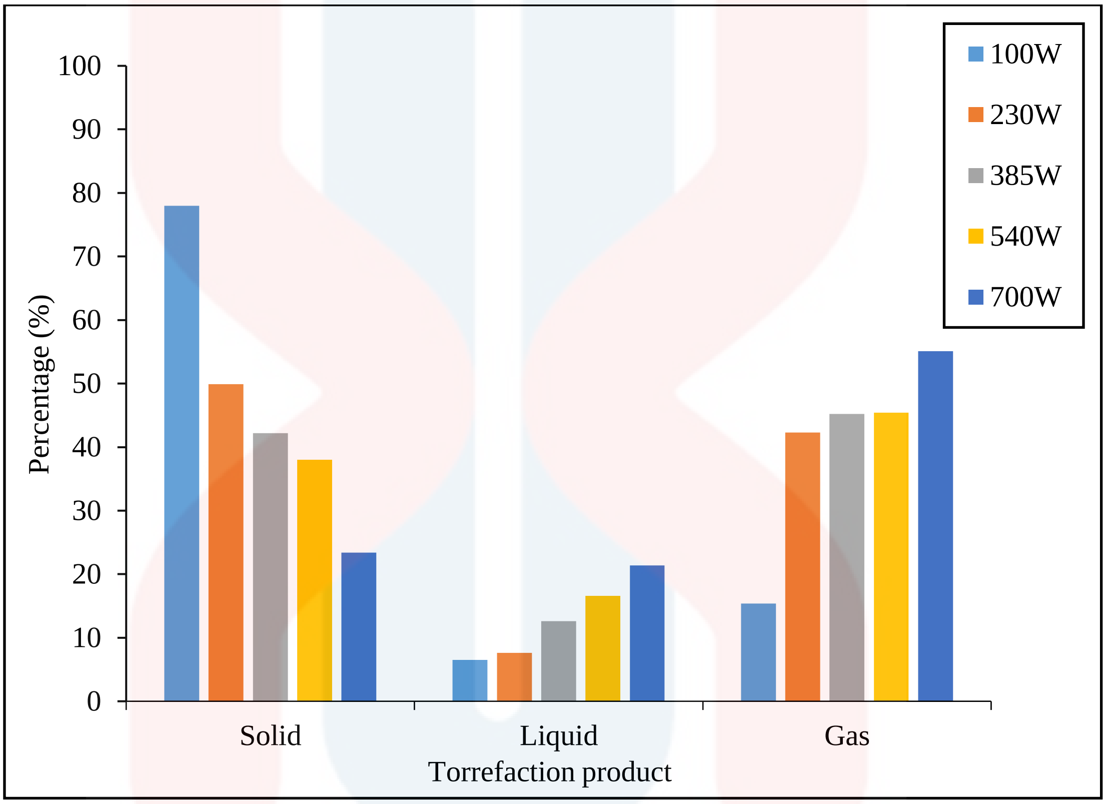

Figure 4.2: Percentage (%) of fractional mass

A comparison yields of solid, liquid and gas were stated in Figure 4.2 resulting from the heating process of different power used. The percentage of fractional mass showed a higher solid percentage during the power of 100 W, unfortunately, the percentage of liquid and gas deposited were lower. Even though a high percentage of solid retain is good, the condensation of liquid and gas should be high to eliminate moisture content and made lignocellulose to be decomposed. Meanwhile, higher power generated showed that the percentage of solid was lowered than the others. This is because at higher power, more ashes were formed as discussed by Saleh et al. (2013).

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4.1.2 Temperature profile

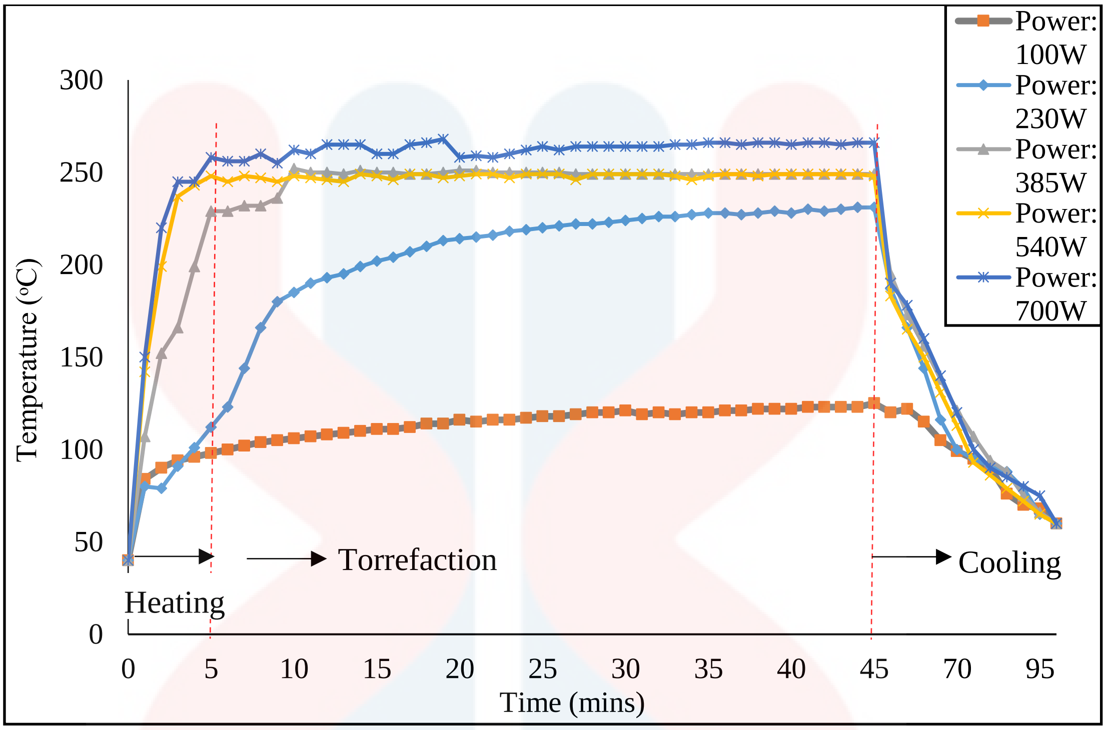

Figure 4.3: Temperature profile of torrefaction time

Figure 4.3 showed the temperature profile against time under a different power generated. Heating of EFB took place for about first 5 minutes before torrefaction process took place for 40 minutes then, cooled to 60 ℃. From the figure, 100 W produced a very low temperature profile of 107 ℃ and hold before cooled down. This condition also applied to 230 W, as the heating took a longer time up to 5 minutes. Not only that the torrefaction process took place at low temperature, the EFB was only being dried up and not fully torrefied. Meanwhile, power generated of 385 W, 540 W and 700 W, respectively boosted to high temperature at the first 5 minutes. The only different was during the holding time of torrefaction process. 385 W showed a consistent holding time whereas 540 W, fluctuated down for nearly 5 minutes causing its heating rate to be a little bit low as well as the torrefaction process started slowly. On 700 W, it exceeds the desired temperature of nearly 250 ℃ but kept on fluctuated made it unsuitable due to more ash content.

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4.1.3 Study of magnetron

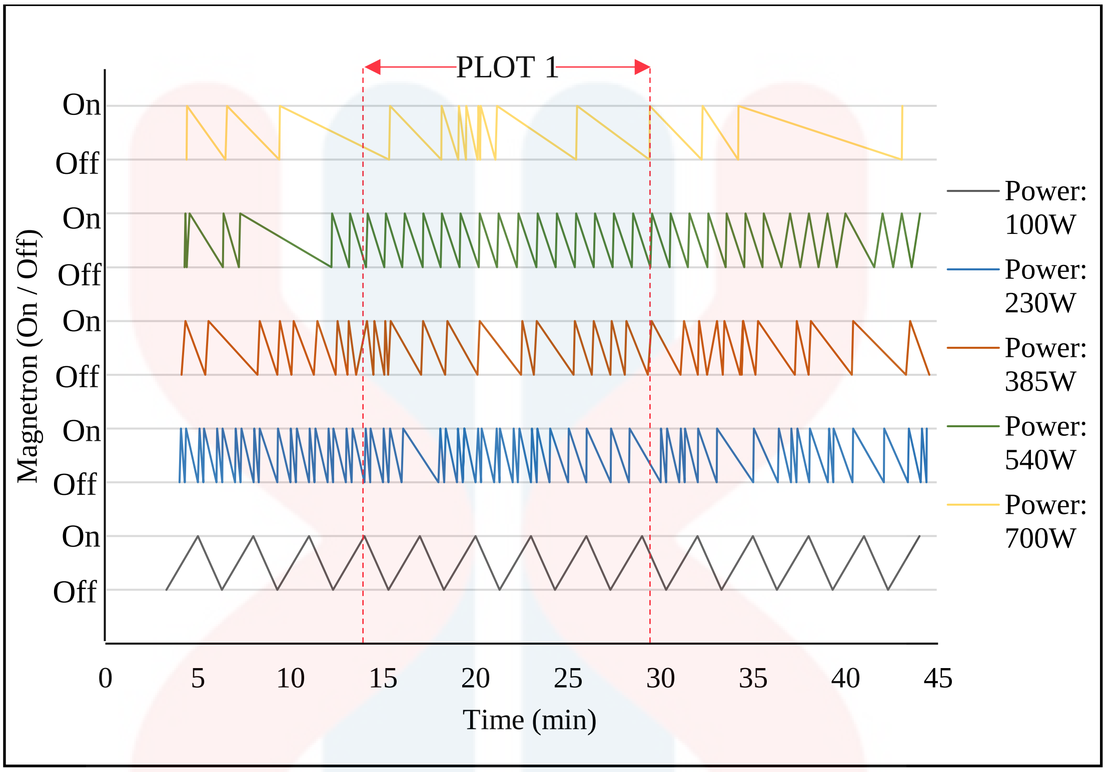

Figure 4.4: Magnetron (On / Off) against Time

Figure of magnetron as illustrated in Figure 4.4 was plotted to show how frequent the electrons were released inside the microwave as torrefaction process took place. A consistent but huge peak was produced for 100 W, as the result from frequent released of electrons but on a consistent duration of time. This might happen due to the low power. During 230 W, electrons released were more frequent but with inconsistent duration, as sometimes they were rapidly released but 15 to 20 minutes, they took time to release. For the power generated of 385 W and 540 W, the electrons were released in quite consistent frequencies indicate the torrefaction took place well. On the other hand, 700 W showed huge gap for the electrons released which was not ideal for product of torrefaction because the process occurred in an ambient condition inside the microwave.

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4.2 Surface Morphology of Empty Fruit Bunch (EFB)

Structure of EFB was observed under the scanning electron microscope (SEM) under magnifications of ×700, ×2000 and ×10000 were shown in Figure 4.5, Figure 4.6, Figure 4.7 and Figure 4.8, respectively.

Raw EFB as shown in Figure 4.5, showing a smooth surface with sharp edges at the end (Figure 4.5 a and b). An unorganized and irregular fibers arrangement could be seen at its cross-sectional area (Figure 4.5 b and c). There were possibilities of pores, voids or inter-particle gaps presence in the structure of raw EFB but they were not clearly determined due to the irregular fiber arrangements.

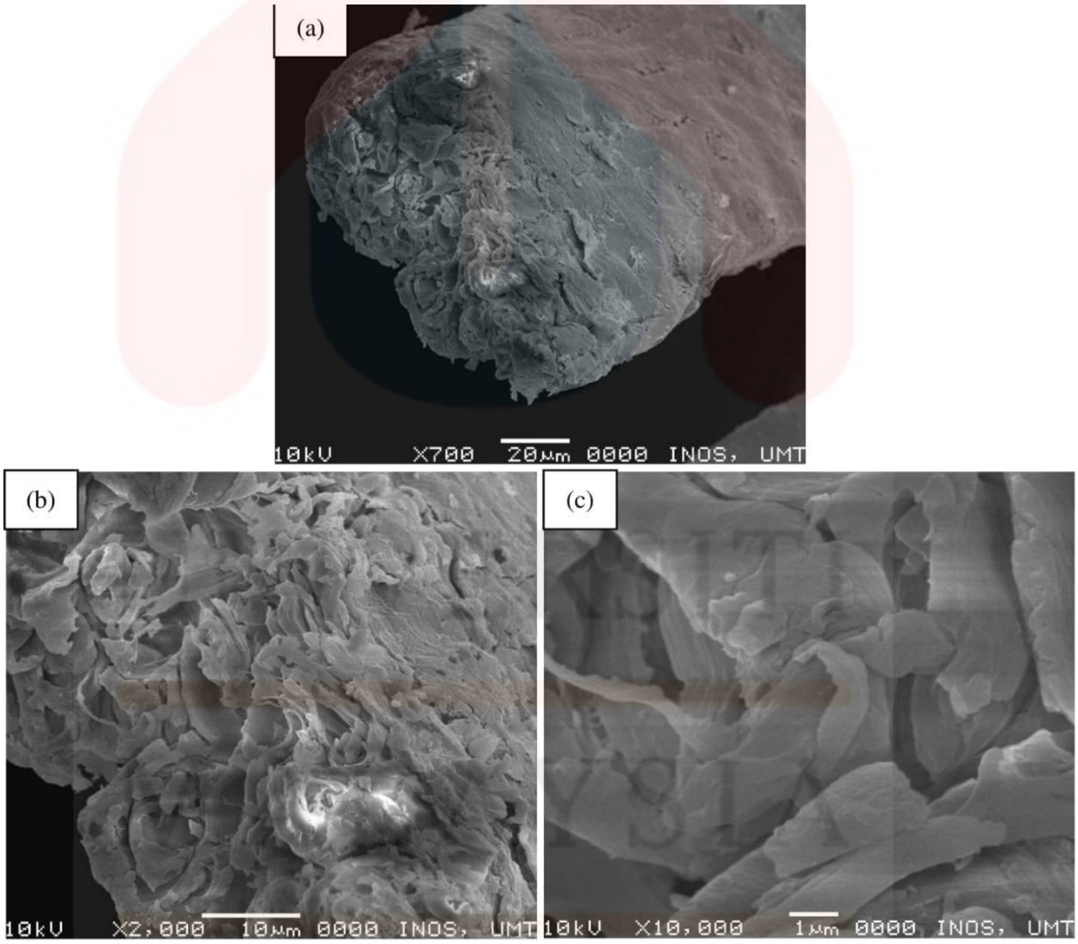

Figure 4.5: Structure of Raw EFB sieved 500 µm. (a) magnification ×700, (b) magnification ×2000 and (c) magnification ×10000

When compared to the torrefied EFB shown in Figure 4.6, Figure 4.7 and Figure 4.8, respectively, spongy-like structure were shown as the effect of torrefaction process. Figure 4.6 showing the 20 minutes' residence time of torrefaction process where at the formation of porous were observed and determined. In Figure 4.6 (a, b and c), only torrefied EFB at 200 ℃ showing a similar structure as raw EFB. This was because of the low temperature was used in the torrefaction process which almost unaffected the structure. Started at temperature of 225 ℃ as shown in Figure 4.6 (d, e and f), interparticle gaps were formed but the sharp edge between the gaps were not completely diminished. The sharp edge became completely flattened at the torrefaction temperature of 250 ℃.

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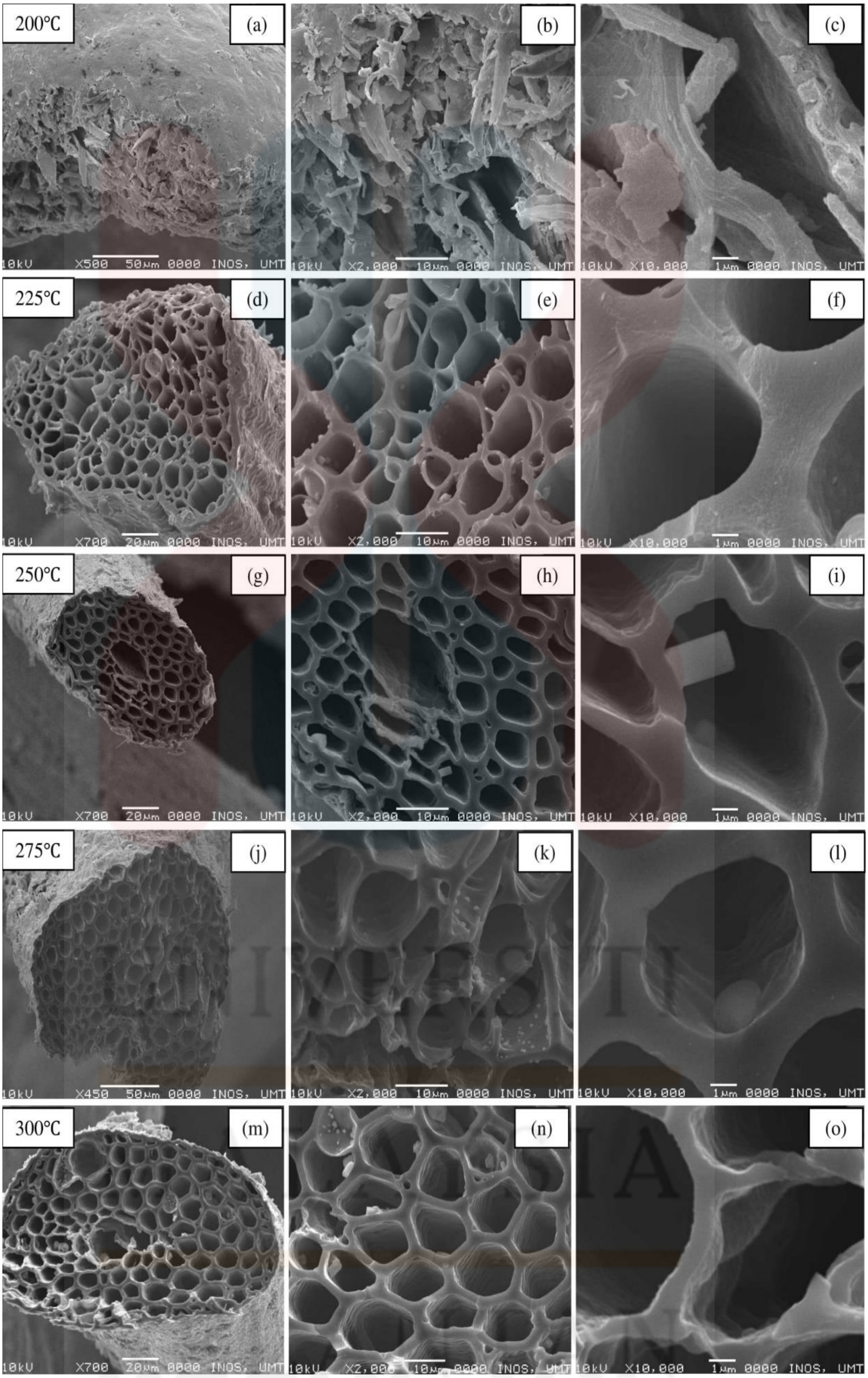

Figure 4.6: Structure of Torrefied EFB on 20 minutes of torrefaction at respective temperatures with magnifications of ×700 (a, d, g, j and m), ×2000 (b, e, h, k and n) and ×10000 (c, f, i, l and o)

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As the temperature increased corresponded with residence time upon torrefaction process take place, the structures of EFB became fully porous as shown in Figure 4.7 and Figure 4.8. On a high magnification of ×2000 and ×10000, tiny particles could be clearly saw which were attached to the porous-structure of EFB. These were happening due to the particles of alumina crucible which were crack as the effect from thermal expansion happened inside it, causing alumina unable to retain strength as temperature increased upon time. Mertke and Aneziris (2014) claimed that alumina crucible could retained strength when the thermal shock was 14.51 MPa at relatively high maximum temperature of 500 °C. However, frequently used alumina crucible could triggered the crack formation. Hence, causing its particles to attached to the structure.

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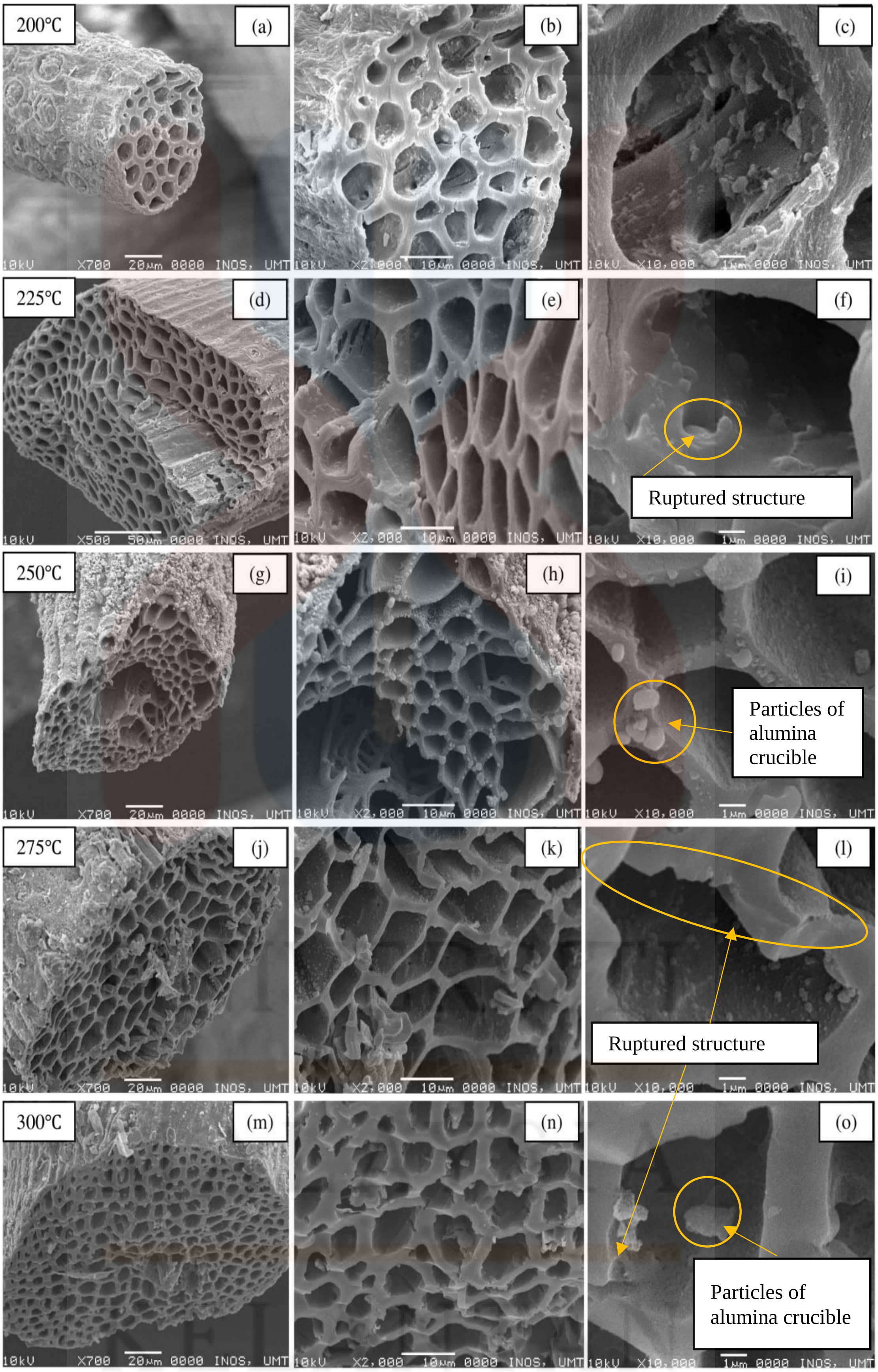

Figure 4.7: Structure of Torrefied EFB on 40 minutes of torrefaction at respective temperatures with magnifications of ×700 (a, d, g, j and m), ×2000 (b, e, h, k and n) and ×10000 (c, f, i, l and o)

Figure 4.8 exposing a clear image of ruptured at the surface structure of the EFB from cross-sectional area's point of view. The ruptured images could be seen during 40 minutes of residence time (Figure 3.3) but not as clear as residence time of torrefaction was 60 minutes. The stretching of ruptured surface structures was caused by the degradation of lignocellulose compared to during the low temperature at low residence time. The clause of breakage of fibre wall surface was contributed by the rupture impact as the decomposition of hemicellulose and cellulose take place.

Not only the ruptured images contributed to the degradation of lignocellulose, the porosity also participated in the degradation. High porosity indicates high degradation of lignocellulose. Longer residence time causing a large amount of hemicellulose and cellulose were destroyed. Since the cellulose was degraded, the fiber wall became thinner as residence time was longest, 60 minutes. This was because of cellulose tend to lose its strength. The longer the residence time, the faster the degradation happened as well as the temperature.

When looked up closely at ×10000 magnification, the fiber was torn apart. Each of hemicellulose, cellulose and lignin were depleted under a certain temperature of torrefaction (Chen & Kuo, 2011; Wen et al., 2014) whereby hemicellulose was decomposed at very low temperature started at 200 ℃ and completely disappeared as it almost reached 300 ℃ (Chen & Kuo, 2011b). Temperature above 250 ℃ was placed for cellulose degradation whereas for lignin was on high temperature of 300 ℃. Decomposition of lignin take place at high temperature and residence time, respectively because of its complex structure consists of phenolic polymer that made them strong and durable against an enzymatic attack (Gomez et al., 2008). Thus, lignin was hard to be degrade.

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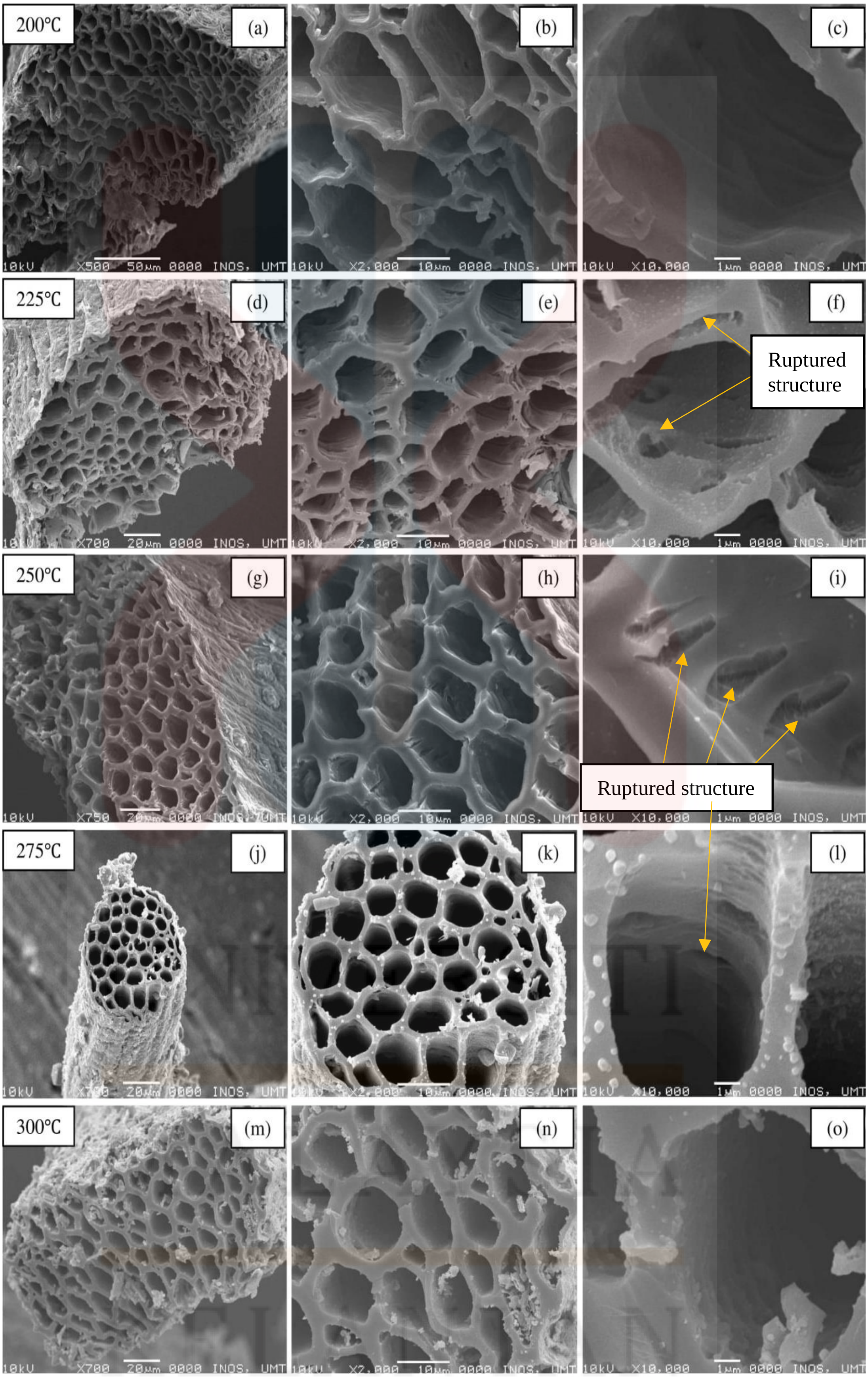

Figure 4.8: Structure of torrefied EFB on 60 minutes of torrefaction at respective temperatures with magnifications of ×700 (a, d, g, j and m), ×2000 (b, e, h, k and n) and ×10000 (c, f, i, l and o)

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4.3 Fourier transform infrared spectra

Fourier Transformation Infrared (FTIR) spectrometry was used in this studied to distinguish the change in chemical structure for both raw and torrefied EFB by analysed the functional groups which later on, will affected the degradation of hemicellulose, cellulose and lignin as the impact of torrefaction. Generally, studying the functional group helped in determined the frequency of each lignocellulose to be degenerated.

Figure 4.9, Figure 4.10 and Figure 4.11 showing the functional groups presence during 20 minutes, 40 minutes and 60 minutes of torrefaction, respectively for selected torrefied EFB at temperature of 200 ℃, 250 ℃ and 300 ℃ and being compared to the raw EFB. According to Nasri et al. (2013), absorption bands around 850 - 890 cm^{-1} indicated the deformation of C-H bending, meanwhile bands between 1045 up to 1055 cm^{-1} corresponded to the increasing of C-O stretching whereas peaks ranged at 1430 - 1440 cm^{-1} showing rapid stretch of C-H bond. On these bands, $C-H_2$ shearing and benching present. Bands of 1730 - 1750 cm^{-1} stating the C=O stretching take place. Both C-H and $C-H_2$ bonds appeared around bands of 2930 – 2950 cm^{-1} but the bonding was stretched as the temperature of torrefaction increasing whereby the reaction of C-H was almost complete. The bands around 3420 – 3435 cm^{-1} were assigned for O-H stretching with rapid decreasing intensity. C-O-C stretching bond also identified at the band of 1260 cm^{-1}.

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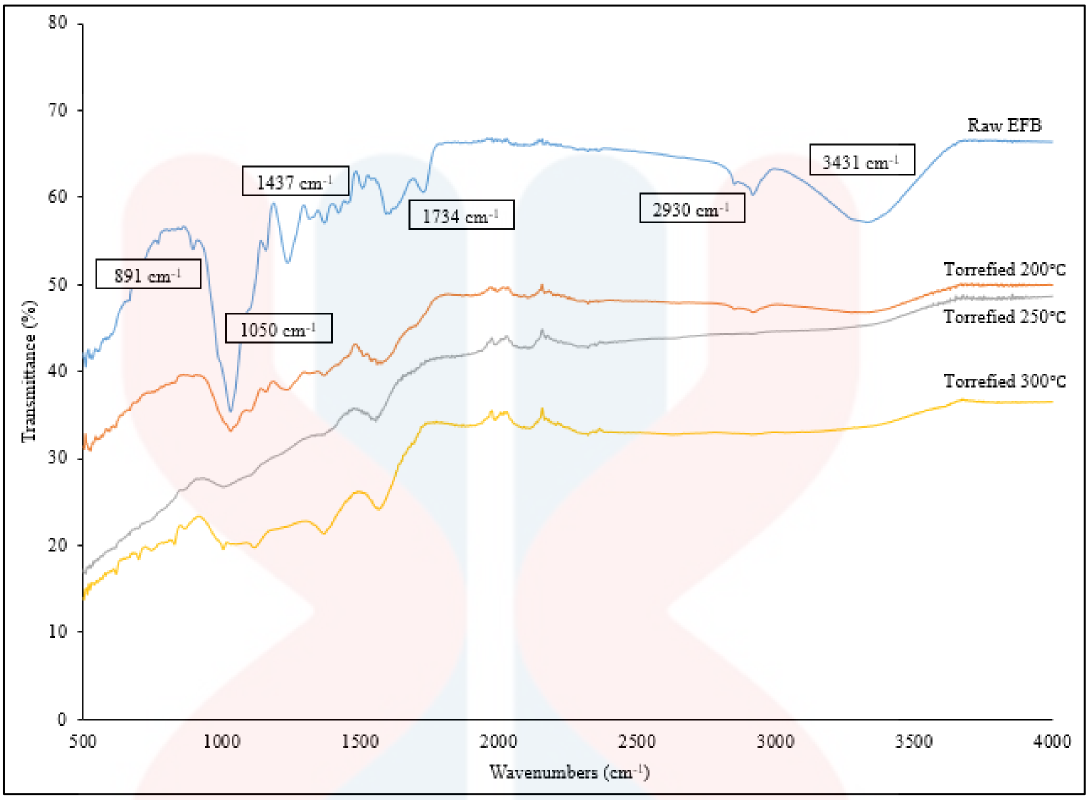

Figure 4.9: Functional groups presence during 20 minutes

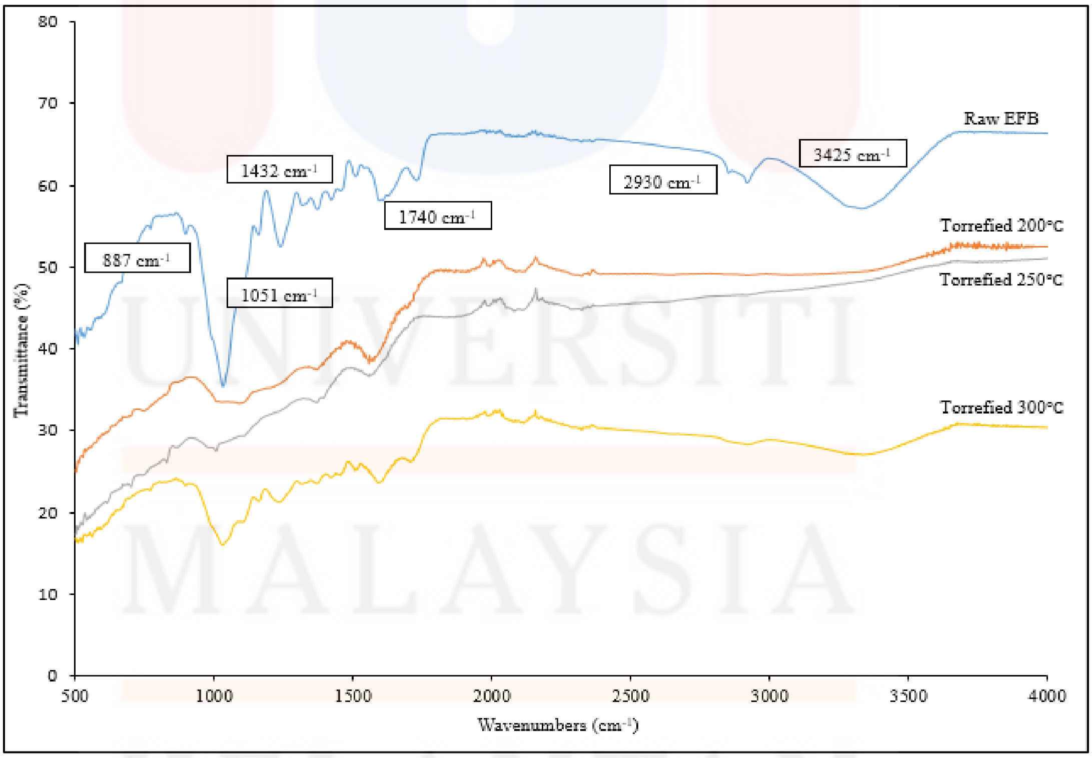

Figure 4.10: Functional groups presence during 40 minutes

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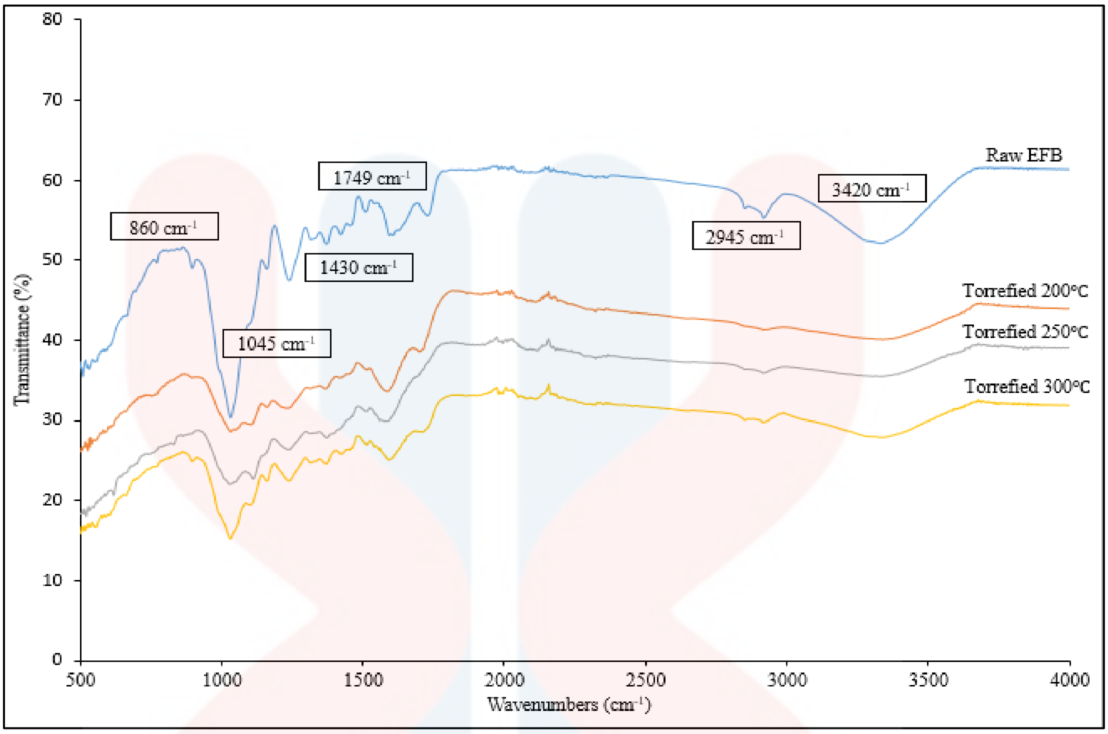

Figure 4.11: Functional groups presence during 60 minutes

Based on these wavenumbers of infrared spectra, the degradation of hemicellulose, cellulose and lignin could be determined as stated by Zakaria et al (2013). Hemicellulose started to decomposed at the absorption band of 1050 cm^{-1} and 1734 cm^{-1} while cellulose takes place simultaneously on band 1050 cm^{-1} and at 1437 cm^{-1} which the C-H$_2$ bond was shearing. In extend, lignin was breakdown at lower infrared bands of 891 cm^{-1} as well as at 1256 cm^{-1} and 1437 cm^{-1} which similar to the decomposition of cellulose but the bond was benched.

Hemicellulose decomposed at a relatively long wavelength compared to cellulose and lignin because the hemicellulose does not require a longer time of torrefaction (60 minutes) to be deformed. It was already take place during 40 minutes of residence time at lower temperature as could be seen in Figure 4.7 by the formation of rupture structure. Meanwhile, for cellulose, the structure was degraded at relatively lower wavelength than of hemicellulose indicating the process take place at high temperature alongside high residence time between 40 minutes up to 60 minutes. Since

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the structure of lignin was complex (Gomez et al., 2008), its deformation took place at a lower wavelength with not fully detached explaining that it required relatively high temperature of 250 – 500 ℃ to be degraded whereby temperature above 300 ℃ was for pyrolysis process (Chen & Kuo, 2011a). Thus, the degradation of lignin could only be appeared at lower wavenumbers.

Longer residence time would cause lignocellulose to be decomposed. In other words, as the time of torrefaction increasing, the intensity of bands whereby the degradation of hemicellulose took place was decreasing as can be seen in Figure 4.9, Figure 4.10 and Figure 4.11, respectively. It was not only for hemicellulose, both of cellulose and lignin were also affected by the time but not fully caused degradation to be happen. Lignin; which has been mention in previous paragraph, did not fully detached its structure made the its band to showed only a slightly decrease in bands.

Summary of FTIR analysis with respective functional groups presented on particulars bands associates with their compounds were stated in Table 3.1.

Table 4.2: FTIR functional groups present at particular wavenumbers with their compound

Wavenumber (cm^{-1})	**Functional group (Phenomenon)**	**Compound class**
3420 - 3435	O-H stretching	Phenols, alcohols
2930 - 2950	C-H and C-H_2 stretching	Alkanes
1730 - 1750	C=O stretching	Carbonyl (aldehydes, carboxylic acids, ketones)
1430 - 1440	C-H stretching, C-H_2 shearing and benching	Alkanes
1260	C-O-C stretching	Aromatics

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Table 4.2 (Continued)

1045 - 1055	C-O stretching	Esters, ethers
850 - 890	C-H bending (deformation)	Aromatics

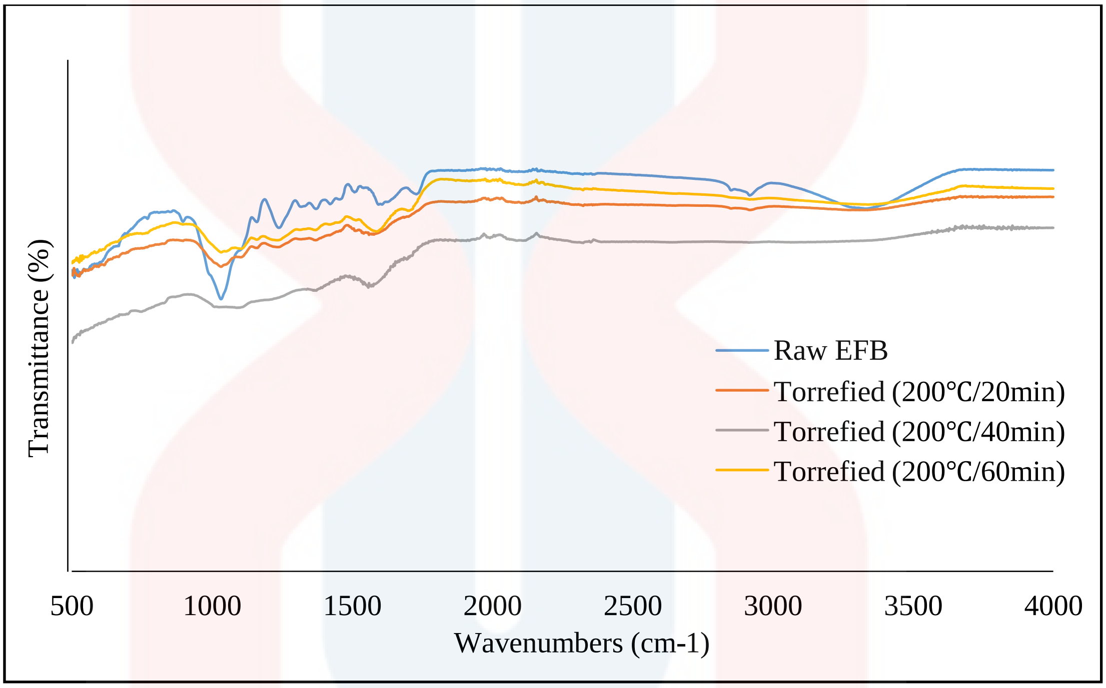

Figure 4.12: Functional groups presence for 200℃

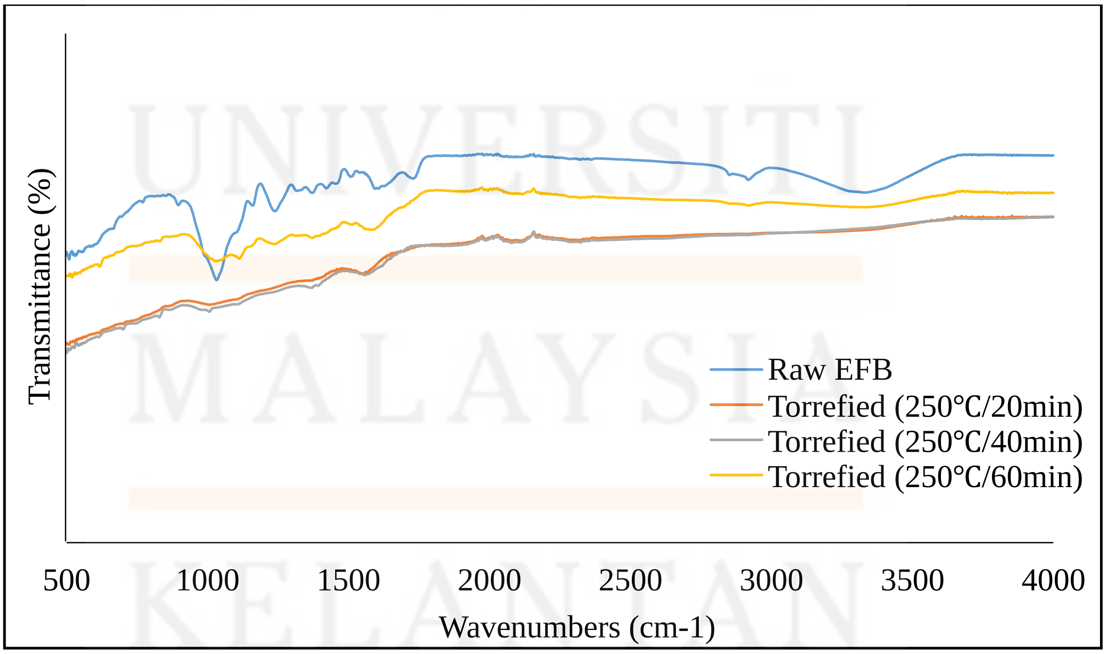

Figure 4.13: Functional groups presence for 250℃

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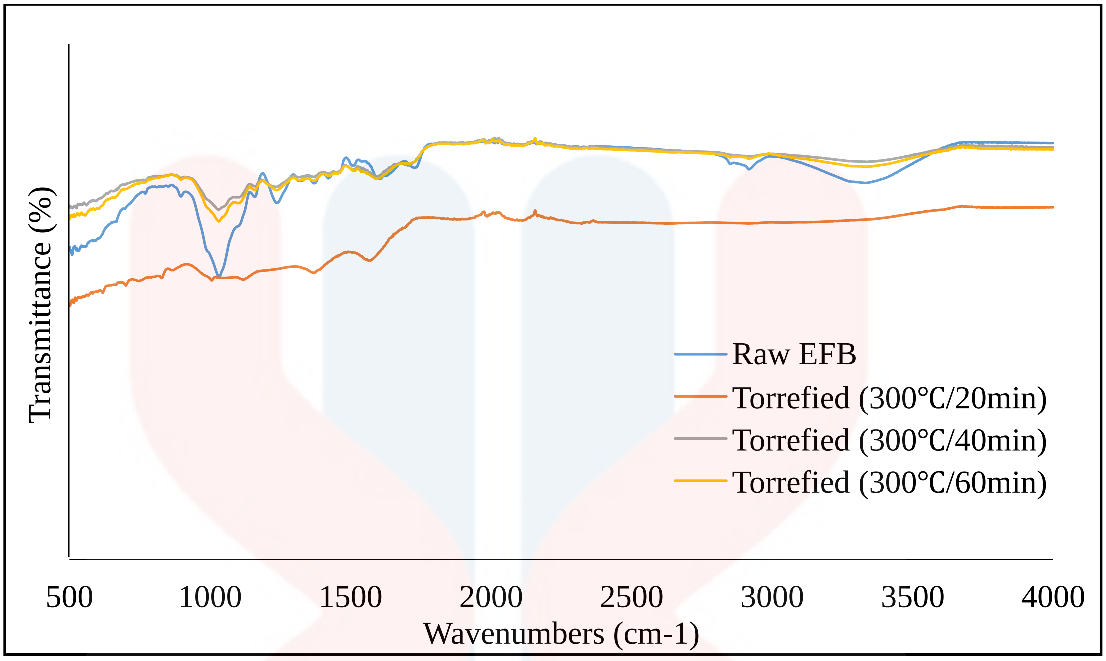

Figure 4.14: Functional groups presence for 300℃

In this next figures were showing the shifting of functional groups upon the different in time of torrefaction process. The differences were not huge as in the first three figures. But, the suitable residence time torrefaction process could be decided based on Figure 4.12, Figure 4.13 and Figure 4.14 whereby both of Figures 4.12 and Figure 4.13 showing that during 40 minutes of residence time for respective 200 ℃ and 250 ℃, the difference in the absorption bands were clearly showed whereas in Figure 4.14 indicates 20 minutes as the time showing most changes of functional groups occurred for 300 ℃.

This might be because of hemicellulose and cellulose degraded faster on lower temperature despite the time. In the case of Figure 4.14, the temperature was already high, thus it was essential for the hollocellulose to be decomposed.

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CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Before jumping to the conclusion of choosing the best power, after taking consideration not only in terms of mass remain of EFB, liquid and gas loss, the thermal expansion need to be reconsidered. Therefore, the best power generated is 385 W by which the mass retain was relatively reasonable with great condensation of liquid and gas as well as less thermal expansion occurred. Nevertheless, the magnetron profile showed 385 W has the most frequent and stable electrons released.

Furthermore, both morphology and bonding behaviour of the torrefied EFB are influenced the degradation of lignocellulose; hemicellulose, cellulose and lignin. All structures of torrefied EFB showed that the hemicellulose degraded at lower temperature of torrefaction as well as its spectrum were on between 1050 and 1734 cm^{-1} involving the stretching of C-O and C=O bonds. Meanwhile, cellulose decomposed at a medium temperature of torrefaction involving also stretching of C-O and CH bonds as well as shearing and benching of CH_2 bonds on the spectrums of 1050 cm^{-1} and 1437 cm^{-1}, respectively. The decomposition of lignin is hard to be determined based on the torrefied EFB’s structures, fortunately the FTIR spectra proved it occurred at the band gap of 890 cm^{-1}, 1256 cm^{-1} and 1437 cm^{-1}.

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As the conclusion, the best conditions of torrefied EFB is on 300 ℃ for 60 minutes.

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5.2 Recommendations

There are several recommendations need to be considered and improved in the future study, which are: -

5.2.1 There are few study have been done involving the bonding analysis of empty fruit bunch (EFB) as biomass resources although, the quantity of this biomass is abundance in Malaysia.

5.2.2 During torrefaction process is conducted, the alumina crucible is cracked. Therefore, it is essential to look up for new material that able to withstand high temperature and thermal expansion to prevent thermal shock.

5.2.3 Most of studied only discussing about the time and temperature of torrefaction and less studied focusing on the sample's size whether it is affecting the mass output of torrefied biomass. So, a study of sample's size of biomass either affecting or not the torrefaction process need to be determine.

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