PRODUCTION OF LIGNOCELLULOSES FROM PALM OIL EMPTY FRUIT BUNCH

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A thesis submitted in fulfillment of the requirements for the award of the degree of Bachelor of Chemical Engineering

Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang

APRIL 2010

UNIVERSITI MALAYSIA PAHANG

PSZ 19:16
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SESI PENGAJIAN : 2009/2010

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DEDICATION

Special Dedication to my beloved husband,

my family members, My supervisor, my friends,

my fellow colleague and all faculty members

For all your care, support and believe in me.

ACKNOWLEDGEMENT

I am so thankful to Allah S.W.T for giving me patient and highly spirit completing this research project successfully done. With the mercifulness from Allah therefore I can produces a lot of information in this research project.

To my beloved husband, Shamsul Nizam Bin Jaffar, I am so grateful to have you in my life and giving me full of support in order to finish this project. A lots of thank to my parent for always standing behind me.

I am indebted to my supervisor, Dr Ir Said Nurdin, lecturer from the Faculty of Chemical and Natural Resources Engineering for her advice, insightful comments and support. Thank for your guide and without your guide this research will not be complete and well organized. And not forgetting for my panels, Dr Julios Gimbun and Mdm Junhaslinda Bin Hj Sharifudin for your brilliant ideas that you gave to me, thank you.

My sincere appreciation also extends to all my fellow colleagues and others who have provided assistance at various occasions. Their views and tips are useful indeed. Thank you for the time sacrificed to accompany me. And last but not least, I am grateful to all my family members.

ABSTRACT

Lignocelluloses were a significant new value added product especially for composite industries. The method of alkali and acid treatment was used in order to produces lignocelluloses from palm oil empty fruit bunch. This research obtained to identify the effect of temperature, size of palm oil empty fruit bunch and concentration of acid on production of lignocelluloses yield. The sample of palm oil empty fruit bunch was treated by sodium hydroxide and hydrochloric acid. Sodium hydroxide was used in order to breakdown all the lignin bonding in sample of palm oil empty fruit bunch. Hydrochloric acid was used as a bleaching treatment for lignocelluloses produces. The result shows the smallest size of palm oil empty fruit bunch was resulted high yield of lignocelluloses produces that was 80%. The lowest concentration was decrease the yield of lignocelluloses production that was 40%. Characteristic of lignocelluloses was found using SEM, TGA and FTIR. The morphological structure of lignocelluloses has been analyzing using SEM. The biggest sample of palm oil empty fruit bunch was resulted no breakage and better surface lignocelluloses produces. More stable structure was degrade at higher temperature that was resulted from TGA analyze. There are three functional groups consist in this lignocelluloses that were $N(C_2H_5)_4$, C-C=C and O-H.

ABSTRAK

Lignocelluloses merupakan salah satu hasil penemuan baru yang mempunyai nilai tinggi terutamanya didalam industri composite. Penghasilan lignocelluloses daripada tandan kosong kelapa sawit adalah dengan menggunakan kaedah rawtan alkali dah acid. Kajian ini dapat menemukan kesan terhadap suhu, sais tandan kosong kelapa sawit and kepekatan acid keatas kadar penghasilan lignocelluloses. Bahan kajian tandan kosong kelapa sawit telah dirawat menggunakan sodium hydroxide dan acid hydrochloric. Sodium hydroxide telah digunakan untuk tujuan memecahkan ikatan gentian lignin yang terkandung didalam tandan kosong kelapa sawit. Acid hydrochloric digunakan sebagai rawatan peluntur bagi membersihkan cairan hitam yang terhasil daripada tindakbalas sodium hydroxide didalam menghasilkan lignocelluloses. Hasil kajian menunjukkan penghsilan lignocelluloses adalah paling tinggi 80% apabila sais terkecil digunakan. Kepekatan acid yang paling rendah akan menyebabkan kemerosotan kadar penghasilan lignocelluloses iaitu 40%. Sturuktur permukaan lignocelluloses telah dikaji dengan menggunakan mesin SEM. Semakin besar sais tandan kelapa sawit kosong akan menghasilkan lignocelluloses yang mempunyai permukaan bagus dan tidak akan musnah. Semakin stable structure akan mamerlukan suhu yang tinggi untuk memecahkan ikatan antara molekul lignocelluloses, ini dapat dilihat dengan hasil analysis menggunakan mesin TGA. Lignocelluloses yang dihasilkan mengandungi tiga kumpulan berfingsi iaitu kumpulan alkine, amine dan alcohol.

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LIST OF SYMBOLS/ABBREVIATIONS

NAOH	- Sodium Hydroxide
HCl	- Acid Hydrochloric
SEM	- Scanning Electron Microscope
FTIR	- Fourier Transform Infrared
TGA	- Thermo Gravitation Analyzer
EFB	- Empty Fruit Bunch
wt%	- Weight Percent
^oC	- Degree Calcius
IR	- Infera Red
FPISB	- Felda Palm Industries Sdn Bhd
FKKSA	- Fakulti Kejuruteraan Kimia Sumber Asli
M	- Molar
μm	- Micrometer
POEFB	- Palm Oil Emty Fruit Bunch
pH	- Potential For Hydrogen Ion Concnetration
FKM	- Fakulti Kejuruteraan Mekanikal
Ca	- Concentration
gm	- Gram
N(C2H5)4	- Amine Group
C-C=C	- Alkenes Group
O-H	- Alkohol Group

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Lignocelluloses are the main structural component of wood plant and non wood plant such as palm oil empty fruit bunch. The chemical properties of the components of lignocellulosics make them a substrate of enormous biotechnological value (Howard et al. 2003). In Malaysia, the main sources of lignocelluloses come from palm oil empty fruit bunch as a palm oil residue. Nowadays, no more technology obtained to dispose this residue. Because of that, many palm oil mill use incineration process to dispose this residue. Effect of that, can contribute to the environmental pollution. Today, many researches was run in order to convert the palm oil empty fruit bunch as waste to become various value added product.

This study is to find the production of lignocelluloses palm oil empty fruit bunch. The study of production lignocelluloses from empty fruit bunch is one of process to recycle the palm oil mill residue. The chemical properties of the component of lignocelluloses make them highly potential to become various value added product including biofuels, chemicals, cheap energy sources for fermentation, improved animal feeds and human nutrients. Lignocellulytic enzymes also have significant potential applications in various industries including chemicals, fuel, food, brewery and wine, animal feed, textile and laundry, pulp and paper, and agriculture.

In Malaysia, the palm oil empty fruit bunch is the one of palm oil mill residue. The palm oil empty fruit bunch was produce in the large amount is more than 8 million metric ton per annum (Shawkataly and Rozman, 2004). Much of the lignocelluloses waste is often disposed of by incineration, which is not restricted to developing countries alone, but is considered a global phenomenon. Because of that, it can contribute to the environmental pollution. So one of the way for save our environment is recycle the palm oil empty fruit bunch to become various value added product such as plastic compound filler and lignocelluloses.

This study was focused on the production of lignocelluloses from palm oil empty fruit bunch. The method of alkali and acid treatment was use in order to produce lignocelluloses from empty fruit bunch. The chemical that use in the alkali treatment is sodium hydroxide (NAOH) and hydrochloric acid (HCl) as solvent. The parameters that measure in this experiment is concentration of acid, temperature and also the size of oil palm empty fruit bunch. After experiment was done we will see relationship between the parameters and production of lignocelluloses.

1.2 Problem Statement

Palm oil empty fruit bunch is the one of palm oil mill residues ‘waste’. Malaysia is one of the palm oil producers around the world. But the residue of the palm oil has still not been completely used. Palm oil empty fruit bunch was disposed by incineration process and it directly contributing to the environmental pollution.

Because of that, the purpose of this research is to convert the waste of palm oil empty fruit bunch into the value added product. The research title of my project is production of lignocelluloses from palm oil empty fruit bunch.

According to the previous research it was stated the physical properties of the palm oil empty fruit bunch make it highly potentials to become various value added product.

1.3 Research Goal/ Objectives

i. To produces lignocelluloses from palm oil empty fruit bunch.
ii. To obtained the effect of temperature, size of oil palm empty fruit bunch and concentration of acid on production of lignocelluloses.

1.4 Research Scope

i. The effect of size, temperature and Acid concentration in production of lignocelluloses.
ii. The optimum condition for production of lignocelluloses.
iii. Analyze the lignocelluloses produce by using SEM, FTIR and TGA at the optimum condition.

1.5 Research Advantage

i. Environmental friendly product will produce.
ii. Creating new composite from natural resources of palm oil empty fruit bunch.
iii. Recycle palm oil empty fruit bunch ‘waste’ as the value added product.
iv. Lignocelluloses from palm oil empty fruit bunch as natural composite will produce for industrial needed.

CHAPTER 2

LITERATURE REVIEW

2.1 Palm Oil Empty Fruit Bunch

The Palm oil empty fruit bunch comes from palm oil tree (Elais guineensis) and is ones of by-products generate from palm oil mill. Malaysia is the largest producers of palm oil around the world it about more than twelve million tones every year (Nafpour et al 2007). Effect of that, many residues was generated from this industries and it become abundant of biomass resources. Now days, many researcher was study the potential of palm oil empty fruit bunch as a new profitable product such as ethanol, composite and lignocelluloses. The production of lignocelluloses from palm oil empty fruit bunch is highly potential in Malaysia. It is because, the palm oil empty fruit bunch is one sources to produce lignocelluloses.

2.2 Lignocelluloses Structure

Lignocelluloses or natural fibers are subdivided based on their origins, coming from plants, animals or minerals. All plant fibers are composed of cellulose while animal fibers consist of proteins (hair, silk, and wool). Plant fibers include bast (or stem or soft sclerenchyma) fibers, leaf or hard fibers, seed, fruit, wood, cereal straw, and other grass fibers (John and Thomas, 2007). The main components of lignocelluloses are celluloses, hemicelluloses, lignin, pectin and waxes.

TABLE 1.1 Lignocelluloses Composition from Different Source of Lignocellulsic Materials

Sources	Main Components (Percentage by Dry Weight)				
	Cellulose (%)	Xylan (%)	Hemicellulose (%)	Lignin (%)	Ash (%)
Palm Oil EFB	50.4	-	21.9	10.0	0.5
Sorghum Straw	35.0	19.0	24.0	25.0	-
Sugar Cane Bagasse	38.9	20.6	-	23.9	-
Corn Cobs	31.7	-	34.7	20.3	-
Rice Straw	43.4	20.2	28.0	17.2	11.4

(Source: Najafpour et al 2007)

2.2.1 Cellulose

Cellulose is a natural polymer consisting of D-anhydroglucose ($C6H11O5$) repeating units joined by 1,4-b-D-glycosidic linkages at C1 and C4 position. The degree of polymerization (DP) is around 10,000. Each repeating unit contains three hydroxyl groups. These hydroxyl groups and their ability to hydrogen bond play a major role in directing the crystalline packing and also govern the physical properties of cellulose. Solid cellulose forms a microcrystalline structure with regions of high order i.e. crystalline regions and regions of low order i.e. amorphous regions. Cellulose is also formed of slender rod like crystalline microfibrils. The crystal nature (monoclinic sphenodic) of naturally occurring cellulose is known as cellulose I. Cellulose is resistant to strong alkali (17.5 wt%) but is easily hydrolyzed by acid to water-soluble sugars. Cellulose is relatively resistant to oxidizing agents (John and Thomas 2007).

2.2.2 Hemicellulose

Hemicellulose is not a form of cellulose and the name is a misnomer. They comprise a group of polysaccharides composed of a combination of 5- and 6-carbon ring sugars. Hemicellulose differs from cellulose in three aspects. Firstly, they contain several different sugar units whereas cellulose contains only 1,4–b-D-glucopyranose units. Secondly, they exhibit a considerable degree of chain branching containing pendant side groups giving rise to its non crystalline nature, whereas cellulose is a linear polymer. Thirdly, the degree of polymerization of native cellulose is 10–100 times higher than that of hemicellulose. The degree of polymerization (DP) of hemicellulose is around 50–300. Hemicelluloses form the supportive matrix for cellulose microfibrils. Hemicellulose is very hydrophilic, soluble in alkali and easily hydrolyzed in acids (John and Thomas 2007).

2.2.3 Lignin

Lignin is a complex hydrocarbon polymer with both aliphatic and aromatic constituents. They are totally insoluble in most solvents and cannot be broken down to monomeric units. Lignin is totally amorphous and hydrophobic in nature. It is the compound that gives rigidity to the plants. It is thought to be a complex, three-dimensional copolymer of aliphatic and aromatic constituents with very high molecular weight. Hydroxyl, methoxyl and carbonyl groups have been identified. Lignin has been found to contain five hydroxyl and five methoxyl groups per building unit. It is believed that the structural units of lignin molecule are derivatives of 4-hydroxy-3-methoxy phenylpropane. The main difficulty in lignin chemistry is that no method has been established by which it is possible to isolate lignin in its native state from the fibre. Lignin is considered to be a thermoplastic polymer exhibiting a glass transition temperature of around 90 ºC and melting temperature of around 170 ºC. It is not hydrolyzed by acids, but soluble in hot alkali, readily oxidized, and easily condensable with phenol (John and Thomas 2007).

2.2.4 Pectins and Waxes

Pectins are a collective name for heteropolysaccarides. They give plants flexibility. Waxes make up the last part of fibres and they consist of different types of alcohols.

2.3 Chemical Treatment of Lignocelluloses

Production of lignocelluloses from palm oil empty fruit bunch involved some chemical treatment. Chemical pretreatments have been widely reported as a potential technique for lignocelluloses production using acid and alkaline (Mosier et al., 2005; Rebecca et al., 2007 and Ray et al., 2001). The first one is alkali treatment, Sodium

Hydroxide was used in this step. After done the alkali treatment, the sample will go for the acid treatment. In this treatment Hydrochloric acid was used. There are several sources in order to done the chemical treatment for palm oil empty fruit bunch.

2.3.1 Alkali Treatment

Alkali treatment of cellulosic fibers, also called mercerization, is the usual method to produce high quality fibers (Ray et al. 2001). Alkali treatment improves the fiber-matrix adhesion due to the removal of natural and artificial impurities. Moreover, alkali treatment leads to fibrillation which causes the breaking down of the 12 composite fiber bundles into smaller fibers. In other words, alkali treatment reduces fiber diameter and thereby increases the aspect ratio. Therefore, the development of a rough surface topography and enhancement in aspect ratio offer better fiber-matrix interface adhesion and an increase in mechanical properties. Alkali treatment increases surface roughness resulting in better mechanical interlocking and the amount of cellulose exposed on the fiber surface. This increases the number of possible reaction sites and allows better fiber wetting.

The following reaction takes place as a result of alkali treatment:

Fiber-OH + NaOH — Fiber-O-Na+ + H2O

Alkali treatment influenced the chemical composition of the flax fibers, degree of polymerization and molecular orientation of the cellulose crystallites due to cementing substances like lignin and hemicelluloses which were removed during the mercerization process. Alkali treatment had a lasting effect on the mechanical behavior of flax fibers, especially on fiber strength and stiffness. Several other studies were conducted on alkali treatment. They reported that mercerization led to the increase in the amount of amorphous cellulose at the expense of crystalline cellulose and the removal of hydrogen bonding in the network structure.

The mechanism of NaOH pretreatment is postulated to be by saponification of intermolecular ester bonds crosslinking xylan hemicellulose and other polymeric materials, such as lignin or other hemicelluloses that was study by mission et al (2009).

2.4 Characterization of Lignocelluloses

There are several methods for characterize the lignocelluloses which are using Scanning Electron Microscope (SEM), Fourier Transform Infrared (FTIR) and Thermo Gravitation Analyzer (TGA).

2.4.1 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) is a microscope that uses electrons instead of light to form an image. Since their development in the early 1950's, scanning electron microscopes have developed new areas of study in the medical and physical science communities. The SEM has allowed researchers to examine a much bigger variety of specimens.

The scanning electron microscope has many advantages over traditional microscopes. The SEM has a large depth of field, which allows more of a specimen to be in focus at one time. The SEM also has much higher resolution, so closely spaced specimens can be magnified at much higher levels. Because the SEM uses electromagnets rather than lenses, the researcher has much more control in the degree of magnification. All of these advantages, as well as the actual strikingly clear images, make the scanning electron microscope one of the most useful instruments in research today.

The SEM is an instrument that produces a largely magnified image by using electrons instead of light to form an image. A beam of electrons is produced at the top of the microscope by an electron gun. The electron beam follows a vertical path through the microscope, which is held within a vacuum. The beam travels through electromagnetic fields and lenses, which focus the beam down toward the sample. Once the beam hits the sample, electrons and X-rays are ejected from the sample.

Detectors collect these X-rays, backscattered electrons, and secondary electrons and convert them into a signal that is sent to a screen similar to a television screen. This produces the final image.

2.4.2 Fourier Transform Infrared (FTIR)

FTIR (Fourier Transform Infrared) Spectroscopy, or simply FTIR Analysis, is a failure analysis technique that provides information about the chemical bonding or molecular structure of materials, whether organic or inorganic. It is used in failure analysis to identify unknown materials present in a specimen, and is usually conducted to complement EDX analysis.

The technique works on the fact that bonds and groups of bonds vibrate at characteristic frequencies. A molecule that is exposed to infrared rays absorbs infrared energy at frequencies which are characteristic to that molecule. During FTIR analysis, a spot on the specimen is subjected to a modulated IR beam. The specimen's transmittance and reflectance of the infrared rays at different frequencies is translated into an IR absorption plot consisting of reverse peaks. The resulting FTIR spectral pattern is then analyzed and matched with known signatures of identified materials in the FTIR library.

Unlike SEM inspection or EDX analysis, FTIR spectroscopy does not require a vacuum, since neither oxygen nor nitrogen absorb infrared rays. FTIR analysis can be applied to minute quantities of materials, whether solid, liquid, or gaseous. When the library of FTIR spectral patterns does not provide an acceptable match, individual peaks in the FTIR plot may be used to yield partial information about the specimen.

Single fibers or particles are sufficient enough for material identification through FTIR analysis. Organic contaminants in solvents may also be analyzed by first separating the mixture into its components by gas chromatography, and then analyzing each component by FTIR.

2.4.4 Thermo Gravitation Analyzer (TGA)

Thermal methods investigate changes that occur upon heating a sample. Thermo gravimetric measures changes in the mass of a sample that occur when it is heated. These changes relate to the reactions during decomposition, the loss of volatile material and the reactions with the surrounding atmosphere. Thermo gravimetric analysis (TGA) continuously measures a weight of samples as a function of temperature and time. The sample is place in a small pan or crucible connected to the microbalance and heated in a control manner and or held isothermally for a specified time. The atmosphere around the sample may consist of an inert gas, such as nitrogen, or reactive gas, such as air and oxygen. The heated program may started in an inert atmosphere and then be switched to air at a certain point to complete the analysis. Weight changes observed at specific temperature correlates to vitalization of sample components, decompositions, oxidation or reduction reactions or other reactions or changes. Temperature and mass data are collected and processed by computer dedicated to the system.

The TGA data gives information about such properties as; thermal stability, moisture or solvent content, additive or filler content, oxidation and decomposition temperatures and rate. Thermal events such as melting, glass transition and other changes are not detected because there are no changes in the sample mass associated with this events. Any physical and chemical change involving mass may be studied.

CHAPTER 3

RAW MATERIAL AND EQUIPMENT

3.1 Raw Material and Equipment

The raw material in this research was palm oil empty Fruit bunch. The sample of palm oil empty fruit bunch was collected at FPISB Felda Chini 2 Sdn Bhd. The palm oil empty fruit bunch was got for free. It because, the palm oil mill produce a lot of palm oil empty fruit bunch as a residue ‘waste” and no value.

Figure 3.1: A lot of Palm oil empty fruit bunch produce from Palm Oil Mill.

Figure 3.2 : The Sampling Process.

3.1.1 Chemical

i. Sodium Hydroxide NaoH.

In this research the sodium hydroxide was used as a solvent in the process of alkali treatment. The function Sodium hydroxide is to break down the lignin bonding in the sample. Alkali treatment is the first treatment process to the lignocelluloses. The sample of palm oil empty fruit bunch was immersed in the solution of sodium hydroxide at constant concentration. The pretreatment of sample was carried out using 0.5 M NaOH . Sodium hydroxide was supplied by R and M Chemicals Malaysia to the FKKSA Laboratory.

ii. Hydrochloric Acid.

The sample was treated with hydrochloric acid after finish the pretreatment process. Hydrochloric Acid was used in the bleaching treatment and as a function to remove all the black liquor remained after alkali treatment. The concentration of hydrochloric acid is one of the parameter measured in this research. Concentration of acid was constant at 0.5 M for the parameter of temperature and sample size. In order to measured the parameter of concentration, the sample was run at the different concentration of hydrochloric acid which is 0.3 M, 0.4 M, 0.5 M, 0.6 M and 0.7 M. Hydrochloric acid was supplied by R and M Chemicals Malaysia to the FKKSA Laboratory.

3.1.2 Apparatus

There are list of the apparatus was used in order to done this research:

i. Volumetric flask 1000ml.
ii. Beaker 200 ml.
iii. Stainless steel tray.
iv. Glass rod.
v. Spatula.
vi. Thermometer.
vii. Sieve.
viii. Petri disk.
ix. Filter funnel.
x. Magnetic bar.

3.1.3 Equipment

There are list of equipment that was used in this experiment:

i. Tyler mesh.
ii. Balance.
iii. Stirring hot plate.
iv. Scanning Electron Microscope (SEM).
v. Fourier Transform Infrared Spectroscopy (FTIR).
vi. Thermo Gravitation Analyzer (TGA).

3.2 Methodology

There are several methods used to produces lignocelluloses from palm oil empty Fruit bunch. The overall methods can divided into four sections:

3.2.1 Preparation Sample of Palm Oil Empty Fruit Bunch

The sample will collect in plastic bags and stored in the cold room at 4ºC- 8ºC for one day. After that the sample was washed with water to make sure it free from dust and any contaminate particles. For the next step the sample was dried in an oven at temperature 103ºC until constant weight will obtained. The sample of palm oil empty fruit bunch will cut into small size. The sample will blended to get the small particle range of 45µm until 1000µm. This process was shown in the flow chart as bellow. (Najafpour et al. 2007)

Sample collected and stored in cold room 4-8°C.

Figure 3.2 The sampling process.

↓

The EFB washed to make sure dust free.

Figure 3.3 The cleaning process.

↓

Dried in an oven at 103°C until constant weight obtained.

Figure 3.4 The drying process.

↓

Cut EFB into small size.

↓

Blended sample to make the sample size of 200µm-1000µm

Sample has been sieve.

Particle size of POEFB

Figure 3.5 The process flow of sample preparation.

3.2.2 Treatment Process of Palm Oil Empty Fruit Bunch

Firstly, the process of pretreatment with sodium hydroxide at 0.5 M was done to the sample. The main reason of pretreatment process is to break down the lignin bonding between the lignocelluloses. In this step, the sample was immersed into the solution of sodium hydroxide for the two hour at temperature 30ºC until 70ºC. The stirring hot plate was used in order to maintain the temperature at this range.

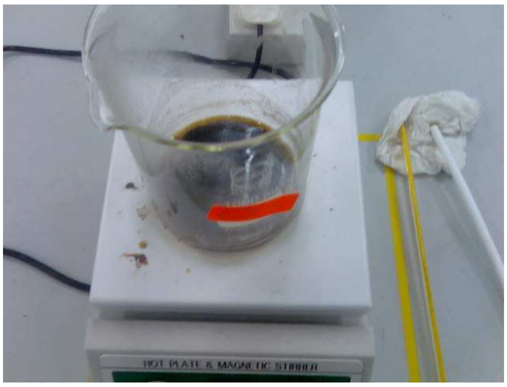

Figure 3.6 Alkaline treatment

Secondly, the sample was done to the process of treatment with Hydrochloric Acid. Hydrochloric Acid was used in order to neutralize the sample and also to remove the black liquor after alkaline treatment. The sample was immersed into the solvent of hydrochloric acid about 2 hour at temperature 80ºC ± 5ºC. After that, Sample was wash with distilled water until pH become neutral.

Figure 3.7 Acid treatment.

Finally, the sample was dried at room temperature to make sure all the moisture removed and lignocellulose was produces. The sample will go to the analyze process in order to determine the characteristic of lignocelluloses produced.

Figure 3.8 Lignocelluloses.

3.3 Yield of Lignocelluloses Determination

Results was obtained by measured the yield percent of lignocelluloses. In order to find the yield percent of lignocelluloses production, we should take the weight of sample before and after treatment was done. After that, the percent of lignocelluloses degradation was illustrated in equation (1) (Misson et al. 2009).

$$\text{Yield of lignocelluloses} = \frac{W1-W2}{W1}x100$$

Where W1 and W2 represent the weight of filler before and after treatment process.

3.4 Lignocelluloses Characterization

There are several analysis for characterize the lignocelluloses which are using Morphological Analysis by Scanning Electron Microscope (SEM), Fourier Transform Infrared (FTIR) and Thermo Gravitation Analyzer (TGA).

3.4.1 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) was used to analyzed the morphological structure of the treated lignocelluloses (Rozman et al. 2000). This process was done at the FKM Laboratory. The SEM analyze was done based on the selected sample only. According to the yield percent, the sample with optimum and minimum yield was analyzed by using SEM.

The small amount of sample of lignocelluloses was put on the aluminum plates. Double side tape was used in order to hold the sample. After that, the sample will put into the SEM analyze. Result of the morphological structure of sample will appears according to the sample condition.

Based on the sample of lignocelluloses the result was appears within 20 minute. The result of the SEM analyze for lignocelluloses was clear, it is because the sample totally dried.

Figure 3.9 Scanning Electron Microscope (SEM)

3.4.2 Fourier Transform Infrared (FTIR)

Fourier Transform Infrared (FTIR) was used to analyze the functional group of the sample (Rozman et al. 2002). Before start the analysis of sample, we must to make sure there are no remaining sample on the aluminum plate and washed the plate by using ethanol.

The small amount of sample was put on the plate. After that, the plate was places into the FTIR to analyze. The start bottom will was press when the item ready was appears on the computer screen. In the few minutes, the result form of graph was appeared on the screen.

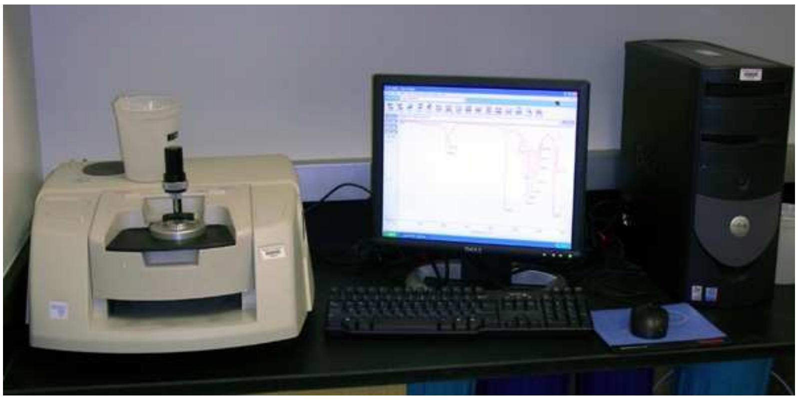

Figure 3.10 Fourier Transform Infrared Spectroscopy (FTIR)

3.3.4 Thermo Gravitation Analyzer (TGA)

Thermo Gravitation Analyzer (TGA) was used to analyze the degradation characteristic of the sample of lignocelluloses after treatment process was done. The small amount of sample of lignocelluloses was put into the pin. After that, the higher temperature for the experiment was setup to 600ºC.

Figure 3.11 Thermo Gravitation Analyzer (TGA)

CHAPTER 4

RESULTS AND DISCUSSION

4.1 The Effect of Size on Lignocelluloses Yield

In this experiment, we are obtained the effect of size into the production of lignocelluloses. The sample size of palm oil empty fruit bunch was variable which is 45, 160, 400, 500 and 800µm. The sieve shaker was used as equipment to find the size distribution for the sample. There are the experiment conditions Ca, NAOH = 0.5M, Ca, HCL = 0.5M, Temperature = 45ºC and Weight of sample before experiment = 2gm.

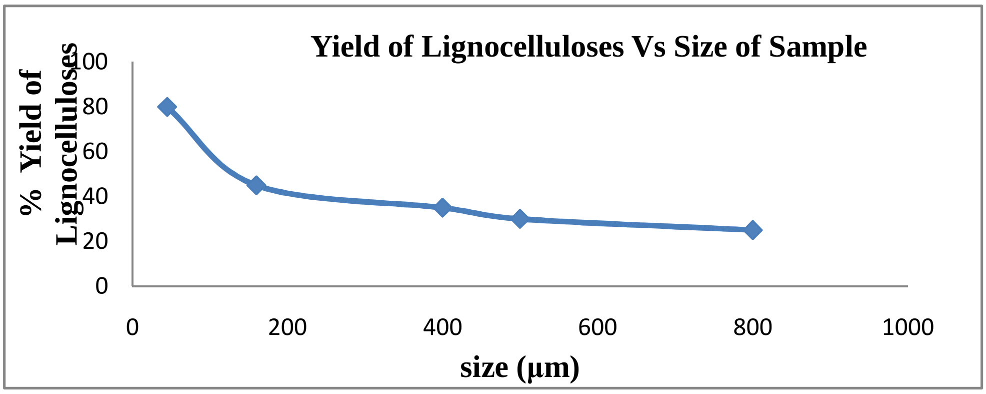

Figure 4.1 The Effect of Size on Lignocelluloses Yield

From the data table, the result of experiment was illustrated into the graph. According to graph the optimum size of sample was at 45µm. At the size of sample 45µm the production of lignocelluloses was high. It was cause by the more surface area of the sample will exposes to the reaction.

The reaction of alkaline and acid to the sample will break down the structure of fiber. Because of that, the more surface area provided for the reaction will produce the best result to the production of lignocelluloses. Rozman et al. (2001) was investigated, on his conclusion the surface area of sample was one of important factor effect to reaction.

4.2 The Effect of Concentration on Lignocelluloses Yield

The second parameter that measured in this study is concentration. In this experiment, we want to obtain the effect of concentration of acid hydrochloric into the production of lignocelluloses. Various concentrations were used in this experiment which is 0.3, 0.4, 0.5, 0.6 and 0.7. These are the experiment conditions Ca, NAOH = 0.5M, Size of sample = 160µm, Temperature = 45ºC, Weight of sample before and experiment = 2gm

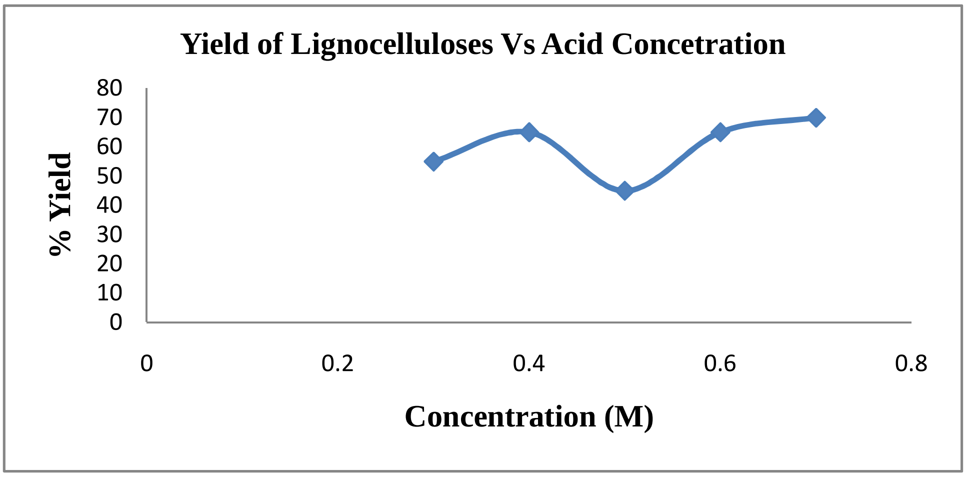

Figure 4.2 The Effect of Concentration on Lignocelluloses Yield

This result was showed the experiment effect of concentration. The high of acid concentration can improved the reaction rate it was done by Najafpour et al. (2007). It was proved that, the concentration is one more important factor affected to the strength of EFB-PU composites.

Increasing acid concentration significantly will improve the lignin degradation ability when temperatures are constant that was studies by Mission et al (2009). According to the graph we can find the optimum acid concentration for this experiment at 0.7M. The graph also had shown when the acid concentration increase the yield percent will increase. At the concentration of acid 0.5M the result of yield will decrease drastically. Increasing the concentration of reactant will increase the frequency of collisions between the two reactants and cause the reaction occurred faster.

4.3 The effect of Temperature on Lignocelluloses Yield

The third parameter that measured in this study is about the temperate condition of the experiments. The effect of temperature on acid hydrolysis was done by Najafpour et al. (2007). It was finding, the possibility of lignin component to decomposed at temperature greater than 55ºC.

This experiment was run with the temperature range 30ºC until 70ºC. These are the experiment conditions Ca, NAOH = 0.5M, Ca, HCL = 0.5M

Size of sample = 160µm, Weight of sample before experiment = 2gm

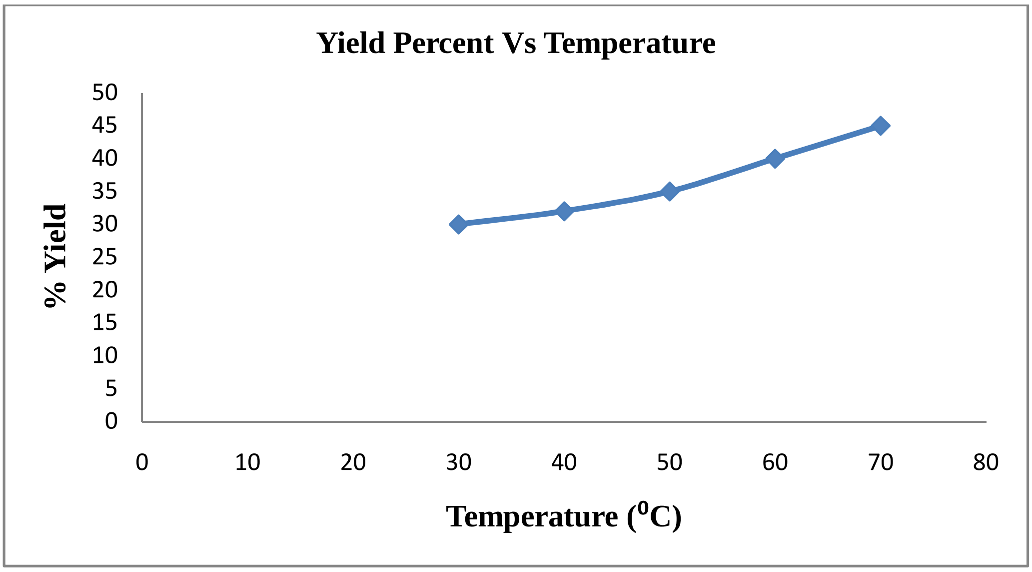

Figure 4.3 The effect of Temperature on Lignocelluloses Yield

This graph was shown the experiment of effect of temperature into the production of lignocelluloses. According to the graph we find that, when the temperature condition of the experiment increases the yield percent also increase. So, optimum temperature for the production of lignocelluloses was at 70ºC.

The increasing of lignocelluloses production was proportional with to the temperature that was proved by (Najafpour et al 2007). It was shown temperature one of the factor to improved the rate of reaction. As a temperature increase the molecules of acid get more kinetic energy, as a result the concentration occur at a greater rate and effectively.

4.4 Optimum Size of POEFB Sample

After all the three experiment of parameter size of sample, concentration and temperate was done. We were found the optimum condition in production of lignocelluloses. The optimum condition was size of sample at 45µm, concentration was 0.7M and temperature at 70ºC.

The experiment was repeated by using the optimum size of sample which is 45µm. The two parameters that were done on 45µm of sample which is effect of temperature and concentration.

4.4.1 The Temperature Effect on 45µm of POEFB

In this experiment, the optimum sample size 45µm was used to obtain the effect of temperature. Based on the optimum size of sample it may give the best and high yield percent result.

The all optimum condition was used in this experiment. These are the experiment conditions Ca, NAOH = 0.5M, Ca, HCL = 0.7M and Weight of sample before experiment = 2gm.

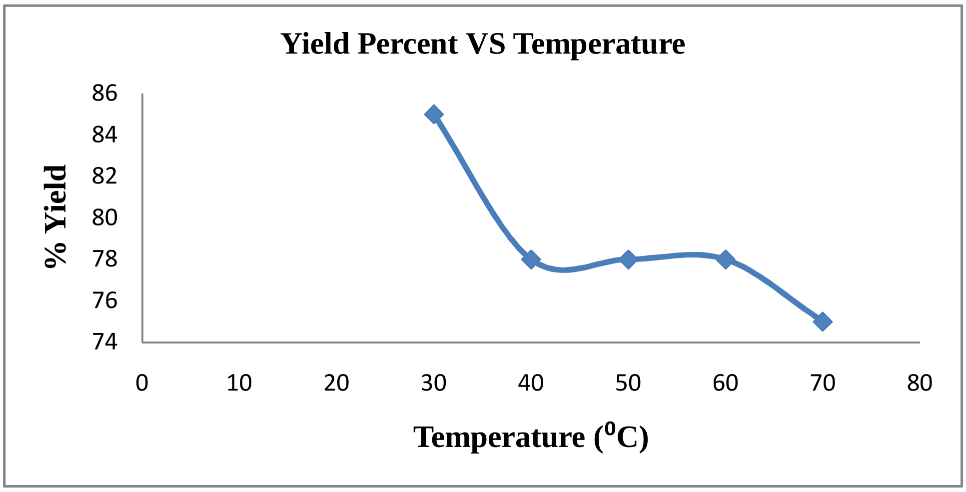

Figure 4.4 The Temperature Effect on 45µm of POEFB.

This graph was shown the result for optimum size 45µm with all the experiment condition at optimum. From the graph we can find the high yield percent was at temperature 30ºC. The lowest yield percent produced was at temperature at 70ºC. According to the graph it also had shown the decreasing of yield percent when the temperature was increase.

4.4.2 The Concentration Effect on 45µm of POEFB

The repeated experiment also was done on the effect of concentration. We want to find the effect of concentration to the optimum size of sample 45µm. In this experiment, we can identify how far the concentration will effect to the yield percent.

The all optimum condition was used in this experiment. These are the experiment conditions Ca, NAOH = 0.5M, Temperature = 80ºC, Size of sample = 160µm and Weight of sample before experiment = 2gm.

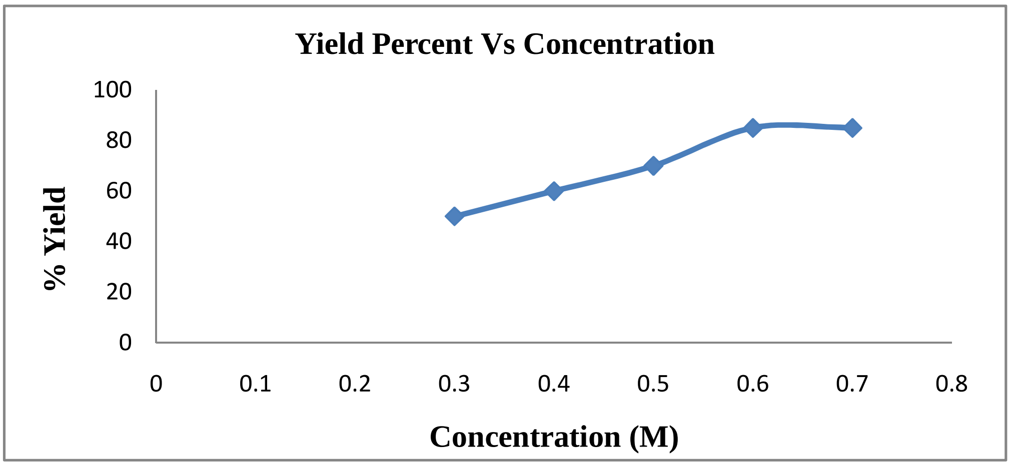

Figure 4.5 The Concentration on 45µm of POEFB.

According to the graph we can find the yield percent was increase when the concentration of acid increase. The yield percent start to constant at the concentration 0.6M.

4.5 Analysis of Lignocelluloses

There are several methods for characterize the lignocelluloses which are using Morphological Analysis by Scanning Electron Microscope (SEM), Fourier Transform Infrared (FTIR) and Thermo Gravitation Analyzer (TGA).

4.5.1 Thermo Gravimetric Analyzer (TGA)

Degradation characteristic of lignocelluloses was determined using TGA. That have been studied by Alvarez et al. (2004).From figure 4.5 and 4.6 we can found the degradation rate of lignocelluloses was increase when the temperature increases. Figure 4.5 was proved that more stable structure will degrade at higher temperature. It was compared with the figure 4.6 which is degradation totally occur at temperature 400^{o}C.

From figure 4.5 the first weight loss was started at temperature 41.23^{o}C and 99.17% weight percent. The lignocelluloses were totally degraded at temperature about 300^{o}C. The increasing in temperature has a moderately beneficial effect on the flexure strength of the lignocelluloses it was investigated by Alvarez et al 2004.

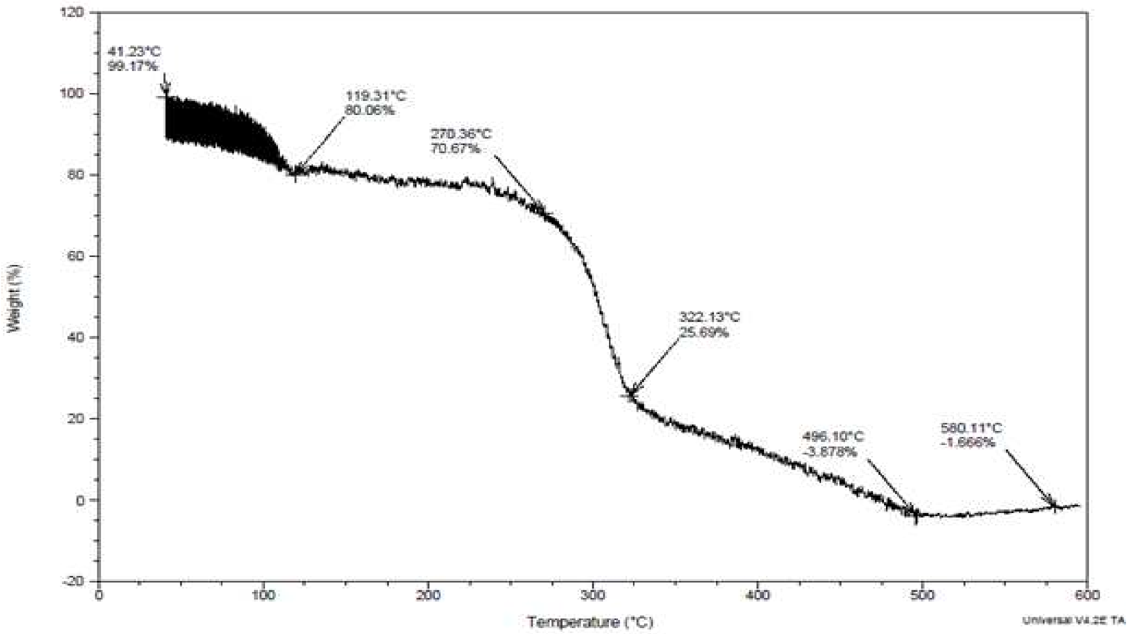

Figure 4.6 Result for the TGA analyze at sample size of 45μm.

The result for TGA analyzed for sample size 160μm was shown by the figure 4.6. According to the figure we can found the temperature for the lignocelluloses started to degrade was at 120ºC. At the maximum temperature of 500ºC there are still have the small weight percent which is 13%. It means the lignocelluloses not completely degrade at temperature 500ºC. The range of temperature should be expended in order to find the possible temperature to totally degrade for lignocelluloses 160μm. There we can see the largest particle size of sample need more energy to breakdown the bonding of lignocelluloses structure.

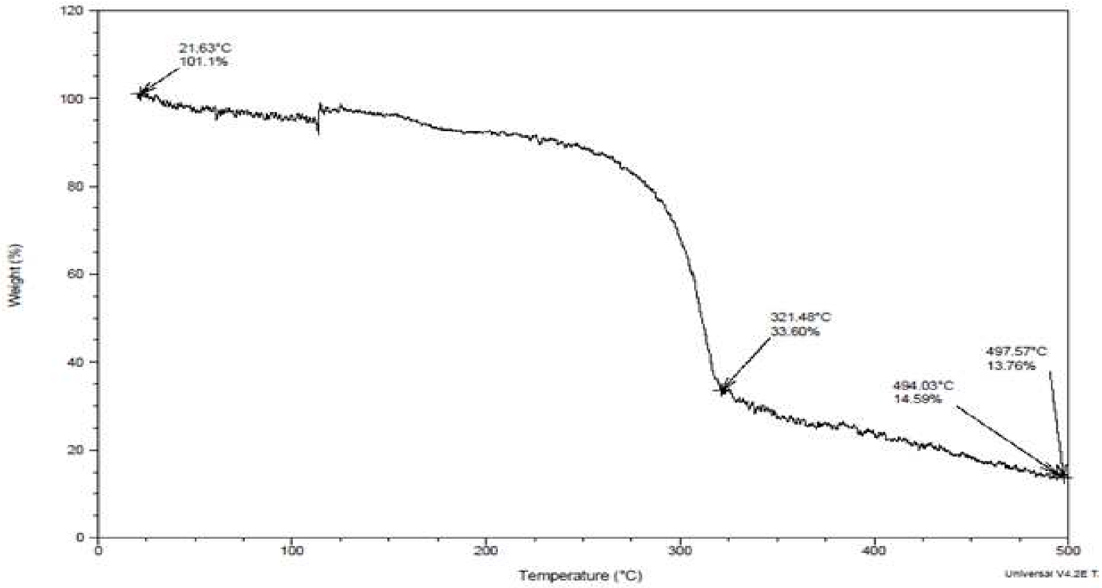

Figure 4.7 Result for the TGA analyze at sample size of 160μm.

4.5.2 Fourier Transform Infrared (FTIR)

FTIR was used in order to find the functional groups consist for the lignocelluloses. From figure 4.7 it was resulted the three functional groups consist in these lignocelluloses. It can been seen that the peak at approximately 431cm^{-1} shown the group of $N(C_2H_5)_4$. The lignocelluloses sample also showed an increase absorption in the C-C=C group at peak of 1645cm^{-1}. There are consist of O-H group, it was the same result that have done by Rozman et al (2002). The hydroxyl group was shown the lignocelluloses material would have swelled the cell wall and subsequently exposed lignocellulosic.

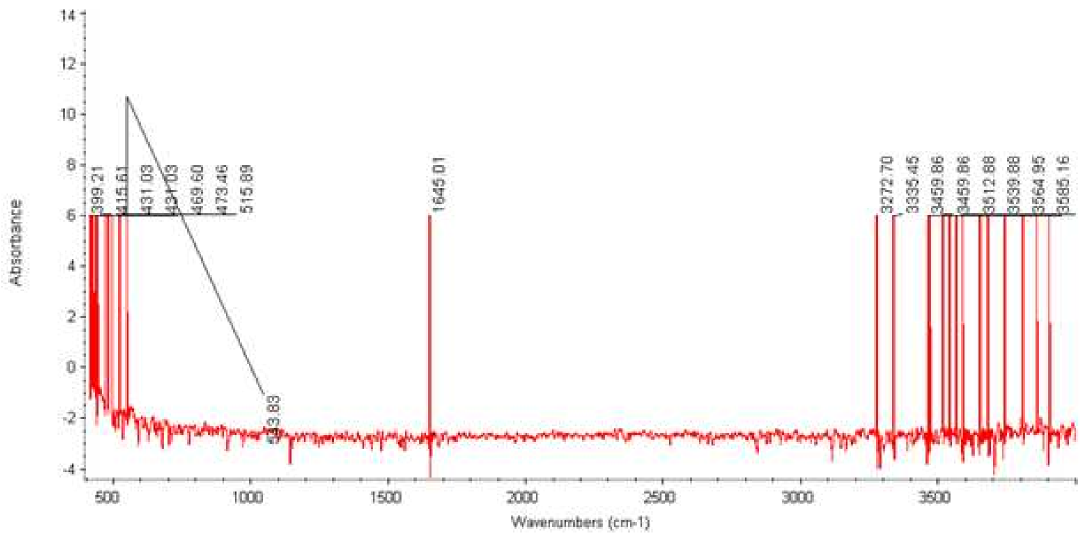

Figure 4.8 Result for the FTIR analyzes.

4.5.3 Scanning Electron Microscope (SEM)

Result for the lignocelluloses analyze using SEM was shown in the figure 4.8 and 4.9. The figure 4 was illustrated the better surface of lignocelluloses and no fiber breakage. It was compared with the figure 4.9, thus is very poor surface and resulted fiber breakage. This was clearly shown that effective lignocelluloses structure has occurred at sample size of 160µm.

The SEM analyze was used by Satyanarayana et al. (2009) to find the synthesis of polymer composites containing lignocellulosic fibers will often result in fibers physically dispersed in the polymeric matrix. They were compare the surface of lignocelluloses treated and no treated in the majority of cases, poor adhesion and consequently poor mechanical properties result. Hence, surface treatment of the fibers is essential.

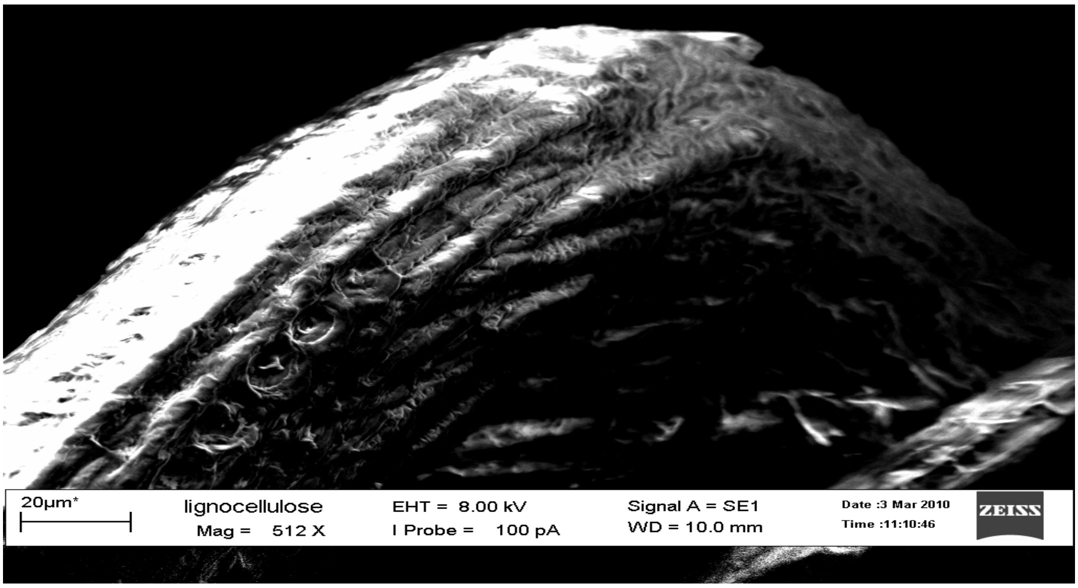

Figure 4.9 Result for the SEM analyze on 160µm sample size.

This result of SEM analyze was shown the morphological structure for the sample size of 45µm. According to the figure 4.9 we can find the structure of lignocelluloses was breakage and was resulted very poor surface of lignocelluloses. It may cause by decreasing the size of sample POEFB resulted the lowest strength of particle. Mission et al 2009 was studies the production of lignocelluloses using hydrogen peroxide and sodium hydroxide was concluded there are the effective chemical pretreatment to degrade lignin in EFB.

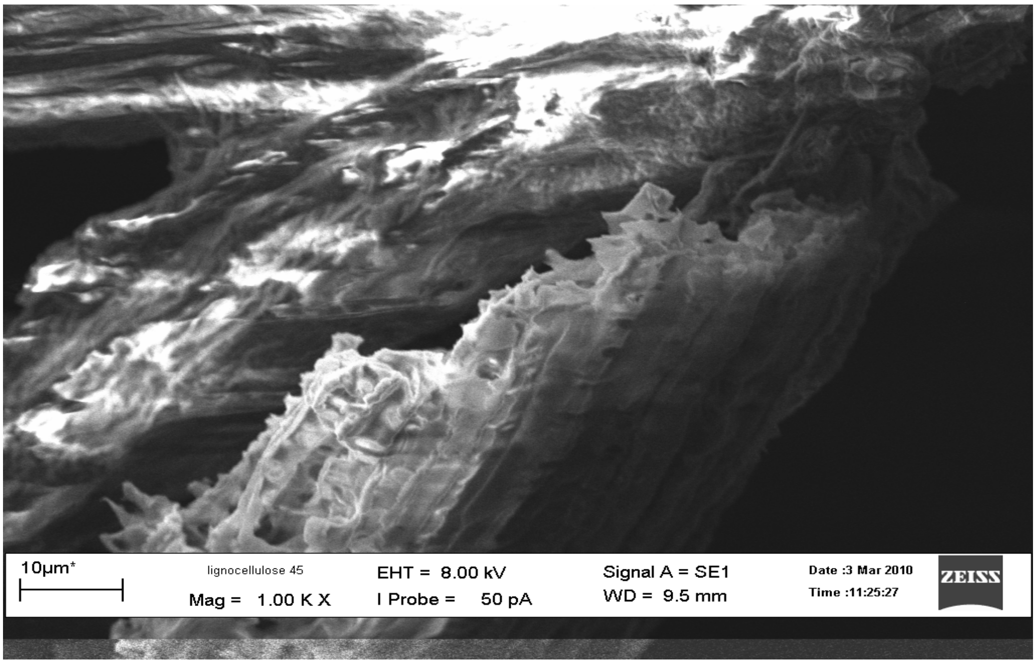

Figure 4.10 Result for the SEM analyze on 45 µm sample size.

CHAPTER 5

CONCLUSION & RECOMMENDATIONS

5.1 Conclusion

Lignocelluloses can be produced from palm oil empty fruit bunch using Alkali and Acid method effectively. The effectively of the method was proved by the experiment result of lignocelluloses production. Production of lignocelluloses from POEFB has feasibility for value added product. The result for effect of temperature, size and acid concentration was determined from this research. Highest temperature 70°C was improved the yield of lignocelluloses. Beside of that, the smallest size of POEFB was resulted high yield of lignocelluloses produces that was 80%. The lowest concentration was decrease the yield of lignocelluloses production that was 40%. Characteristic of lignocelluloses was found using SEM, TGA and FTIR. The morphological structure of lignocelluloses has been analyzing using SEM. The biggest sample of POEFB was resulted no breakage and better surface lignocelluloses produces. More stable structure was degrade at higher temperature that was resulted from TGA analyze. There are three functional groups consist in this lignocelluloses that were $N(C_2H_5)_4$, C-C=C and O-H. For the conclusion, from objective that was stated at the beginning of this a research has been archived successfully. All the parameter that involved in the objective was observe with clearly understanding.

5.2 Recommendations

There are several recommendations that have found according o this research in order to get better result for the future. The several of other process condition can be exploded such as pH and etc. The temperature more than 70^{o}C could be expended to find the precisely optimum effect of temperature on lignocelluloses production. It also has big potential to reuse of POEFB in Malaysia. There are strongly supported by the available of raw material POEFB as a waste.

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