PRODUCTION OF CELLULOSE FIBER FROM OIL PALM FROND USING STEAM EXPLOSION METHOD

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PRODUCTION OF CELLULOSE FIBER FROM OIL PALM FROND USING STEAM EXPLOSION METHOD

KAMARUL AZLAN ABD. HAMID

A Thesis submitted in fulfillment of the requirement for the award of the degree of
Bachelor of Chemical Engineering

Faculty of Chemical Engineering and Natural Resources
Universiti Malaysia Pahang

MAY 2008

“ I declare that this thesis is the result of my own research except as cited references. The thesis has not been accepted for any degree and is concurrently submitted in candidature of any degree.”

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Date : ……………………………..

DEDICATION

Dedicated to my beloved father, mother
Brothers and sisters…

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ABSTRACT

The world's largest producer and exporter of palm oil today is Malaysia, producing about 47% of the world's supply of palm oil. Indonesia is the second largest world producer of palm oil producing approximately 36% of world palm oil volume. Both nations are expanding their palm oil production capacity and the market continues to grow. It is estimate 26.2 million tonnes of oil palm fronds is treated as biomass in Malaysia. Palm frond consist huge amount of fiber while it can be transform because the fiber has cellulose in it. Majorly, fronds are taken from palm oil waste which is usually will be burn. Cellulose is the major constituent of paper, textiles and pharmaceutical products. This research is to prepare and produce the cellulose fiber from oil palm frond. Steam explosion pretreatment was conducted to enhance the accessibility of the oil palm frond lignocellulose. The combination of temperature and time known as the Log Ro in steam explosion process were used as the main parameter in this research. Several steps are involved in the preparation of cellulose such as steam explosion, alkaline treatment and bleaching. Temperature is use as parameter to achieve better product. Result shows that Log Ro giving contribution to the yield obtained. Steam exploded fiber giving yield in range 71 to 90%, alkaline extracted fiber : 70 – 90% yield and bleach fiber : 90 -95% yield from their raw materials.

ABSTRAK

Malaysia merupakan pengeksport terbesat kelapa sawit di seluruh dunia, Malaysia mengeluarkan kira – kira 47% daripada pengeluar minyak sawit dunia. Indonesia pula di tangga kedua dengan mengeluarkan kira – kira 36% minyak sawit. Kedua – dua negara mengembang pengeluaran minyak mereka dan permintaan pasaran semakin meningkat. Dianggarkan kira – kira 26.2 juta tan pelepah kelapa dibuang begitu sahaja di Malaysia. Pelepah kelapa mengandungi fiber yang dapat ditukarkan menjadi sellulosa kerana ia mengandunginya. Kebiasaannya, pelepah kelapa sawit ini di bakar begitu sahaja. Sellulosa merupakan bahan utama dalam pembuatan kertas, kain dan produk – produk farmasi. Kajian ini dijalankan untuk menghasilkan fiber sellulosa daripada pelepah kelapa sawit. Kaedah ledakan stim digunakan untuk meningkatkan keupayaan pelepah kelapa sawit menghasilkan lignosellulosa. Kombinasi penggunaan suhu dan masa tindakbalas yang dikenali Log Ro dalam ledakan stim digunakan sebagai parameter utama kajian. Beberapa kaedah digunakan dalam menghasilkan fiber sellulosa iaitu ledakan stim, pengekstrakan alkali dan proses pelunturan. Suhu digunakan sebagai parameter untuk menghasilkan produk yang lebih baik. Keputusan menunjukkan peratus hasil yang diperolehi dalam proses menghasil sellulosa fiber bergantung kepada Log Ro selepas ledakan stim. Hasil bagi ledakan stim adalah 71 – 90%, pengekstrakan alkali 70 – 90% dan pelunturan 90 – 95% daripada berat kering sample bahan mentah masing – masing.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Recycle lignocellulose waste of palm oil was a challenge to farmer and factory. Lignincellulose waste comprised 2 types which is farm waste and factory waste. This research is limit only for waste that are from palm oil estate or more specifically, palm oil frond.

Palms are one of the most well-known and extensively cultivated plant families. They have had an important role to humans throughout much of history. Many common products and foods are derived from palms, and palms are also widely used in landscaping for their exotic appearance making them one of the most economically important plants. In many historical cultures, palms were symbols for such ideas as victory, peace, and fertility. Today, palms remain a popular symbol for the tropics and vacations

The oil palms (Elaeis) comprise two species of the Arecaceae, or palm family. They are used in commercial agriculture in the production of palm oil. The African Oil Palm Elaeis guineensis is native to west Africa, occurring between Angola and Gambia, while the American Oil Palm Elaeis oleifera is native to tropical Central America and South America.

Mature trees are single-stemmed, and grow to 20 m tall. The leaves are pinnate, and reach between 3-5 m long. A young tree produces about 30 leaves a year. Established trees over 10 years produce about 20 leaves a year. The flowers are produced in dense clusters; each individual flower is small, with three sepals and three petals. The fruit takes five to six months to mature from pollination to maturity; it comprises an oily, fleshy outer layer (the pericarp), with a single seed (kernel), also rich in oil. Unlike other relatives, the oil palm does not produce offshoots; propagation is by sowing the seeds.

Oil palms are grown for their clusters of fruit, which can weigh 40-50 kg. Upon harvest, the drupe, pericarp and seeds are used for production of soap and edible vegetable oil; different grades of oil quality are obtained from the pericarp and the kernel, with the pericarp oil used mainly for cooking oil, and the kernel oil used in processed foods.

For each hectare of oil palm, which is harvested year-round, the annual production averages 10 tonnes of fruit, which yields 3,000 kg of pericarp oil, and 750 kg of seed kernels, which yield 250 kg of high quality palm kernel oil as well as 500 kg of kernel meal. The meal is used to feed livestock. Some varieties have even higher productivities which has led to their consideration for producing the vegetable oil needed for biodiesel.

The African oil palm was introduced to Sumatra and the Malaya area in the early 1900s; many of the largest plantations of oil palms are now in this area, with Malaysia growing over 20,000 square kilometers. Malaysia claims that in 1995 it was the world's largest producer with 51% of world production. Palm oil and its fractions are practical and attractive choice for importers and food manufacturers, especially in 3rd world countries due to its price competitiveness, year-round supply, diversity and versatility for edible and non-edible applications (Mohamad Husin *et al.*, 1986).

The oil palm industry in Malaysia started 80 years ago in a modest way. Today it is the largest in agricultural plantation sector, exceeding rubber plantation by more than double in area planted.

In terms of hectare, the total area under oil palm cultivation is over 2.65 million hectares, producing over 8 million tonnes of oil annually. The oil consists of only

10% of the total biomes produced in the plantation. The remainder consists of huge amount of lignocellulosic materials such as oil palm fronds, trunks and empty fruit bunches. The projection figures of these residues are as follows:

- 7.0 million tonnes of oil palm trunks
- 26.2 million tonnes of oil palm fronds
- 23% of Empty Fruit Bunch (EFB) per tonne of Fresh Fruit Bunch (FFB) processed in oil palm mill

Based on the above figures, Malaysia therefore has a great potential in turning its abundant supply of oil palm industry by-products into value-added products (www.aseansec.org/**7**011.htm).

Under the present scenario, Malaysia can no longer remain idle and complacent in its position as the top grower and supplier of palm oil. In view of the escalating challenge posed by the other oil producing countries, Malaysia has to change its objective of being a world producer of palm oil to amongst others a leader in converting biomass waste into value-added products. Malaysia has therefore to seriously resort in aggressive R&D to support its ambition.

Before embarked into identifying the parameters affecting the overall quality of the fiber, let turn to look into the current utilization of these fibers. Recent report shows that the monocarp fiber and shell are used as boiler to produce steam and to generate power. Whereas, empty fruit bunches are mainly incinerated to produce bunch ash to be distributed back to the field as fertilizer (www.freepatentsonline. com/ 4352341.htm).

The conventional method of burning these residues often create environmental problems in that it generates severe air pollution and is prohibited by the Environment Protection Act. In abiding by the regulations, these residues are becoming expensive to dispose. Nevertheless, looking on the brighter side of things, extensive research has provided us with an alternative way of optimizing the usage of oil palm residues fiber based into value-added product.

1.2 THE OBJECTIVES OF THE RESEARCH.

There are 2 objectives of the research which are to:-

i. Examine correlation between time and temperature in Reaction Ordinate (Ro).
ii. To produce cellulose from waste biomass cellulosic material, oil palm frond

1.3 THE SCOPES OF RESEARCH

There are 3 scopes of the research are to:

i. Use oil palm frond as alternative material to create high quality cellulose.
ii. The influence of temperature and time on the steam explosion method in producing cellulose fiber from cellulosic material (oil palm frond).
iii. The cellulose fiber shall be the main raw material input for pulp, paper and medical product.

1.4 PROBLEM STATEMENT

In the world today, biomass is became a massive problem to many country due to its causes to environment. Many studies have been made by several scientist but not all them can solve the problem of biomass. Utilization of waste material such as palm frond also being studied. The race for producing biodegradable products has increase tremendously. Different approaches have been attempted to use biomass as natural biopolymer for production of biodegradable plastics. It is almost 26.2 million tonnes of oil palm fronds in Malaysia according to MARDI. This biomass has been the main source cellulose fiber. This research is important because of Malaysia total net of importing cellulose is more than RM300 million per year and keep increasing.

So, this research will help palm oil sector to solve the biomass problem of oil palm frond and turn them into valuable product which is cellulose fiber.

CHAPTER 2

LITERATURE REVIEW

2.1 OIL PALM

2.1.1 The tree, history, distribution and habitat.

The oil palm is a tropical palm tree. There are two species of oil palm, the better known one is the one originating from Guinea, Africa and was first illustrated by Nicholaas Jacquin in 1763, hence its name, Elaeis guineensis Jacq.

Figure 2.1 The Fruit Bunch

The fruit is reddish about the size of a large plum and grows in large bunches. A bunch of fruits can weigh between 10 to 40 kilograms each. Each fruit contains a single seed (the palm kernel) surrounded by a soft oily pulp. Oil is extracted from both the pulp of the fruit (palm oil, an edible oil) and the kernel (palm kernel oil, used mainly for soap manufacture). For every 100 kilograms of fruit bunches, typically 22 kilograms of palm oil and 1.6 kilograms of palm kernel oil can be extracted (www.en.Wikipedia.org/wiki/ Oil_palm).

The high productivity of the oil palm at producing oil (as high as 7,250 liters per hectare per year) has made it the prime source of vegetable oil for many tropical countries. It is also likely to be used for producing the necessary vegetable oil for biodiesel, an example being a planned refinery Darwin, Australia which will import the palm oil from Indonesia and Malaysia.

The oil palm originated in West Africa but has since been planted successfully in tropical regions within 20 degrees of the equator. There is evidence of palm oil use in Ancient Egypt.

Figure 2.2 Plantation

In the Republic of the Congo, or Congo Brazzaville, precisely in the Northern part, not far from Ouesso, local people produce this oil by hand. They harvest the fruit, boil it to let the water part evaporate, then they press what its left in order to collect the reddish, orange colored oil.

The world's largest producer and exporter of palm oil today is Malaysia, producing about 47% of the world's supply of palm oil. Indonesia is the second largest world producer of palm oil producing approximately 36% of world palm oil volume. Both nations are expanding their palm oil production capacity and the market continues to grow.

Worldwide palm oil production during the 2005-2006 growing season was 39.8 million metric tons, of which 4.3 million tons was in the form of palm kernel oil. It is thus by far the most widely-produced tropical oil, and constitutes thirty-four percent of total edible oil production worldwide. Oil palm is one of the most abundant, unutilised waste biomass from plantation in South-Asia (www.en.wikipedia.org/wiki/Oil_palm**).**

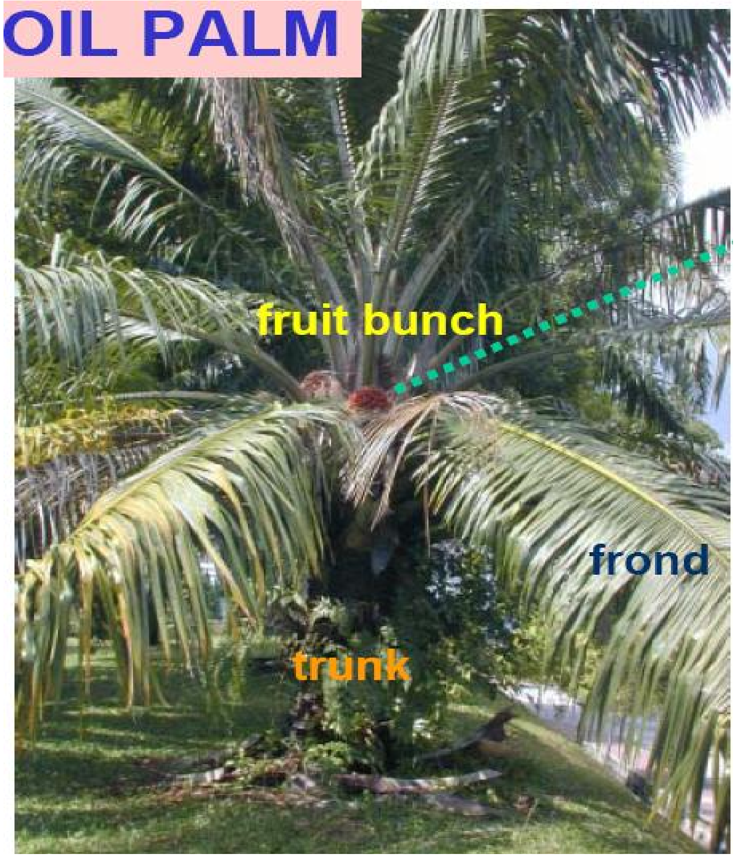


Figure 2.3 The tree

2.1.2 Palm Frond

A palm branch (or palm frond or palm stem), usually refers to the leaves of the Arecaceae (sometimes known by the names Palmae).

The palm branch was a symbol of triumph and victory in pre-Christian times. The Romans rewarded champions of the games and celebrated military successes with palm branches. Jews followed a similar tradition of carrying palm branches during festive times. Early Christians used the palm branch to symbolize the victory of the faithful over enemies of the soul, as in the Palm Sunday festival celebrating the triumphal entry of Jesus into Jerusalem.

In Judaism, the date palm represents peace and plenty, and is one of the Four Species (Lulav) used in the daily prayers on the feast of Sukkot. It is bound together with the hadass (myrtle), and aravah (willow) The palm may also symbolize the Tree of Life in Kabbalah. While in Islam, Muhammad is said to have built his home out of palm, and the palm symbolizes rest and hospitality in many cultures of the Middle East. The first muezzin climbed palm trees to call the faithful to prayer, from which the minaret developed.

The cell walls of OPF are composed mainly of cellulose, hemicelluloses and lignins. In addition to those three main components, several percent of ash and extractives are included. Among those components, cellulose and hemicelluloses are polysaccharides, which are easily decomposed and metabolized by wood-rot fungi. In the constituent analysis of wood, when lignin is selectively removed, what is obtained is holocellulose. Holocellulose can be thought of as the total of cellulose and all hemicelluloses (KOZUMAYosei *et al.*,).

Fiber property	Oil Palm Frond
Mean fiber length (mm)	
Arithmetic	0.59
Length weighted	1.13
Weight weighted	1.54
Coarness (mg/m)	0.098
Fiber dimensions(μm)	
Fiber diameter (D)	19.6
Lumen Width (L)	11.66
Cell wall thickness (T)	3.97

* Source : Law and Jiang (2001)

Table 2.1 Fiber Characteristics of Oil Palm Fronds

Lignin of oil palm frond was characterized by the presence of significant amounts of esterified p-hydroxybenzoic acid together with small amounts of etherified *p*-hydroxybenzoic acid. Vanillic and siringic acids were esterified or etherified to lignin. Some extents of these ester bonds and *beta*-O-4 interunit linkages of lignin were cleaved during steam explosion, in addition to great condensation of guaiacyl nuclei, as revealed by ^{1}H- and ^{13}C-NMR spectra of isolated lignins from the steam exploded pulps, of which yields were quite high, suggesting that lignin has been released from other wall polymers. Wall polysaccharides of oil palm frond are composed of cellulose and significantly high concentration of arabinoxylan, which produced great abundance 5-hydroxymethyl-furfural and furfural during steam explosion, respectively and even hot pressing at 125 ^{0}C to prepare binderless boards. It is suggested that released lignin and furfural derivatives generated during steam explosion contribute to self-binding of the steam exploded pulps. However, severe conditions of steam explosion caused great damages in lignin macromolecules, and gave poor quality of binderless board.

Cmponent	Oil Palm Frond
Lignin	15.2
Holocellulose	82.2
Alpha cellulose	47.6
Ash	0.7
Polysaccharide Composition	
Arabinose	1.5
Mannose	2.2
Galactose	0.9
Glucose	66.6
Xylose	28.9

* Source : Law and Jiang (2001)

Table 2.2 Chemical Compositions of Oil Palm Fronds

Bioconversion of lignocellulosic waste materials to chemicals and fuels are receiving interest as they are low cost, renewable and widespread in nature. Malaysia is well known for its potential in renewable resources such as oil palm waste, sugar cane bagasse and rice straw. At present Malaysia is the largest exporter of palm oil in the international market. In the process of extraction of palm oil from oil palm fruit, a lignocellulosic material oil palm empty fruit bunch (OPEFB) and palm frond is generated as a waste product. Approximately 15 million tons of OPEFB and frond biomass waste is generated annually throughout Malaysia by palm oil mills. In practice this biomass is burned in incineratorsby palm oil mills which not only creates environmental pollution problems in nearby localities but also it offers limited value to the industry. The OPEFB and frond biomass contains cellulose, hemicellulose and lignin. It is estimated that OPEFB biomass is comprisedof 24% xylan, a sugar polymer made of pentose sugar xylose. This xylose can be used as substrate for production of a wide variety of compounds by chemical and biochemical processes (Azemi *et al.*, 1996).

2.1.3 Conservation Status

Optimal plant density is 58 trees/acre with triangular patterns about 30 ft apart. During the first 3 years, little or no fruit is obtained and plantations are often intercropped with staple crops. Pruning and Training - none, old leaves are pruned off to facilitate access to the bunch at harvest. When palms reach heights of 20-30 ft, they become difficult to harvest, and are often injected with an herbicide to kill them or bulldozed down. New trees are planted among the dead and rotting trunks

While it is still unclear as to the status of the oil-palm plantations in central Borneo, the Friends of the Earth report lays out a set of recommendations for making better ecological and economic use of the rainforests of Kalimantan. If the government does intend to carry on with the proposed project, then it should first focus on increasing productivity in existing plantations, not on clearing new land for palm..

This can be done using improved seeds and doing a better job of adopting harvesting practices from other parts of the world, while encouraging replanting of abandoned and degraded plantations.

2.1.4 Harvesting of the frond

It is an agricultural plant, which originates from West Africa and cultivated in Malaysia for its oil producing fruit. Besides palm oil, the industry also generates massive amounts of lignocellulosic residues such as trunks, fronds and the empty fruit bunches (EFB), with an estimated amount of 30 million tonnes (MPOB, 2001). The suitability of this abundant, inexpensive and renewable raw material for papermaking resource has been explored using a variety of pulping methods. Majorly, fronds are taken from palm oil waste which is usually will be burn.

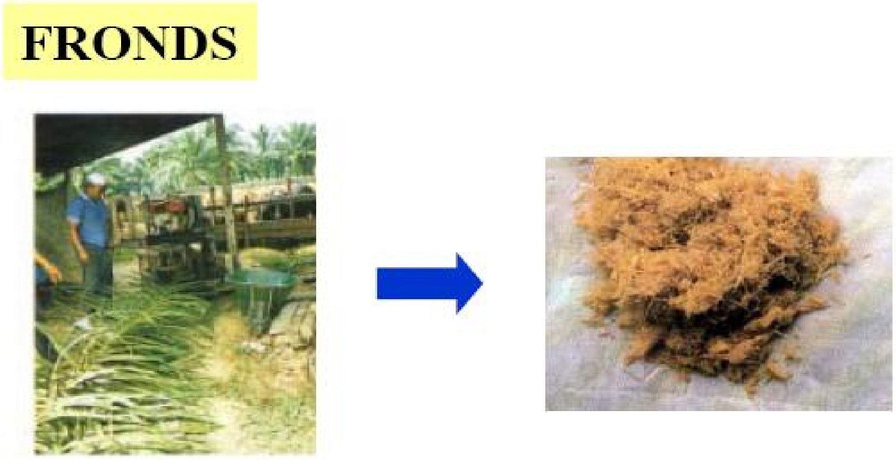


Figure 2.4 From fronds to fiber

2.2 FROND PROPERTIES, CHEMICAL COMPOSITION

Component	**Oil palm fronds**	**EFB**	**Hardwood**	**Softwood**
Lignin (%)	20.5	17.2	25.2	21-37
Holocellulose (%)	83.5	70	72.4	60-80
α-Cellulose (%)	49.8	42.7	44.7	31-60
Alcohol–benzene extractives (%)	1.4			
Ash (%)	0.7		< 1	< 1
Alkali solubes (%)		17.2	13.6	
Pentosans (%)		27.3	12.9	

* Source : Law and Jiang (2001)

Table 2.3 Chemical Compositions of Oil Palm

2.2.1 Cellulose

Cellulose is an organic compound with the formula (C6H10O5)n. It is a structural polysaccharide derived from beta-glucose. Cellulose is the primary structural component of green plants. The primary cell wall of green plants is made of cellulose; acetic acid bacteria are also known to synthesize cellulose, as well as many forms of algae, and the oomycetes. Cellulose was discovered and isolated in the mid-nineteenth century by the French chemist Anselme Payen with an estimated annual production of 1.5x109 Tonnes.

Some animals, particularly ruminants and termites, can digest cellulose with the help of symbiotic micro-organisms - see methanogen. Cellulose is not digestible by humans and is often referred to as 'dietary fiber' or 'roughage', acting as a hydrophilic bulking agent for feces.

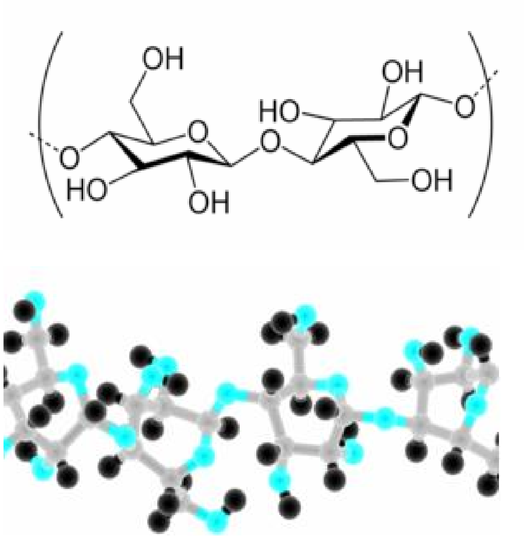


Figure 2.5 Molecule Structure of Cellulose

Cellulose is derived from (β-glucose), which condense through β(1→4)-glycosidic bonds. This linkage motif contrasts with that for α(1→4)-glycosidic bonds present in starch and other carbohydrates. Cellulose is a straight chain polymer: unlike starch, no coiling occurs, and the molecule adopts an extended rod-like conformation. In microfibrils, the multiple hydroxyl groups on the glucose residues hydrogen bond with each other, holding the chains firmly together and contributing to their high tensile strength. This strength is important in cell walls, where they are meshed into a carbohydrate matrix, conferring rigidity to plant cells.

In contrast to starch, cellulose is also much more crystalline. Whereas starch undergoes a crystalline to amorphous transition at 60 -70 °C in water (as in cooking), cellulose requires 320 °C and 25 MPa to become amorphous in water (www.en.wikipedia.org/wiki/Cellulose).

2.2.2 Commercial Product

Cellulose is the major constituent of paper and textiles made from cotton, linen, and other plant fibers. Cellulose can be converted into cellophane, clear rolling papers made from Viscose film, rayon, and more recently cellulose has been used to make Modal, a bio-based textile derived from beechpulp cellulose. Cellulose is used within the laboratory as the stationary phase for thin layer chromatography, and cotton linters, is used in the manufacture of nitrocellulose, historically used in smokeless gunpowder. Rayon is an important fiber made out of cellulose and has been used for textiles since the beginning of the 20th century.

Cellulose is also mainly use for pharmaceutical which is almost 70% of this type of product use cellulose fiber. As it growth year after year, cellulose is one of the businesses that have to be watch in coming years, this is because cellulose :

i. **Completely Renewable**

   Cellulose is the major part of all plants, including trees. As long as trees grow, cellulose fiber products are renewable.

ii. **A Natural Product**

   It grows in our pristine forests and is as pure as a natural product can get.

iii. **Not Petroleum Based**

   Unlike the plastics that have populated our lives over the past three decades. Cellulose fibers have never had a connection to fossil fuels.

iv. **Bio-Degradable**

The products are natural cellulose fibers and therefore return to the earth once is been used.

v. **Versatile**

As noted before, it can be used in making newspapers, writing paper, wrapping paper, toothbrush handles, ice cream thickeners, diapers for babies, pillow cases, gaskets, filters, food additives and coatings for pills, among other things. Future uses are constantly being explored.

vi. **Recyclable**

Many products on the market today are made from fiber based products which have already experienced one lifecycle as office paper, boxes, wrapping paper, etc. and are now returning to bring new value to the consumer.

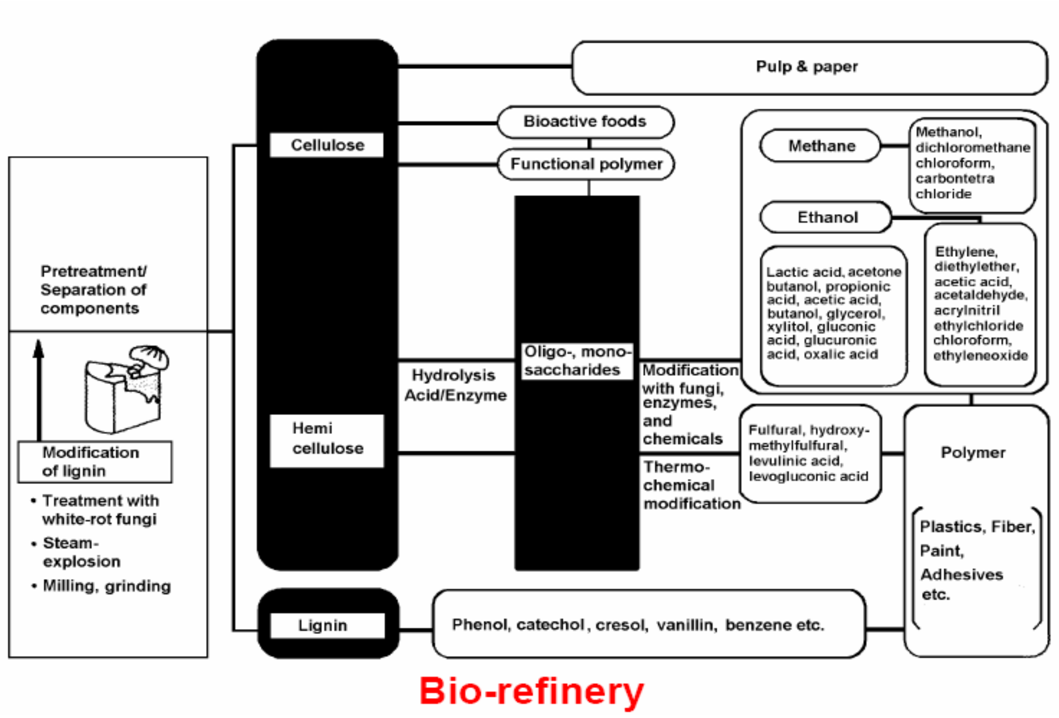


Figure 2.6 Sample Process of Derivatives Cellulose

2.2.3 Lignocellulosic

Lignocellulosic biomass is biomass composed primarily by lignin and cellulose. It consists principally of lignin, cellulose, hemicellulose and extractives. Pulpy biomass is about 45-50% cellulose, 20-25% hemicellulose and 20-25% lignin. Herbaceous materials have lower cellulose, lower lignin and higher hemicellulose contents. Examples of lignocellulosic biomass are all types of trees, grasses, agricultural residues such as corn stover, sugarcane bagasse, straw, etc.Research suggests that lignocellulosic ethanol could proivde an eco-friendly alternative to gasoline, cutting down on Greenhouse Gas emissions by as much as 85%.

2.2.4 Lignin

Lignin is a polymer of three closely-related phenylpropane moieties. Unlike cellulose and hemicellulose, lignin cannot be depolymerized by hydrolysis. Cleavage of the principal bonds in lignin require oxidation.

Lignin is formed by removal of water from sugars to create aromatic structures. These reactions are not reversible. There are many possible monomers of lignin, and the types and proportions depend on the source in nature. Some typical monomers are shown in the sketch :

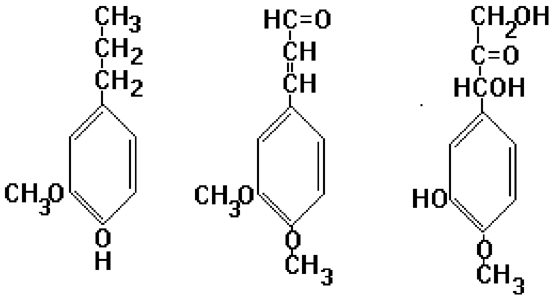


Figure 2.7 Monomer of Lignin

The OH groups (either the alcoholic OH's on the chains or the phenolic OH's on the aromatic rings) can react with each other or with the aldehyde or ketone groups. When an OH reacts with another, an ether linkage is formed. As we have seen, an OH reacts with an aldehyde to form a hemiacetal. The reactions of OH groups with ketones forms ketals. An early stage in the condensation of various monomers to form lignin is shown in the next sketch :

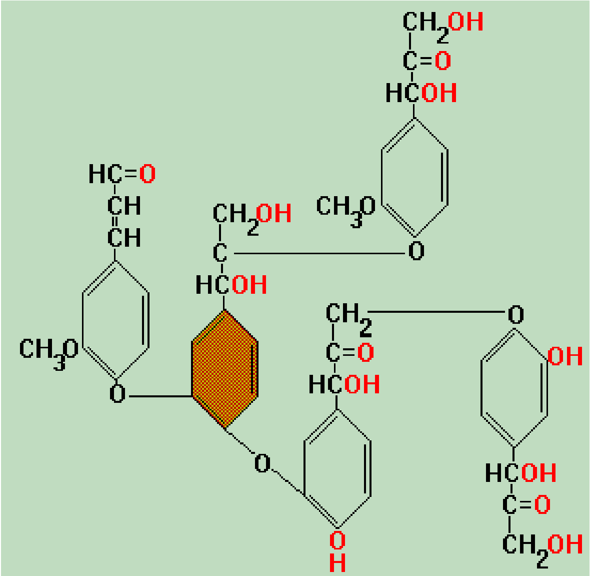


Figure 2.8 Condensations of Various Monomers

There are several groups shown in red that can react further. Some will simply extend the polymer while others would establish cross linking. The monomer that is shaded in orange has three of its functional groups linked to other monomers, so it is starting a branch or cross link. The large lignin molecules fill three dimensions and are heavily cross linked. Sometimes lignin is isolated as a brown powder, but more often it is a gummy mixture of lignins with a wide range of molecular weights.

Lignin resists attack by most microorganisms, and anaerobic processes tend not to attack the aromatic rings at all. Aerobic breakdown of lignin is slow and may take many days. Lignin is nature's cement along with hemicellulose to exploit the strength of cellulose while conferring flexibility.

2.2.5 Hemicellulose

Some monomers of hemicellulose are shown in the sketch :

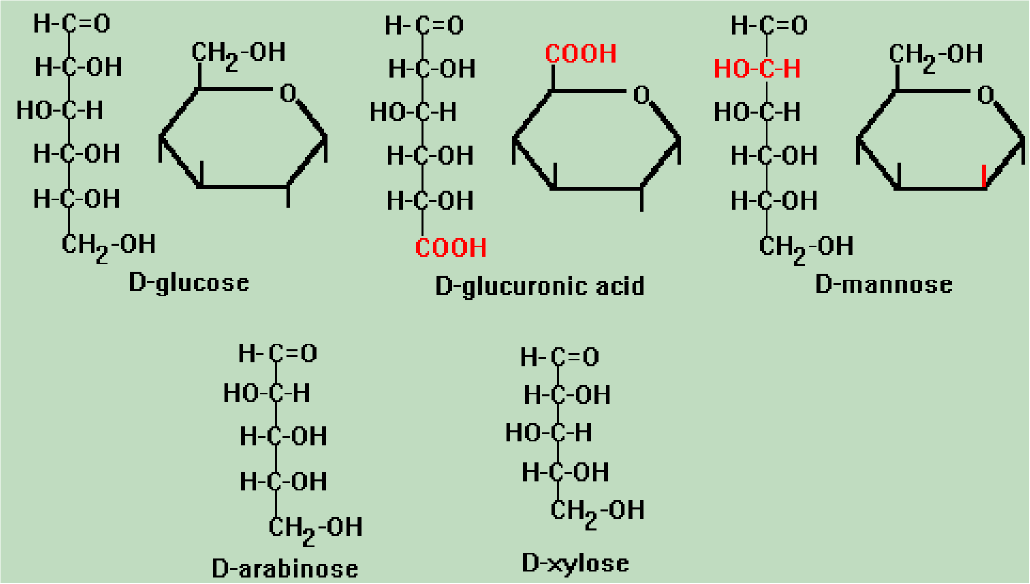


Figure 2.9 Monomers of Hemicellulose

Usually, all of the pentoses are present. There may even be small amounts of L- sugars. Note that there are hexoses as well as acids formed by oxidation of sugars. Mannose and mannuronic acid tend to be present, and there can be galactose and galacturonic acid.The groups that are different from glucose are shown in red on the six-carbon structures. The pentoses are also present in rings (not shown) that can be 5-membered or 6-membered.

In contrast to cellulose that is crystalline, strong, and resistant to hydrolysis, hemicellulose has a random, amorphous structure with little strength. It is easily hydrolyzed by dilute acid or base, but nature provides an arsenal of hemicellulase enzymes for its hydrolysis. These enzymes are commercially important because they open the structure of pulp for easier bleaching, and older methods of bleaching consume larger amounts of chemicals such as chlorine that are bad for the environment.

2.3 METHOD USE

2.3.1 Steam Explosion

Steam explosion processing of pulp is a reactive method that changes the hierarchical arrangement, chemistry and morphology of pulp's bio-polymer components. Mechanical disruption, hydrolysis, and free radical reactions have been previously shown to contribute to the changes. Because of growing interest in pulp-thermoplastic composites, this processing method was investigated as a novel way to modify pulp fiber with thermoplastics. Specifically, the processing technique was applied to co-refining pulp chips and polyolefin thermoplastics such as polyethylene and isotactic polypropylene. The co-steam-explosion experiments explored variables of polyolefin type, molecular weight, polyolefin loading and gaseous vessel atmosphere reactivity, while keeping the processing time and steam temperature constant. .

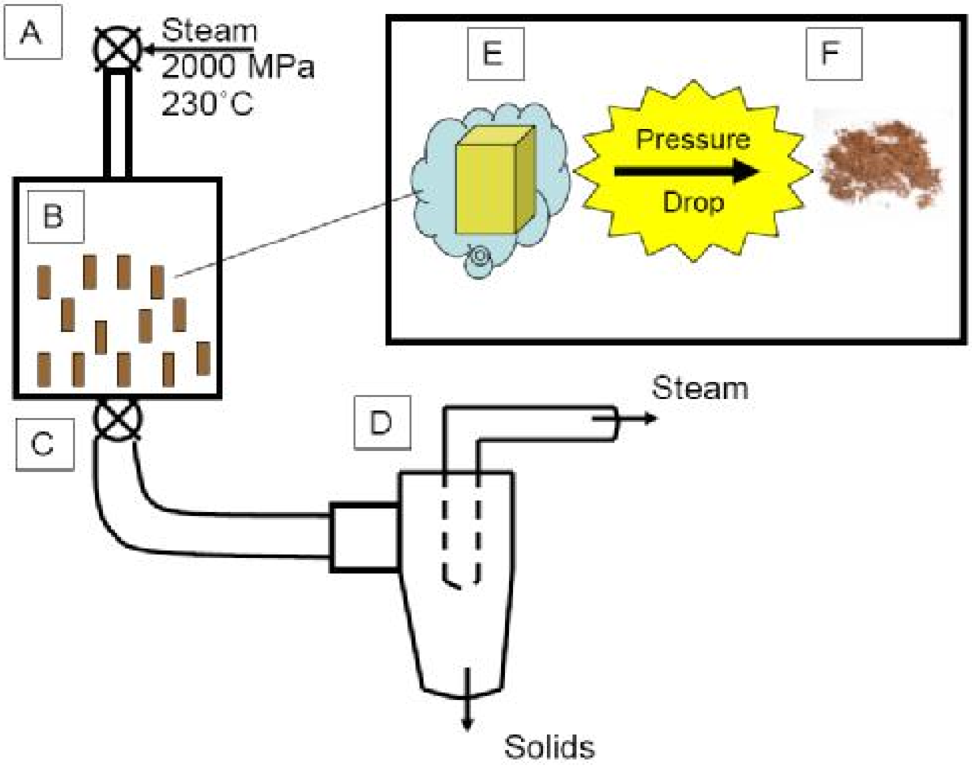


Figure 2.10 Sample Preview of Steam Explosion

2.4 METHOD REVIEW AND HISTORY

2.4.1 The inventor, Mason method

Explosion pulping was invented by Mason (1928) in the early 1930s. In this process, the chips are fed from a chip bin through a screw loading valve in a masonite gun. The chips are then steam heated at a very high temperature, about 285°C, and at a pressure of 3.5 MPa for about 2 min. The pressure is increased rapidly to about 7 MPa for about 5 s, and the chips are then discharged through restricted orifices (slotted port) and explode at atmospheric pressure into a pulp. Two stages of low consistency atmospheric refining result in a dark pulp of about 75% yield suitable for the manufacture of high-density fiberboard. This process is very effective as a means of fiber separation with low energy consumption. However, Koran and coworkers (1978) concluded that masonite pulp is very coarse in texture and dark in color. The fibers are mostly uncollapsed, rigid, and degraded and display a highly lignin-rich surface structure, which is unsuitable for papermaking.

2.4.2 Asplund Method

Asplund (Asplund *et al.*, 1953) refined chips at elevated temperature and pressure to produce a coarse, dark fiber for board manufacturing. They attempted to avoid the fiber degradation, inherent in high-temperature processes, through the application of different chemicals. Thus, steam was replaced with ammonia (O’Connor, 1972) and liquid sulfur dioxide (Mamers and Grave, 1973). The pulp produced was of acceptable quality, but the technical and economic problems associated with the recycling of the liquefied gases precluded any commercial applications (Mamers *et al*., 1981).

An alternative approach to explosion defibration was used with aqueous systems at a relatively moderate temperature of 200°C with further pressurization of the digester to 13.8 MPa by injection of compressed nitrogen. The fibrous material was explosively discharged through a specially designed multiple bar nozzle

(Mamers *et al.*, 1976, 1979, 1981). The pulp properties obtained were comparable to those of conventional CTMP and CMP processes but had lower brightness. As pointed out by Stamm (1965), the major problem to overcome is oxidation and hydrolytic degradation of fibers, which leads to brightness and yield losses.

2.4.3 Vit and Kokta Method and Overend and Chornet

Vit and Kokta (Vit and Kokta, 1986; Kokta and Vit, 1987) were able to produce ultra-high-yield pulps suitable for papermaking. The process consists of chemical impregnation of chips, short-duration saturated steam cooking at a temperature varying from 180 to 210°C sudden pressure release, refining, and bleaching. Later, Kokta (1987, 1989) modified and improved this explosion process, referred to as the steam explosion pulping process (SEP). The negative effect of wood hydrolysis and oxidation on yield and brightness owing to high temperature cooking was minimized by optimizing the impregnation and cooking conditions (Kokta, 1989). Kokta and his co-workers conducted many studies on explosion pulping (Vit and Kokta, 1986; Kokta, 1987; Kokta *et al.*, 1987, 1987, 1988, 1989, 1990, 1990, 1991, 1992, 1993,).

In addition, modeling based on combining the effects of time, temperature, and concentration into one single parameter was used to develop a linear model expressing the relationship between pretreatment severity and lignin reduction or xylan solubilization. Overend and Chornet (1987) initially defined this severity parameter to relate temperature and time for steam explosion pretreatment based on the assumption that pretreatment affects follow first-order kinetics and obey the Arrhenius equation.

Using this relationship they defined a reaction ordinate (R_0, min)

$$R_0 = t * \exp\left[\frac{(T_r - T_b)}{14.75}\right]$$

Where t is the residence time (min), T_r is the reaction temperature (°C), T_b is the base temperature (100 °C) and 14.75 is the conventional energy of activation assuming the overall reaction is hydrolytic and the overall conversion is first order. The logarithm of the reaction ordinate defines the severity during steam explosion pretreatment such that severity is equal to log (R_0).

CHAPTER 3

METHODOLOGY

3.1 MAIN METHOD, STEAM EXPLOSION

Steam explosion of lignocelluloses materials has been largely studied in the last 10 to15 years, 1 to 14 with a view to increasing the conversion yields of the polysaccharides into monosaccharide by enzymatic hydrolysis, the monosaccharide being initially used as starting material for nearly all fermentation products.

Figure 3.1 Steam Explosion Reactor

In the steam explosion method, steam, and additives when neccessary, under pressure and with increased temperature, penetrates the space between fibers of the bast fiber bundle. In this way, the middle lamella and the fiber adherent substances are elementarized "softly" and made water soluble. These can be then removed by subsequent washing and rinsing. According to the desired quality of the elementarized fibers, the pressure of the steam can also be reduced suddenly to atmospheric, and blown out along with the substrate, to a settling chamber.

The combination of chemical and mechanical treatment produces an effective loosening of the rigid bast fiber structure and thereby a further splitting into single fibers.

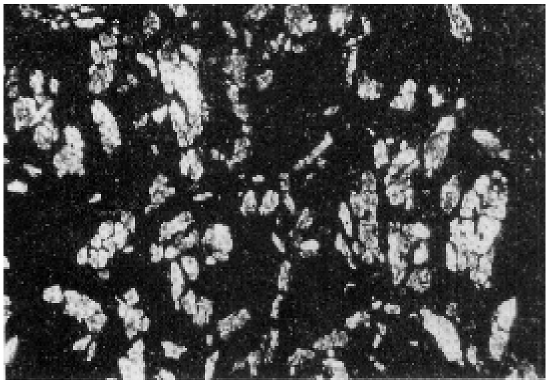


Figure 3.2 Microscopic cross section of OPF fibers before steam explosion

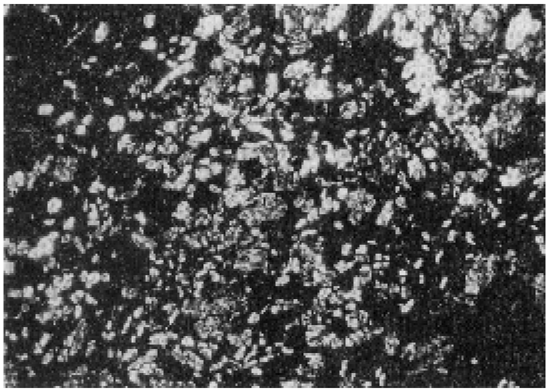


Figure 3.3 Microscopic cross section of OPF fibers after steam explosion

3.2 SAMPLE PREPARATION

3.2.1 Harvesting

Oil palm frond usually being an abundant biomass and will be burn. Oil palm frond(OFB) can be taken free directly from oil palm plantation. This is because OFB is not having a value as it treated as biomass. Oil palm plantation usually cut their frond twist a year to maintain the tree growth.

3.2.2 Chipping

Chipping done by hand or by a simple machine which consists of a driven disc with radial chipping slots fitted with cutting blades. The Malaysian type consists of a heavy rotating circular steel plate about 12 mm thick and 1m in diameter to which six blades (1-1.5 mm steel plate) are attached

The chipping wheels are usually mounted in wooden frames incorporating feed hoppers and driven by petrol, diesel, kerosene or electric motors. But in this research, all the sample are chipping already.

Figure 3.4 Chipping Frond

3.2.3 Drying

Natural drying is done on cement floors which are sometimes painted black for better absorption of radiant energy or on trays for artificial drying factors that affect OFB drying time are the geometry (shape and size) of the OFB chips, the chip loading per unit drying area, air speed, temperature, humidity, radiation, as well as dry matter content of the fresh chips.

In artificial heat dryers, all these parameters can be optimized to minimize the drying time and guarantee a high quality product.

But in this research, sample are dried up in oven at 105^{0}C for about 8 hours. The dry matter content of fresh OFB is affected by several factors such as the variety, harvesting age, and the agronomic conditions, but in general, it below 10% humidity.

$$\% \text{ humidity} = \frac{\text{initial weight} - \text{final weight}}{\text{final weight}} \times 100\%$$

3.2.4 Steam Explosion

The steam explosion process is done using mass reactor volume 1 liter that being fabricated by Sabutek (M) Sdn. Bhd. The reactor is placed in Teluk Intan which is located in Fiber X Sdn. Bhd. The reactor is supplied by high steam boiler that produced high pressure steam about 15-25kPa.

The sample weight of 100g is immersing in water to get 50% humidity before being feed into steam explosion reactor. Steam is entered into the reactor for about 1 minute to remove air bubble. After that, reactor is closed and steam is entered until get the temperature and pressure that needed.

The combination of time is use as parameter in this experiment. After steam explosion method, the fiber is placed in temperature plat for 60^0C for about a day.

Figure 3.5 Steam Explosion

3.2.4 Pretreatment of the Fiber

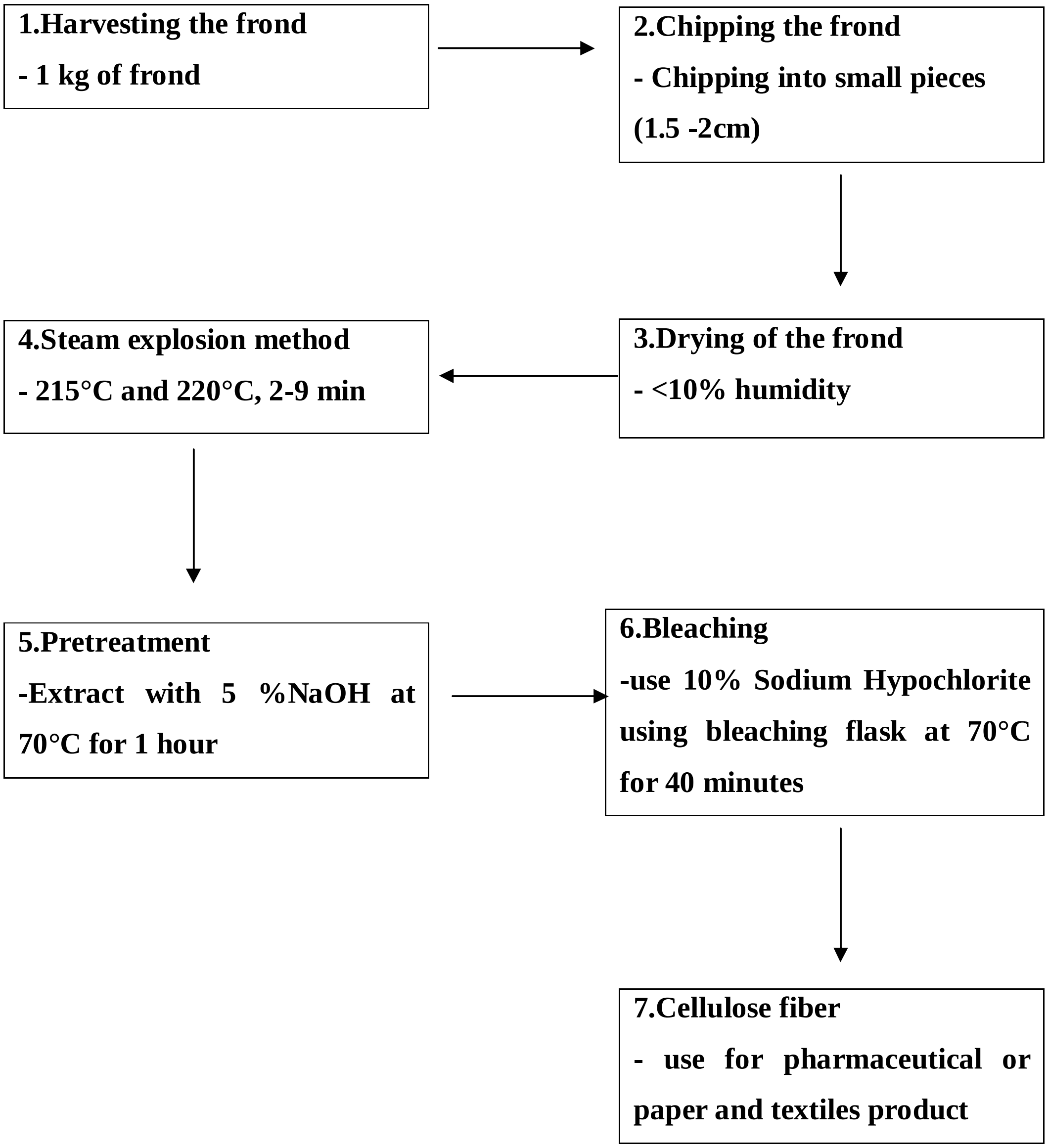
3.2.4.1 Alkaline Extraction

About 5g of sample is use in this step. The concentrated of NaOH is 0.125M from the dry weight of the fiber. The consistency that is used is from fraction 1:10 (fiber : water). The NaOH solution is diluted from stock 2.5M that were prepared early. The extraction time is 1 hour with temperature of 70^0C.

3.2.4.2 Bleaching Treatment

Bleaching in this research is done by using chlorine based bleaching which is Sodium hypochlorite that has concentrated of 10% that has been prepared by staff in Fiber X Sdn. Bhd. Sample is take after alkaline treatment is to weight about 3g. The consistency of sample are 10% (fiber : liquid) which is used in this steps.

3.3 PROCESS FLOW DIAGRAM



CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

In this method. 3 steps before having cellulose is done which is steam explosion, alkaline treatment and bleaching. Weight of sample of these 3 steps is calculated as different method produce different result. For steam explosion, the result is fiber with lignocellulose, after alkaline treatment, the lignin wall of cell will break to get alpha cellulose and holocellulose. And after bleaching to remove colour and odor, the acid will eliminate alpha cellulose to get only holocellulose which product that needed.

4.2 After Steam Explosion

4.2.1 Result

After Steam Explosion (Sample of 100g)

Temperature (°C)	Time (min)	Log Ro	Weight (g)	% Result
215	**3**	**3.86**	**89.46**	**89.46**
215	**6**	**4.16**	**69.96**	**69.96**

Table 3.1 Result After Steam Explosion

$$Ro = t.\ e^{(T-100/14.75)}$$

$$\% \text{ Result} = \frac{\text{net weight of result}}{\text{net of original weight}} \times 100\%$$

Figure 3.6 After Steam Explosion Log Ro 3.86

Figure 3.7 After Steam Explosion Log Ro 4.16

4.2.2 Discussion

Percentage of result after steam explosion shows that the increasing of value of Log Ro will produce low yield of product which is fiber with lignocellulose. This observation same with result that get by Overend & Chornet, (1990). As form the picture, the increasing of Log Ro also will increase the brownies colour of the fiber. It also increases the intensity odor of acid organic.

4.3 After Alkaline Extraction

4.3.1 Result

After Alkaline Extraction (Sample of 5g)

Temperature (°C)	Time (min)	Log Ro	Weight (g)	% Result
215	3	3.86	4.475	89.5
215	6	4.16	3.495	69.9

Table 3.2 Result After Alkaline Extraction

$$\% \text{ Result} = \frac{\text{net weight of result}}{\text{net of original weight}} \times 100\%$$

4.3.2 Discussion

After alkaline treatment, it shows that there were different variations between results of sample based on Log Ro. Log Ro is inversely proportional to the result. The higher different of results that shows that Log Ro 4.16 get 15% of weight which is the highest different compared to Log Ro 3.86.

Figure 3.8 After Alkaline Treatment

4.4 After Bleaching

4.4.1 Result

After bleaching (sample of 3g)

Temperature (°C)	Time (min)	Log Ro	Weight (g)	% Result
215	3	3.86	2.658	88.6
215	6	4.16	2.796	93.2

Table 3.3 Result After Bleaching

$$\%\ \text{Result} = \frac{\text{net weight of result}}{\text{net of original weight}} \times 100\%$$

4.4.2 Discussion

Bleaching is the most critical part in producing cellulose fiber from lignocellulose ingredient. Experiment is done by using Sodium Hypochlorite. Research is based on percentage of producing holocellulose which is the main product of this research. Based on the result above, mainly lignin component is being removed from the fiber. This cause percentage of having holocellulose is increasing with Log Ro of steam explosion. This result shows that when longer time and higher temperature of steam explosion occurs will result higher Log Ro that will increasing the yield of holocellose after bleaching.

Figure 3.9 After Bleaching

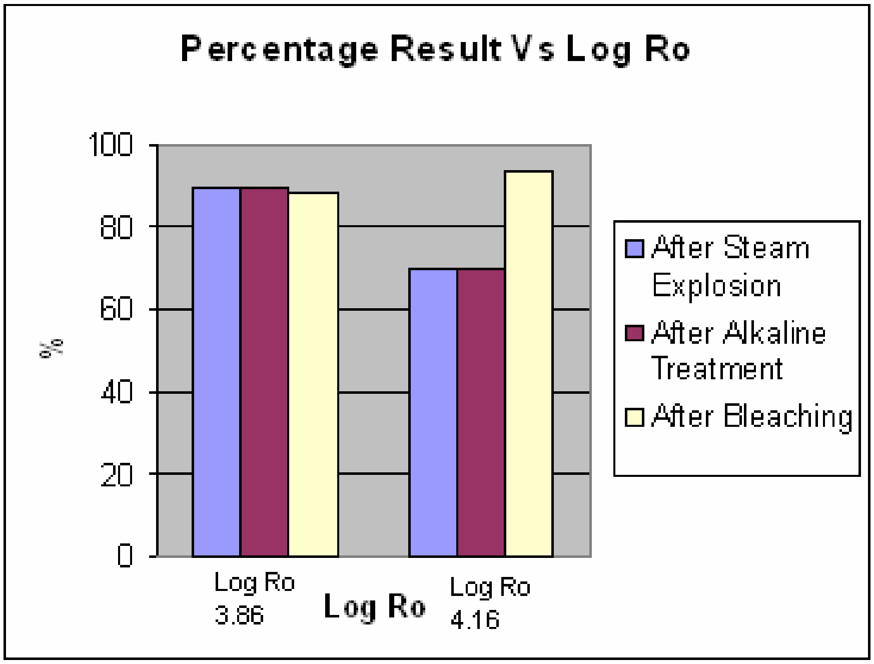


Figure 3.10 Graph Percentage of Result In 3 Stages Versus Log Ro

CHAPTER 5

5.1 CONCLUSION

This research has successfully shows that steam explosion method could increase the ability to produce cellulose fiber. The steam explosion can help simplify the process of purifying the cellulose from palm frond lignocellulose. The product has the same functional and physical property compared to other material that also has cellulose fiber like from corn plant and sugarcane.

The highest result collect from steam explosion fiber is Log Ro 3.86 which is 89.5% and lowest result is Log Ro 4.16 which is 69.9%. Product which is fiber is produced less when log Ro is high. Steam explosion fiber also change into smaller fiber which has higher water solubility compared to its original weight.

Result for alkaline extraction fiber also shows same criteria which is the highest Log Ro 4.16 produced less result which is 69.9% while lowest Log Ro 3.86 is vice versa which is produced 89.5% result. Alkaline extraction shows high lignin content which mean it has potential to re use as extraction of lignin later.

While in bleaching treatment, the production of holocellulose shows different criteria. Log Ro 3.86 give lowest result which is 88.6% compared to 93.2% with Log Ro 4.16. This situation shows that bleaching reaction eliminate more lignin and hemicellulose that not been removed after alkaline extraction.

5.2 RECOMMENDATION

Based on the result form this research, there were some recommendation which is :-

1. A research must be conducted for using catalyst in steam explosion like using acetic acid. Use of catalyst might reduce the pressure in steam explosion reactor.

2. Reseacrh must also be conducted in using bleaching reactant that environmental friendly like using ozone, oxygen or hydrogen peroxide. Method that been used in this experiment while bleaching must be avoid because it use chlorine based bleach that has bad effect to environment.

3. Universiti Malaysia Pahang (UMP) especially FKKSA should fabricate the complete set of equipment for steam explosion for further study. This is because many areas in steam explosion that not been discovered and need more perfection of the system.

REFERENCES

Azemi et al., 1996 Simatupang, M.H. Harun, S & Norma N.M.N : Steam Assisted Fractionation of oil palm Trunk (OPT), (1996) Proceedings of 1996 PORIM International Palm Oil Congress, Palm Oil Research Institute of Malaysia

Anon. 2000. Editorial on "Demand for wood and agriculture fibers". plastics BRIEFreinforced plastics, 24(23):1-2.34

Alcaide et al., 1990 L.J. Alcaide, I.S. Parra and F.L. Baldovin, Characterization of Spanish agricultural residues with a view in obtaining cellulose pulp, Tappi J. 73 (1990) (8), pp. 173–177.

Alen, 2000 R. Alen, Basic chemistry of wood delignification. In: P. Stenius, Editor, Papermaking Science and Technology Series vol. 3, Fapet, Helsinki (2000), pp. 58–104.

Atchinson, 1989 J.E. Atchinson, Bagasse. In: M.J. Korucek and C.F.B. Stevens, Editors, Pulp and Paper Manufacture vol. III, Joint Textbook Committee of Paper Industry, Tappi Press, Atlanta (1989), pp. 58–60.

Brooks, S.H.W. 1999. Steam explosion, a uniquely new method of producing a wood plastic-molding compound. Unpublished technical report to client. Brooks Associates, Ltd., 247 Winry Drive, Rochester Hills, MI 48307-1161.

Boudet, 2000 M. Boudet, Lignin and lignification: selected issues, Plant Physiol. Biochem. 38 (2000), pp. 81–96.

Chornet, E. and R. Overend. 1991. Phenomenological kinetics and reaction engineering aspects of steam/aqueous treatments, In Steam Explosion Techniques Fundamentals and Industrial Applications, B. Focher, *et al.*, Eds., Gordon and Breach Science Publishers, Amsterdam:21-58.

Clark, 1985 J. d'A Clark, Pulp Technology and Treatment for Paper (2nd ed.), Miller Freeman Inc., San Francisco (1985).

Hansen, C. and A. Bjorkman. 1998. The ultrastructure of wood from a solubility parameter point of view. Holzforschung, 52(4):335-344.

Johnson, J. (1999 July 20). Technology turns old bits and pieces into new parts. Roanoke Times- New River Valley Current, pp. 1-3.

KOZUMAYosei Center for Archaeological Operations, National Research Institute for Cultural Properties, Nara Independent Administrative Institution

Law and Jiang, 2001 K.N. Law and X. Jiang, Comparative papermaking properties of oil-palm empty fruit bunch, Tappi J. 84 (2001) (1), p. 13.

Law et al., 1986 K.N. Law, M. Lapointe and J.L. Valade, Chemithermomechanical pulping of mixtures of different species of hardwoods, Tappi J. 69 (1986) (7), pp. 80–83.

Mohamad Husin, Abd. Halim Hassan and Ahmad Tarmizi Mohamad (1986) : Availability and potential Utilization of Oil Palm Trunk and fronds Up to the Year 2000, PORIM Occasional Paper, No. 20 : 1-17

Overend and Chornet, 1987 R.P. Overend and E. Chornet, Fractionation of lignocellulosics by steam-aqueous pretreatments, Philos. Trans. R. Soc. Lond. 321 (1987), pp. 523–536.

Pearson, R. 2000. Introduction to the Toughening of Polymers, In Toughening of Plastics. Advances in Modeling Experiments, R. Pearson, *et al.*, Eds., Oxford University
Press, Washington D.C.:1-11.

Razi, P., R. Portier, and A. Raman. 1999. Studies on polymer-wood interface bonding: effect of coupling agents and surface modification. Journal of Composite Materials, 33:1064-1079.

Shook, S. and I. Eastin. 2001. A characterization of the U.S. residential deck material
market. Forest Products Journal, 51:28-36.

U.S. Environmental Protection Agency, Office of Compliance, Sector Notebook Project, Standard Industrial Classification 261–265, Profile of the Pulp and Paper Industry, Washington, D.C.,
September 1995.

www.aseansec.org/7011.htm

www.en.wikipedia.org/wiki/Cellulose

www.freepatentsonline.com/4352341.htm

APPENDIX A

No	Month	July		August		September		October		November		December		January		February		Mac		April	
1	Planning / Week	2	4	6	8	10	12	14	16	18	20	22	24	26	28	30	32	34	36	38	40
2	Reviewing Literature:																			■	■
3	a.) Search The Information	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■
	b.) Extract The Information		■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■
	c.) Visit to MPOB		■																	■	■
4	Proposal preparation*		■	■	■															■	■
5	Presentation(Seminar 1)*					■														■	■
6	Visit to Sabutek													■						■	■
7	Visit to UniKL													■	■					■	■
8	Lab Analysis														■					■	■
9	Lab Experiment															■	■			■	■
10	Final Lab Analysis																	■		■	■
11	Presentation (Seminar 2)*																	■		■	■
12	Thesis writing*														■	■	■	■	■	■	■

* Submit proposal 27.07.2007

*Seminar I Presentation on week 9

* Progress report presentation on week 31

*Seminar II Presentation on week 39

*Submission thesis on week 42

*Submission technical paper and poster on week 43

Appendix B

$$Ro = t.\ e^{(T-100/14.75)}$$

For Temperature = 215 and Time = 3

$$Ro = 3e^{(215-100/14.75)}$$

$$= 7297.0287$$

so. Log Ro = Log 7297.0287

= 3.86

$$\%\ \text{Result} = \frac{\text{net weight of result}}{\text{net of original weight}} \times 100\%$$

$$= \frac{89.96}{100} \times 100\%$$

$$= 89.96\%$$