CHARACTERISATION OF THE ALKALINE PEROXIDE PULPS FROM THE OIL PALM EMPTY FRUIT BUNCH AND THEIR POTENTIAL FOR CELLULOSE ACETATE PRODUCTION

NURUL HASANAH KAMALUDIN

UNIVERSITI SAINS MALAYSIA

2013

CHARACTERISATION OF THE ALKALINE PEROXIDE PULPS FROM THE OIL PALM EMPTY FRUIT BUNCH AND THEIR POTENTIAL FOR CELLULOSE ACETATE PRODUCTION

NURUL HASANAH KAMALUDIN

Thesis submitted in fulfillment of the requirements for the degree of
Master of Science

JULY 2013

ACKNOWLEDGEMENT

All praise and thanks only to Allah swt for all His guidance, caring, encouragement and strength throughout the journey in completion of my study.

The author would like to express the greatest gratitude to her supervisor Dr Arniza Ghazali for her guidance, ideas, comments, critics, advises, and assists throughout this study. Without her moral supports and encouragements the completion of this study would have not been possible. Special gratitude is due to co-supervisors, Prof. Wan Rosli Wan Daud and Dr. Leh Cheu Peng for their constructive comments and valuable suggestions.

Grateful acknowledgements are extended to the staffs of Division of Bioresource, Paper and Coatings especially to staffs of Paper Testing Laboratory and Papermaking Laboratory for their assistance and guidance. Acknowledgements are due to the School of Biological Sciences, particularly to the staffs of Microscopy Laboratory for their assistances and guidance that made this study success. Tremendous appreciation goes to lecturers and staffs of School of Industrial Technology especially to fellow colleagues: Rosnah, Yunita, Hafiz, Azli and Amin for being such a good listener, supporter and source of courage. Much appreciation also goes to her other numerous friends and fellow postgraduates for their kindness with much inspirations and guidance.

Last but not least, the author wishes her deepest appreciation to her dearest parents and family for their encouragement, motivation, inspiration, love, understanding and sacrifices along the completion of her study. They are the source of light in the darkest places.

TABLE OF CONTENTS

Contents **Page**

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

APMP	Alkaline Peroxide Mechanical Pulping
APP	Alkaline Peroxide Pulping
AP	Alkaline peroxide
APP-O	oxygen bleached pulp
APP-OZ	oxygen-ozone bleached alkaline peroxide pulp
APP-OZP	oxygen-ozone-peroxide bleached alkaline peroxide pulp
APP-P	peroxide bleached alkaline peroxide pulp
APP-Z	ozone bleached alkaline peroxide pulp
CA	cellulose acetate
OCA	cellulose acetate synthesized from oxygen bleached alkaline peroxide pulp
DPCA	cellulose acetate synthesized from dissolving pulp
DP	dissolving pulp
DP	degree of polymerisation
OZCA	cellulose acetate synthesized from oxygen-ozone bleached alkaline peroxide pulp
OZPCA	cellulose acetate synthesized from oxygen-ozone-peroxide bleached alkaline peroxide pulp
PCA	cellulose acetate synthesized from peroxide bleached alkaline peroxide pulp
ZCA	cellulose acetate synthesized from ozone bleached alkaline peroxide pulp
CCA	commerciallised cellulose acetate
SEM	Scanning Electron Microscopy
XRD	X-ray Diffraction
DSC	Differential Scanning Calorimetry
DS	degree of substitution

OH	hydroxyl group
$COCH_3$	acetyl group
NaOH	sodium hydroxide
H_2SO_4	sulphuric acid
P	peroxide
O	oxygen
Z	ozone
EFB	empty fruit bunch
$KMnO_4$	potassium permanganate
KI	potassium iodide
FTIR	Fourier Transform Infrared Spectroscopy
TEC	triethyl citrate
HCl	hydrochloric acid
ASTM	American Society for Testing and Material
CH_3COOH	acetic acid
$(CH_2CO)_2O$	acetic anhydride
TAPPI	Technical Association of The Pulp and Paper Industy
KHP	potassium hydrogen phthalate
TCF	totally-chlorine-free bleaching
CSF	Canadian standard freeness
RH	relative humidity
FQA	fibre quality analysis
MPOB	Malaysian Palm Oil Board
OPT	oil palm trunk
MDF	medium density fibreboard
OPF	oil palm frond
KBr	potassium bromide

K	kappa number

PENCIRIAN PULPA PEROKSIDA BERALKALI TANDAN BUAH KOSONG KELAPA SAWIT DAN POTENSINYA BAGI PENGHASILAN SELULOSA ASETAT

ABSTRAK

Dalam fasa pertama kajian ini, kertas telah dihasilkan daripada pulpa peroksida beralkali (APP) dan kajian ini diikuti oleh proses dua kali kitaran semula kertas yang telah dihasilkan. Lapan set kertas yang berbeza dalam tempoh tindak balas antara tandan buah kosong (EFB) dan peroksida beralkali telah dihasilkan. Komponen pulpa dan halusan yang telah diperolehi telah dikaji danpotensi penggunaan gentian pulpa halus di dalam kertas juga dinilai.. Perlu dinyatakan bahawa halusan yang dikumpul di atas jaringan *400-mesh* (R400) boleh meningkatkan kekuatan tegangan kertas sebanyak 100%. Kitaran semula pulpa berkenaan, walaubagaimanapun, menunjukkan kemerosotan sifat-sifat mekanikal kertas.Hal ini disebabkan fenomena 'hornification' gentian dan ikatan antara gentian yang lemah akibat daripada membasahkan semula dan peyepaian yang berulang pada kertas. Ekoran penurunan kapasiti tersebut, ujikaji penulenan pulpa yang tidak dapat dikitar semula telah dijalankan bagi menyediakan pulpa yang lebih baik sebagai bahan mentah bagi penghasilan selulose asetat (CA). Keputusan penulenan bebas klorin sepenuhnya (TCF) yang dikenakan terhadap pulpa peroksida beralkali dengan urutan oksigen-ozon-peroksida (OZP), menunjukkan permukaan yang paling licin terhasildaripada pulpa peroksida beralkali dilunturkan melalui proses oksigen-ozon-peroksida selulosa asetat (OZPCA) dengan penambahan pemplastik. Samada dengan atau tanpa pemplastik, selulosa-selulosa asetat (CAs) yang terhasil menunjukkan persamaan sifat termal selulosa asetat komersial (CCA). Dengan pembuktian dengan kalorimeter pengimbasan kebezaan (DSC), keseluruhan keputusan menunjukkan potensi penghasilan CA daripada pulpa peroksida beralkali bersumberkan tandan buah kosong (EFB). Hal ini memberikan satu demonstrasi bagi penambahbaikan pengurusan akhir-hayat produk berasaskan pulpa daripada sistem mesra pemulpaan perosida beralkali EFB.

CHARACTERISATION OF THE ALKALINE PEROXIDE PULPS FROM THE OIL PALM EMPTY FRUIT BUNCH AND THEIR POTENTIAL FOR CELLULOSE ACETATE PRODUCTION

ABSTRACT

The first phase of this study involved the making and analyses of hand sheet made from the fibers of the oil palm empty fruit bunches via the process called the alkaline peroxide pulping (APP). This was followed by a two-stage paper recycling process Eight sets of hand sheets were made from the fibers extracted from the varying EFb-alkaline peroxide (AP) reaction duration The pulp and fines from the process were studied and the possibility of incorporating the fines in paper was also assessed. It is noteworthy that the fines collected as materials retained on the 400-mesh screen (R400) could improve hand sheet tensile strength by 100%. Recycling of this pulp, however, showed a declining mechanical properties of hand sheets. This is attributable to fibre hornification and weak inter-fibre bonding as a consequence of re-wetting and repeated disintegration of the hand sheets. Owing to the lost papermaking capacity, pulp purification was therefore attempted to upgrade the pulp to become raw material for cellulose acetate (CA) production. As a result of totally chlorine-free bleaching (TCF) purification of the alkaline peroxide pulp by oxygen-ozone-peroxide (OZP) sequence, CA powders and films show the smoothest oxygen-ozone-peroxide cellulose acetate (OZPCA) film surface produced by incorporation of plasticiser. With or without plasticiser, the produced CAs show thermal characteristics that are similar to those of the commercial CA. By verification with differential scanning calorimetry (DSC), the overall results show the possibility of CA production from AP pulp of empty fruit bunch

(EFB). This further defines an improved end-of-life management of the pulp produced from the benign alkaline peroxide pulping system of EFB.

CHAPTER ONE

INTRODUCTION

1.1 General background

Asia is an influential source of non-wood pulp from annual fiber crop, agricultural residue (or agro-waste) and non-plant fibrous mass such as algae, rags and animal waste. In the course of palm oil milling in Malaysia, for instance, 19.3 million tonnes of empty fruit bunch (EFB) is generated each year as the fruits are used for oil extraction (Hoggard, 2011). Valuable pulp was successfully extracted from this cellulosic material via eco-friendly pulping system such as alkaline peroxide mechanical pulping (APMP). Besides the proven low environmental hazard, APMP is also simple and fast as the system combines the pulping and bleaching stages in one process, with pulp quality comparable to chemical pulps. (Fang *et al.*, 2000j; Ghazali, 2006; Ghazali *et al.*, 2009). The fact that pulping and bleaching occur in a single process, eliminates the need for a separate bleach plant and the ensuing operation and maintenance costs.

As EFB pulp is gaining worldwide acceptance as blended pulp in many industrial paper and packaging products, the knowledge on characteristic and potential use of the waste material in EFB APMP is especially important. Study was not only made on the characteristic of the EFB alkaline peroxide pulping (APP) pulp but also the co-generated fines materials. Furthermore, the end-of-life of this pulp has yet been defined. Thus, the recyclability of the EFB APP pulp was conducted and to prevent the cradle-to-grave type of end-of-life, the possibility of CA production from EFB APP was attempted to define a more profitable end-of-life of the pulp.

Cellulose acetate (CA) is an ester type of cellulose derivative. Commonly, it is derived by an acetylation process involving treating cotton linter or wood pulp with acetic anhydride, in the presence of acetic acid as the medium and the presence of sulphuric acid as the catalyst. Commercially, CA is made from processed wood pulp. The sources of CA are from renewable resources such as wood pulp or cotton linters having high cellulose content. Wide range applications of CA attracted most researchers to explore and understand the behavior of this special product due to their biocompatible (material for biomedical applications) and biodegradable properties.

For the acclaimed strength, the principles of APMP were therefore adapted to EFB. Compensations were made between maximisation of AP power and maximisation of available equipment and this was attempted on the abundant industrial residue, the oil palm empty fruit bunch. This was carried out on the bases outlined in the subsequent section.

1.2 Problem Statement

Pulping is the second most polluting industrial process for several important reasons.

a) Wood as raw material is a controversial environmental impact. This study focuses on industrial waste as raw material.

b) Downstream residues are often discharged to water bodies causing organic pollutant although the APMP is chlorine and sulphur-free. This study demonstrates the potential use of the fibrous mass from the discharge.

c) Non-recyclable paper products due to its poor paper properties or not suitable for recycle often define the grim end-of-life of paper pulp at the dump site and blown into air as airborne contaminant. This study demonstrates the possibility of CA production from the pulp.

1.3 Objectives

Based on the introduced issues, this research project has been planned to achieve the following objectives:

- To characterise the alkaline peroxide pulp as accepts and the fines generated and escaped as the refining discharge and investigate the potential use of fines in paper quality enhancement
- To examine recycling effects of empty fruit bunch (EFB) alkaline peroxide pulp as a way of gauging its reusability
- To explore the possibility of high-value use of empty fruit bunch (EFB) alkaline peroxide pulp (as way of redefining its end-of-life) in cellulose acetate (CA) production.

CHAPTER TWO

LITERATURE REVIEW

2.1 Non-Woody as a Precursor Material for Pulp and Papermaking

Biomass material composed of a mixture of organic elements (containing hydrogen, oxygen, nitrogen etc.). It provides feedstock for a variety of bio-energy end products and end uses after pre-processing by various conversion technologies (thermal, chemical and biochemical conversion). It can be divided into two broad categories: woody and non-woody. Forests are the sources of woody material. The main sources of non-woody materials are agricultural residues including sugar, starch, oils/fat and lignocellulose materials (cereal straw, bagasse, bamboo, flax, kenaf, jute, sisal) (Williams, 2011).

Non-woody plants offer several advantages including short growth cycles, moderates irrigation requirement and low lignin content which in principle would result in reduced energy and chemicals consumption during pulping process (Hurter & Riccio, 1998). The use of non-woody materials is an alternative to woody materials for pulp and paper production in developing countries. Non-woody fibres are mainly used for the production of specialty papers, i.e., tea bags, filter papers, bank notes, etc. in developed countries (Marques *et al.*, 2010) due to the wide variety among the length of the fibres and of different species. The giant reed fibres has short length (1180 µm in length) while fibres like flax and hemp has remarkable length (28000 µm in length) which is suitable for bank notes sheet incoroporation to enhance general strength characteristics and they have traditionally used as the primary furnish of cigarette paper (burning tube). There is a growing need to move agricultural industry based on food production to other industrial sectors such as paper and textiles. Therefore, non-woody materials could become important materials in this transformation (Moore, 1996; Paavilainen, 1998).

In terms of environmental concern, the utilisation of non wood material nowadays in papermaking, contributes to ecological balance besides control of deforestation and this could become a serious threat when having to cater the rapid growth in worldwide population, which results in the rapid demand of papermaking raw materials. In addition, pulping of non woody material requires less chemical and energy in contrast with wood material. In fact, non-wood fibres are potential source of pulping material. The wide variety of physical and chemical properties offered by non-wood plant fibres provides virtually endless opportunities for papermaking. However, these fibres have tremendous variations in chemical and physical properties as compared to wood fibre. Chemical composition of non-wood plant fibres varies widely depending on the fiber growth condition, botanical classification of fibre (Han, 1998), leave, fruit and stalk (Hurter, 2012) and processing procedures (storage and pretreatment). Chemical composition of non-woody plants basically contain of cellulose, lignin, pentosan and ash (Parham & Kausftinen; 1974). Table 2.1 shows the chemical composition of some common non-wood fibre compared to wood fibre.

Table 2.1: Chemical Composition of Some Common Non-wood Fibre Compared to Wood Fibre

Fibre type	Chemical composition (%)			
	Alpha Cellulose	Lignin	Silica	Ash
Straw[a]				
Rice[a]	28 - 36	12 - 16	9 - 14	15 - 20
Wheat[a]	38 - 46	16 – 21	3 - 7	5 - 9
Oat[a]	31 - 37	16 - 19	4 - 7	6 - 8
Cane[b]				
Sugar[b]	32 - 48	19 - 24	27 - 32	1.5 - 5
Bamboo[b]	26 - 43	21 - 31	15 - 26	1.7 - 5
Bast[b]				
seed flax[b]	43 - 47	21 - 23	24 - 26	5
kenaf[b]	44 - 57	15 - 19	22 - 23	2 - 5
Core[b]				
Kenaf[b]	37 - 49	15 - 21	18 - 24	2 - 4
Jute[b]	41 - 48	21 - 24	18 - 22	0.8
Baggase[a]	32 - 44	19 - 24	1 - 4	2 - 5
Softwoods[c]	40 - 45	26 - 34	NA	<1
Hardwoods[c]	38 - 48	23 - 30	NA	<1

Key:
[a] source of Parham and Kausftinen; 1974
[b] source of Gonzalo *et al.,* 1998
[c] source of Ward *et al.,* 2008

2.1.1 Use of Non-wood as Pulp and Papermaking in Asia

The pulp and paper industry around the world has been growing rapidly. As a result there has been a huge demand for pulp and paper making raw material. It leads to the increase in usage of non-wood fibres as a raw material for this purpose. Although some of the non-wood fibres used for papermaking are used because of their fine paper making qualities, majority of non-wood fibres is used to overcome the shortage of wood fibres. As a result their use is more widespread in countries with shortage of wood.

Worldwide, the forest products industry is a dynamic, vital and growing enterprise. Demand for global paper and paperboard is 402 millions tonnes per annum and it has doubled in 20 years from 1990 to 2011. Paper production has been forecasted to grow. By 2021, per annum paper consumption is projected to reach 521 million tonnes with Asia producing nearly 44 % (177 million tonnes) while the rest of the world produces 56 % (225 millions tonnes) (Kulkarni, 2013).

Growth in pulp and paper production entails massive felling of trees, which in turn leads to deforestation. Increasing competition for wood supplies coupled with gradually rising costs of wood have generated renewed interest in the use of non-wood plant fibres for papermaking in the highly industrialized countries (Sabharwal & Young, 1996). The use of agricultural-residues in pulping and papermaking might be desirable because it averts the need for disposal, which currently increases farming costs and causes environmental deterioration through pollution, fires, and pests.

There has been a great increase in global non-wood pulping capacities since 1970's. In 1970, non-wood pulp production was only 6.7% of the worldwide pulp production. By 1996, it had risen to less than 10% of the total pulp and paper production worldwide (El-Sakhawy *et al.*, 1996) and is expected to rise to less than 20% of the other more than 80% demand of wood pulp, by the end of 2013 (Kamago Omar *et al.*, 2013). Table 2.2 presents the global pulp for papermaking production. It is important to examine the potential benefits and problems associated with the use of non-wood plant fibres in pulp and paper industry since the increasing of non-wood usage and opportunities for further substitution for wood based fibres.

Table 2.2: Global Pulp for Paper Production (FAOSTAT, 2013)

Type of pulp	Production (million tons)							
	2005	2006	2007	2008	2009	2010	2011	2012
Chemical Wood Pulp	126.1	128.4	133.4	131.6	119.6	127.9	131.4	131.2
Mechanical Wood Pulp	35.8	35.5	34.9	33.4	28.9	30.7	29.4	28.9
Other Fibre Pulp	17.8	18.1	18.6	18.4	17.1	18.3	17.8	16.1
Semi-Chemical Wood Pulp	8.9	8.4	9.2	8.9	8.3	8.5	8.7	8.9

The demand for non-wood plant fibres for papermaking is expected to increase in the highly industrialized nations of Europe and North America due to the above environmental concerns like depleting forest resources and disposal of agricultural residues. Europe has an additional problem with the shortage of short fibered hardwood pulp, which can be replaced by some non-wood fibres. This will require knowledge of the processes and developments already in place in the countries already using these raw materials in the paper industry. Already, a number of non-wood fibres are commonly used in many countries for papermaking. Baggase are by far the largest source of non-wood fibres followed by bamboo and straw (Table 2.3).

Table 2.3: Worldwide Non-Wood Papermaking Pulping Capacities (FAO, 2010)

Raw material	Total papermaking pulp capacities (million metric tons(air dry)/ year)			
	2009	2010	2011	2012
1. Straw	20	27	27	27
2. Bagasse	654	689	690	711
3. Bamboo	149	149	149	149
4. Miscellaneous raw materials	277	278	278	279

Most of the increased use of non-wood plant fibres has been attributed to the tremendous demand in paper and increase in non-wood pulping capacities in People's Republic of China. At present, China produces more than two thirds of the world non-wood pulp followed by India. These two countries account for about 80 % of the total non-wood pulp production (Jahan, Gunter & Ataur Rahman, 2009). Table 2.4 gives a list of the top 20 countries utilizing non-wood fibres. These countries account for 99% of the total non-wood fibre used in papermaking.

Table 2.4: Leading Countries in Non-Wood Papermaking Pulp Production (FAOSTAT, 2013)

Country	Production '000 tons		Changes in percentages
	2006	2011	
1. China	12900	12400	-3.88
2. India	1995.3	1995.3	0.00
3. Spain	850	900	5.88
4. Pakisatan	350	370	5.71
5. USA	245	245	0.00
6. Italy	180.7	203	12.34
7. Colombia	176	158.7	-9.83
8. Thailand	155	131	-15.48
9. Argentina	131	236	80.15
10. Egypt	145	120	-17.24
11. Mexico	140	30	-78.57
12. Indonesia	105	105	0.00
13. South Africa	103	79	-23.30
14. Vietnam	74	30	-59.46
15. Japan	60	28	-53.33
16. Brazil	70	56	-20.00
17. Iran	57	67	17.54
18. Turkey	53	53	0.00
19. DPR Korea	50	56	12.00
20. France	56	88.36	57.79

There is an abundance of non-wood fibers potentially available for the paper industry. Around 12,000 tons use of non wood raw materials were recorded in 2003 and this increased to 850, 000 tons in 2010 (Lopez *et al.*, 2010). Nonwood fibres have been used in many countries like China, India, Spain, Italy, Turkey, Brazil and Egypt (Yilmaz, 1995). There are three sources of non-wood plant materials and these are agricultural by-products, industrial crops and naturally growing plants. Agricultural by-products are the secondary products of the principal crops such as cereals and grains. Industrial crops are the products with high quality pulp such as hemp, kenaf and sugarcane while naturally growing plants such as bamboo and elephant grass can also produce high quality pulp (Sridach, 2010). Figure 2.1 presents the global consumption of non-wood pulp in paper production.

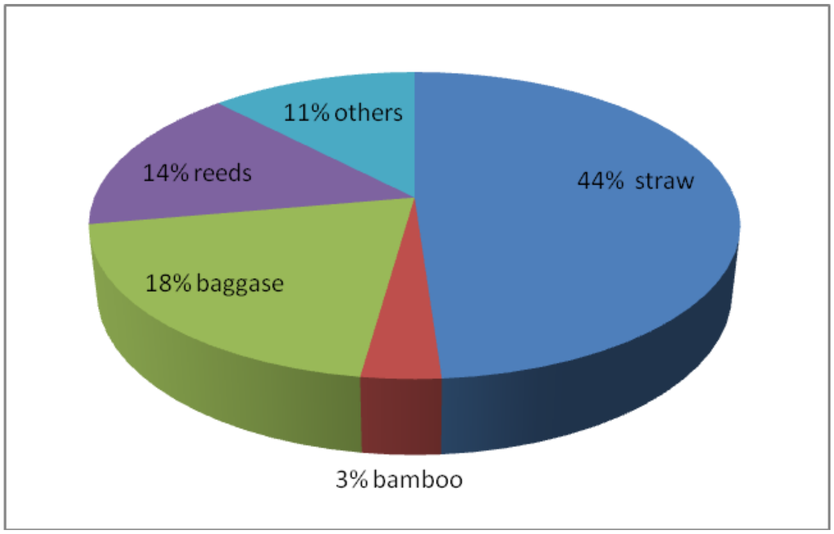

Figure 2.1: Global consumption of non wood pulp in paper production (Sridach, 2010).

Any grade of paper can be produced from several non-wood plant fibers and in blends with wood fibers. Some grades of paper have been made from 100% non-wood fibers. Table 2.5 shows the products of non-wood plant fibres in pulp and papermaking.

Table 2.5: Products of Non-Wood Plant Fibres in Pulp and Papermaking (Atchison, 1989)

Non-wood plant fibre	Products	Percent of non-wood plant fibre (%)
Abaca	Superfine, lightweight, bond, ledger, tea bags, filter	10-80
	Non-wovens	10-50
	Linerboard, wrapping and bag	10-30
Bagasse	Woodfree printing and writing	20-100
	Mechanical printing and writing	20-50
	Tissue	60-90
	Glassine and greaseproof	30-90
	Duplex and triplex	20-70
	Corrugating medium	50-90
	Linerboard	50-80
	Wrapping and bag papers Multiwall sack	50-85
	Newsprint substitute	30-70
		80-90
Bamboo	Woodfree printing and writing	70-100
	Mechanical printing and writing	40-60
	Bristol board	50-100
	Duplex and triplex	30-80
	Linerboard	60-100
	Wrapping and bag	80-100
	Multiwall sack	80-100
	Newsprint substitute	50-70
Cotton	High grade bond, ledger, book and writing	25-100
Esparto	Woodfree printing and writing	30-100
	Lightweight papers	50-70
Flax	Writing and book	20-60
	Lightweight printing and writing	20-80
	Condenser	20-60
	Cigarette paper	70-100
Hemp	Cigarette paper	50-100
	Lightweight printing and writing	20-80
	Condenser	20-60
Jute	Printing and writing	20-80
	Tag, wrapping and bag	40-60

Kenaf Bast fibre Stalk	Woodfree printing and writing	20-100
	Mechanical printing and writing	20-50
	Newsprint	20-30
	Multi-sack	50-100
	Linerboard	50-100
	Cigarette paper	50-100
	Tissue	60-90
	Bleached paperboard	50-100
	Newsprint	80-90
	Woodfree printing and writing	20-80
	Mechanical printing and writing	20-50
	Corrugating medium	50-100
	Linerboard	40-50
	Multi-wall sack	20-40
	Tissue	50-60
	Bleached paperboard	40-50
Reed	Woodfree printing and writing	20-100
	Mechanical printing and writing	20-50
	Duplex and triplex	20-70
	Corrugating medium	50-90
	Linerboard	50-70
	“B” grade wrapping	50-60
Straw, cereal and rice	Woodfree printing and writing	20-100
	Mechanical printing and writing	20-50
	Glassine and greaseproof	30-90
	Duplex and triplex	20-70
	Corrugating medium	50-90
	Strawboard	80-100
	“B” quality wrapping	50-60
Sisal	Superfine, lightweight, bond and ledger	10-80
	Non-wovens	10-50
	Publication paper	10-15
	Linerboard, wrapping and bags	10-30

2.1.2 The Advantages and Disadvantages of Using Non-wood for Pulping and Papermaking

There are a lot of issues and problems related to perspectives of using non-wood fibres as pulp for papermaking industry although there is growing interest in non-wood fibres. Concerning to this, it is realized to see the potential use of non-wood fibres due to demand and supply in paper making industries. Nowadays, wood is the domain supply for papermaking industry. However, a problem occurred regarding the long term fibre supply that has been faced by industry about two centuries ago in which the consumption of paper during the 18th century had increased dramatically (Hurter, 1998). Owing to high demand in pulp and paper making industry and shortage of wood supply, a few aspects are expressed regarding the positive and negative matter of using the non-wood plant fibre for pulp and paper production. The advantages and disadvantages of using the non-wood plant fibre for papermaking industry are listed as follows.

Advantages and the disadvantages of non wood fibres for papermaking (Hurter, 1998):

- Non-wood plant fibres are derived from fibre crops and agricultural residue such as kenaf and sugarcane baggase. These plant fibres are growing rapidly than wood plant and produce substance amount annually, thus it can resolve in fibre shortage. However, the disadvantages of it is that they put depends on annual residues and need large storage is required to ensure year round supply.

- In term of production, non-wood plant fibre has lower proportion of lignin compared to wood therefore less energy required to bleach fibre but there will be less amount of lignin to be used as fuel.
- Higher proportion of silica is found in non-wood plant and it will cost on machinery maintenance and makes the conventional chemical recovery inappropriate, but it will open more opportunity to researcher in utilisation of pulping bleach liquor.
- Less appropriate to make good furnish of paper, but development in new technology make it possible to used non-wood as raw material for papermaking.
- Industry is dominated by wood pulp producers and is conservative. Therefore, alternative pulping system such as APMP which reduce the need for chemical recovery may produce useful by-products. However, alternative pulping tend to use more expensive chemicals and only a little interest, research or investment in non-wood fibre by industry due to the high risks that may required change son machinery to enable used of non-wood fibres.

2.2 *Elaeis guineensis*

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. *Elaeis guineensis* does not grow in primeval forest. It flourishes in habitats where forests have been cleared. It requires a relatively open area to grow and reproduce itself and thrives best when soil moisture is maintained. It reaches a height of 20 m or more at maturity. The trunk is characterized by persistent, spirally arranged leaf bases and bears a crown of 20 to 40 massive leaves. The root system consists of primaries and secondaries in the top 140 cm of soil. Leaves numerous, erect, spreading to drooping, long, reaching 3-5 m in adult trees; leaf stalks short with a broad base (Anon. n.d.).

The major components of an oil palm tree are illustrated in Figure 2.2. The main residue generated by the milling operation of oil palm is the empty fruit bunches while the fronds come from maintenance pruning and replanting and the trunks are available only during the replanting operation. These residues were reported to have many characteristics that are equal or superior to peat in growing media (Lim & Ratnalingam 1980). All these residues can be utilized into value added products such as cooking oil, plywood, fibreboard and become as an energy resource.

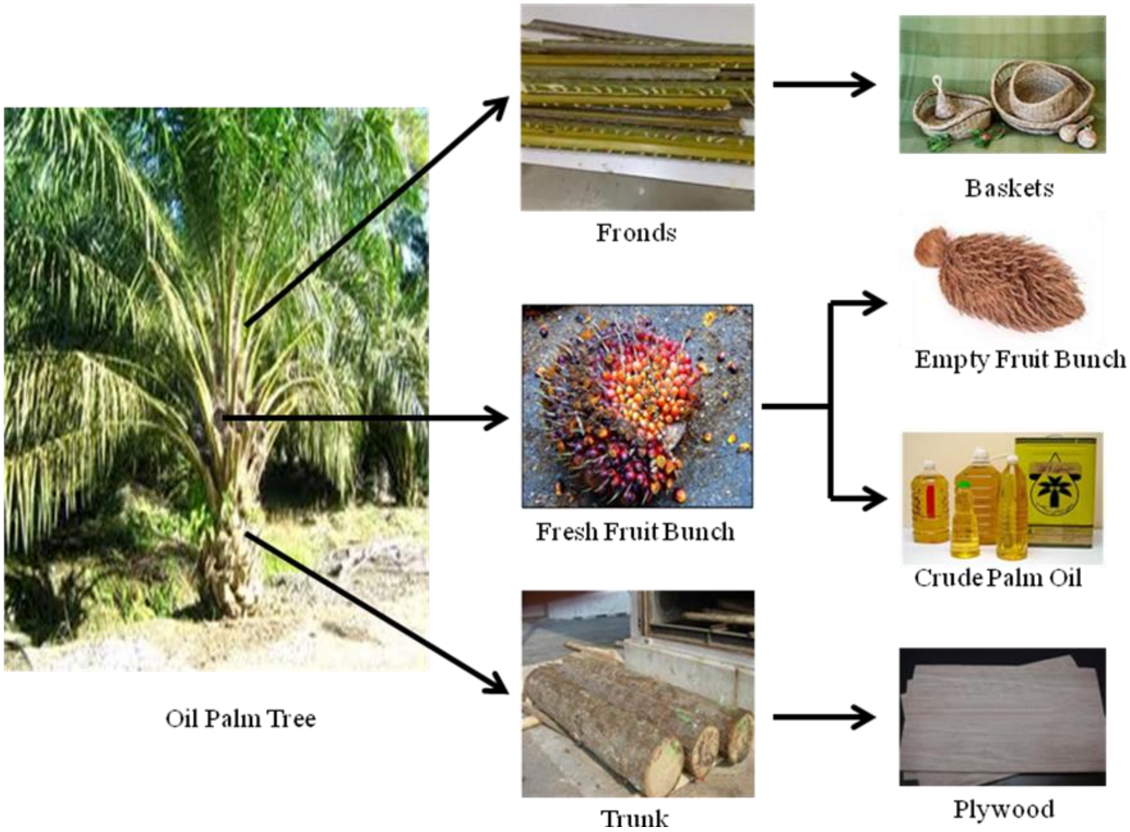

Figure 2.2: Biomass / products from oil palm tree (Anon, 2012).

2.2.1 Empty Fruit Bunch (EFB)

The empty fruit bunches (EFB) are waste by-products that are being investigated for further uses. Many studies have been carried out on the utilization of EFB, such as in particle boards (Harun *et al.*, 1997), medium density fiberboard (MDF) (Chooi, 1997), pulp (Mott *et al.*, 1997), and composites (Abdul Khalil *et al.*, 2001; Mohd Ishak *et al.*, 1998; Rozman *et al.*, 2001). Owing to its lower lignin content relative to oil palm fronds (OPF) and oil palm trunk (OPT), high cellulose content, moderate level of extractives and acceptable level of starch, EFB has been the most suitable raw material for pulping and paper making as compared to palm derivatives, OPF and OPT.

2.2.2 Availability of Empty Fruit Bunch in Malaysia

The oil palm was first introduced into Malaya (later known as Malaysia) in 1870 when it was the regarded as an ornamental plant. It was almost fifty years later (early 1920's) that its commercial viability as an estate crop was recognized and the first commercial oil palm plantation was started at Tennamaran Estate, Batang Berjuntai in Selangor (Anon., 2012). Oil palm is one of the most important commercial crops in Malaysia and become the world's largest producer and exporter of palm oil in 1980's. However, Indonesia overtook Malaysia as the world's largest in palm oil production since mid 2006. In 2006, Indonesia and Malaysia produced 15.90 million tonnes (44% world total) and 15.88 million tonnes (43% of world total) of oil palm, respectively (Anon. 2007).

In December 2011, the plantation area of oil palm in Peninsular Malaysia is 2.55 million ha, while in Sabah and Sarawak are 1.43 million ha and 1.02 million ha, respectively. Table 2.6 shows the oil palm planted area until June 2012. The oil palm plantations in Malaysia are owned by various categories of companies. Plantation ownership are grouped into various categories, namely the private estates, government owned agencies/schemes such as Federal Land Development Agency (FELDA), Federal Land Consolidation and Rehabilitation Authority (FELCRA) and Rubber Industry Smallholders Development Authority (RISDA), state schemes and smallholders as shown in Figure 2.3.

Table 2.6: Oil Palm Planted Area as at June 2012 (Hectares)

Region	Mature	%	Immature	%	Total	%
P. Malaysia	2, 190, 435	85.6	369, 237	14.4	2, 559, 672	50.8
Sabah	1, 279, 961	89.6	148, 339	10.4	1, 428, 300	28.4
Sarawak	851, 044	81.1	198. 943	18.9	1, 049, 987	20.8
Malaysia	**4, 321, 440**	**85.8**	**716, 519**	**14.2**	**5, 037, 959**	**100.0**

Source: MPOB (2012)

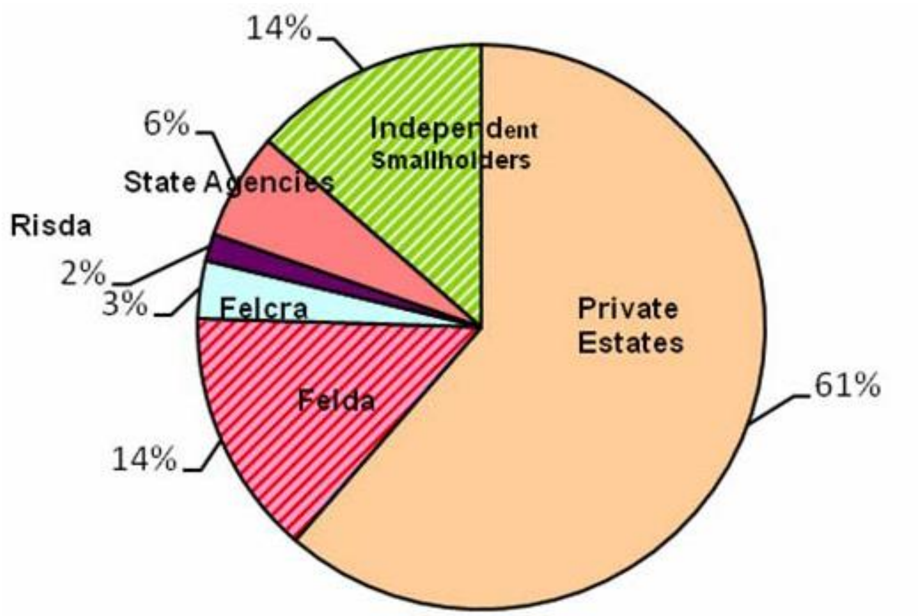

Figure 2.3: Ownership of oil palm planted area as at June 2012 (Hectares) (MPOB, 2012).

The highest area of oil palm plantation belongs to private estates which are about 61%. These include companies such as Sime Darby, KLK, IOI, Tabung Haji and others.

The continued high growth of oil palm plantations has generated enormous amounts of vegetable waste, creating problems in replanting operations and tremendous environmental concerns. The palm oil milling sector in Malaysia generates millions of metric tonne of oil palm waste annually. Malaysia produced about 70 million tonnes of oil palm biomass including trunks, fronds, and EFB in 2006 (Yacob, 2007). In 2009, Malaysian palm oil industry produced approximately 95.3 million tonnes of dry lignocellulosic biomass based on planted area of 4.69 million ha (Wahid, 2010) and a production rate of dry oil palm biomass of 20.336 tonnes per ha / year (Lim, 1998). This figure is to increase substantially when the total planted hectarage of oil palm in Malaysia could reach 4.74 million ha in 2015 (Basiron & Simeh, 2005). To maintain paper industry growth, governments as well as industry executives have to establish and implement policies and plans to ensure a sustainable fibre supply, including reforestation programs, plantation management, recycling and development of non-wood fibres or alternative fibres (Wan Rosli & Law, 2011). Besides reducing environmental impacts from waste accumulation, non-wood utilization such as EFB for pulp production can also help Malaysia to be independent of imported fibre (Fang *et al.*, 2000).

2.2.3 Empty Fruit Bunch Characteristics

Physical and mechanical properties of natural fibre vary according to shapes, sizes, orientations and thickness of the cell walls of individual fibre (Satyanarayana *et al.*, 1990). Table 2.7 and 2.9 show the physical and mechanical properties of oil palm EFB, respectively.

Table 2.7: Physical Properties of Oil Palm Empty Fruit Bunch (EFB) (Mohamad *et al.*, 1985; Law & Jiang, 2001)

Fibre	Length of fibre (mm)	Diameter of fibre (µm)
Oil palm EFB	0.89-0.99	19.1-25.0

Table 2.8: Mechanical Properties of Oil Palm Empty Fruit Bunch (EFB) (Abdul Khalil *et al.,* 2008)

Fibre	Density (g/cm³)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
Oil palm EFB	0.7-1.55	248	3.2	2.5

Amounts of parenchyma cells present in the fibre of oil palm EFB influence the fibre length and diameter of the fibre. Oil palm EFB have a much thicker cell wall when compared with those of wood, yielding a substantially higher rigidity index. Oil palm EFB fibres are lignocellulosic fibres where the cellulose and hemicellulose are reinforced in a lignin matrix similar to that of other natural fibres. This oil palm EFB consists of high cellulose content and is a potential natural fibre resource, but its applications account for a small percentage of the total biomass productions. Cellulose, hemicelluloses and lignin that form major constituents of the oil palm EFB fibre might differ depending on plant age, environment, soil condition, weather effect and testing method used (Ndazi *et al.,* 2006). Chemical compositions of EFB fibre from different researchers are shown in Table 2.9

Table 2.9: Chemical Composition of Empty Fruit Bunch (EFB)

Cellulose (%)	Hemi-cellulose (%)	Lignin (%)	Location	References
48	22	25	Malaysia	Hill & Abdul Khalil (2000)
47.9	17.1	24.9	Malaysia	Rozman *et al.,* (2005)
65	-	19	India	Sreekala (1997)

2.3 Pulping

Pulp is a fibrous material and made from wood or other lignocellulosic materials. It was prepared by chemically or mechanically separating fibres and breaks down the raw materials. Pulping is a process to produced pulp. There are three types of pulping process including chemical pulping, mechanical pulping or combined chemical and mechanical action called semichemical pulping process. All pulping processes suited for wood while non-wood materials are either chemically or semichemical. Basically, pulping processes break down the fibres structure by separating cellulose from the lignin and other non-cellulosic material (Peel, 1999).

Chemical pulps are produced from lignocellulosic material which is treated by means of appropriate chemical solution with presence of elevated temperature and pressure to remove mainly lignin and retain most of modified hemicelluloses and cellulose (Peel, 1999). Most of the lignin is removable by dissolution in chemical dry digestion process and the yield is around 40-50 % (Smook, 1992).

Meanwhile, mechanical pulping is contrary from chemical pulping that it only applies mechanical action to cut off the fibres. The mechanical action such as grind, press and refine involves energy causes the fibres to be heated (EPA, 1997; Peel, 1999). As a result, the fibres sheared to only limited damages which retained the original constituent like lignin. Therefore, papers made from mechanical pulps easily discoloured when exposed to light (Casey, 1980a).

The oldest method used in mechanical pulping (MP) is stone groundwood (SGW) process since 1960 in which the blocks of wood is pressed against wetted and roughened grinding stone (Smook, 1992). It uses only mechanical actions from machinery, pressing and water. Water was used to wash away the fibre slurry that remains on the stone surface. The concept of these mechanical actions is to break apart the structure or defiberated the block (bolt) from the wood. After around three decades, mechanical pulps were being produced by refiner methods and called as refiner mechanical pulp (RMP). The wood chips involved shredding and defibering between rotating disc in the presence of water. It was being developed by new installation of MP which employed heat such as steam pretreatment and the resultant product, called thermomechanical pulp (TMP). In fact, the heat (thermal) was therefore gave a function to presoftening the wood chips and help to loosen the fibres with very little screen reject materials and is significantly improved paper properties. Mechanical pulps are commonly used softwood due to its greater fiber properties which was larger, broader and longer than hardwood. Hardwood fibers are more severely damaged during conventional MP. Recently, chemi-thermomechanical pulping (CTMP) process as a modification of the TMP process, is being developed and applied which enabled to use certain hardwood suitable for blending into a variety of papermaking furnishes. CTMP involves presteaming with chemical treatment and first-stage refining at more than 100°C. This process improves the bonding properties of the pulp and lowers the debris content and produces high brightness pulps at reasonable costs as well.

Combining the chemical and mechanical pulping concepts, a hybrid pulping or semi-chemical or chemi-mechanical pulping is established. Semi-chemical pulps are usually prepared with pre-cooked chemical treatment followed by drastic mechanical actions such as pressing and refining. The fibres are partially delignified, eradicate extractives and some hemicelluloses content. The pulp property will depend on pulp's