UNIVERSITI PUTRA MALAYSIA

EFFECT OF HEAT TREATMENT ON CELLULOSE CRYSTALLINITY OF EMPTY FRUIT BUNCH OIL PALM FIBER AND ITS RELATION TO THE DIMENSIONAL STABILITY OF MEDIUM DENSITY FIBERBOARD

SITI MUNAWARAH BINTI ABDUL HAFID

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By

SITI MUNAWARAH BINTI ABDUL HAFID

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia in Fulfillment of Requirement for the Degree of Master of Science

November 2005

Specially dedicated to

Abang
Mak and Bapa , Ma and Ayah
My daughter and My son,
My brothers and sisters

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science.

EFFECT OF HEAT TREATMENT ON THE CELLULOSE CRYSTALLINITY OF EMPTY FRUIT BUNCH OIL PALM FIBER AND ITS RELATION TO THE DIMENSIONAL STABILITY OF MEDIUM DENSITY FIBERBOARD

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November 2005

Chairman: Professor Mohd. Hamami Sahri, PhD

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Oil Palm (*Elaeis guinensis*) is an important commercial agriculture plant in Malaysia generating a vast amount of residues, in the form of oil palm trunk (OPT), oil palm frond (OPF) and empty fruit bunch (EFB) which have high potential as raw material in wood-based industry. However, these lignocellulosic materials have poor dimensional stability i.e., high thickness swelling and water absorption. Cellulose crystallinity has been reported to influence the physical properties of the fibers. This study attempts to increase the cellulose crystallinity of oil palm EFB fiber via heat treatment and aimed i) to evaluate the effect of heating temperature on the cellulose crystallinity of oil pal EFB fiber ii) to determine the effect of cellulose crystallinity on thickness swelling (TS) and water absorption (WA) of Medium Density Fiberboard (MDF) made from heat treated oil palm fibers and iii) to establish relationship between cellulose crystallinity and TS and WA.

Oil palm EFB fibers were heated for two hours at different temperatures, 150°C 160°C, 170°C, and 180°C. Untreated samples were used as control. The crystallinity and crystallite size of the cellulose molecules were investigated using X-ray diffraction (XRD) technique. Chemical analyses were also conducted on both heat treated and untreated EFB fibers to determine the proximate amount of lignin, cellulose and hemicellulose. MDF with the size of 130mm x 130mm x 5mm were produced from these fibers. The density of the MDF was 700 Kgm^{-3}. The effects of cellulose crystallinity to the TS and WA of board were examined.

The proximate amount of chemical components in the EFB fiber in this study was found comparable with those of conventional wood. The lignin content was in the range of 20-27%, cellulose content was 40-47% and hemicellulose content was 23-25%.

The results shows that crystallinity of oil palm EFB cellulose were 21.05% for untreated fiber, 27.09% for fiber heated at 150°C, 26.68% at 160°C, 26.53% at 170^{o}C and 30.84% at 180^{o}C. The corresponding crystallite sizes of the cellulose sample were 3.22 nm, 3.42 nm, 3.64, 3.38 nm and 2.93 nm respectively. Heat treatment was found to increase the cellulose crystallinity of EFB fiber but did not markedly affected the crystallite size.

In this study, MDF made from heat treated EFB of oil palm fiber was found to have relatively high TS and WA and failed to pass the Japanese Industrial Standard (JIS A 5906 – 1983). The TS were 15.9% in the MDF from untreated fiber,16.5% for the MDF from fiber heated at 150°C,16.3% at 160°C, 15.3% at 170°C and 15.6% at 180°C. Meanwhile the WA of the MDF boards was 77.7%, 81.0%, 79.5%, 83.2% and 71.8%, respectively. It was found that the increase in temperature in this study gave no effect to the TS but reduce the WA only at temperature of 180°C. This study found that there were no trend found in the relatioship between cellulose crystallinity with TS and WA at various temperatures as the R-squares were very small. There was also no direct effect of increase in cellulose crystallinity to the TS and WA of MDF as the R-squares were very small.

Even though the cellulose crystallinity of EFB fiber increased by heat treatment, it was not sufficient to impart dimensional stability to the MDF board.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KESAN RAWATAN SUHU KE ATAS KRISTALIT SELULOSA GENTIAN TANDAN KELAPA SAWIT DAN KAITANNYA TERHADAP KESTABILAN DIMENSI MEDIUM DENSITY FIBERBOARD

Oleh

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Kelapa sawit (*Elaeis guinensis*) adalah merupakan tanaman komersil pertanian di Malaysia yang menghasilkan banyak sisa-sisa dalam bentuk batang (OPT), pelepah (OPF) dan tandan (EFB) yang mempunyai potensi sebagai bahan mentah di dalam industri berasaskan kayu. Walaubagaimanapun, bahan-bahan lignoselulosa ini mempunyai kestabilan dimensi (pembengkakan dan penyerapan air) yang rendah. Kristalit selulosa dipercayai mempengaruhi ciri-ciri fizikal gentian. Oleh itu, kajian ini adalah bertujuan untuk meningkatkan kristalit selulosa gentian tandan kelapa sawit melalui rawatan suhu dan juga untuk i) menilai kesan pemanasan ke atas kristalit selulosa gentian kelapa sawit ii) menentukan kesan-kesan kristalit selulosa ke atas pembengkakan dan penyerapan air oleh MDF yang dibuat dari gentian kelapa sawit yang telah dirawat dengan suhu dan iii) menghasilkan hubungan di antara kristalit selulosa dengan pembengkakan dan penyerapan air.

Gentian tandan kelapa sawit dirawat dengan suhu pada tahap yang berlainan iaitu 150°C, 160°C, 170°C and 180°C selama dua jam.Sampel tanpa rawatan suhu juga disediakan sebagai kawalan. Kristalit dan saiz kristalit molekul selulosa di dalam gentian kelapa sawit dikaji menggunakan teknik ‘X-ray diffraction (XRD)’. Analisis kimia juga dijalankan ke atas gentian tandan kelapa sawit tersebut untuk menentukan kandungan lignin, selulosa dan juga hemiselulosa. Papan MDF bersaiz 130mm x 130mm x 5mm dan mempunyai 700 Kgm^{-3} ketumpatan juga dihasilkan dari gentian tandan kelapa sawit tersebut. Kesan kristalit selulosa ke atas pembengkakan dan penyerapan air oleh papan juga dikaji.

Kandungan komponen kimia gentian tandan kelapa sawit yang dianggarkan melalui kajian ini didapati lebih kurang sama dengan kayu biasa. Kandungan ligninnya adalah di antara 20-27%, kandungan selulosa adalah 40-47% dan kandungan hemiselulosanya pula adalah 23-25%.

Hasil daripada kajian menunjukkan kristalit di dalam selulosa gentian kelapa sawit adalah 21.05% di dalam gentian tanpa rawatan, 27.09% untuk gentian yang telah dirawat pada 150°C, 26.68% pada 160°C, 26.53% pada 170°C dan 30.84% pada 180°C. Saiz kristalit sampel selulosa masing-masing adalah 3.22 nm, 3.42 nm, 3.38 nm dan 2.93 nm. Rawatan suhu telah meningkatkan kristalit selulosa gentian tandan kelapa sawit tetapi tidak memberi kesan ke atas saiz kristalit.

Kajian ini juga menunjukkan bahawa MDF daripada gentian tandan kelapa sawit mempunyai pembengkakan dan penyerapan air yang tinggi dan tidak dapat memenuhi keperluan yang digariskan oleh Japanese Industrial Standard (JIS A 5906 1983). Pembengkakan ialah 15.9% bagi papan MDF yang dibuat daripada gentian yang tidak dirawat,16.5% pada papan MDF dari gentian yang telah rawat pada 150°C, 16.3% pada 160°C, 15.3% pada 170°C dan 15.6% pada 180°C. Manakala penyerapan air papan MDF masing-masing adalah 77.7%, 81.0%, 79.5%, 83.2%dan 71.8%. Peningkatan suhu didapati tidak mempunyai kesan ke atas tahap pembengkakan papan MDF tetapi tahap penyerapan air papan didapati berkurang pada suhu 180°C. Kajian ini mendapati bahawa hubungan antara kristalit selulosa dengan tahap pembengkakan dan penyerapan air papan MDF pada kebanyakan tahap suhu tidak mempunyai tren kerana R-square yang diperolehi terlalu kecil. Kajian ini juga mendapati tidak ada hubungan di antara peningkatan kristalit selulosa dengan pembengkakan dan penyerapan air papan MDF kerana R-square yang diperolehi juga terlalu kecil.

Kristalit selulosa gentian kelapa sawit meningkat dengan rawatan suhu tetapi, tidak dapat memberikan kestabilan dimensi kepada papan MDF.

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I certify that an Examination Committee has met on 22 November 2005 to conduct the final examination of Siti Munawarah Binti Abdul Hafid on her Master of Science thesis entitled "Effect of Heat Treatment on Cellulose Crystallinity of Empty Fruit Bunch Oil Palm Fiber and Its Relation to the Dimensional Stability of Medium Density Fiberboard" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not previously or concurrently submitted for any degree at UPM or other institutions.

SITI MUNAWARAH BINTI ABDUL HAFID

Date:

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LIST OF ABBREVIATIONS

Å	Angstrom
BS	British Standard
CaO	calsium oksida
Cm^2/g	centimeter per square gram
EMB	European Stamdard
g	gram
IB	Internal Bond
JIS	Japanese Standard
Kg/m^2	Square kilogram per meter
K_2O	kalium oksida
Kg/m^3	Cubic kilogram per meter
MOE	Modulus of Elasticity
MOR	Modulus of Rupture
MPa	Mega Pascal
MgO	Magnesium Oksida
N	Newton
N_2	Nitrogen
NPA	National Particleboard Association

CHAPTER 1

INTRODUCTION

1.1 General

Oil Palm (*Elaeis guinensis*) is an important commercial agriculture plant in Malaysia and originated from West Africa. The main product from this plant is crude palm oil, which can be further processed into vegetable oil and other industrial and food products. Malaysia is among the highest producer of palm oil production and this is attributed to the climate and good management arising from research and development (R&D) (Yusof and Chan, 2004). The other producing countries are Indonesia, Colombia, Ivory Cost and Papua New Guinea (Yusof and Mohd. Arif, 2005).

Malaysia has over 3.6 million hectares of land covered by oil palm cultivation and the oil only consists about 10% of total biomass produced in the plantation (Mohamad Husin *et al*., 2002). Therefore, the oil palm plantation generating a large amount of residue in the form of trunk (Oil Palm Trunk), fronds (Oil Palm Frond) and fruit bunches (Empty Fruit Bunch). Mohamad Husin *et al.* (2002) estimated that there were about eight million tones of oil palm trunk (OPT), 22 million tones of oil palm frond (OPF) and about five million tones of empty fruit bunch (Table 1.1). This large amount of residues consists of lignocellulosic materials (Mohd. Zin and Imamura, 1989; Yusof and Muhamad Husin., 1997) and therefore provide an alternative resource for the wood industry especially in wood composite board industry to complement of impending shortage of rubberwood supply.

Table 1.1: Estimated Oil Palm Biomass Supply in Malaysia in 2002

Oil Palm Biomass	**Supply (t/y dry weight) (million tones)**
Oil Palm Trunk (OPT)	8
Oil Pal Frond (OPF)	22
Empty Fruit Bunch (EFB)	5

Source: Mohamad Husin *et al.*, (2002)

Generally, wood is made up of three important organic compounds called cellulose, hemicellulose and lignin. Among these three components, cellulose is the most important as it constitutes slightly less then one half the weights of both hardwoods and softwoods. Cellulose is also the main chemical constituent in most wood (Sjöström, 1981).

The reactivity of cellulose is affected by its two-phase morphology i.e crystalline and amorphous regions. Sjöström (1981) indicated that this morphology played major roles on cellulose reactivity. The hydroxyl groups located in the amorphous regions were highly accessible and react readily while those in the crystalline regions with closely packed and strong interchain bonding can be completely inaccessible. Therefore, this morphological nature may affect the composite board in term of its dimensional changes. Crystallinity is very important factor, which in turn affect the physical properties of the board (Wadsworth and Cuculo, 1978).

Realising the importance of cellulose crystallinity, a number of researches have been conducted which covers many aspects. Crystallinity index, is one of the importances which related to the degree of crystallinity can be determined and evaluated. There are many techniques to determine the proportion of crystalline and non-crystalline (amorphous) in the cellulose molecules. There were three main methods of determinations, i.e. the physical, swelling and non-swelling methods are available to measure the degree of crystallinity in the cellulose molecules (Wadsworth and Cuculo., 1978). The physical method, i.e. x-ray diffraction method is the most popular to determine the cellulose crystallinity. With x-ray, it is possible to determine the finer structure in crystallite materials (Stamm, 1964). Krässig (1993) also stated that it is the most versatile method of determining the structure characteristics of cellulose substrate in the small as well as in the wide angle region. In the wide angle region (beyond $2\theta=6^{\circ}$), the x-ray diffraction allows the determination of the degree of crystallinity, the degree of orientation and from the width at half maximum of the meridional and equatorial reflections, the crystallite length and width dimension respectively (Krässig, 1993).

1.2 Justification

A large amount of lignocellulosic residues from oil palm tree will be available in the future. These residues include trunks, leaf, fruit bunches and fronds consist of solid and fibrous raw materials. Oil palm trunks, which considered being readily available non-wood lignocellulosic material, may be used as a compliment or a substitute to the timber resources especially for production of wood composite products.

Generally, oil palm residues or oil palm by-products can be converted into fibrous strands and can be utilised in the production of composite board such as cementboard, particleboard, fibreboard, blockboard and others. Mohd. Husin *et al*. (1985) noted that oil palm fibre is short and thin but it is comparable in length to the fibre of rubberwood, the conventional source of raw material in the wood industry (Table 1.2). Salleh (1994) indicated that the oil palm strands also have comparable cellulose content to the average Malaysian hardwoods. Although, the mean fibre length of oil palm strands is much shorter than most Malaysian hardwoods, the fibres width is almost similar with those of medium density or heavy density hardwoods and the cell wall thickness is similar to that of light density hardwood. Therefore oil palm fibre provides an alternative source of raw material in the wood composite industry.

Table 1.2: Fibre dimension of oil palm trunks, fronds and bunches in comparison to rubberwood

Dimension	**Part**			
	Trunks	**Fronds**	**Bunches**	**Rubberwood**
Length (mm)	1.22	1.52	0.89	1.50
Width (mic)	35.3	-	25.0	40.0
Cell wall thickness (mic)	4.5	-	2.8	0.50

Source: Mohamad. Husin *et al*. (1985)