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OIL PALM TRUNK PLYWOOD PRODUCTION USING COPOLYMERIZED AND MODIFIED PHENOLIC RESINS

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By

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OIL PALM TRUNK PLYWOOD PRODUCTION USING COPOLYMERIZED AND MODIFIED PHENOLIC RESINS

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Chairman: **Prof. Luqman Chuah Abd, PhD**

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Nowadays, the use of waste material such as, saw dust, rice husk, coconut coir, empty fruit brunch (EFB), oil palm mass and oil palm stem (OPT) as alternative material for wood-based industry in producing various commercial product have been extensively explored. Nevertheless, the used of OPT as raw material replacing hardwood species in plywood production has been in practice for the past 10 years. However, high resin consumption and low mechanical properties in OPT plywood are still the limitation. Hence, in this study we explored the potential of a new resin treatment approach using LmwPF in order to produce high grade OPT plywood. In this work, the effects of several factors such as properties such as, thickness swelling, water absorption, hot-press pressure, bonding integrity, density, the modulus of rupture (MOR) and modulus of elasticity (MOE).

The LmwPF resin treatment of OPT in plywood production indicated that with this new resin treatment method (1st part of pilot scare study), improvement of $\geq$ 200% in strength, $\geq$ 259% in stiffness, dimensional stability ($\geq$ 6% thickness swelling and $\geq$ 36% water absorption) as well as, 28% and 80% greater in dry and WBP shear under hot-pressing pressure at 20 bar at the first 5 min and increased to 50 bar for the next 9 min, respectively as compare to the conventional method of commercial OPT plywood. While the 2nd part of pilot scale study showed that, the mechanical properties and bonding performance of the pre-preg OPT plywood were influenced by the different pressing time (14, 16, 18 and 20 min). The high grade OPT plywood with improved at least 227 % MOR and 348 % MOE compared to commercial OPT plywood, with greater in MOR (31 %) and MOE (12 %) higher compared than the commercial tropical mix light hardwood (MLHW) plywood at 20 min hot-pressing time with most optimum hot-pressing pressure obtained from previous study.

Moreover, the performance of the formaldehyde emission, some mechanical properties and bonding quality of oil palm trunk (OPT) plywood treated with low molecular weight phenol-formaldehyde (LmwPF), as affected by resin concentration. The mechanical properties are affected by different of amount resin contain used. The OPT veneer were treated at either 40 %, 32 %, 23 % or 15 % of resin concentration and 12 mm thickness of 3-ply plywood panel were manufactured for

each group. In this study the formaldehyde emission, modulus of rupture (MOR), modulus of elasticity (MOE) and bonding quality (shear strength) of OPT plywood were determined. The results revealed that the resin-treatment method tended to significantly improved the mechanical properties of the OPT plywood panel in which increased solid absorption gives better mechanical properties. Apparently, high mechanical properties were obtained for panel manufacturer from veneer treated with 32 % and 40 % resin content. However, mechanical properties of the resin-treated OPT plywood were drastically decreased with increasing the water substitution. Formaldehyde emission content of OPT panels decreased upon reduction of resin content into treatment process and were significant at resin concentration. The resin-treated OPT panels at 32 % solid content provided a reasonable amount of free formaldehyde (0.359 mg/l) which attained F**** according to Japanese Agriculture Standard (JAS).

The matrix-assisted laser desorption/ionisation time of flight (MALDI-TOF) mass spectrometry (MS) and ^{13}C nuclear magnetic resonance (NMR) spectroscopic technique were used to characterize synthesis Phenol-Urea-Formaldehyde (PUF) resin. The MALDI-TOF-MS illustrated and confirmed a series number of the phenol-urea co-condensates repeating unit exit in the prepared PUF resins which corroborated well with the mechanical properties (Modulus of Elasticity and Modulus of Rupture), bonding quality (Dry test and WBP) and physical performance test results. A series of PF, UF and PUF resins oligomers forms repeating unit up to 1833 Da were identified. Besides that, the Solid Stated ^{13}C NMR interpretation indentified that the signal at 44-45 ppm and 54-55 ppm corresponding to methylene bridges was co-condensed in between phenol to urea in the PUF resin system. The ^{13}C NMR investigation showed that the synthesis process of PUF resin had no free formaldehyde elements. In addition, the proportion of urea and methylolureas in the mixture to synthesis PUF resin are sufficient and well corporated into the formulation by reacting with LmwPF units to form co-condensed methylene bridges.

The output of this pilot scale study proved that high performance OPT plywood could produced through pre-treatment method in the current plywood mills in which provides broader area of applications compared with conventional OPT plywood. For instant, the pre-preg OPT plywood which is suitable for structural application, concrete formwork, heavy duty interior structuring board, load bearing plywood, marine grade plywood, was obtained, thus consequently increases the price of OPT plywood panels.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
Sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PENGHASILAN PAPAN LAPIS BATANG POKOK MINYAK KELAPDA SAWIT DENGAN MENGGUNAKAN POLIMERISASIKAN DAN UBAHSUAI RESIN FENOLIK

oleh

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November 2014

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Pada masa kini, penggunaan bahan buangan seperti habuk, sekam padi, sabut kelapa, tandan kelapa sawit (TKS), jisim kelapa sawit dan batang kelapa sawit (BKS) sebagai bahan alternatif untuk industri berasaskan kayu dalam menghasilkan pelbagai produk komersial telah diterokai secara meluas. Walau bagaimanapun, BKS digunakan sebagai bahan mentah menggantikan spesies kayu keras dalam pengeluaran papan lapis telah diamalkan sejak 10 tahun yang lalu. Walau bagaimanapun, penggunaan resin yang tinggi dan sifat mekanikal rendah pada BKS papan lapis masih terbatas. Oleh itu, dalam kajian ini kita menerokai potensi pendekatan rawatan resin baru menggunakan LmwPF untuk menghasilkan gred tinggi BKS papan lapis. Dalam karya ini, kesan beberapa faktor seperti ciri-ciri, ketebalan, penyerapan air, tekanan panas-tekan, integriti ikatan, ketumpatan, modulus pecahan dan modulus kekenyalan .

Rawatan resin LmwPF pada BKS untuk pengeluaran papan lapis menunjukkan bahawa dengan ini kaedah baru rawatan resin (kajian pertama yang berskala besar), peningkatan > 200 % dalam kekuatan > 259 % dalam ketegangan, kestabilan dimensi (> 6% pembengkakan ketebalan dan > 36 % penyerapan air) dan juga, 28 % dan 80 % lebih besar dalam keadaan kering dan WBP dalam tekanan masa panas iaitu 20 bar pada 5 minute yang pertama dan menaikan kepada 50 bar pada 9 minute yang kemudian, masing-masing berbanding dengan kaedah konvensional BKS papan lapis komersial. Sebaliknya, kajian kedua yang berskala besar menunjukan Hasil kajian menunjukkan bahawa, sifat-sifat mekanikal dan prestasi ikatan pra-preg BKS papan lapis telah dipengaruhi oleh tekanan masa (14 minute, 16 minute, 18 minute and 20 minute). Gred tinggi BKS papan lapis dengan peningkatan MOR sekurang-kurangnya 227 % dan 348 % berbanding dengan MOE BKS papan lapis komersial, dengan lebih tinggi dalam MOR (31 %) dan KPM (12 %) berbanding daripada komersial tropika campuran kayu keras (MLHW) pada tekanan masa panas selama 20 minute bersama dengan tekanan optima daripada keputusan kajian berskala besar yang sebelum itu.

Selain itu, pelaksanaan pelepasan formaldehid, beberapa sifat mekanik dan kualiti ikatan batang kelapa sawit (BKS) papan lapis dirawat dengan berat molekul yang rendah pada fenol-formaldehid (LmwPF), yang dipengaruhi oleh kepekatan resin. Sifat-sifat mekanikal dipengaruhi oleh berlainan jumlah kandungan resin yang digunakan. Lapisan BKS dirawat sama ada 40 %, 32 %, 23 % atau 15 % daripada kepekatan resin dan ketebalan 12 mm 3-lapis papan lapis panel telah dihasilkan untuk setiap kumpulan. Dalam kajian ini pelepasan formaldehid, modulus pecahan (MOR), modulus kekenyalan (MOE) dan kualiti ikatan (kekuatan ricih) BKS papan lapis telah ditentukan. Keputusan menunjukkan bahawa kaedah resin rawatan telah cenderung untuk menambah baik sifat-sifat mekanik panel papan lapis BKS di mana peningkatan penyerapan yang kukuh dengan ketara memberikan sifat mekanikal yang lebih baik. Rupa-rupanya, sifat mekanik yang tinggi diperolehi bagi panel pengeluar dari lapisan yang dirawat dengan 32 % dan 40 % kandungan resin. Manakala, sifat mekanik resin BKS papan lapis yang dirawat telah secara drastik mengurangkan peningkatan penggantian air. Kandungan pelepasan formaldehid panel BKS menurun apabila pengurangan kandungan resin ke dalam proses rawatan dan pada kepekatan resin. Panel BKS resin dirawat pada 32 % kandungan pepejal dengan syarat bebas formaldehid (0.359 mg/l) yang dicapai F **** berdasarkan Japanese Agriculture Standard (JAS 233).

Matrix dengan bantuan laser desorption / pengionan penerbangan (MALDI-TOF) spektrometri jisim (MS) dan ^{13}C resonans magnetik nuklear (NMR) teknik spektroskopi telah digunakan untuk mencirikan resin sintesis Fenol-Urea-Formaldehyde (PUF). MALDI-TOF-MS digambarkan dan mengesahkan beberapa siri fenol-urea bersama kondensat mengulangi unit keluar dalam resin PUF yang disokong dengan baik dengan sifat-sifat mekanikal (Modulus Keanjalan dan Modulus pecah), kualiti ikatan (ujian kering dan WBP) dan keputusan ujian prestasi fizikal. Satu siri PF, UF dan PUF resin oligomers bentuk unit berulang sehingga 1833 Da telah dikenal pasti. Di samping itu, dinyatakan ^{13}C NMR tafsiran pepejal dikenalpasti bahawa isyarat pada 44-45 ppm dan 54-55 ppm bersamaan dengan ikatan metilena telah bersama kondensat di antara fenol kepada urea dalam sistem resin PUF. Siasatan ^{13}C NMR menunjukkan bahawa proses sintesis resin PUF tidak mempunyai unsur-unsur bebas formaldehid. Di samping itu, peratusan urea dan methylolureas dalam campuran untuk sintesis resin PUF yang mencukupi dan juga sesuai di dalam penggubalan tindak balas dengan unit LmwPF untuk membentuk ikatan metilena.

Daripada hasil kajian ini pada skala yang besar membuktikan bahawa prestasi tinggi papan lapis BKS boleh dihasilkan melalui kaedah pra-rawatan di kilang-kilang papan lapis sedia ada dalam yang menyediakan kemudahan yang cukup berbanding dengan papan lapis OPT konvensional. Bagi pra-preg OPT papan lapis sesuai untuk aplikasi struktur, acuan konkrit, penstrukturan dalaman besi yang keras, ujian kekuatan papan lapis, papan lapis gred marin, telah diperolehi, dan seterusnya meningkatkan harga panel papan lapis KBS.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the Doctor of Philosophy. The members of the Supervisory Committee were as follows.

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LmwPF	Low molecular weight phenol formaldehyde
UF	Urea formaldehyde
PF	Phenol Formaldehyde
MALDI-TOF	Matrix-assisted lasers desorption/Ionization time of flight
MS	Mass spectroscopy
DP	Degree of polymerization
OPT	Oil palm trunk
MW	Molecular weight
NMR	Nuclear magnetic resonance
MOE	Modulus of elasticity
MOR	Modulus of rupture
Da	Dalton
PDI	Polydispersity index
ANOVA	An analysis of variance
MDF	Medium density fibreboards
WPC	Wood plastic composite
hr	hr
min	minute
sec	second
L	litre
ml	millilitre
g	gram
mg	milligram
µl	micro litre
%	percent
cm	centimetre
mm	millimetre

MPa	Mega pascal
˚C	Degree celcius
MLHW	Mixed light hardwoods
–OH	Hydroxyl group
Kg	Kilogram
WPG	Weight percent gain

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Plywood industry in Malaysia

The plywood industry has been the 2nd largest wood-base industry in Malaysia after the wooden furniture industry which has contributed approximately RM 6.52 Billion (28 % of total timber export) through worldwide exports. In year 2010, the plywood industry in Malaysia contributed up to RM 5.15 Billion export revenue based on the total plywood production which was amounted at 480,000 m3. Statistically reported by Malaysia Timber Industry board (MTIB, 2010), the major country of plywood exporter includes, Japan (42 %), Korea (14.1 %), Taiwan (10 %), Yewen (2.4 %), USA (4.4 %), UAE (2.4 %), United Kingdom (4.4 %), Egypt (2.1 %), Philippines (2.3 %) and others (13.7 %). At local market, the traded price of plywood is currently about RM 16.10, RM 25.60 and RM 36.50 for the plywood thickness at 4 mm, 6 mm and 9 mm per piece respectively. Meanwhile, shuttering boards of 12 mm of thickness were traded at RM 45.50 per piece. The commonly used types of wood species in Malaysia's plywood industry are Mempisang, Kedondong, Chengal, Meranti (*Shorea* spp.), Keruing, Jelutong, Seseduk, Senduduk and etc.

Oil palm plantation in Malaysia

The total oil palm planted area in Malaysia is shown in Figure 1.1. Based on the Figure 1.1, the total oil palm plantation in Malaysia is grown by 16 % to 5.8 million hectares in 2012 compared to 5.0 million hectare in 2011. The expansion of oil palm plantation in drastically mainly in Sabah and Sarawak then followed by Peninsular Malaysia. The total plantation area Sabah and Sarawak growth of 2.7 % or 65,477 hectares compared to 0.4 % or 11,343 hectares in Peninsular Malaysia. No doubt, Sabah is the largest oil palm planted state with 1.44 million hectares or 28.4 % of the total planted area followed by Sarawak with estimated 1.08 million hectares (21.2 %), Johor - the largest oil palm in Peninsular Malaysia (0.71 million hectares or 14.4 % and Pahang state is planted 0.70 million hectares (13.8 %).

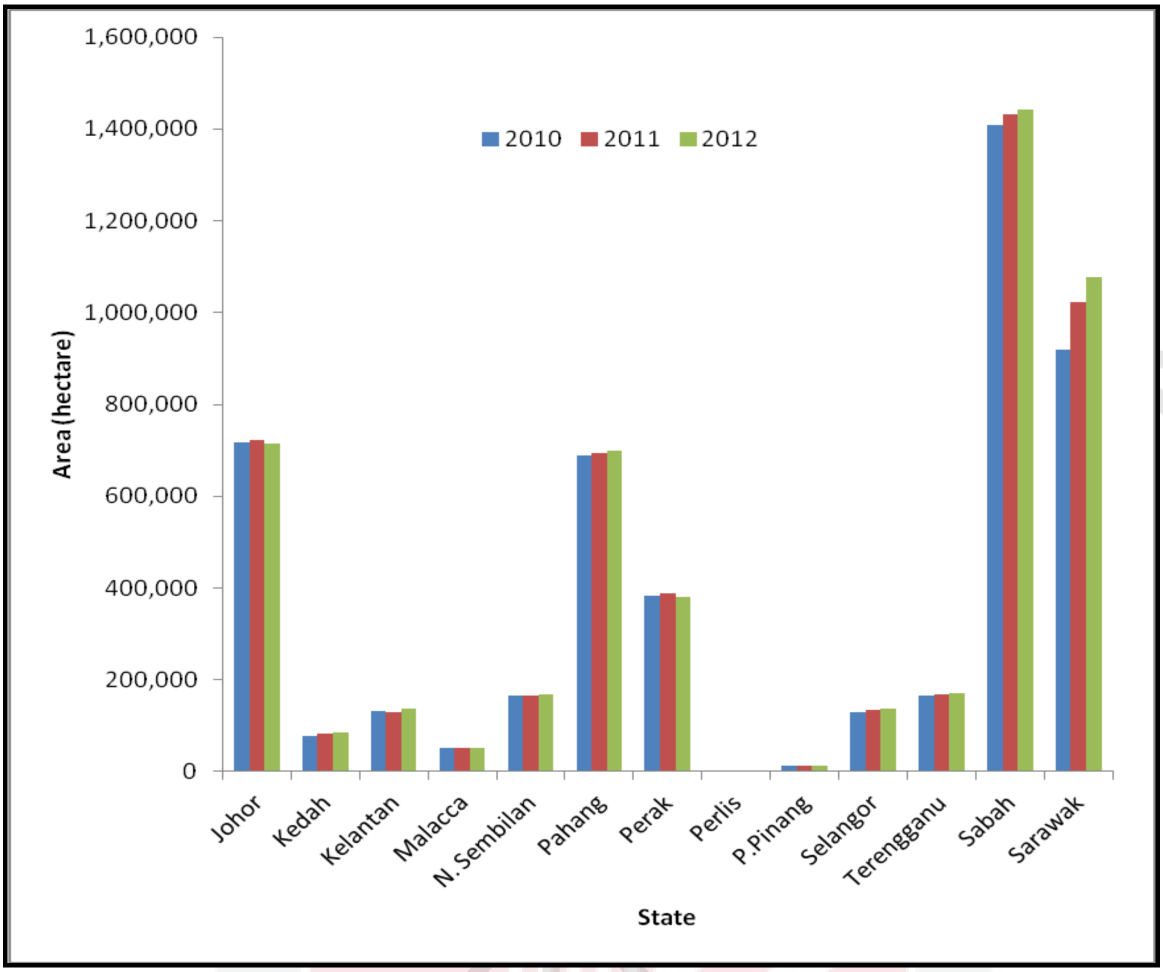

Figure 1.1. Distribution of oil palm plantation area in Malaysia in year 2010 to 2012. *Source*: (MPOB, 2010)

Based on the Figure 1.2 and data from Malaysian Palm Oil Board (MPOB), the expansion in planted area in 2011 was mainly attributed by private estate and smallholders with growth of 7.1 % and 3.5 %. Form the ownership perspective, the private estates covered 3.04 million hectares (14.1 %) followed by the independent smallholder with 0.70 million hectares (14.0 %). The State Schemes owned 0.32 million hectares (6.4 %), 0.16 million hectares or 3.2 % owned by FELCA and RISDA 0.08 million hectares (1.6 %).

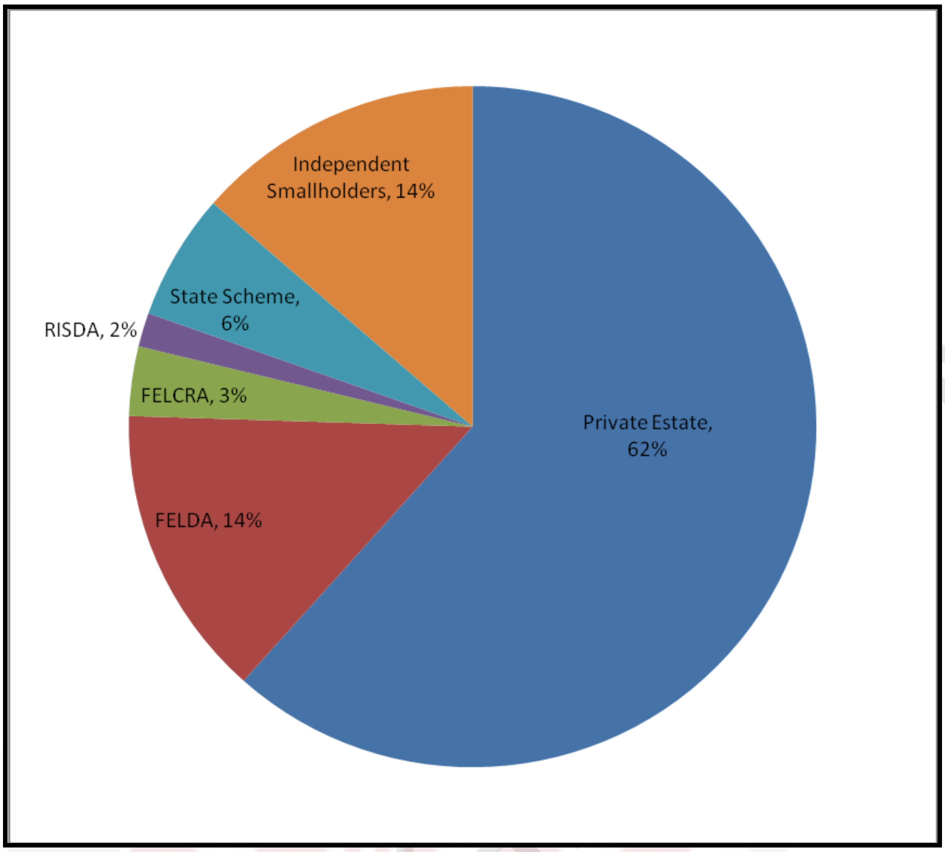

Figure 1.2. Oil palm planted area according to category in year 2012 (hectares). ***Source*****: (MPOB, 2010)**

Available of oil palm trunk

No doubt, oil palm tree is an ingenious resource which can be obtained at any plantation in Malaysia. Oil palm trees were felled every 25 years due to the reduction in fruit oil production. In general, these agriculture residues were chopped into small disk and left in plantations for natural degradation which serves the purpose biomass as fertilizer for next replanting rotation. Base on the annually statistic published data (Figure 1.3) of replanting activity in oil palm plantation by MPOB, its tend to compute and project the total oil palm tree that have reach replanting age at 25 year.

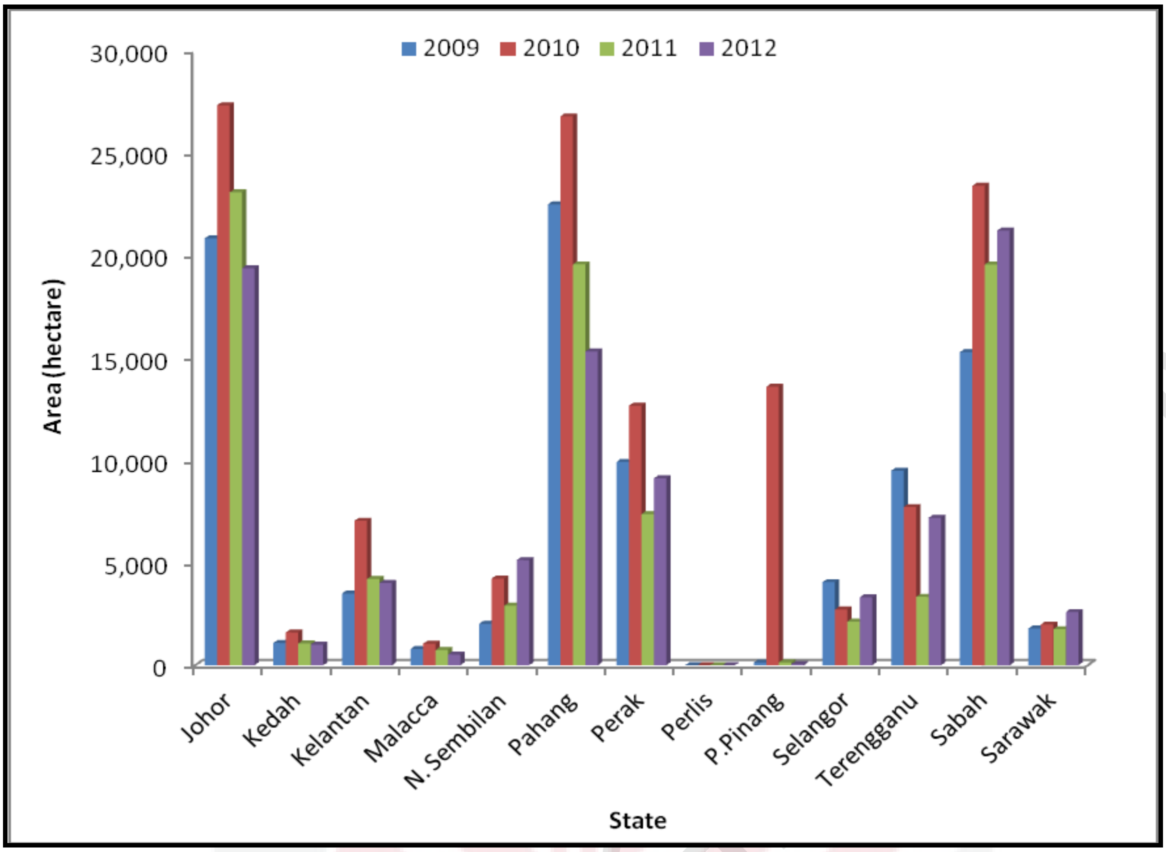

Figure 1.3. Replanting areas in each state of Malaysia from year 2009 to 2012.
***Source*: (MPOB, 2010)**

The estimation is base on the current practical that a hectare of plantation is consisted of an average of 140 trees. Referring to the estimation of the replanting area, the amount of available of OPT in Peninsular Malaysia is ranging from 12 million tree (2014 year) and 4 to 9 million tree at the year from 2015 to 2026. In year 2024, Malaysia is estimated about 32 million of oil palm tree is generate from the replanting activities (Figure 1.4)

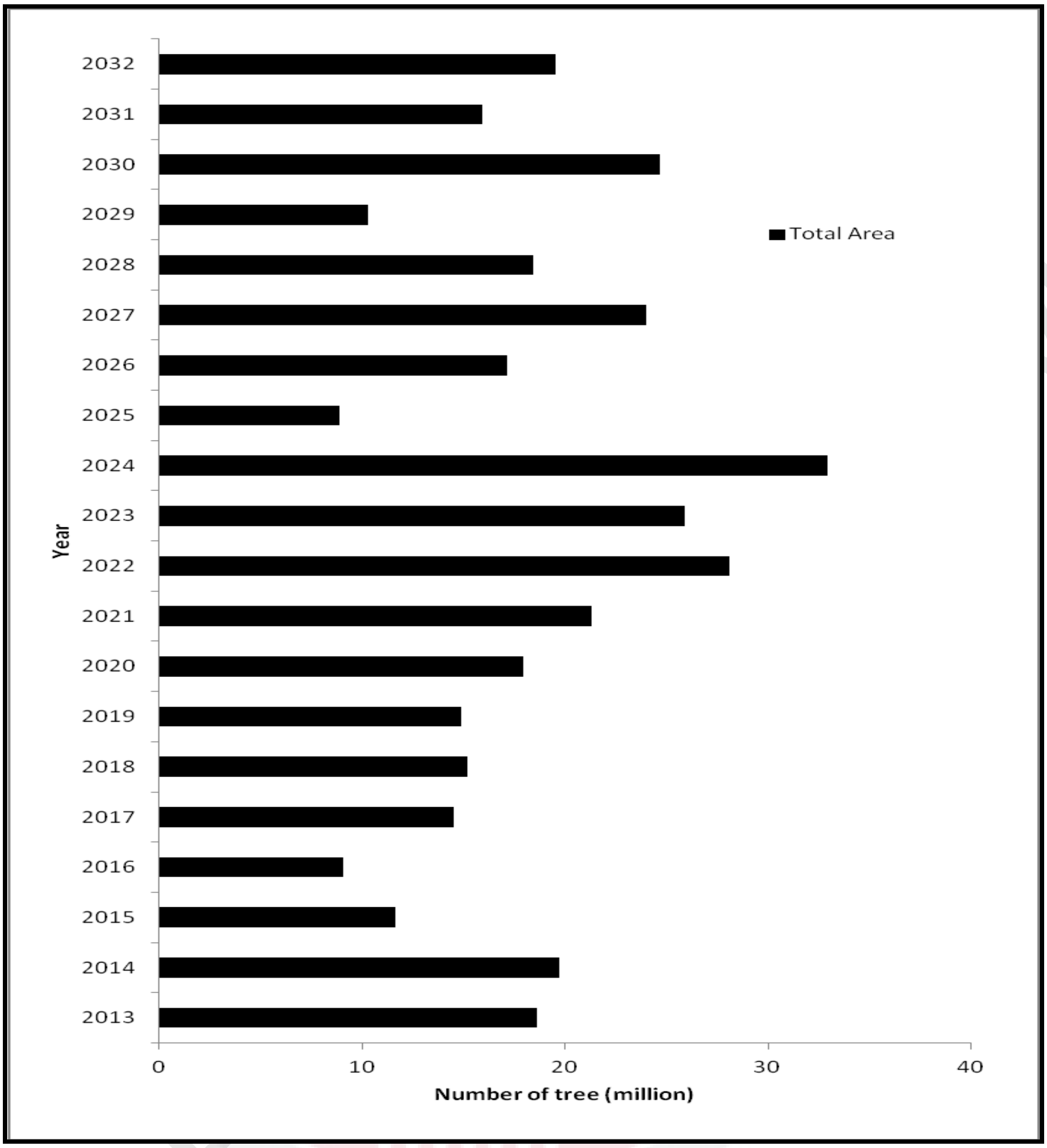

Figure 1.4. Total estimated replanting area from year 2011 to 2032. (MPOB, 2010)

Oil palm trunk (OPT)

One of the most potential raw materials for local plywood industry which is abundant and readily available in Malaysia is OPT as shown in Figure 1.5. Oil palm *(Elaeis guineensis*) tree are one type of palms that are grown for oil production. The trees after 20-30 years old where the oil production is declining, there are considered uneconomic and have to be replanted. The replanting activities generate huge amount of oil palm stems.

From the annual report (MPOB, 2010), Malaysia produced about 21.63 million cubic meters of oil palm biomass, including trunks, fronds, and empty fruit bunches. This figure is expected to increase substantially when the total planted hectare of oil palm in Malaysia will reach 5.10 million hectares in 2020. The total oil palm planted area in Malaysia has expanded from merely 1.7 million hectares in 1990 to 3.37 million hectares in 2002 to 4.3 million hectares in 2006 and to 5.0 million hectares in 2012. The annual availability of OPT is estimated to be around 13.6 million logs based on 100,000 hectares of replanting each year. Under controlled processing conditions, this amount of OPT could be converted into approximately 4.5 million m^3 of plywood each year.

Figure 1.5. Oil palm trunk from plantation.

1.2 Problem statement and justification

The matter of fact, oil palm trunk has been used as an alternative raw material in wood-based industry since last 10 years due to shortage of forest timber. However, these alternatives are not so wildly utilized at wood based industry in Malaysia due to its natural poor mechanical properties. Apparently, there are 5 OPT plywood manufacturers in Peninsular Malaysia (Central Kedah Plywood Factory Sdn. Bhs, Plus Interest Sdn. Bhd., Nippon Palm Sdn. Bhd., Gerbang Mahsur Sdn. Bhd. and KamSeng Enterprise) with very low market competitive and approximately production of 200-1000 m^3/month. In fact, if compared with the LDHW plywood, the usage of oil palm veneer and other form of oil palm fiber has always been down-graded to the production of non-structural materials. This problem may be associated with density variations inside the stem itself as well as the cell structure found in OPT fibers. The distribution of vascular bundles and parenchyma ratio the outer zone of the OPT is consolidated by 51 % of high density vascular bundles; whilst the center zone is comprised with 70 % of soften parenchyma tissues, which are low in density (Bakar et al., 1998). In addition, the research finding also reported that the bottom-outer part of OPT of more dense than the top-outer part along the OPT. Even though several companies have initiated commercial production of OPT based products (mainly veneer-based), the problem of mechanical properties and dimensional stability during post production has slowed down full commercialization.

The resin treatment of wood by phenolic resin to enhance the mechanical properties, dimensional stability, bonding properties of final product has been studied by those researchers (Ab Wahab et al., 2012; Loh et al., 2011a; 2011b; 2011c, Nor Hafizah et al., 2009; Paridah et al., 2006). The low molecular weight resin has relatively, short chain, smaller molecules, and can easy penetrate onto wood cell, once its cured, thus improved the mechanical strength. Nevertheless, the main drawback of treatment with LmwPF resin is a much longer pressing time during plywood manufacturing. The LmwPF resin contains a high number of methylol groups in the main polymer chain compared to commercial PF resin, thus a longer time is required to cure the resin and production cost still remain high or uneconomically. Another drawback if the treatment method with LmwPF resin might generate high emission of unbond or free formaldehyde due to, the resin is in "half-cook" condition, and presented in a very large amount of short molecular chain which needed longer hot-pressing time to fully polymerization. Formaldehyde gas is emitted from wide range of natural and human-made products. Formaldehyde is an organic compound and can be toxic, irritating, allergenic, and carcinogenic cause to human (IARC, 2006). Formaldehyde has been significant effluent with human health problems either short or long term exposure to the gas. Nonetheless, the effect of important setting parameter including hot-pressing pressure, hot-pressing time and resin solid content of the resin treatment methods on the mechanical properties, bonding quality as well as formaldehyde emission from the OPT plywood are not being reported by using resin treatment methods.

1.3 Objective

The main objective of this study was to investigate the OPT as raw material for plywood production and improve this material through the wood treatment method. The study comprised two aspects: (1) Process optimization and (2) Development new method of properties enhancement. In this task, the phenolic-based resin is used as treatment agent to enhance the physical properties, mechanical properties, bonding quality and formaldehyde emission content of the resin treated OPT plywood. The specific objectives were:

Specific objectives for process optimization:

1. To investigate the effect of moisture content, resin uptake, solid content, of OPT veneer through pre-treatment with phenolic-based resins.
2. To determine the optimum condition of hot pressing pressure and hot temperature for producing phenolic resin treated OPT plywood.
3. To evaluate the effect of resin solid content on formaldehyde emission, strength and bond integrity of resin-treated OPT plywood.

Specific objectives for develop new treatment method:

4. To develop new resin synthesis method and its effect on bonding integrity, and mechanical properties of the treated OPT plywood.

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