



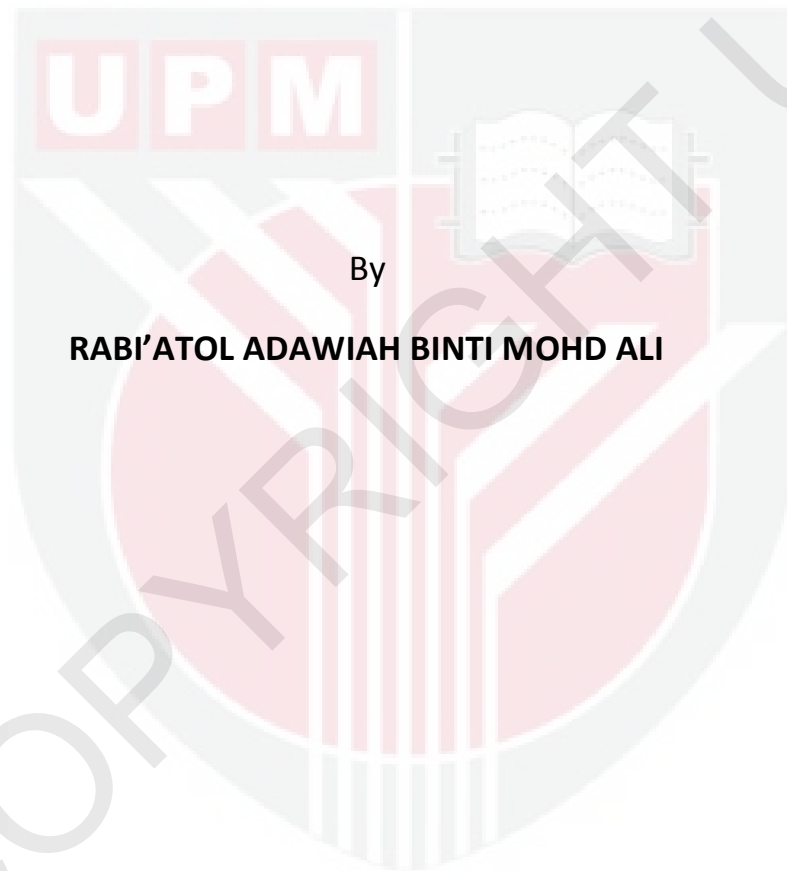
UNIVERSITI PUTRA MALAYSIA

***FORMALDEHYDE EMISSION AND PROPERTIES OF COMPREGNATED
WOOD TREATED WITH LOW MOLECULAR WEIGHT PHENOL
FORMALDEHYDE WITH ADDITION OF UREA***

RABI'ATOL ADAWIAH BINTI MOHD ALI

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TREATED WITH LOW MOLECULAR WEIGHT PHENOL FORMALDEHYDE WITH
ADDITION OF UREA**



By

RABI'ATOL ADAWIAH BINTI MOHD ALI

June 2012

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

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Phenol formaldehyde (PF) resin impregnation and compression at considerable high hot pressing pressure of jelutong and sesenduk is attractive for the improvement of strength properties, dimensional stability and decay durability. The formaldehyde emission (FE) from the treated materials is, however, very high especially when low molecular weight resin is used. Attempt to reduce the formaldehyde emission was made by introducing urea in the treating solution. In this study,

urea was used to scavenge the excess formaldehyde produce from the PF-compregnated wood. The urea was preferable as scavenger since it is efficient in reducing formaldehyde emission and low manufacturing cost. The experimental design of this study included impregnation of jelutong (*Dyera costulata*) and sesenduk (*Endospermum diadenum*) strips with 20 %, 30 % and 40 % low molecular weight phenol formaldehyde (LmwPF, M_w 600) mixed separately with urea (30% based on solid PF), pre-curing at 60°C for certain periods (7 h, 8 h and 9 h) and subsequently compressed to a compression ratio (CR) of 80 % in a hot press temperature of 150°C for 20 min. The formaldehyde emission, physical and mechanical properties and durability of the *compreg* wood for each treatment combination was analyzed. Addition of urea into PF resin successfully reduced formaldehyde emission by 97 % for both *compreg* woods. The results also showed the dimensional stability of the *compreg* jelutong and sesenduk increased by 60 % and 52 % in anti swelling efficiency (ASE), respectively. When compared between strips treated with and without the presence of urea, the efficiency in preventing swelling of *compregs* treated with PF solution admixed with urea was more successful. Based on the *compreg* properties evaluated, the optimum treatment combination of fabricating jelutong and sesenduk was using 30 % PF mixed with 30% urea (based on solid PF), precuring at 60°C for 8 h followed by compressing to 80 % CR at 150°C for 20 min. This treatment combination was then applied to produce *compreg* laminates. Three layers laminated *compreg* jelutong and sesenduk were fabricated by assembling the impregnated strips perpendicular (cross) or parallel to each other, followed by compression in a hot press. Compared to the untreated solid wood of the same size, both parallel and cross laminated *compreg* wood had superior properties. The density of the *compreg* wood increased by 78 % to 117 % from its original densities. The modulus of rupture (MOR) and modulus of elasticity (MOE) were increased by 24 %, except for the parallel laminates of sesenduk which had lower values than the untreated solid wood. The study also revealed that shear strength at the bonding glueline was higher for the parallel than for the cross laminated *compreg* wood. Parallel *compreg* laminates had bonding shear strength comparable to that of solid wood. The treatment used in the study also rendered *compreg* laminates to be highly resistance towards towards white rot fungus (*Pycnoporous sanguineus*).

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

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ADDITION OF UREA**

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Penerapan dan pemampatan melalui resin phenol formaldehid (PF) pada suhu yang tinggi adalah menarik untuk memperbaharui sifat kekuatan, kestabilan dimensi dan daya ketahanan kayu menentang pereputan. Walau bagaimanapun, pelepasan formaldehid daripada bahan yang dirawat adalah sangat tinggi terutama apabila menggunakan resin yang berat bermolekul rendah. Percubaan untuk mengurangkan pelepasan formaldehid telah dilakukan dengan memasukkan urea ke dalam

larutan rawatan. Di dalam kajian ini, urea telah digunakan untuk mengurangkan formaldehid yang berlebihan daripada kayu yang diserap mampat dengan PF. Urea telah dipilih kerana efisien mengurangkan pelepasan formaldehid dan kos pembuatan yang bersesuaian. Pemboleh ubah eksperimen termasuklah penyerapan oleh kepingan jelutong (*Dyera costulata*) dan sesenduk (*Endospermum diadenum*) melalui 20 %, 30 % dan 40 % phenol formaldehid yang mempunyai berat molekul rendah (berat molekul 600) dicampur secara berasingan dengan urea (30 % berdasarkan berat pepejal PF), pra pengeringan pada 60°C selama beberapa jam (7, 8 dan 9 jam) dan seterusnya dimampatkan kepada purata kemampatan 80 % pada suhu 150°C selama 20 minit. Pelepasan formaldehid, sifat fizikal dan mekanikal dan ketahanan kayu yang telah diserap mampat telah dianalisis. Penambahan urea ke dalam PF berjaya mengurangkan pelepasan formaldehid sebanyak 97 % bagi kedua-dua kayu serap mampat. Hasil kajian juga menunjukkan kestabilan dimensi kayu serap mampat jelutong dan sesenduk telah meningkat sebanyak 60 % dan 52 % dalam mengurangkan pengembangan. Apabila dibezakan antara kepingan yang dirawat dengan atau tanpa penggunaan urea, kecekapan dalam mengurangkan pengembangan oleh kayu serap mampat yang dirawat dengan larutan PF ditambah urea adalah lebih berjaya. Berdasarkan sifat kayu serap mampat yang telah diuraikan, gabungan rawatan yang optimum untuk menghasilkan kayu jelutong dan sesenduk ialah 30 % kepekatan PF dicampur dengan 30 % urea (berdasarkan pepejal PF), 8 jam pra pengeringan pada 60°C disusuli oleh pemampatan kepada 80 % purata kemampatan pada 150°C selama 20 minit. Gabungan rawatan ini telah digunakan untuk menghasilkan lapisan kayu serap mampat. Tiga lapisan kayu jelutong dan sesenduk telah dihasilkan melalui penyatuan kepingan secara bertentangan dan selari antara satu sama lain, diteruskan dengan pemampatan oleh pemampatan panas. Apabila dibandingkan dengan kayu tanpa rawatan yang tidak dirawat yang mempunyai saiz yang sama, kedua-dua kayu lapis serap mampat mempunyai sifat yang lebih cemerlang. Ketumpatan kayu serap mampat telah meningkat sebanyak 78 % hingga 117 % daripada kayu yang tidak dirawat. Modulus pemecahan “MOR” dan modulus elastik “MOE” telah meningkat sehingga 24 %, kecuali lapisan selari sesenduk yang mempunyai nilai yang lebih rendah berbanding kayu yang tidak dirawat. Kajian juga telah memperlihatkan kekuatan kericihan di garisan pelekatan adalah tinggi bagi kayu serap mampat selari

berbanding bertentangan. Tambahan lagi, kayu serap mampat selari mempunyai kekuatan kelucutan setanding kayu pejal tidak dirawat. Rawatan yang telah digunakan dalam kajian ini telah mempunyai rintangan yang tinggi menentang kulat putih (*Pycnoporous sanguineus*).



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I certify that a Thesis Examination Committee has met on 26th June 2012 to conduct the final examination of Rabi'atol Adawiah Binti Mohd Ali on her thesis entitled "Formaldehyde Emission and Properties of Compregnated Wood Treated with Low Molecular Weight Phenol Formaldehyde with Addition of Urea" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the degree of Master of Science.

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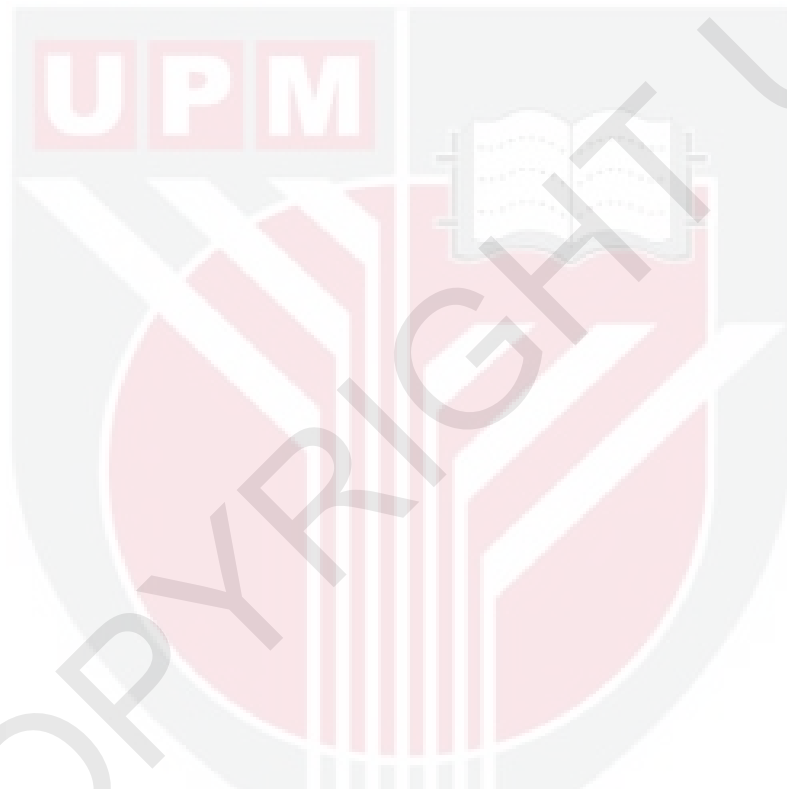
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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.



RABI'ATOL ADAWIAH BINTI MOHD ALI

Date: 26 June 2012

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
ASE	Anti-swelling Efficiency
ASTM	American Standard Test Method
CR	Compression ratio
FE	Formaldehyde Emission
FRIM	Forest Research Institute Malaysia
FTIR	Fourier Transform Infrared
IARC	International Agency for Research on Cancer
LMW	Low Molecular Weight
LVL	Laminated Veneer Lumber
LMWPF	Low Molecular Weight Phenol Formaldehyde
MC	Moisture Content
Min	Minutes

MMA Methyl Methacrylate

MOR Modulus of Rupture

MOE Modulus of Elasticity

MS Malaysian Standard

PDA Potato Dextrose Agar

PF Phenol Formaldehyde

PPM Part Per Million

RH Relative Humidity

SPSS Statistical Package for Social Science

TS Thickness Swelling

UF Urea Formaldehyde

UPM Universiti Putra Malaysia

WA Water Absorption

WHC Water Holding Capacity

WL

Weight Loss

WPG

Weight Percent Gain



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CHAPTER 1

INTRODUCTION

1.1 Background of study

Nowadays, human implement various sophisticated technologies to develop high quality forest products and maintaining sustainable forest management, simultaneously. In line with the increasing of human population and economic development especially in developing countries, global demand for timber products is rising. However, just few of us realized the population of natural forest has become distracted caused by the continuous development. Natural wood resources have been decreased year by year caused by illegal logging and unsustainable forest management. Wood technologist has been forced to develop new composites technology with good quality properties like particle board, oriented strand board, plywood, laminated veneer lumber and hardboard to optimize the using of wood. Regardless of some better properties than untreated wood, the products still has similar problem like solid wood.

An alternative in optimizing forest wood resources is to fully utilise the wood species available including low density wood. We always heard about timber named as teak, meranti, and chengal. They are the most valuable timber for Malaysian timber trade and wood products. Usually, exclusive and good quality furniture made from those materials. However, low density wood species which usually non durable like jelutong and sesenduk need to be treated in order to enhance its performance. Kempas (*Koompassia malaccensis*) a commercial

timber which once was considered as nondurable species, has succeed through chemical preservation. Its service life now is six times longer than normal life span through preservative treatment (Stubbs 1967).

Another way to cater the lack of natural wood resources is by using chemically engineered material. The engineering material is preferable rather than solid wood due to its uniform strength and stability. Wood is not preferable because it's poor dimensional stability due to hygroscopicity towards moisture, low durability when attacked by biodeterioration agent and relatively low mechanical properties. Permanent stabilization of the dimensions of wood is needed for specialty uses. This can be achieved by depositing bulking agents within the swollen structure of the wood fibres. The deposition of the resin is called as impreg which mean the wood treated with thermosetting resin, fibre penetrating resin and cured without compression.

Impregnation involves treating wood or cellulosic material with monomer solution that diffuse into cell wall, and then followed by polymerization. The monomer solution usually diffuses into cell wall and ended with polymerization. Besides, Hill (2006) defines impregnation as method resulting in the filling of wood with inert material to obtain a greater performance product. The mechanisms of impregnant fixation occur through two mechanisms like monomer impregnation (subsequent polymerization within the cell wall) and diffusion of a soluble material into the cell wall (immobile). The resins that can be used in impregnation process are PF, UF, MMF and MF.

Successful treatment can be achieved by taking some factors into consideration (Hill, 2006):

- i. The impregnant molecules must be smaller in size, so that it can swell the cell wall
- ii. Choose molecules with a greater tendency to form/break hydrogen bonds
- iii. The molecules can be introduced in carrier liquids, which swell the cell wall to a greater extent
- iv. Allow sufficient time for the impregnant molecules to diffuse into the intracellular spaces
- v. The impregnant molecules is nonleachable

For example, bamboo that was impregnated with resin caused increasing in strength properties and greater dimensional stability (Deka and Saila, 2000). Other than that, Ryu *et al.*, (1991) found that after applying PF resin above 40%, there was a little change in antishrink efficiency (ASE) values.

The physical and biological properties of wood could be enhanced through few mechanisms:

- i. The material swell the cell wall
- ii. The material occupies the cell wall, reduces hygroscopicity of wood
- iii. The blocking of cell wall micropores reduce diffusion of water and other molecules into the cell wall
- iv. Masking of hydroxyl content in the cell wall
- v. Crosslinking might occur between bulking agent and cell wall constituents

Compressed wood products from low density species on have started in 1980s in order to utilize fast growing trees (Wang *et al.*, 2000). *Compreg* is the process of impregnation of wood with certain monomer solution followed by compression under specific temperature and pressure to obtain a good connection between wood and synthetic resin polymer (Mclean, 1949). According to Stamm (1964), *compreg* product usually made of treated veneer in few layers and then being compressed to specific gravity and desire thickness. In contrast to impregnation process, the resin cures after hot pressing at high temperature and pressure. Before the compression begins, the resin is pre-cured upon medium temperature heat around 60 to 70 °C, providing semi polymerized resin in preventing a lot of resin squeezed out during the compression process. Gabrielli and Kamke (2009) stated that during compression process, less of the PF resin squeezed out from the samples caused by the slowed movement of the larger molecular size resin.

The most popular resin used in *compreg* process is PF resin since many researchers discovered excellent properties was bring out from wood treated with PF. At the softened stage, the compression applied will not rupture the cell wall while the resin will cure during the compression densification, simultaneously (Yano *et al.*, 1997). For the wood veneer that has more than 3mm thick, it supposed to be dried to less than 2 percent moisture content to prevent checking of product. Compared to impreg, the advantage of *compreg* is its natural lustrous finish that can be developed on any surface by sanding with fine grit paper and buffing. It also has ability to mold by gluing blocks of resin treated wood at about 150⁰C.

The compreg product usually has mechanical properties such as modulus of rupture (MOR), modulus of elasticity (MOE) and hardness greater than untreated wood mainly due to their high specific gravity (Stamm, 1964 and Kumar, 1994). Compreg product is also known as high resistant to decay, termites and mariner borers. Compare to untreated wood, it is more acid, electrical and fire resistance due to its greater density. Recently, addition of urea into PF has been introduced to improve curing, to lower the content of free formaldehyde and cost efficiently in minimizing resin (He and Riedl, 2003).

Compreg product is very resistant to decay, termites and marine borers. Thermal process can improve decay resistance by few ways such as formation of toxic compounds, chemical modification of wood components and breakdown of hemicelluloses (Weiland and Guyonnet, 2003). It could give better properties of wood like affecting drying speed, equilibrium moisture content, hygroscopicity, dimensional stability, surface quality, surface abrasion strength and shear modulus of wood (Welzbacher *et al.*, 2008). It also has better fire resistance compare to untreated wood due to greater density.

Compregnation of wood with PF at considerably high pressure has been proved can enhance the bending strength, dimensional stability and durability against fungal attack (Yano *et al.*, 2001). Wood treatment using low molecular weight resin is the most successful and reported can improve the dimensional stability of composite products (Stamm and Beacheler, 1960).

Compreg product normally has mechanical properties (Modulus of Rupture (MOR), Modulus of Elasticity (MOE) and hardness greater than untreated wood due to higher specific gravity (Galperine *et al.*, 1995). However, high pressure treatment needs high investment which has limited the application of this method in the wood industry. The final products are more

expensive than other treated wood or wood based panel because of high production cost (Rowell and Konkol, 1987).

Even though PF resin is very well known in wood treatment industry, drawback of the formaldehyde emission released especially from low molecular weight phenol formaldehyde has become a worldwide major concern. Formaldehyde is a chemical made up of oxygen, carbon and hydrogen. It is colourless but yet producing strong smelling gas. It occurs naturally in the environment by decay process or emitted by all timber species. Naturally, formaldehyde present in air at background level of 0.03 ppm and up to 0.08 ppm in outdoor urban air. Formaldehyde is usually being used as ingredient in synthetic resin, industrial chemicals, preservatives and production of paints and varnishes.

Internal Agency for Research on Cancer a division of World Health Organization has classified formaldehyde to a known carcinogen. The properties of formaldehyde that can cause cancer are only evident at very high concentrations which are hundreds times greater than emission from plywood and laminated veneer lumber products. Formaldehyde emission has been established by IARC where formaldehyde emitted below 0.1 ppm is undetectable by smell. Formaldehyde emitted between 0.1 ppm to 0.5 ppm is detectable by smell and caused slightly irritation to eyes, nose and throat. Emission between 0.5 ppm to 1.0 ppm could irritate eyes, nose and throat to most people. Extremely discomfort could appear if the emission is above 1.0 ppm.

1.2 Problem statement and justification

The world today, especially Malaysia, is lacking of timber supply due to unsustainable forest management and illegal logging. One of the methods to solve the lacking of natural timber is by improving the mechanical properties of low density wood such as mahang, jelutong and sesenduk.

There is a need to enhance its mechanical properties before it can be used for value added products. *Compregnating* using low molecular weight Phenol Formaldehyde (PF) in low density wood can improve dimensional stability and some strength properties (Zaidon, 2009). Previous research has low molecular weight PF resins has become the best method to increase dimensional stability in wood products like plywood and laminated veneer lumber (Wan and Kim, 2006). It easily penetrates the wood cell and ultimately bulks the wood material to a greater extent, providing greater stability (Rowell, 1991).

Other than using PF itself, the using of urea as formaldehyde scavenger has been proven succeed to reduce the formaldehyde emission. Kim (1996) has added 20 % urea into PF resoles to treat the wood based composites. The analysis done showed that the urea added has lowered the formaldehyde emission, increased degree of polymerization, decrease water soaking but internal bonding and curing rates was decreased as the urea level increased. Furthermore, the cost manufacturing using urea is much lower than other formaldehyde scavenger.

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