UNIVERSITI PUTRA MALAYSIA

DECAY RESISTANCE OF RUBBERWOOD (Havea brasiliensis Müll.Arg.) MODIFIED WITH LINEAR CHAIN CARBOXYLIC ACID ANHYDRIDES AGAINST BASIDIOMYCETES

NURAISHAH BINTI HASSAN

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By

NURAISHAH BINTI HASSAN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Master of Science

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Abstract of thesis presented to senate of Universiti Putra Malaysia in fulfillment of the requirement for the Master of Science

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April 2018

Chairman : Norul Hisham Hamid, PhD
Faculty : Forestry

The general objective of this study is to improve the basic properties of rubberwood. Rubber trees were cut to the dimensions 100 cm x 14 cm x 25 mm (L x W x T) and kiln-dried (10% to 12% moisture content, MC). The specimens (5 mm x 20 mm x 20 mm) (L x W x T) were prepared, and a Soxhlet extraction with toluene/methanol/acetone (4:1:1 by volume) was performed for 8 h. The specimens were oven-dried (103 °C for 24 h) and cooled (gel silica). Then, vacuum impregnation was conducted, and reactions with acetic, propionic, and butyric anhydrides took place for 0.25 h, 1 h, 4 h, 8 h, 10 h, 15 h, 24 h, 30 h, 36 h, and 48 h at 120 °C. The chemical bonding was confirmed by Fourier transform infrared (FTIR) analysis. The specimens were leached in deionized water and exposed to brown rot (*Coniophora puteana*) and white rot (*Trametes versicolor*) in an incubation room at 22 °C for 16 weeks. The reaction of rubber wood with acetic, propionic and butyric anhydrides did not damage its cell walls as shown in SEM images, but the cells shape changed from oval to elliptic. The reaction rate of rubberwood was fastest in ascending order are acetic, propionic and butyric anhydrides. The thickness swelling of rubberwood after submerged for four weeks was not significantly different with anhydrides. However, the propionylated rubberwood at 10.4 WPG gave the lowest thickness swelling. The rubberwood modified with acetic anhydride at 15 WPG gave the lowest decay protection threshold against *C. puteana* and *T.versicolor*, than those of propionic and butyric anhydrides. This showed that acetylation gave the best protection to rubberwood against both fungi. The final moisture content had a positive correlation with the weight loss following decay for both *C. puteana* and *T. versicolor*. The scanning electron microscope (SEM) images confirmed that the *C. puteana* and *T.versicolor* hyphae penetrated the cells in both untreated and modified rubberwood at all levels of WPG.

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

KERENTANGAN KULAT BAGI KAYU GETAH (*Havea brasiliensis* Müll.Arg.) YANG DIMODIFIKASIKAN DENGAN RANTAIAN LINEAR ASID KARBOSILIK ANHIDRA BASIDIOMYCETES

Oleh

NURAISHAH BINTI HASSAN

April 2018

Pengerusi : Norul Hisham Hamid, PhD
Fakulti : Perhutanan

Objektif umum kajian ini adalah untuk meningkatkan sifat asas kayu getah. Pokok getah dipotong kepada dimensi 100 cm x 14 cm x 25 mm (L x W x T) dan dikeringkan sehingga kandungan lembapan dalam kayu mencapai 10-12 %. Spesimen 5 mm X 20 mm X 20mm (L x W x T) telah disediakan, dan pengekstrakan 'Soxlet' dengan toluena / methanol / aseton (4: 1: 1 berdasarkan isipadu) dilakukan selama 8 jam. Kemudian dikeringkan dengan ketuhar (103°C selama 24 jam) dan disejukkan dengan agar-agar silika. Pengisaran vakum telah dijalankan dan tindak balas dengan anhidrida asetik, propionic, dan butirik pada waktu 0.25 jam, 1 jam, 4 jam, 8 jam, 10 jam, 15 jam, 24 jam, 30 jam, 36 jam, dan 48 jam pada 120 °C menggunakan 'oil bath'. Ikatan kimia disahkan dengan analisis 'Fourier transform infrared' (FTIR). Spesimen – spesimen direndam di dalam air yang disuling dan didedahkan kepada kulat reput perang (*Coniophora puteana*) dan kulat reput puith (*Trametes versicolor*) di dalam bilik inkubasi pada 22 °C selama 16 minggu. Kayu getah yang dirawat dengan anhidrida asetik, propionik, dan butirik tidak merosakkan dinding sel seperti yang ditunjukkan dalam imej pengimbasan mikroskop elektonik (SEM) tetapi bentuk dinding sel berubah dari bulat ke bujur memanjang. Pengembangan tebal kayu getah selepas direndam selama 4 minggu tidak menunjukkan perbezaan antara anhidrida. Bagaimanapun kayu getah dengan propionik pada 10.4 peratus pertambahan berat (WPG) memberikan pengembangan yang paling rendah. Kayu getah yang diubahsuai dengan anhidrida asetik pada 15 peratus pertambahan berat (WPG) memberikan ambang perlindungan kerosakkan kulat paling rendah berbanding dengan anhidrida propionik dan butirik. Kandungan lembapan akhir mempunyai korelasi positif dengan penurunan berat selepas kerosakkan kulat untuk *C. puteana* dan *T. versicolor*. Imej pengimbasan mikroskop elektornik (SEM) mengesahkan bahawa hifa *C. puteana* dan *T. versicolor* menembusi sel- sel kayu getah untuk kedua- dua kayu getah yang dirawat dan tidak dirawat pada semua peringkat WPG.

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I hope you will enjoy reading my master thesis about "Decay resistance of Rubberwood (*Havea brasiliensis* Müll.Arg.) modified with linear chain carboxylic acid anhydrides against basidiomycetes". Thank you very much.

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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 Master of Science. The members of the Supervisory Committee were as follows:

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CHAPTER 1

INTRODUCTION

1.1 Background

Wood is a renewable material and been used for many application including the building materials. It is compose of three major dimensional metrics of cellulose, hemicellulose, and lignin elements. These elements influence the dimensional stability, strength, machining, and decay resistance against bio deterioration. The global prediction of timber consumption until 2050 was estimated around 3 billion m^3 per year while the timber production from natural forest can only supply with about 1.9 to 2.2 billion m^3 per year until 2020. The shortage of timber will be sourced from the managed plantation forest, which now being established at a rate of over 3 million ha per year (Hill, 2006). Malaysia is one of timber exporter faced a same problem; this is due to decrease of total production forest from 5.89 to 3.22 million ha in Peninsular Malaysia for 2011.

Outdoors wood degraded by ultraviolet radiation and happens principally in the lignin, which is in charge for color changes. The cellulose fiber is bond together in the cell wall because of the lignin as an adhesive. In contrast with lignin, cellulose is a great deal less powerless to ultraviolet light degradation. After the lignin has been debased, the inadequately fortified starches rich filaments dissolve effectively from the surface, which uncovered new lignin for further degradation (Rowell 2006). The issue increased because of wood degradation could be settled by response the wood with chemical, in light of the fact that the basic properties of wood can be changed by altering the fundamental science of the cell wall polymers (Rowell, 2006). There are a few ways to deal with cell wall modification, contingent upon what property is to be adjusted. For instance, it can be achieved by reducing the hydrophilic system in the cell wall and replace it with hydrophobic groups which can enhance the water repellence or the wood hence increase dimensional stability. The cell wall can be modified by chemical bonding or cell wall polymer parts cross-linked together to confine cell wall extension, or gatherings can be reinforced that diminish hydrogen holding or increase hydrophobicity.

Modification either through chemical, mechanical or physical treatments can improve wood properties including low quality timber. Modification systems include impreg, compreg, stypak, staybwood, wood polymer composite and chemical modification processes (Hill, 2006). Selection of any treatment is dependent on the natural properties of the substrate material itself such as anatomy, density, chemical composition, permeability, and durability. The final properties which can be achieved rely on the types of treatment itself. Hill (2006) has detailed wood modification in terms of chemical, thermal and processes. Among the wood treatments, acetylation offers excellent dimensional stability and decay protection without affecting the mechanical properties.

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The modification of wood with acetic anhydride or other linear chain carboxylic anhydrides gives an advantage in term of environment aspect, because the chemical is attached covalently with the amorphous cellulose, hemicellulose and lignin of wood. This will avoid the leaching of the toxic material when dispose into the landfill. The chemically modified wood has a potential to substitute the toxicity of chopper chromium arsenate (CCA) preservative treated wood which was banned by US Environmental Protection Agency for many equipment. The European Commission (EC) has also banned the use of CCA preservative treated wood far all residential constructions, marine water installation, agricultural uses, fencing or structural uses (Hill, 2006).

1.2 The Rubberwood Industry

The rubberwood is the main wood resources after the natural forest timber. In 2009, there are about 1.0 million ha of rubber trees planted in Malaysia (Teoh *et al*., 2011). The research and development of rubberwood is well established. However, the uses of rubberwood are limited mainly for the furniture and composite products. The use of rubberwood as building material particularly for construction industry is not suitable due to its high starch and sugar contents and these make rubberwood prone to microbial attack. Rubberwood is also prone to attack by fungi and wood borers in dry and wet condition (George, 1985).
The table 1.1 shown the rubber plantation been diminishing from 2006 was 1283.6 hectares and 20166.5% smaller in the total of plantation developing in Malaysia rubber plantation.

Table 1. 1: Rubber Plantation Areas (Hectares)

Year	Estate	Smallholdings	Total ('000 ha)
2006	54.2	1209.4	1283.6
2007	52.7	1194.7	1199.6
2008	50.9	1196.1	1072.4
2009	48.5	1196.1	857.0
2010	49.9	965.3	939.2
2011	49.9	963.9	996.2
2012	65.9	993.8	922.8
2013	77.4	747.6	826.5
2014	80.1	747.6	668.6
2015	76.8	747.6	722.0
2016	77.4	747.6	637.5

Source: Department of Statistics (19 December 2017).

* Starting 2013, the rubber planted area for smallholding refers to the 2013 RISDA Smallholders Census which covers areas under RISDA, FELDA, FELCRA, KESEDAR, Sabah Rubber Industry Board and Department of Agriculture Sarawak.

The plantation areas from smallholders are Rubber Industry Smallholder Development Authority (RISDA), Federal Land Development Authority (FELDA) and Federal Land Consolidation and Rehabilitation Authority (FELCRA) still

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indicated a better execution in rubber development, but it has been declining each year (Ratnasingam *et al*., 2011). In March 2018, natural rubber production was 46, 082 tonnes, dropped by 27.1 percent as compared to 63,246 tonnes in Febuary 2018. For year on year, the production also showed a decrease of 43.4 percent. Export of natural rubber amounted to 57,658 tonnes, rose by 16.2 percent than previous month. Five main destination of rubber natural exports were China, Germany, Finland, Turkey, and U.S.A. stock of natural rubber recorded at the end of March 2018 showed a decrease of 2.5 percent from 285,671 tonnes to 278,602 tonnes (Department of Statistic, Malaysia, March 2018). Therefore, longing on in the natural forest has been controlled by forest plantation in Malaysia because of the demanding for rubberwood product (Shigematsu *et al*., 2011).

1.3 Problem Statements

The exploration of rubberwood as other building materials particularly for construction industries as well as to use it as outdoor furniture is limited by its low dimensional stability, low decay resistance, low insect resistance and extreme weather. Both rubber logs and sawn rubber wood are extremely susceptible to staining fungi as well as insect attack. Thus, if there is any delay (which should not be more than 2-3 days) in conversion or processing, chemical preservatives containing a fungicide and an insecticide could be applied by spraying or end coating the log for temporary or by dipping the board in a mixture of fungicide and insecticide. For long term protection, treatment processes such as conventional bethel process using borax compound is used (Lim *et al*., 2003). The diffusible nature of borates is an advantage in penetrating refractory timber species, such as spruce or Douglas fir (Gentz and Grace, 2006).

The Rubberwood needs to undergo pre-treatment processes to make it possible to be used especially outdoor applications. For example, rubberwood need to be treated with wood preservatives such as copper chrome arsenate (CCA) and creosote to improve its decay resistance, dimensional stability, strength and durability. In addition to environment issue, these may generates a future problem because this treatment is not suitable for outdoor and it’s not long lasting for such application and lack of performance. This is because this method is not chemically bonded within the wood structure. In reality, it just coating or finishing on the surface over time, small amounts of chemicals may leach from CCA-treated timber. Wood that is freshly treated with CCA has a greenish tinge which fades over time. Other wood treatments may also have a green colour. Unless your structure has been built with hardwood or cedar, it is possible it was built with CCA treated wood.

Therefore, the method for modifying rubberwood with linear chain carboxylic anhydride is aligned with the Malaysia government green technology policy towards the status of developed country. Through the years, many other catalyst have been tried, using both liquid and vapor system. Acetylation of wood using acetic anhydride has been done mainly as a liquid phase reaction. Acetic anhydride has been widely used in chemical modification experiments and the results compared to other chemical were also better while wood treated with anhydride leave no harm to the environment (Rowell, 2006). Wood treated with anhydride provides consistent

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and optimum result of biological resistance. The reaction with acetic anhydride result in esterification of the accessible hydroxyl groups in the cell wall with the formation of by-product of acetic acid (Rowell, 2006). The by-product of acetic acid requires additional time and cost to remove from wood cells. Therefore, reactions of wood with other anhydrides with a similar performance are vital to counter this problem. Therefore it is necessary to study the effect of carboxylic acid anhydride on the decay resistance of rubberwood against basidiomycetes; so that the properties can be determine and indirectly the modified rubberwood can be used beyond its limitation without give any harm to the environment.

1.4 Research Objectives

The general objective of this study is to improve the basic properties of rubberwood. The specific objectives of the study are:

1. To evaluate the reactivity of rubberwood with acetic, propionic and butyric anhydrides without the use of catalyst.

2. To investigate the microstructure of rubberwood after reaction with acetic, propionic and butyric anhydrides using scanning electron microscope.

3. To evaluate the dimensional stability of acetylated, propionylated and butyrylated rubberwoods using water soaking method.

4. To evaluate the effectiveness of acetic, propionic and butyric anhydrides to protect rubberwood from *Coniophora puteana* and *Tramestes versicolor*.

1.5 Significance of Research

The rubberwood is a main wood resource after natural forest timber in Malaysia. However, a pre-treatment is necessary to ensure that their performance is highly acceptable for any high end-multipurpose product applications to balance its extra processing time and investment. Chemical modification of wood with anhydride using conventional heating could give a uniform penetration and preserving both the environment and product from any negative toxic impact.

The purpose of this research is to evaluate whether the basic dimensional stability of rubberwood can be improved by anhydride modification and whether the modified rubber wood could achieve durability class 1. This is vital to expand the limitation of plantation tree.

This study will also be beneficial to the Malaysia economic growth by introducing a new highly durable rubber wood for a special application such as outdoor use of house frame, decking, furniture and door/window frame, fence and others to

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maximizing the utilization of rubberwood. The brown and white rot fungi are selected in this study because of its most aggressive colonization for any wood species during in service. The modified rubberwood will be exported to European countries (temperate climate) due to its higher price and is more competitive in terms of currency exchange.

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