UNIVERSITI PUTRA MALAYSIA

OPTIMIZATION OF OIL HEAT TREATMENT PROCESS TO ENHANCE RUBBERWOOD PROPERTIES USING RESPONSE SURFACE METHODOLOGY

S.S. UMAR IBRAHIM

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By

S.S. UMAR IBRAHIM

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

March 2015

DEDICATION

I specially dedicate this thesis to

My Supervisor
Prof. Dr. Zaidon Bin Ashaari

My Mother
K.M. Sirajunisha

My Father
Dr. S.E.S.A. Khader

My Wife
K.S. Ayisha Siddhiqua

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

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March 2015

Chairman: Professor Zaidon Bin Ashaari, PhD
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Rubberwood is an eco-friendly wood. Natural rubber is considered as the excellent agricultural product and it was utilized in many industries. The natural durability is very low in rubberwood. In dry as well as in green condition, it can be affected by wood borers and fungus. The objectives of this study are to determine the effect of resistance to white rot fungus, to assess the significant changes on the physical properties, chemical properties and mechanical properties of rubberwood after the heat treatment (172 - 228°C) in palm oil and to optimize the heating variables to enhance the properties of rubberwood treated with oil using response surface methodology. The colour of oil heat-treated rubberwood becomes uniformly darker. Hydrophobicity, dimensional stability and fungal resistance were improved by the heat treatment with respect to increase in treatment temperature. However, the mechanical properties of treated rubberwood were reduced compared to the untreated wood. The treatment resulted in changes to the wood chemical constituents, mainly the degradation of hemicelluloses which is believed to be principal reason for alterations in wood properties. The oil heat treatment reduced the chemical constituents by 11.7 % in total in the rubberwood. The depolymerization of hemicellulose results in reduction on bending strength of wood. The maximum reduction in Modulus of Rupture, Modulus of Elasticity, Compression and Shear were approximately 47%, 8%, 21% and 33% respectively compared to the control specimen.

This study shows that there is some reduction in density and equilibrium moisture content and positive high in moisture excluding efficiency values. The density of rubberwood was decreased gradually from 629 kgm^{-3} (untreated) to about 591 kgm^{-3} (at 228°C/180min). The Equilibrium Moisture Content also decreased from 12.42 % to 7.97 % (at 228°C/180min). The density reduction was probably due to the hemicelluloses and cellulose degradation. The higher moisture excluding efficiency value indicated that the wood is stable and excludes water after oil heat treatment process. The treated wood has moisture excluding efficiency value in the range between 28-50% depending upon the treatment temperature and time. The fungal resistance effect on the treated rubberwood was examined by exposing the wood to white rot fungi *(Pycnoporus sanguineus)* for 12 weeks and the weight loss was determined. The samples were tested at 13 different treatment conditions. Among those conditions, the lowest weight loss (8.23 %) was observed at 228°C and 180 min, with the decay by *Pycnoporus sanguineus* for rubberwood species. This research helps in improving various properties of rubberwood and biological durability by oil heat treatment process. Thus with the outcome of this research, the optimized heat treatment conditions could be suggested by the response surface methodology models to enhance the quality of rubberwood.

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

PENGOPTIMUMAN PROSES RAWATAN HABA MINYAK UNTUK MENINGKATKAN SIFAT-SIFAT KAYU GETAH YANG MENGGUNAKAN KAEDAH PERMUKAAN TINDAK BALAS

Oleh

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Kayu getah adalah kayu yang mesra alam. Getah asli dianggap sebagai produk pertanian yang sangat baik dan ia digunakan dalam banyak industri. Ketahanan semulajadi adalah sangat rendah di kayu getah. Dalam kering serta dalam keadaan hijau, ia boleh dipengaruhi oleh kayu borers dan kulat. Objektif kajian ini adalah untuk menentukan kesan rintangan kepada kulat putih reput, untuk menilai perubahan ketara pada sifat-sifat fizikal, sifat-sifat kimia dan sifat-sifat mekanik kayu getah selepas rawatan haba (172 - 228°C) di minyak sawit dan mengoptimumkan pembolehubah Pemanas untuk meningkatkan sifat-sifat kayu getah yang dirawat dengan minyak yang menggunakan kaedah permukaan tindak balas. Warna kayu getah heat-treated minyak menjadi seragam gelap. Hydrophobicity, dimensi kestabilan dan rintangan kulat telah bertambah baik dengan rawatan haba terhadap peningkatan suhu rawatan.

Walau bagaimanapun, sifat-sifat mekanik kayu getah yang dirawat telah dikurangkan berbanding kayu tidak dirawat. Rawatan yang menyebabkan perubahan kepada juzuk kimia kayu, terutamanya penurunan kualiti hemicelluloses yang dipercayai menjadi sebab utama perubahan dalam sifat-sifat kayu. Rawatan haba minyak berkurangan juzuk kimia sebanyak 11.7% secara keseluruhan di dalam kayu getah. Depolymerization hemicellulose mengakibatkan pengurangan pada lenturan kekuatan kayu. Pengurangan maksimum Modulus pecah, Modulus keanjalan, mampatan dan ricih adalah kira-kira 47%, 8%, 21% dan 33% masing-masing berbanding dengan spesimen kawalan. Kajian ini menunjukkan bahawa terdapat beberapa pengurangan ketumpatan dan keseimbangan kelembapan kandungan dan positif tinggi lembapan yang tidak termasuk nilai-nilai kecekapan. Ketumpatan kayu getah adalah menurun secara beransur-ansur daripada 629 kgm^{-3} (tanpa rawatan) kepada kira-kira 591 kgm^{-3} (pada 228°C / 180 min). Kandungan lembapan keseimbangan juga menurun daripada 12.42% to 7.97% (pada 228°C/180min). Pengurangan ketumpatan adalah mungkin disebabkan oleh hemicelluloses dan kemerosotan selulosa. Kelembapan tinggi tidak termasuk nilai kecekapan menunjukkan bahawa kayu adalah stabil dan tidak termasuk

air selepas proses rawatan haba minyak. Kayu yang dirawat mempunyai kelembapan yang tidak termasuk nilai kecekapan dalam julat antara 28-50% bergantung pada rawatan suhu dan masa.

Kesan rintangan kulat kayu getah yang dirawat telah diperiksa dengan mendedahkan kayu untuk kulat putih reput (Pycnoporus sanguineus) untuk 12 minggu dan penurunan berat badan yang telah ditentukan. Sampel yang telah diuji di 13 rawatan berbeza keadaan. Antara syarat itu, kehilangan berat badan paling rendah (8.23%) ini dilihat di 228° C dan 180 min, dengan kerosakan oleh Pycnoporus sanguineus spesies kayu getah. Kajian ini membantu dalam meningkatkan sifat-sifat pelbagai kayu getah dan ketahanan biologi dengan proses rawatan haba minyak. Oleh itu hasil kajian ini, syarat-syarat yang optima rawatan haba boleh dicadangkan oleh model permukaan kaedah maklum balas untuk meningkatkan kualiti kayu getah.

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I certify that a Thesis Examination Committee has met on 30.3.2015 to conduct the final examination of S.S. Umar Ibrahim on his thesis entitled “Optimization of oil heat treatment process to enhance the properties of rubberwood using Response Surface Methodology” 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 Master of Science.

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
ASE	Anti Swell/Shrink Efficiency
ASEAN	Association of SouthEast Asian Nations
ASTM	American Society for Testing and Materials
BS	British Standard
CCD	Central Composite Design
DES	Design Expert Software
EMC	Equilibrium Moisture Content
FAO	Food and Agriculture Organization
FRIM	Forest Research Institute Malaysia
FSP	Fiber Saturation Point
IRRDB	International Rubber Research and Development Board
ITTO	International Timber Trade Organization
ITC	International Trade Centre
MC	Moisture Content
MDF	Medium Density Fibreboard
MEE	Moisture Excluding Efficiency
MOE	Modulus of Elasticity
MOR	Modulus of Rupture
NMA	Nmethylolacrylamide
OHT	Oil Heat Treatment
RH	Relative Humidity
RSM	Response Surface Methodology
SEA	South East Asia
SG	Specific Gravity

TAPPI	Technical Association of Pulp and Paper
TEA	Triethylamine
USA	United States of America
UV	Ultra Violet
WL	Weight Loss
WWF-GFTN	World Wildlife Fund - Global Forest and Trade Network

CHAPTER 1

INTRODUCTION

1.1. General Background

Forests provide an extensive variety of services that are needed for living things. According to FAO (2006), plantation forests have increased comprehensively in the most recent years. Wood is produced mostly from forest plantations (FAO 2000, 2004; Bevege 2005). Wood is the main financial outcome from forests. Various kind of wooden products are exported by many countries and earn a good market. By 2030, half of the global timber demand will be supplied by plantation forests (WWF-GFTN 2001) and 75% by 2050 (FAO 2001a). There is a great demand for sawn wood and logs in India and China (ITTO 2013).

Globally there is a demand for good quality woods. The government of many developing countries had restricted for logging to preserve the world existing forests. In 1989, Thailand government has restricted commercial logging. Wood is a renewable resource. Hardwood and softwood are the two types of wood. Strength and dimensional stability are lacking in softwood (Rashmi et al. 2002). Balsa wood is a hardwood but it is softer than other commercial softwoods (Wikipedia 2015).

Rubberwood is a hardwood. Rubber trees are native to the Amazon valley of South America, and then later on, they were smuggled and introduced to other countries which were the parent stock planted in Southeast Asian counties (FAO 2015a). In the 19th century, Brazil was playing a major role in supplying hevea latex. Now many countries in Southeast Asia planted rubber tree as a plantation crop. The key producers of rubber are in Southeast Asia now.

Rubber tree is a tropical tree grows to a maximum of 600 meters above sea level, but normally in plantations it may grow up to 20 - 30 meters at the temperature between 20°C to 28°C with annual rainfall about 1800mm to 2000mm (IRRDB 2005). When the rubber trees attain the age of 5 or 7, the tree is tapped. The tapping process is carried on till 25 to 30 years of its age. The trees are removed when they attain the age of 30 because after that the latex production gets declined then they are supplanted with new seeds (FAO 2014).

According to Lim et al. (2003), the trunk is usually up to the height of 3 to 10 m with free of branches. When the trunks of the hevea trees are cutted, it oozes liquid which is called as latex that was collected to make rubber. Rubberwood and latex are the raw materials produced by the rubber tree plantation. In 1839, Charles Goodyear unintentionally dropped rubber and sulfur on a hot stovetop, this caused it to char like leather, but it remained plastic and elastic, this process is called vulcanization (Wade Davis 1996). Then the latex was used for various industrial applications and manufacturing products.

Hong (1995a) and Arshad (1996) stated that traditionally rubberwood was considered as waste material due to the complexity in preserving wood after milling. Traditionally, the removed trees were used as fuel for locomotive motor, burning bricks and for curing the latex (FAO 2015a). In recent years, the utilization of rubber wood has rapidly grown with various industrial applications (Yamamoto 1997, Kiam 2002, Hong 1995b, Varmola and Carle 2002) due to the development in technology of various wood treatment techniques (ITTO 2005a,b, Killmann 2001), but still the rubber plantations look latex as the main product and wood as secondary by-product. Rubberwood is widely used in furniture industry because of its static strength, dimensional stability and machinability. Ratnasingam and Scholz (2009) stated that rubberwood is used in steel industry as charcoal.

The rubberwood timber is produced by three steps. They are sawing of rubberwood logs, chemical impregnation and kiln drying. Where kiln drying is the most crucial process, time and energy consuming, and it is may degrade the lumber (Srivaro et al. 2008). High effective drying is helpful to enhance rubberwood quality. Rubberwood is not rubbery. It is hard, stiff and its characteristics are same as that of ash or maple. It has slight tendency to crack. It is also known as parawood, heveawood, Malaysian ash or Malaysian oak, but its appearance and properties are not same as ash or oak (Zachariah 2008). Fungus and insects can easily attack the hevea wood in both green and dry conditions (ITTO 2000).

1.2. Justification

Rubberwood is non-durable. According to Chew (1993) and Lew (1992), the rubberwood is used as a raw material in manufacturing many wooden products and it is very versatile wood. Rubberwood needs wood modification treatment to prolong the service life of wood. Heat treatment (thermal treatment) is one of the wood modification technique, which applies heat to wood to improve various wood properties in environment-friendly way (Hill 2006) and therefore the wood quality also increased. The dimensional stability and biological durability can be improved and hygroscopicity of wood can be reduced, these are the advantages of using thermal treatment. There are other wood treatments using preservatives and chemicals also exist, but the thermal modification of wood is preferable due to the environmental benefits of being pesticide-free.

The properties of various wood species can be strengthened by heat treatment process so that their applications can be extensively improved. There are various heat treatment processes available. They are Plato process, Retification process, Le-Bois Perdure, Thermowood process and Oil heat treatment (OHT) Process. These processes involves in improving the biological durability, dimensional stability, weather resistance, mechanical properties etc. and increases the wood's application globally by providing a high quality valuable timber. Existing literature discussed on various heat treatment methods and its effects on various properties of wood. Almost no literature reported on oil heat treatment of rubberwood and its effects on properties specifically. The aim of this study is to investigate various properties of rubberwood after oil heat treatment using palm oil. The oil heat treatment is a green process and is free of toxic chemicals. It is one of the most environment friendly methods of heat treatment of wood. Various vegetable oils are used in the oil heat treatment (OHT) process, which involves in chemical transformation. The chemical constituent of vegetable oil varies and it changes the wood's properties. The OHT treatment provides fast, homogeneous heat transfers with greater intensity. The advantage of using OHT is the cooling time needed for the treatment is lesser than the other treatments. The disadvantage in using other treatments is at the end there will be loss of heat and the heating medium, a special method is incorporated in recovering them. But in OHT process, there can be recycling of heat and heating medium in continuous processing.

1.3. Objectives

The specific objectives of the study are:

1. To optimize the heating variables to enhance the properties of rubberwood treated with oil using Response Surface Methodology (RSM).

2. To determine the effects of oil heat treatment on physical, chemical and mechanical properties of rubberwood.

3. To investigate the resistance of oil heat treated rubberwood against white rot fungal decay.

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