



Faculty of Resource Science and Technology

**DETERMINATION OF MONOMER-IMPREGNATED
WOOD USING ULTRASONIC WAVE**

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**Master of Science
2008**

**Pusat Khidmat Maklumat Akademik
UNIVERSITI MALAYSIA SARAWAK**

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ULTRASONIC WAVE**

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1000246102

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A thesis submitted

in fulfillment of the requirements for the degree of Master Science

**Faculty of Resource Science and Technology
UNIVERSITI MALAYSIA SARAWAK
2008**

**Dedicated to my dearest husband, daughter, son
and family.**

ACKNOWLEDGEMENTS

I am truly thankful to my supervisor, Dr. Sinin Hamdan, for his guidance and providing invaluable support and help throughout this research. I wish to express my heartiest gratitude to him for his patience, understanding and for spending his time in the discussion of this research work.

I wish to thank the TRTTC staff, Mr. Andrew Nyorik, Mr. Tan Jui Liah, Mr. Chan Chong Siew and Mr. Willies Chin in providing, preserving and cutting the wood samples for me. I also wish to express my gratitude to Mr. Lai Jiew Kok, Mr. John Sammy and Mr. Balton in advising and helping in the technical work for wood treatment. Special thanks also go to Mr. Leong Fad Weng and Mr. Johny Kon in identifying the wood in this research.

Not forgetting my colleagues from UiTM, Mr. Foo Kien Kheng, Ms. Leong Siew Hoe and Ms. Lau Ung Hua in helping me in the analytical work. I wish to express my heartiest gratitude to them for their patience and sharing their precious time with me in discussing the analysis problems which I faced during this research. I also would like to thank Madam Cindy Wee in proof-reading my thesis.

Finally, I wish to thank my dearest husband for his advice, support and help during my research work.

ABSTRACT

Plasticization suggests the changes in the characteristics of wood and result in greater pliability. Many chemicals have been used for plasticizing wood for example, anhydrous ammonia (NH_3) with the addition of glycerine and tetra ethylene pentamine to lock into the dimensional changes. In this study, Methyl Metha Acrylate (MMA) was used as the monomer to polymerize or impregnated into the wood samples by adding 2% of hydrogen peroxide catalyst and 5% of ethylene glycol dimetha acrylate as cross-linkers.

The objective of this study was to determine the capability of MMA, when applied to wood in monomer form and polymerized "in situ" to penetrate the wood cells. The penetration of the monomer was then evaluated by using ultrasonic wave to determine the treatabilty of the wood samples used. Ultrasonic velocity in the wood samples is calculated based on the data collected. The correlations between the ultrasound velocity (UV) and moisture content (MC), correlations between UV and density (ρ), and the correlations between UV, MC and ρ are investigated. The treated wood samples are tested for its dimensional stability. The treated wood samples are also tested in DMTA (Dynamical Mechanical Thermal Analysis).

The results found are very encouraging, the wood samples which are penetrated by MMA and the results was shown by the MMA retention. The increase on the ultrasonic velocity before and after treatment for all of the species used also showed the presence of MMA in wood samples. This study also found that there are correlations between ρ and UV but the correlations between UV and MC is weak. The stability of wood samples is found but the result could not be compared because there is no previous research done on the same samples.

The DMTA study showed that the mechanical properties of the treated wood had changed with the present of MMA in wood. The Young's Modulus of the woods also increased due to the presence of MMA in wood samples.

ABSTRAK

Pemplastikan mencadangkan perubahan sifat pada kayu yang menghasilkan lebih kelenturan kepada kayu. Banyak bahan kimia telah digunakan untuk pemplastikan kayu, contohnya ammonia (NH_3) dengan penambahan gliserin dan tetraetilen pentamin untuk mengelakkan perubahan dimensi. Dalam kajian ini, Metil Metha Acrylite (MMA) telah digunakan sebagai monomer untuk dipolimerkan ke dalam sample kayu dengan penambahan 2% hydrogen peroksida sebagai mangkin dan 5% ethylen glicol dimetha acrylite sebagai penyambung silang.

Objektif kajian ini adalah untuk menentukan kebolehan MMA, yang diaplikasikan ke dalam kayu dalam bentuk monomer dan polimerisasi dalam bentuk asal untuk menembusi sel kayu. Penembusan monomer ini dinilai dengan menggunakan ultrasonik untuk menentukan kebolehwatan sampel kayu yang dikaji. Halaju ultrasonik yang bergerak di dalam sampel kayu dikira berdasarkan data yang dikumpulkan. Korelasi antara halaju ultrasonik dan kandungan kelembapan (MC), korelasi antara halaju ultrasonik dan ketumpatan, dan korelasi antara halaju ultrasonik, kandungan kelembapan dan ketumpatan adalah dikaji. Sampel kayu yang dirawat dikaji kestabilan dimensinya melalui proses perendaman dalam air. Sampel kayu yang dirawat juga dikaji sifat mekanik dengan menggunakan analisis dinamik mekanik secara terma.

Keputusan yang diperoleh dari kajian ini sangat menggalakkan, yang mana sampel kayu yang ditembusi oleh MMA menunjukkan nilai pembendungan MMA yang positif dalam sel kayu.

Peningkatan halaju ultrasonik sebelum dan selepas rawatan untuk semua spesies juga mengimplicasikan kehadiran MMA dalam sampel kayu. Daripada kajian ini, didapati terdapat korelasi antara ketumpatan dan halaju ultrasonik dan korelasi antara kelembapan dan halaju ultrasonik adalah lemah. Kestabilan sampel kayu yang dirawat MMA telah ditentukan tetapi keputusan yang diperolehi tidak dapat dibanding kerana tidak terdapat hasil kajian yang serupa ke atas sampel kayu yang sama yang dilakukan oleh penyelidik lain. Kajian mekanik dengan analisis dinamik mekanik secara terma menunjukkan bahawa sifat mekanik kayu telah berubah dengan wujudnya MMA dalam kayu. Modulus Young, salah satu sifat elastik pada kayu juga berubah dengan kehadiran MMA dalam sampel kayu.

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CHAPTER 1

INTRODUCTION

1.1 Wood

Wood is a renewable resource, and one of the most fascinating materials because of its complex structure and its wide application on earth. Concise Encyclopedia of Wood and Wood-Based Materials defined wood as “the hard, fibrous tissue that comprises the major part of stems, branches and roots of tree, belonging to the plant groups known as the gymnosperms and dicotyledonous angiosperms”.

Dry wood is made up chiefly of cellulose (40 - 44%), lignin (18 - 25%), hemicelluloses (15 - 35%), and minor amounts (approximately 6 %) of extraneous materials (Haygreen and Bowyer, 1996). Cellulose, which is the major constituent, is a high - molecular weight linear polymer consisting of chains of bonded glucose monomers. The cellulose molecules are arranged into ordered strands called fibrils during the growth of the tree. The fibril is then organized into the larger structural elements comprising the cell wall of the wood fibers. Wood fibers have great commercial value. It can be formed into paper, synthetic textiles, films, lacquers and explosives.

Lignin is a three-dimensional phenyl-propane polymer, occurs in the wood throughout the cell wall, but it is concentrated throughout toward the outside of the cells and between cells. The structure and distribution of the lignin in wood are still not fully understood. High pressures or high temperatures or vigorous reagents might be needed to remove lignin from

wood on a commercial scale. By theory, lignin might be converted to a variety of chemical products. In fact, a large percentage of the lignin is removed from the wood during the pulping process. One commercial use for lignin is in the formulation of oil-well drilling muds. It is also used in rubber compounding and also in concrete mixes. Lesser amounts of lignin is processed to yield vanillin for flavoring purpose and to produce solvents.

The hemicelluloses are associated with celluloses and are polymers built from several different kinds of sugar monomers. The relative amount of these sugars varies with species. Hemicelluloses play an important role in fiber- to-fiber bonding in the papermaking process. The sugar components of hemicelluloses are of potential interest for conversion into chemical products.

The extraneous materials are not structural components of woods. They are both organic and inorganic. The organic component contributes to the properties of wood such as color, odor, taste, decay resistance, density, hygroscopicity and flammability. Tannins and other polyphenolics, coloring matters, essential oil, fats, resins, waxes, gums, starch and simple metabolic intermediates contributed to the extractives components. These components are classified as extractives because it can be removed from wood by extraction with such solvents for example, water, alcohol, acetone, benzene and ether. The inorganic component of the extraneous material comprises of calcium, potassium, magnesium, phosphorus, sodium, iron, silicon, manganese, copper, zinc and perhaps a few others are also usually present. Wood properties, for example, the wood density (ρ), stiffness, and hardness vary according to different species because of the variation in characteristics and volume of the four chemical substances in wood (Anon, 1987).

Water is also present in wood. Living trees are said to have large quantities of water and the moisture content of wood is defined as the weight of water in wood expressed as a fraction, usually as a percentage of the weight of oven-dry wood (Anon, 1987). Moisture content in trees may range from about 30 – 200% of the wood substance. Moisture is existed in wood as water or water vapor in cell lumens (cavities) and as water “bound” chemically within the cell walls. In green woods, cell walls are completely saturated with water; however, green wood usually contains additional water in the lumens. Fiber saturation point (FSP) is said to be happened when the cell walls are completely saturated (or all “bound” water) but no water exists in cell cavities. The FSP of most of the woods is approximately 30% of the oven-dry weight. As the green condition wood dries, water in the wood is removed. The MC in wood tends to come to equilibrium relative to the ambient humidity. Shrinkage starts to occur when the MC in wood is reduced below the FSP.

According to Gurfinkel (1973), two classes of the wood-producing species in the industries are recognized: hardwood and softwood. Some hardwoods however, are softer than some of the softwoods and some softwoods are harder than the hardwoods. Sarawak woods are categorized based on their density and are classified according to four classes: heavy, medium and light hardwood and softwood (Anon, 1999).

Both hardwoods and softwoods are being utilized in various fields. For example, furniture, construction work, framework for boats, flooring, pallets, plywood and so on. Structural wood, despite its valuable properties, it is also has many natural deficiencies or undesirable properties, such as poor dimensional stability and high moisture adsorption. Moisture adsorption is of high concern because fungi will attack wood if the change of MC in

wood is higher than 20%, (Dirol 1985; Griffin 1977). The moisture adsorption occurs on living trees and even on a piece of wood for construction.

In tropical areas, these effects are more pronounced because the woods are exposed to sunlight and are highly hygroscopic. Wood polymerization can reduce these deficiencies and prolong the usefulness of the wood life. In the wood polymerization, the wood is impregnated with a suitable chemical which is a liquid borne preservative or a monomer that can be polymerized in situ using either a catalyst-heat technique or an irradiation method to yield wood-polymer composite (WPC). In WPC, the void spaces of gross wood are partially or completely filled with polymer and caused the changed in the properties of wood (Noah and Foudjet, 1988).

1.2 Wood Polymer Composite (WPC)

In recent years, there has been a rapid application of chemical and physical principles to the improvement of wood properties. Wood treatment which was introduced from the year 1930 to 1960 included acetylation of the hydroxyl group, cross-linking of the cellulose with formaldehyde, ethylene oxide addition to the hydroxyl group, ozonolysis, propiolactone grafting of side chains, polyethylene glycol bulking of the cell walls. Hydroxyl groups in wood react with some of the condensation type of monomers to form covalent bond cross-links, or branches. The cell walls are simply bulked by polyethylene glycols by replacing the moisture in the green wood. The cellulose chain is degraded by the acid and base catalysts used with some treatments and cause brittleness of the composite.

Another class of chemicals containing one or more double bonds is introduced to perform wood treatment during the early 1960s, called vinyl type monomers. These vinyl type monomers can polymerize into solid polymer by means of free radical catalysts (Siau *et al*, 1965). This vinyl polymerization was an improvement over the condensation polymerization reaction because the free radical catalyst did not cause the wood to degrade. Unlike condensation polymerization, the free radical catalyst does not cause the wood to degrade. In addition the reaction does not leave behind a reaction product that must be removed from the final composite. Examples of vinyl monomers are styrene, vinyl chloride, vinyl acetate, acrylonitrile, ethylene oxide and many acrylates, especially methyl methacrylate (MMA), *t*-butyl styrene and chlorostyrene.

Since the non-polarity of most of the vinyl monomers, there is a little, if any interaction with the hydroxyl groups attached to the cellulose molecule. Generally vinyl monomers simply fill the capillaries, vessels and other void space in the wood structure (Timmons *et al*, 1971).

There are two ways to generate free radicals for polymerization: catalyst-heat curing and gamma radiation curing. Each has its own characteristics. Many complications arise when gamma radiation is used as a source of free radicals, including government regulations. In this study, catalyst-heat curing is used to polymerize the wood.

Vinyl monomer could not swell the cell structure of the wood. There is a little opportunity for the monomer to penetrate the cell wall and form branches on the cellulose. The changes of the physical properties of the wood-polymers are small produced by the

catalyst heat process and radiation curing process. Different vinyl monomers have been used to produce wood polymers since the past 35 years (Langwig *et al*, 1969). MMA is the preferred vinyl monomer for both the catalyst-heat and radiation curing. All types of vinyl monomer in liquid form can polymerize with free radical catalyst (Meyer, 1981)

To prevent the premature polymerization of MMA from heat and natural background during transportation and storage, polymerization inhibitors are added deliberately. In wood, natural inhibitors are also present in the form of extractives. Monomers which are applied will extract the soluble fractions from the wood structure. With the repeated use of polymerization inhibitors, natural inhibitors content build up in the monomer until there is not enough catalyst to start the polymerization.

When the polymerization of vinyl monomer occurs, a certain amount of heat is released. It is an exothermic reaction. When the inhibitors contained in monomer and wood are eliminated, temperature will rise to the maximum value, which corresponds to the peak of the exothermic polymerization reaction. Time needed to achieve this peak or maximum temperature depends on the catalyst, the type of monomer, cross-linker and ratio of the mass of monomer to the mass of wood.

Vinyl monomer which is added with cross-linker (1% to 5%) will increase the peak exothermic temperature, decrease the polymerization time, and prevent the monomer from expanding out from the wood and also increase the molecular weight to the point where the new thermosetting polymer will decompose before melting. The characteristic of non-melting

of wood-polymer composite is important in the machining and sanding of the final products (Meyer, 1968).

When the heat of polymerization is released quickly in wood-monomer composite, high temperature will increase the vapor pressure of the moisture in the cell walls and as a result the moisture will be driven out of the wood. Volume changes of the cell wall due to shrinkage and distortion of the original shape causes the changes of the dimensions, (Meyer, 1981).

Anti shrinkage efficiency (ASE) and ASE' is used to discuss the dimensional stability and it is done by solvent exchange reaction in the internal swelling volume of wood polymer composite. ASE of wood-polymer composites is normally 10% to 15%, which implies that there is penetration of monomer in the cell wall structure. For untreated wood, ASE is 0%.

Water sorption mechanism of dry wood proceeds in two steps: water enters dry wood in vapor form sorbs into the cell wall and forms hydrogen bonds with the cellulose (Meyer, 1981). This mechanism causes the cell wall to swell, and increases the dimension of the wood. When the cell wall swollen state reaches to the maximum, additional water will condense in the capillaries of the other non-cell wall void spaces in the wood until it is fully filled.

WPC only contains polymer in the void spaces, and a little if any, in the cell walls. Loading the capillaries with polymer can reduce the rate of water vapor diffusion into the cell walls. At high humidity and given enough time, water can still reach the cell walls and cause the volume swelling as in untreated wood (Langwig *et al*, 1969).