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PHYSICAL PROPERTY IMPROVEMENT OF RUBBERWOOD WITH POLYMER BASED ON DRYING OIL

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อโณชา ดีชัยยะ : การปรับปรุงสมบัติทางกายภาพของไม้ยางพาราด้วยพอลิเมอร์จากน้ำมันชักแห้ง (PHYSICAL PROPERTY IMPROVEMENT OF RUBBERWOOD WITH POLYMER BASED ON DRYING OIL) อ.ที่ปรึกษาวิทยานิพนธ์หลัก : รศ.ดร.สุรชัย พรภคกุล, 103 หน้า.

ศึกษาผลของการบำบัดทางความร้อน และการเตรียมไม้ยางพาราบำบัดด้วยกรดไขมันในน้ำมันทัง น้ำมันลินซีด น้ำมันทานตะวัน น้ำมันปาล์ม และน้ำมันอิพอกซิไดส์ ศึกษาผลของการบำบัดทางความร้อนที่อุณหภูมิ 100-200 องศาเซลเซียส เป็นเวลา 1-8 ชั่วโมงที่มีต่อค่าน้ำหนักที่หายไป ลักษณะทางภายนอก และสมบัติทางกายภาพของไม้ยางพารา ในการศึกษนี้ใช้ไม้ทางการค้า และไม้สดในการทดลอง ได้เปลี่ยนภาวะต่างๆในกระบวนการเตรียมไม้ ได้แก่ เวลาในการแช่ ชนิดของน้ำมัน และกระบวนการบำบัดแล้วเปรียบเทียบค่าการดูดซับน้ำ ค่าการพองตัว ค่ามุมสัมผัสระหว่างหยดน้ำ ค่าแรงดัดของไม้ ค่าโมดูลัสการยืดหยุ่น ของไม้ยางพาราที่ไม่ผ่านการบำบัดและไม้ยางพาราที่ผ่านการบำบัด ผลการศึกษาพบว่าการบำบัดทางความร้อนที่อุณหภูมิสูงขึ้น ทำให้ได้ไม้ยางพาราจะมีความไม่ชอบน้ำที่มากขึ้น การบำบัดทางความร้อนไม้ยางพาราทางการค้าที่อุณหภูมิ 160 องศาเซลเซียส และไม้ยางพาราสดที่อุณหภูมิ 180 องศาเซลเซียส มีค่าการดูดซับน้ำดีกว่าการบำบัดที่อุณหภูมิอื่น และทำให้ความคงตัวของขนาดไม้ดีขึ้น การบำบัดด้วยน้ำมันโดยการแช่ไม้ให้ชุ่มแบบลดความดันด้วยน้ำมันทัง น้ำมันลินซีด น้ำมันทานตะวัน น้ำมันปาล์มและน้ำมันอิพอกซิไดส์สามารถปรับปรุงสมบัติค่าการดูดซับน้ำ ค่าการพองตัว ค่ามุมสัมผัสระหว่างหยดน้ำกับผิวสัมผัส ค่าแรงดัดของไม้ และค่าโมดูลัสการยืดหยุ่นได้

สาขาวิชาปิโตรเคมีและวิทยาศาสตร์พอลิเมอร์ ลายมือชื่อนิสิต..

ปีการศึกษา 2553 ลายมือชื่อ อ.ที่ปรึกษาวิทยานิพนธ์หลัก........................

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The effect of heat treatment on rubberwood and preparation of treated rubberwood based on fatty acids in tung oil, linseed oil, sunflower oil, palm oil and epoxidized oil has been studied. The effect of heat treatment at 100-200ºC for time spans ranging 1-8 h on the mass loss (%), the appearance of rubberwood and the physical properties was investigated. Commercial rubberwood and fresh rubberwood were used for this study. Various conditions in preparation process including soaking time, type of oil and treatment process were varied and the water absorption, dimension stability, water contact angle, flexure stress and modulus of elasticity of untreated rubberwood and treated rubberwood were compared. Results showed that higher temperature of heat treatment of rubberwood gave more hydrophobic property. The treated commercial rubberwood at 160ºC and the fresh rubberwood at 180ºC gave water absorption better than other temperatures of the heat treatment and dimension stability of heat treated woods were improved. The oil treatment by impregnation procees with tung oil, linseed oil, sunflower oil, palm oil and epoxidized oil improved the water absorption, volume swelling, water contact angle, flexure stress and modulus of elasticity of rubberwood.

Field of Study : Petrochemistry and Polymer Science   Student's Signature ....
Academic Year : 2010   Advisor's Signature ....

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LIST OF ABBREVIATIONS

°C : Degree Celsius
cm : Centimeter
g : Gram
h : Hour
m : Meter
mol : Mole
ml : Milliliter
*C*st : Centi-stroke
PO : Palm oil
EPO : Epoxidized palm oil
SFO : Sunflower oil
ESFO : Epoxidized sunflower oil
LO : Linseed oil
ELO : Epoxidized linseed oil
TO : Tung oil
ETO : Epoxidized tung oil

CHAPTER I

INTRODUCTION

1.1 The Statement of Problem

Wood and wood products derived from trees have been important in the human economy since earlier times. Wood is a major material for many applications such as construction materials, furniture, kitchen equipments etc. Nowadays, a shortage of wood supplied for the domestic industrial purposes has been discussed. Therefore, a search for the substitute materials for wood has been developed. Rubberwood, in particular, is considered as a promising alternate raw material, because of its fast growth rate and short rotation rate.

Rubberwood is a common name for timber of *Hevea brasiliensis*. Good qualities of rubberwood for usage, namely its homogeneous texture, pleasant appearance and beautiful grains make it suitable for numerous end uses. It is an ideal wood for making furniture and toys. However, it has limited uses in a number of different applications due to its low durability, dimension stability and fungi and insect resistance. Therefore, freshly sawn wood must be treated with wood preservative.

Wood preservatives are broadly classified as either water-borne preservative or oil or oil borne preservative, depending on the chemical compositions of the preservatives and the carrier used during the treatment process. Unfortunately, these substances are harmful to the environment and toxic to the human health. However, some types of oil can be used as environmental preservatives.

Natural oil that comes from renewable resources is a good choice as raw material for producing a variety of products such as coatings, paints, lubricants and etc. Drying oil, mostly composed of the mixture of triglycerides, is natural fatty oil which forms solid film after a period of exposure to air. It is the ester formation between one molecule of glycerol and three molecules of various linear fatty acids. For more complex molecules such as polymers, copolymers and their composites, the oil must be modified chemically. Epoxidation of long chain olefins and unsaturated fatty acid derivatives are increasingly attractive. Therefore, in this research, vegetable oil and epoxidation oil were used to improve the properties of the rubberwood by cold soaking and impregnation process.

1.2 Objectives of the Research Work

1.2.1 To improve antifungal and water repellent properties of rubberwood.

1.2.2 To study physical and mechanical properties of rubberwood after improving the antifungal and water repellent properties.

1.3 Scope of the Research Work

1.3.1 To study properties of rubberwood before and after treatment including SEM of microstructure, water absorption, volume swelling, water contact angle, antifungal, modulus of elasticity and compressive of parallel to grain.

1.3.2 To prepare epoxidation palm oil, sunflower oil, linseed oil and tung oil.

1.3.3 To study effect of the temperature and time on the properties of rubberwood on the following parameters: mass loss, failure of wood, water absorption, volume swelling and water contact angle.

1.3.4 To study effect of types of oil on the properties of rubberwood on the following parameters: water absorption, volume swelling, water contact angle, anti-fungal, modulus of elasticity and SEM of the microstructure.

1.3.5 To study the effect of the treatment process on the properties of rubberwood on the following parameters: oil loading, water absorption, volume swelling, water contact angle and SEM of the microstructure.

CHAPTER II

THEORY AND LITERATURE REVIEWS

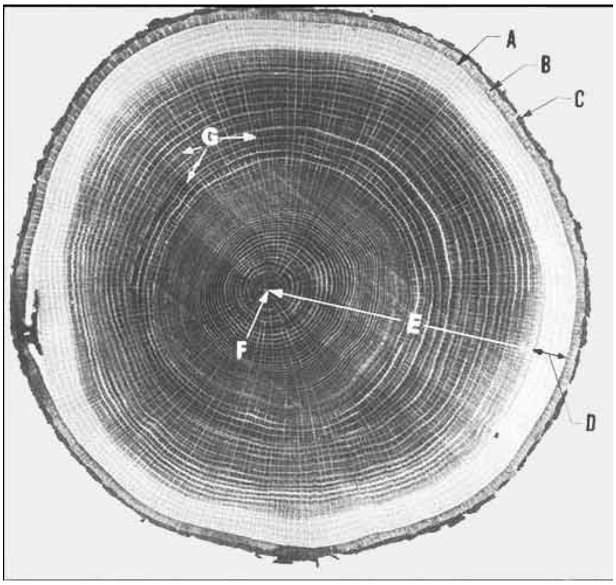
2.1 Wood [1]

For many centuries, wood has been a natural material for many applications such as construction for homes and other building, furniture and etc. During wood have desirable properties such as high specific strength, low heat conductivity, electric insulation, ability of controlling atmospheric humidity, beautiful appearance. However, wood also has undesirable properties. So wood should be modified the properties, especially for uses.

2.1.1 Wood formation

A cross section of a tree (Figure 2.1) shows several different features. From the outside, there is first the bark-both the outer corky dead part and the inner thin living part. Next is the wood, which in the characteristic sizes of most species can be differentiated into sapwood and heartwood. Finally, there is the pith, a small central core which represents the primary growth in a stem or a branch.

Between the bark and the wood is a layer of thin-walled cells called the cambium. New wood cells are formed on the inside of the cambium and new bark cells on the outside. Thus growth in diameter results from the formation of new cells, not from growth of the wood already formed. Similarly, growth in the length of branches and stems is the result of the addition of cells at the ends of the stem and the branches.



A.) Cambium layer
B.) Inner bark
C.) Outer bark
D.) Sapwood
E.) Heartwood
F.) Pith
G.) wood ray

Figure 2.1 Cross section of tree trunk.

Wood cells are of various sizes, shapes, and functions. The major of cells are elongated and pointed at the ends and are called fibers or tracheids, they impart strength to the wood. In addition to fibers, hardwoods contain a cell of large diameter called vessels, which function as the main arteries for the longitudinal movement of sap. In softwoods, the same function is performed by the tracheids. Other type of cells, some arranged horizontally in the tree, perform specialized functions.

2.1.2 Sapwood and heartwood

The outer portion of the tree stem, the sapwood, located next to the cambium, function primarily in the storage of food and the mechanical transport of sap. It varies in thickness, is usually light-colored, and is less resistant to decay than is the heartwood. The heartwood in many species is darker than the sapwood, its cells have become inactive, various materials (commonly called extractives) have become deposited in its cell, and it functions mainly in the mechanical properties between heartwood and sapwood, that is, the strength of a piece of wood is determined principally by the condition which exist when the wood is formed, not by the changes that take place during the growth of the tree.

2.1.3 Hardwood and softwood

Tree species generally are divided into two large groups commonly referred to as hardwood and softwood. The hardwoods (Figure 2.2) are heterogeneous in their structure, having a greater complexity of cell form, associated with a more marked division of labor in the living tree than is the case in the softwoods. Ascent of sap in the stem is facilitated by tube like structures known as vessels. Mechanical support is provided by fibers. Food storage and conduction are functions to the rays and also of the wood parenchyma cells, which are quite well developed in some species.

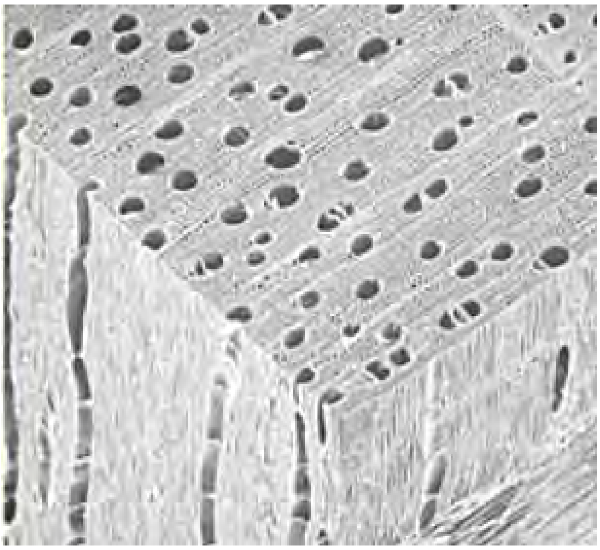


Figure 2.2 Magnified three-dimensional of hardwoods.

The softwoods (Figure 2.3) are comparatively homogeneous, consisting essentially of two main elements or cell forms: tracheids and ray. Tracheids concern in the living tree with the dual function of sap conduction and mechanical support. And, ray serve chiefly for storage and horizontal conduction of food materials. In certain softwoods, the wood is characterized by the presence of vertical and radial intercellular canals or resin duct. Wood parenchyma cells, other than the epithelial parenchyma which surrounds and limits the resin ducts, also occur in some softwood species but are sparsely developed.

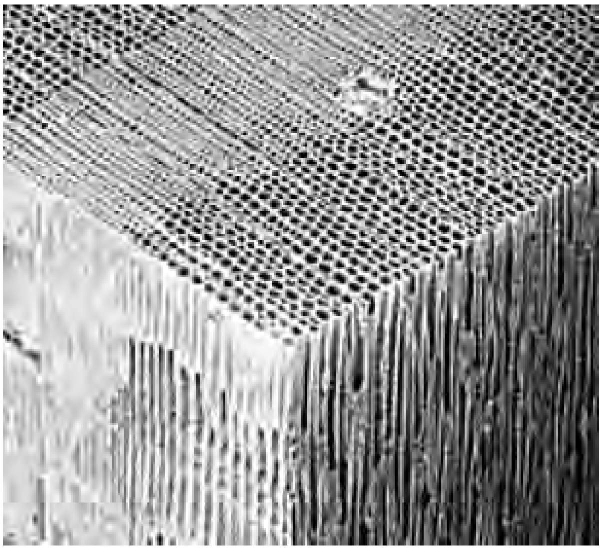
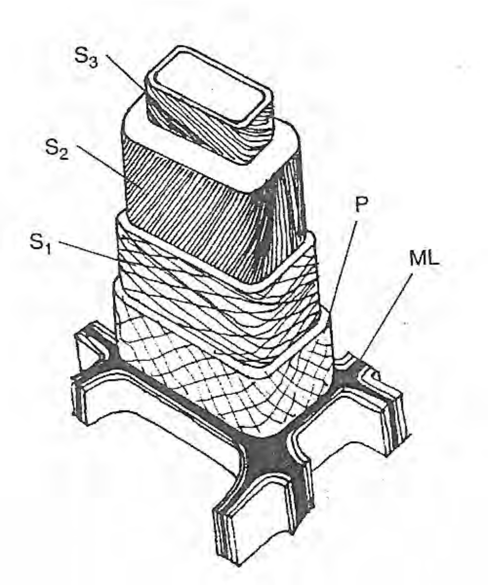


Figure 2.3 Magnified three-dimensional of softwood.

2.1.4 The cell wall of wood [2]

The cell wall of wood is composed of a number of layers (Figure 2.4). These are divided into the primary (P) and second (S) layers. The secondary layer is further subdivided into the S_1, S_2, and S_3 layers. The primary layer is the first to be laid down when the cell is formed and is composed of microfibrils, which have an essentially random orientation that allows for expansion of the cell to occur as cell growth takes place. The secondary layer is subsequently formed, with each of the sub-layers exhibiting different patterns in the way the microfibrils are oriented, as showed in Figure 2.4. Of these, the S_2 layer occupies the greatest volume of the cell wall and, as a consequence, is the layer that has the greatest influence on many of the properties of the cell and hence the macroscopic properties of the wood. The S_2 layer exhibits a definite microfibrillar orientation, and is itself composed of many lamellae consisting of a great many closely associated morifibrils that exhibit a helical winding pattern.



P.) Primary layer
S.) Secondary layer
ML.) Middle lamella

Figure 2.4 A schematic of the wood cell wall, showing the middle lamella, the main cell wall layers and the associated microfibrillar orientation.

2.1.4.1 Cell wall microporosity

The space between the microfibrils is occupies by the hemicelluloses and lignin. However, the incomplete filling of the intermicrofibrillar region results in the existence of what are usually referred to as micropores (or microvoids) in the cell wall. These have diameters of the order of nanometers and thus technically should be referred to as nanopores, but since the term ‘micropores’ is the most commonly used in the literatures, it will be used throughout this book.

When the cell wall is fully swollen, the micropores are open and the interior of the cell wall can be accessed by entities that are smaller than the diameter of the micropores. When wood is dried from a water-saturated condition, as water is removed the lumen and other macrovoids, and then subsequently the cell wall, lose moisture. As the water is removed, the micropores begin to collapse, and this process continues until the wood is dry.

The determination of the accessibility of the cell wall interior is of importance for a number of reasons:

- In many cases, modification with chemical reagents requires reaction with OH groups inside the cell wall.
- In some classes of modification, it is necessary for the modifying agent to penetrate the cell wall, which requires that these agents have dimensions no greater than the diameter of the cell wall micropores.
- Improvement of some properties (such as dimension stability) requires that, in some cases, the modifying agent resides within the cell wall micropore structure.
- Blocking of the cell wall micropores may be a mechanism in explaining some of the properties of modified wood (e.g. decay resitance).
- Modification resulting in the formation of cross-links within the cell wall prevents the micropores from opening when the wood is exposed to moisture.

2.1.5 The chemical constituents of wood [2]

Many of the physical, chemical and biological properties of wood can be understood by referring to the polymeric chemical constituents. In many cases of wood modification, these polymeric components are modified to some extent. The three structural polymeric components of the wood cell wall are cellulose, hemicelluloses and lignin.

2.1.5.1 Cellulose

The cellulose content of wood varies from about 40% to 50%. Cellulose is composed of β-D-glucopyranose monomeric units that are alternately inverted to form cellobiose dimeric units, which are 1.03 nm in length. This results in the cellulose backbone being linear. The cellobiose units link together via glycosidic linkages to form the polymer cellulose. The degree of polymerization (DP) of cellulose has been determined as about 10^4 in wood, although this is likely to be an underestimate, since some degradation will inevitably occur when cellulose is isolated from the cell wall. A number of cellulose chains are closely associated via extensive H-bonding networks to form the microfibril, which is the primary reinforcing element in the cell wall. The microfibrils have associated with them crystalline and amorphous components with associated OH groups. Because of the highly crystalline nature of the microfibrils, the cellulose component of wood is relatively unreactive and thermally stable. It appears that the crystalline region is associated with the core of the microfibril, with the exterior corresponding to the amorphous cellulose content.

2.1.5.2 Hemicelluloses

Like cellulose, hemicelluloses are polysaccharides, but they are composed of a number of different sugar units. The DP of hemicelluloses is the order of 200-300, and they are generally less ordered than cellulose, although some can form crystalline units. They also differ from cellulose, in that some of the OH content is naturally acetylated, and there are also carboxylate groups associated with the structures. Unlike cellulose, the main backbone of hemicelluloses structures also has short branches of sugar units attached. Because of the generally amorphous nature of the hemicelluloses, they contain the greatest proportion of the accessible OH content of the cell wall, react more readily, and are less thermally stable than cellulose or lignin. Hemicelluloses appear to act as interfacial coupling agents between the highly polar surface of the microfibrils and the much less polar lignin matrix. Hemicellulose degradation results in wood becoming brittle and rigid, indicating the important role that they have in imparting viscoelastic properties to wood. Hardwood hemicelluloses are generally found in higher proportions compared with softwoods, with a higher proportion of pentosans and a higher degree of acetylation.

2.1.5.3 Lignin

Lignin is a highly amorphous phenolic polymer of indeterminate molecularweight. Lignification of the wood cell wall involves the diffusion of the phenylpropane monomer units and polymerization to produce a random three-dimensional network, via a free-radical mechanism. Due to the random nature of the polymerization reaction, there is no definitive structure to lignin, although the frequency of individual bond types is well established. As with all structures, the two-dimensional representation does not give an accurate impression of the true complexity of the structure. The random nature of the lignin polymer network is the main factor in determining the complex geometry of the cell wall micropores. Lignin is responsible for providing stiffness to the cell wall and also serves to bond individual cells together in the middle lamella region. Although lignin is relatively rigid at room moisture in the cell wall opens up the structure of lignin. Lignin has a low concentration of OH groups compared to the polysaccharide components. Hardwood lignins have a syringyl content varying from 20% to 60%, whereas softwood lignins have very low syringyl content.

2.1.5.4 Extractives

In addition to its major, structructure components, cellulose, hemicelluloses and lignin, wood also contains an exceedingly large numer of other compounds. Among the more important ones are the terpenes, and wood resin. Most of the extractives are located in the heartwood, the present of some of them being the source of general darkening of this portion of tree. Depending on the species, wood can contain levels of extractible material ranging from approximately 0.5% to around 20% by weight, depending on the species.

2.1.5.5 Hydroxyl accessibility

The OH groups associated with the cell wall polymeric constituents are very important in determining many of the properties of wood. Many of the material properties of wood depend upon the presence of these groups and, in particular, hydrogen bonding interaction between the molecular components of the cell wall. Moreover, the OH groups are also responsible for absorption of moisture (leading to dimensional instability) and permit biological attack of the material. On a molecular level, the cell wall OH groups represent the most important component influencing the performance of wood.

Hydroxyl groups are associated with cellulosed, hemicellulose and lignin in the cell wall. Cellulose is the molecular component of the reinforcing elements of the cell wall (the microfibrils) and a proportion of the cellulosic OH groups are involved in hydrogen bonding interactions with sorbed water molecules. The majority of the accessible OH groups of the cell wall are associated within the hemicellulosic constituents; the lignin less so, due to its relatively low abundance of OH groups.

2.1.6 Anisotropy [1]

The arrangement of fibers in wood, with the long axes of the fibers parallel to the axis of the trunk, suggests that wood may have different characteristics in the various directions within itself. This is indeed true, and thus wood is anisotropic rather than isotropic, as are many other materials such as metals. Specifically, wood is considered to be orthotropic, having unique and independent properties in the direction of three mutually perpendicular axes. The axes (Figure 2.5) are longitudinal, or parallel to the length of the fibers, radial, or perpendicular to the length of the fibers and in a direction radial to growth rings and, tangential, or perpendicular to the fibers length and in direction tangential to the growth rings The strength, the modulus of elasticity, and other characteristics such as shrinkage and swelling differ in the three direction.

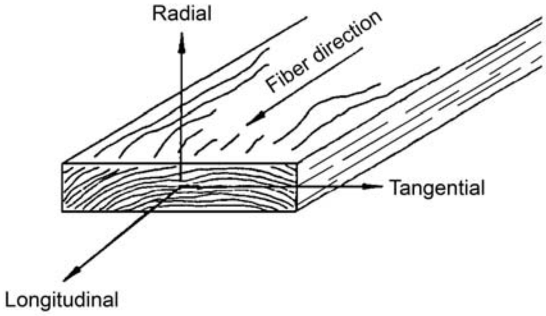


Figure 2.5 Three principal axes of wood with respect to grain direction and growth rings.

2.1.7 The wood-water relationship

Wood is a hygroscopic material, due to the fact that the cell wall polymers contain hydroxyl groups. In an environment containing moisture, dry wood will absorb moisture until it is in equilibrium with the surrounding atmosphere. Similarly, saturated wood, when is placed in an atmosphere of lower relative humidity (RH), will lose moisture until equilibrium is attained. If the wood is placed in an environment where the RH is stable, it will attain constant moisture content (MC), known as the equilibrium moisture content (EMC). At this point, the flux of water molecules into the cell wall is exactly balanced by the outward flux into the atmosphere.

Wood responds to its environment; in particular, it changes dimension in respond to changes in atmospheric RH. As water vapor enters the wood cell wall, it occupies space causing the dimensions of the cell wall to increase, and as a consequence the wood increases in dimension. These changes in dimension are anisotropic and are greatest in the tangential direction, then the radial and least in the longitudinal direction.

2.2 Rubberwood [3]

Rubberwood is the timber of *Hevea brasiliensis*. The genus *Hevea* is a member of the family Euphorbiaceae and comprises 10 species, of which the rubber tree. *Hevea brasiliensis* is quick-growing, erect tree with a straight trunk and bark which is usually grey and fairly smooth, but varies somewhat in both color and surface. It is the tallest species of the genus and in the wild trees may grow to over 40 m and live for over 100 years, but in plantation they rarely exceed 25 m because growth is reduced by tapping and because they are usually replanted after 25-30 years when yield fall to an uneconomic level.

2.2.1 Anatomy of rubberwood [4, 5]

In the center cylinder of rubber tree is surrounded by bark, the two tissues being separated by a thin layer of vascular cambium which generates xylem tissues on the inside and phloem tissues on the outside. The wood (xylem) consists mainly of fibres, tracheids and vessels, the latter two being responsible for the upward transport of water and nutrients take up form the soil by the roots.

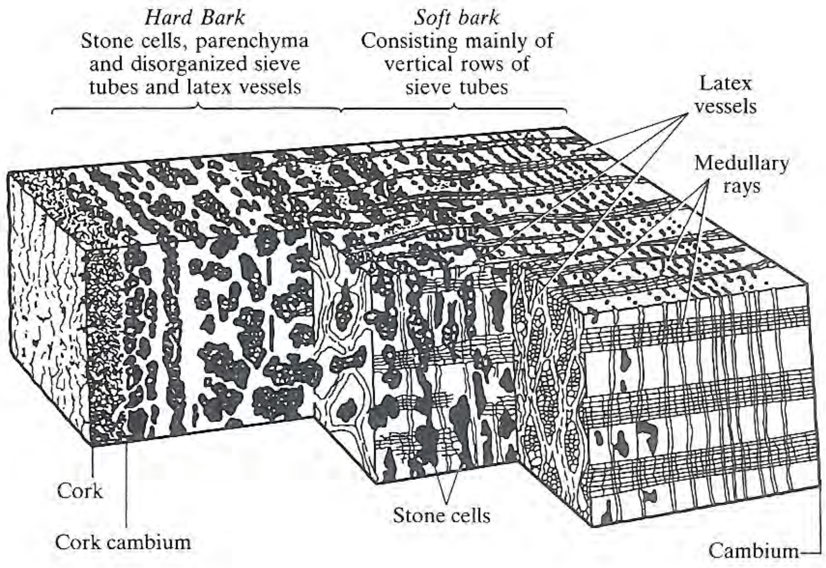


Figure 2.6 Three-dimensional diagram of the bark of *Hevea brasiliensis.*

In Figure 2.6 the stem consists of the texture of wood which is the center of stem called “Central axis or Pith (medulla)”. The next layer is wood or xylem, the next one is ring of cells or called “cambium”, then the next of cambium is soft bark which contains phloem. The next of soft bark is hard bark and the inner of hard bark, combined with soft bark, contains latex vessels that twisted on the right hand. And then, it is cork cambium and cork respectively.

The texture of the wood is fairly even with moderately straight and slightly interlocking grain. It is whitish yellow when freshly cut, but the wood turns to light brown dryness. Latex vessels can be found with characteristic smell in some parts of the wood. The wood is soft to moderately hard with an average weight of 515 kilograms per cubic meters at 12% moisture content. Pores on the cross section are diffused and of medium to large size, mostly solitary but sometimes in short multiples of two to three, filled with tyloses, vessel tissues are conspicuous in radial and tangential faces and are of the order of about 200 microns in diameter. Wood parenchyma are abundantly visible to the naked eye appearing as narrow, irregular and somewhat closely spaced bands forming a net like pattern with rays. The rays of the wood are moderately broad, rather few and fairly wide spread. The pits found between the vessels and rays are haft-bordered with narrow width. The length of the fibers is more than 1.0 millimeters on the average and the width is about 22 microns when dry. The cell wall thickness when dry is about 2.8 microns. [4]

There is insignificant heart wood formation and no transition appears between sapwood and heart wood, which is confined near the pith. Growth rings or annual rings are not visible in rubber in rubber wood, unlike many other woods (ring porous wood). However, concentric false rings sometimes appear on the wood, depending on the presence of tension wood (gelatinous cells) which is fairly common in most of the clones. Maximum numbers of such rings are found in the basal portions with decreasing number towards the top. The tension wood may vary from 15 to 65% and such erratic distribution and variation are supposed to be responsible for some of the commonly observed defects that may occur during drying and processing. The other characteristics of rubber wood are summarized in Table 2.1.

Table 2.1 Properties of rubber tree bark wood elements

Basic wood density (kg m^{-3})	543.7
Basic bark density (kg m^{-3})	620.8
Double bark thickness (mm)	11.6
Bark proportion (%)	7.7
Fibre length (mm)	1.189
Fibre proportion (%)	58
Vessel proportion (%)	8.5
Ray proportion (%)	22.0
Parenchyma (%)	11.5

Since the mid 1980 rubberwood is one of the most popular timbers for making furniture and other wood based products and is an important resource for the timber industry and has now become one of the major money earners for this industry. The main reason for the popular of rubberwood rest with its light color, beautiful grain, even properties, low shrinkage rate, and good dimensional stability. The strength of the lumber is also good, and it is easy to process, such as sawmilling, peeling, drilling, adhesion and painting.

The disadvantages are also evident in rubberwood.

1. Fast biodegradation and susceptibility to insect infestations after felling, the rubberwood logs must be sawn as soon as possible or preservation.
2. Comparison to other species, the dimension of rubberwood lumber is small.
3. Drying defects are easily occurred, such as warp, twist, bow and splitting.

Because of rubberwoods are susceptible to stain fungi, which discoloration of the wood, and insects which reduce the quality and durability of timber. So rubberwood should be treated, especially for furnisher making.

2.3 Wood modification methods [2]

Modification of wood can involve active modifications, which result in a change to the chemical nature of the material, or passive modifications, where a change in properties is effected, but without an alteration of the chemistry of the material.

2.3.1 Heat treatment

The application of heat to wood results in degradation associated which chemical changes in the material, the physical and biological properties of the wood are altered. These changes include the following belows:

- Improvement in dimension stability, depending on the treatment condition
- Reduced hygroscopicity
- Improved resistance to microbiological attack
- A reduction in impact toughness, modulus of rupture and work to fracture
- Reduced abrasion resistance
- A tendency for cracks and splits to form, knot to come loose
- A darkening of the material

The properties of treated wood are highly dependent upon the heat treatment employed. If carefully controlled, the property changes that are obtained due to heat treatment can be used for certain applications.

2.3.1.1 The effect of temperature and the duration of treatment

As wood is heated, there is a decrease in weight of the material initially, due to the loss of bound water and volatile extractives, with less volatile extractives tending to migrate to the surface of the wood. As the temperature is increased, chemical changes to the macromolecular components of the cell wall occur, accompanied by further weight loss and color changes.

2.3.1.2 The effect of the treatment atmosphere

Treatment can be performed in air, in a vacuum or under an inert atmosphere. In some early studies, air was excluded by heating the wood under molten metal. Clearly, treatment in oxygen results in oxidative process occurring, and there are significant differences in chemistry of degradation as well as the material properties obtained.

2.3.1.3 Effect of species

Differences have been noted between species in the way in which they respond to heat treatment, but most notably between hardwoods and softwoods. Thermal, hydrothermal or hygrothermal treatment of various woods results in weight losses that are generally found to be higher for hardwood compared to softwood species.

2.3.1.4 Chemical changes in wood due to thermal modification

The heating of wood will lead to a variety of processes occurring, which depend upon the heating regimen employed. It is accepted that hemicelluloses are degraded to a greater extent than the other macromolecular components, but the relative stability of the cellulose and lignin is harder to determine.

2.3.2 Wood preservative process [6, 7]

Wood preservatives are chemical substances. When wood preservatives are applied to the wood, make it resistant to attack by fungus, insects, or marine borers. Preservatives may be either pure or mixtures chemical compounds. They vary widely in character, cost, and suitability for use under different method. A wood preservative should be toxic to the wood destroyers, permanent, penetrative, safe to handle and use, colorless, odorless, non-swelling, fire resistant, moisture repelling, and economical or have certain combination of these properties.

Preservatives are classified in various types, according to their chemical and physical characteristics such as oil and oil-borne preservatives and water-borne preservatives

Oils an oil-borne preservatives

In this group are the by-product preservative oils in general use, including coal-tar creosotes, creosote-coal-tar solutions, and creosote-petroleum solutions. It also comprises by-product oils in less common use, such as the wood oils water-gas tar and creosotes, oil-tar creosotes, lignite-tar oils, and others. Preservatives prepared by dissolving toxic chemicals in low-cost solvent oils are also contained in this group. The solvents may be heavy petroleum fuel oils. Light fuel oils, or any other oil that is cheap enough and has required properties. The principal toxic chemical used in this way at present is pentachlorophenol.

The principal advantages of coal-tar creosote as a wood preservative are (a) its marked toxicity to wood-destroying fungi, marine borers, and insects; (b) its relative insolubility in water and low volatility, to which the oil owes its high degree of permanence under the most varied service conditions; (c) its ease of application; (d) the facility with which its depth of penetration can be determined; and (e) its general availability and relatively low cost.

Water-borne preservatives

Water has several advantages as a solvent for wood preservatives, chief of which are cheapness and availability. It also penetrates wood wall and is free from fire, explosion, and health hazards. Its chief disadvantages are that wood impregnated with it swells upon treatment, requires redrying for most purposes, and shrinks upon drying. Furthermore, water-borne preservatives afford no protection against weathering or mechanical wear. Because of their solubility in water, the older preservatives of this kind are subject to leaching from treated wood whenever it is in contact with wet soil or water. As a class, therefore, such water-borne preservatives have been found less suitable than those of the oil-borne type for use in wet places. On the other hand, when leaching is not a factor they should remain in the wood and protect it indefinitely. Wood treated with water-borne preservatives is especially suitable for use in buildings because of its cleanness, freedom from odor, lack of preservative fire hazard, low increase in weight (after drying), and relatively low cost.

Most wood preservertive system now in use may be classified roughly as either nonpressure processes, carried out without use of artificial pressure, or pressure process, which wood is placed in a treating cylinder and impregnated with preservative under considerable force.

2.3.2.1 Steeping and cold soaking

Cold soaking is generally applied to use of the oil solution while steeping use to soaking of wood in water solution of preservative. The preservative solution usually allowed to remain at atmospheric temperature, although more rapid penetration would be obtained if it were heated somewhat. When treatment is applied to seasoned timbers preservative it contain soak into the wood.

Absorption is most rapid during the first 2 or 3 days but will continue at a decreasing rate for an indefinite period. Consequently, the wood can be left in the solution, the better treatment it will receive. If soaking times were continued large enough. The advantage of this process are numerous such as simple, low cost, achieve excellent preservation penetration and loading provided that adequate time is available.

2.3.2.2 Impregnation Process

The preservation of wood by pressure process is prefered commercial approach because of its greater efficiency and effectiveness. The wood is placed in a pressure vessel, usually called a cylinder or autoclave. A preliminary vacuum is applied to the cylinder and held for a period up to on hour. Then, with out releasing the vacuum, the cylinder is filled with preservative solution and maintained until the desired absorption is reach. During releasing of air from the wood followed by admission of preservative solution, creates a slight preservative pressure on the wood.

The principle impregnation process, is to impregnate the cell wall of wood with a chemical, or a combination of chemicals, that then react so as to form a material that is locked into the cell wall. For this to occur, it is necessary that during the impregnation phase that the cell wall is in a swollen state. It is self-evident that the molecular components of the impregnate should be small enough that they can gain access to the cell wall interior.

A very important factor in ensuring that full cell wall penetration has occurred is to allow sufficient time for the impregnant molecules to diffuse into the intracellular spaces. Many workers allow several days (weeks in some cases) for this to occur. It is important to emphasize that pressure treatment will aid penetration of larger wood samples, but will not in any way result in cell wall penetration, which is a purely diffusion-controlled process.

2.3.3 Chemical Modification

Most of the chemical modification methods investigated to date have involved chemical reaction of a reagent with the cell wall polymer hydroxyl groups. The chemical nature of the cell wall polymer is thus changed, which may be responsible for the new properties of wood. Chemical modification of wood has been the subject of a number of reviews see in literature survey.

2.4 Wood degradation by decay fungi [8]

Subject to the necessary conditions, wood is susceptible to degradation by microbiological organisms. In order for fungal attack to occur, three components are required: water, oxygen and a source of nutrients. One of the best ways of preventing fungal attack is to prevent the wood from reaching sufficiently high moisture content, and good design of wood structures should ensure that this is the case.

2.4.1 Brown rot

Brown rot is caused by Basidiomycetes, which metabolize the carbohydrate cellulose and hemicelluloses of the woody cell wall by non-enzymatic and enzymatic action and leave the lignin almost unaltered.

Brown-rot fungi do not produce lignin-degrading enzymes. There is however reports of lignin peroxides and manganese peroxidase in some brown-rot fungi, and lignin loss or metabolization by brown-rot fungi have been reported. Particularly in later stages of decay, the highly lignified middle lamella/primary walls were observed to undergo attack. Also, the penetration of the wood cell wall by bore holes removes lignin in the process, all suggesting that low molecular weight lignin degrading agents and potentially even lignin degrading enzymes max occur in some brown-rot fungi, at least with localized activity.

2.4.2 White rot

White rot is means the degradation of cellulose, hemicelluloses, lignin usually by Basidiomycetes and rarely by Ascomycetes, Kretzschmaria deusta and Xylaria hypoxylon. White rot has been classified by macroscopic characteristics into white-pocket, white-mottled, and white-stringy, the different types being affected by the fungal species, wood species, and ecological conditions. From microscopic and ultrastructural investigations, two main types of white rot have been distinguished.

In the simultaneous white rot (“corrosion rot”), carbohydrates and lignin are almost uniformly degraded at the same time and at a similar rate during all decay stages. Typical fungi with simultaneous white rot are Fomes fomentarius, Phellinus igniarius, Phellinus robustus, and Trametes versicolor in standing trees and stored hardwoods.

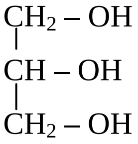
2.4.3 Soft rot

Soft-rot fungi differ from brown-rot and white-rot Basidiomycetes by growing ainly inside the woody cell wall. The wood is colonized via the wood rays. In conifers, the fungi penetrate, starting from the tracheidal lumina, by means of thin perforation hyphae of less than 0.5 µm thickness into the tertiary wall and re-orientate then as thin hyphae after L-bending in one direction or after T-branching in both directions along the microfibrils in the secondary wall.

2.5 Drying oils [9, 10]

Oils used in coatings are classified broadly as vegetable oils and marine oils. Chemically they are triglycerides, i.e., compounds of one molecule of glycerin and three molecules of long chain fatty acids. Fats are also triglycerides. They differ from oils in that they are solids rather than liquids at normal temperatures. Drying oils are converted by the oxygen of air to dry, hard, insoluble resinous materials. The oils commonly used in coating today are tung, linseed, soybean, safflower, fish, and castor oils.

Triglycerides have the following composition:

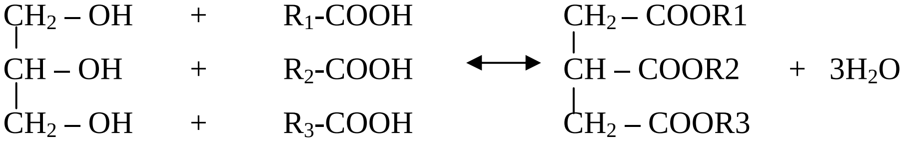


$$\begin{array}{l} \mathrm{CH_2 - OH} \\ | \\ \mathrm{CH - OH} \\ | \\ \mathrm{CH_2 - OH} \end{array}$$

$CH_3\text{-}CH_2\text{-}CH_2\text{-}CH_2\text{-}CH_2\text{-}CH_2\text{-}CH_2\text{-}CH_2\text{-}COOH$

Glycerol Fatty Acid

Fatty acid chain length can vary from C_9 to C_{22}



$$\begin{array}{lclcl} \mathrm{CH_2 - OH} & + & \mathrm{R_1\text{-}COOH} & & \mathrm{CH_2 - COOR1} \\ | & & & & | \\ \mathrm{CH - OH} & + & \mathrm{R_2\text{-}COOH} & \longleftrightarrow & \mathrm{CH - COOR2} \quad + \quad \mathrm{3H_2O} \\ | & & & & | \\ \mathrm{CH_2 - OH} & + & \mathrm{R_3\text{-}COOH} & & \mathrm{CH_2 - COOR3} \end{array}$$

Glycerol Fatty Acid Triglyceride Water

In the formulas shown above, R_1, R_2 and R_3 stand for fatty acid chains. If R_1, R_2, and R_3 are the same, a simple triglyceride is results. If they are not the same, the triglyceride is mixed. Triglycerides occurring in nature are usually of this type. This is a reversible reaction so that if we hydrolyze oil we obtain glycerine and fatty oils.

The nature of the fatty acid present in oil determines its characteristics. The fatty acid consists of a carboxyl group attached to a hydrocarbon chain. Saturated fatty acids have hydrocarbon chains containing no double bonds, each carbon having at least two hydrogen atoms. Fatty acids with chains containing double bonds are termed unsaturated. They may have one, two, three or more double bonds, whose position in the chain may vary. Two double bonds separated by a single bond are called conjugated.

The double bonds in unsaturated fatty acids are chemically reactive sites. The reaction of oxygen with the oil molecule at the double bond results in drying; usually the greater the unsaturation, the better is the drying. Saturated fatty acids are nondrying. Fatty acids with a single double bond are essentially nondrying. Fatty acids with three double bonds dry the most rapidly. However, in addition to the number of double bonds, the position of the double bond is important. Conjugated double bonds polymerize and dry more rapidly than isolated double bonds. Oils vary in drying properties depending on the degree of unsaturation of the fatty acid that will ologomerize or polymerize when exposed to the oxygen in air. The result is an increase in the molecular weight including cross-linking [11].

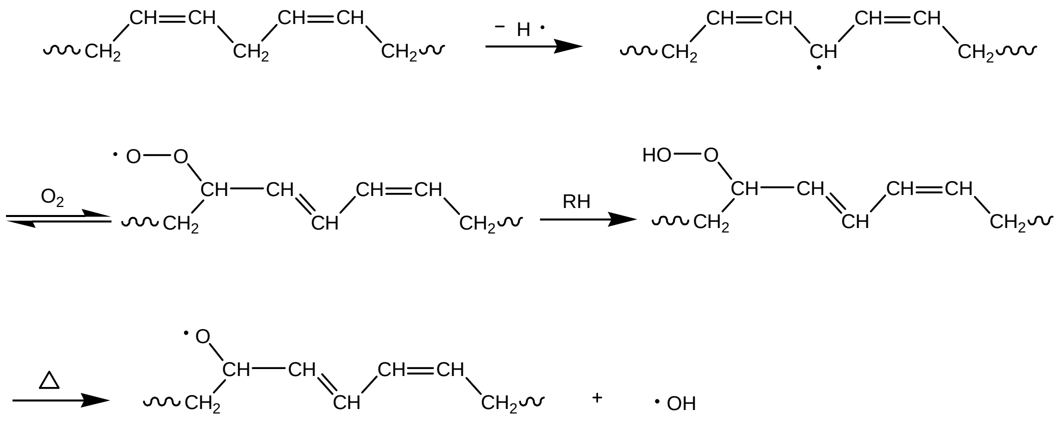


Figure 2.7 Cross-linking mechanism of linolenic acid on linseed oil.

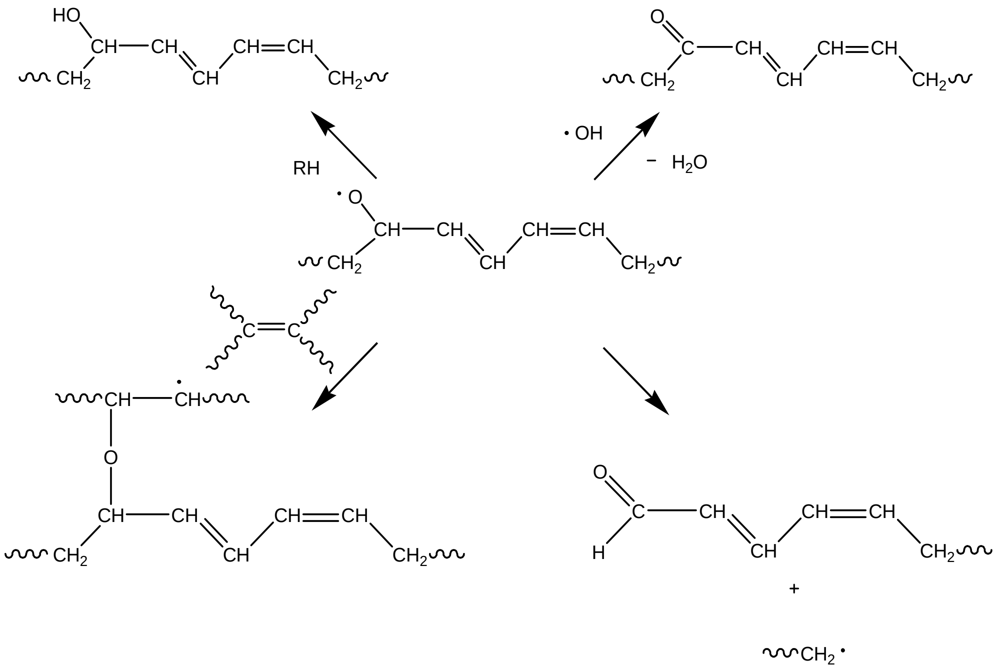


Figure 2.7 Cross-linking mechanism of linolenic acid on linseed oil (contined).

2.6 Tung Oil

Tung oil or china wood oil is obtained from the nut of the tung oil tree (*Aleurites fordii*), now grown in China. The oil content is about 50%. Tung oil contains about 80% eleostearic acid (Table 2.2) which is conjugated oil. Tung oil is pale yellow or darker liquid with valuable drying and polymerization properties because of its high content of eleostearic acid. Tung oil dries rapidly to a hard film which has good water and alkali resistance and good durability.

Figure 2.8 Nut of the tung oil tree.

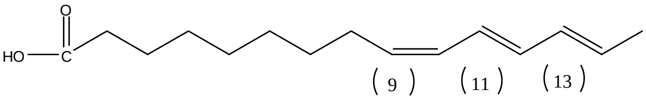


Figure 2.9 The chemical structure of eleostearic acid.

Tung oil will dry in the air to form a solid film. This drying is a result of polymerization by action of atmospheric oxygen (autoxidation).

Table 2.2 Fatty acid composition of oils [12, 13]

Fatty acid	**Tung oil**	**Linseed oil**	**Sunflower oil**	**Palm oil**
Lauric (C12:0)				0.5
Miristic (C14:0)			<0.1	1.5
Palmitic (C16:0)	8	5.5	6.9	45.5
Stearic (C18:0)	1	6	6	4
Oleic (C18:1)	8	21	26.5	38
Linoleic (C18:2)	4	14	66.5	10
Linolenic(C18:3)	3	53.5		0.5
Eleostearic (C18:3)	80			

2.7 Linseed Oil

Linseed oil is obtained from the seed of the common flax plant, *Linum usitatissimum*, *L*. Flax seed (Figure 2.10) has an average oil content of 35%. Linseed oil, historically, has been the most important oil in the coatings industry. Although its use has decreased because of other materials, linseed oil still leads in volume in the coatings industry. Linseed oil has a moderate drying rate. Because of its high linolenic acid content (Table 2.2) and its high iodine value, linseed oil yellows on aging. It is used as the principal vehicle in house paints and also as the drying oil in some alkyds.

Figure 2.10 Seed of flax plant.

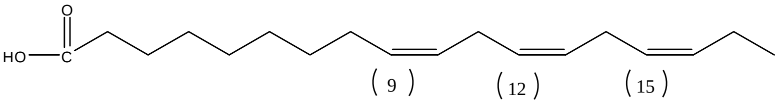


Figure 2.11 The chemical structure of linolenic acid.

2.8 Sunflower oil [14]

Sunflower oil is obtained from the seed of the (*Helianthus annuus*). The major composition of oil is linoleic acid (Table 2.2). There are three types of sunflower oil available; Traditional (Oleic 20-30%), Mid-Oleic (Oleic 55-75%) and High Oleic (Oleic 80-90%) sunflower oil. All are developed with standard breeding techniques. They differ in oleic levels and each one offers unique properties. With three types of sunflower oil available, sunflower oil meets the needs of consumer and food manufacturers alike for a healthy and high performance non-transgenic vegetable oil. It is light in taste, and sunflower oil provides more Vitamin E than all other vegetable oils.

Figure 2.12 Seed of Sunflower.

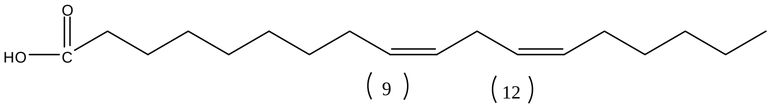


Figure 2.13 The chemical structure of linoleic acid.

2.9 Palm oil

Palm oil is obtained from the fruit and seed of the *Arecaceae elaeis* (Figure 2.14). Palm oil is one of the few vegetable oils relatively high in saturated fats and thus semi-solid at room temperature. Palm oil contains palmitic acid, oleic acid, linoleic acid and stearic acid (Table2.2). The oil is widely used as cooking oil and a component of many foods. For non-food application, it is also an important component of many personal care products and a feedstock for biodiesel.

Figure 2.14 Seed of palm.

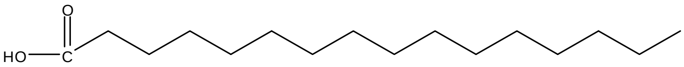


Figure 2.15 The chemical structure of Palmitic acid.

2.10 Epoxidized Oils [10]

Epoxides of all kinds of plant oils are well known commercially since they can undergo many important reactions. Epoxidized oils are produced by the reaction of unsaturated oils with peracetic acid (Figure2.16). The double bonds are converted to epoxy or oxirane group. Quantitative conversions of double bond to epoxide are expected if the reaction time is long enough. However, side reactions become competitive after a certain period of time. Such side reaction involves the ring-opening of oxiranes by acetic acid or water (by product). The efficiency of these epoxides is directly related to the amount of epoxy groups present in the molecule, the oxirane index being related to the iodine index of the starting oil. The epoxidized oil has potential uses as an environmentally friendly and they present a low toxicity for human. The use of epoxidized oils lies in the versatility of epoxide rings. Due to high reactivity of the oxirane ring, epoxides also act as a reactant raw material for a variety of chemicals, such as alcohols, glycols, alkanolamines, carbonyl compounds, olefinic compounds and polymers like polyesters, polyurethanes (PU) and epoxy resins. Then, epoxidation of long chain olefins, and unsaturated fatty acid derivatives such as palm oil and other plant oils is carried out on an industrial scale.

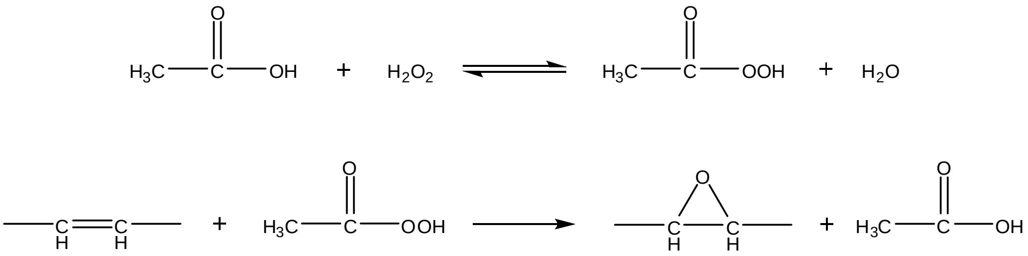


Figure 2.16 The reaction of fatty acid in vegetable oil with peracetic acid.

2.11 Literature Survey

In 1997, Rozman H.D. *et al.* [15] prepared WPC of rubberwood by impregnating the wood with glycidyl methacrylate (GMA), combination of glycidyl methacrylate (GMA-DAP) or diallyl phthalate (DAP) alone. Polymerization was carried out by catalytic heat treatment. The results showed significant WPC based on GMA exhibited greater dimension stability, flexural, compressive, and impact properties for all the samples were improved, especially for those with higher chemical loading.

In 2001, Sailer M. *et al.* [16] studied the wood treatment process by heat treatment and hot oil treatment. For the hot oil treatment, the wood samples were treated at 180-260°C with linseed oil and rape oil in an oil bath with exclusion of oxygen for 4.5 h. For heat treat, the wood samples were treated at 180-260°C. By thermal action the wood substance was converted, so that some properties of these materials were altered. Dimension stability and resistance to brown-rot decay fungus (*Coniophora puteana*) were improved.

In 2003, Honary L. A. T. [17] studied the improvement of wood by impregnation with soybean oil or a mixture of vegetable oils with soybean oil, which oil is caused to polymerize within the wood. Pressure and vacuum was applied in process to promote impregnation, and heat, blowing air, oxygen, ultraviolet light, and other agents was employed to promote polymerization of the treated wood. Impregnated samples showed significant improvement in to prevent pest infestations and the growth of fungi.

In 2005, Wang J.Y. *et al.* [18] studied in the fluence of oil type, temperature and time on moisture properties of white spruce wood. Wood samples were treated in commercial palm oil, soy oil and slack wax at 200°C and 220°C for 2 and 4 h in an oil bath. Wood samples were placed in an oven at 103°C for 24 h to obtain constant weights. The result showed significant improvement in antishrink efficiencies (ASE), water absorption and lower hygroscopicities for the high temperature treatments. Slack wax was better than palm oil or soy oil in improving the moisture performance of thermally treated wood and 220°C was superior to 200°C, with 4 h being generally better than 2 h treatment. The oil absorbed by wood benefits the performances of treated wood as well, especially by reducing water absorption.

In 2007, Korkut D.S. *et al.* [19] studied the influence of on physical properties and surface roughness of red-bud maple (*Acer trautvetteri Medw.*) wood. The wood samples were treated at 120°C, 150°C, and 180°C for 2, 6, and 10 h under atmospheric pressure. The result showed that density, swelling and surface roughness were decreased with increasing temperature and time treatment.

In 2007, Honghai D. *et al.* [20] studied the kinetics of epoxidation of soybean oil (SBO) by peroxyacetic acid (PAA) in the presence of sulphuric acid catalyst. Epoxidation with higher conversion of unsaturated carbon and lower oxirane cleavage can be attained by the *in situ* technique. The epoxidation of SBO by PAA generated *in situ* can be carried out at moderate temperatures with minimum epoxide degradation. The rate constant for epoxidation of SBO was found to be of the order of 10^{-6} $mol^{-1}{\cdot}s^{-1}$ and activation energy of epoxidation of 43.11 $kJ{\cdot}mol^{-1}$. The enthalpy, entropy and free energy activation were 40.63 $kJ{\cdot}mol^{-1}$, -208.80 $kJ{\cdot}mol^{-1}$ and 102.88 $kJ{\cdot}mol^{-1}$, respectively. The kinetic and thermodynamic parameters of epoxidation obtained indicate that an increase in the process temperature would promote the rate of epoxide formation.

In 2008, Kocaefe D. *et al.* [21] studied the influence of heat treatment on the wettability of white ash and soft maple. Wood samples were heated to 160–250°C in a thermogravimetric analyzer heating rate of 20 °C/h. The experiments were carried out under a gas atmosphere without the presence of oxygen. Contact angle measurements before and after heat treatment showed a significant increase in wood hydrophobicity. It was observed that this increase was more significant in the axial direction compared to the increase in the radial and tangential directions for both species. The contact angles in the radial and tangential directions were found to be similar.

In 2009, Boren H. [22] prepared wood and wood products by impregnating with a wood preservative containing vegetable oil without drying agents or agents causing polymerization. Wood samples were heated to a temperature of over 50°C before impregnated with a wood preservative and after impregnation wood samples were heated to a temperature of over 100 °C at least 75 min for removing wood preservative from the surface layers of wood.

In 2009, Matan N.*et al*. [23] studied the influence of peppermint oil, eucalyptus oil and their main components (menthol and eucalyptol) against molds (*Aspergillus niger*, *Penicillium chrysogenum*, and *Penicillium* sp.) and a white-rot decay fungus (*Trametes versicolor*) on rubberwood by vacuum process. The broth dilution method and the agar diffusion technique were employed to determine the minimal inhibitory concentration (MIC) and the minimal fungicidal concentration (MFC) using the concentration of substances between 100 and 800 $\mathrm{ml \cdot ml^{-1}}$. The result showed that the activities of peppermint oil and its main component, menthol, against molds on agar and rubberwood were stronger than those of eucalyptus oil and its main component, eucalyptol. Both peppermint oil and eucalyptus oil at the MICs of 300 $\mathrm{ml \cdot ml^{-1}}$ and 600 $\mathrm{ml \cdot ml^{-1}}$, respectively, was showed moderate resistance to white-rot fungal decay and high resistance to termite attack on rubberwood.

In 2009, Nakayama F.S. *et al*. [24] studied the influence of kukui oil (*Aleurites moluccana*) for controlling termite southern yellow pine (Pinus spp.) wood by impregnation process. Oil was obtained by pressing of the nut from the kukui plant and diluted with acetone to obtain oil concentrations ranging from 6.5, 12.3, 24.8, 50.0, and 66.0% (w/w) for impregnation into the wood blocks. The result showed that the oil treated wood was increased the resistant to termite damage when the wood contained >27% kukui oil by weight. Results also indicated that the oil acted primarily as a feeding deterrent and not a toxic agent.

CHAPTER III

EXPERIMENTAL

3.1 Materials

3.1.1 Rubberwood

Commercial rubberwood was obtained from wood industry and it was treated by copper-chrome-arsenic (CCA) as wood preservative. Fresh wood was prepared from freshly cut rubberwood obtained from eastern part of Thailand. The samples used in this study were sawn into required dimension of specimens for testing.

3.1.2 Chemicals

1. Palm oil
2. Linseed oil
3. Sun Flower oil
4. Tung oil
5. Hexane (commercial grade)
6. Glacial acetic acids 99 % (Merck)
7. Ethyl acetate (commercial grade)
8. Sulphuric acid, H_2SO_4, 98% purity (Merck)
9. Sodium carbonate, Na_2CO_3 (Merck)
10. Sodium chloride, NaCl (commercial grade)
11. Ethanol (Merck)
12. Chloroform-d, $CDCl_3$ (NMR spectroscopy grade (Merck))
13. Vanillin solution (containing vanillin (1%) and conc. H_2SO_4 (4.5 %) in ethanol)
14. Methanol (Merck)

3.2 Glassware and equipments

1. Oil bath
2. Hot plate with magnetic stirrer
3. Reflux condenser set
4. Rotary evaporator
5. Separation funnel with stand and clamps
6. Thermometer
7. 1000 ml two-necked round bottom flask
8. Beaker
9. Filter paper
10. Vacuum chamber
11. Vacuum pump
12. Autoclave

3.3 Instruments

1. Fourier transform infrared spectrophotometer (FT-IR)
2. Nuclear magnetic resonance (NMR)
3. Scanning electron microscope (SEM)
4. Universal testing machine (UTM)
5. Goniometer

3.4 Experimental procedures

3.4.1 Preparation of rubberwood sample

Rubberwood samples used for testing were cut into dimension required of test by using electric saw. The rough surface of samples was rubbed by sandpaper in order to remove the woolly fiber and make the smooth surface. Thus, precise measurement of the dimension can be made.

3.4.2 Preparation of epoxidized tung oil, linseed oil, sunflower oil and palm oil [20]

Epoxidation of oil was carried out by a procedure reported by Honghai D. *et al*. [20.]. Oil (150 g, 0.172 mol) was placed in a 1000 mL two-necked round bottom flask equipped with a reflux condenser and thermometer. Peracetic acid was prepared *in situ* by reacting 99.5% glacial acetic acid (35g, 0.58 mol) and 30% H_2O_2 (165g, 1.46 mol) in the presence of concentrated sulphuric acid (0.1 mL) for about 3 hours and then added slowly to the oil in the round-bottom flask. The reaction mixture was stirred and heated to maintain the reaction temperature at 80°C.

At the end, the mixtures were quenched by cooling in ice bath to stop the epoxidation reaction and then poured into a separating funnel. The aqueous layer was drawn off and the oil layer was washed with saturated sodium carbonate solution until the pH was neutral. The oil layer was washed again with saturated sodium chloride solution and distilled water, respectively. The oil layer was dried over anhydrous magnesium sulfate and then filtered.

3.4.3 Heat treatment [25]

Effect of heat treatment was studied on commercial wood and fresh wood. The rubberwood samples were dried in the oven at 100, 120, 140, 160, 180 and 200°C until constant weight was obtained. Weight and appearance of samples were measured every hour to investigate mass loss and failure of wood. Mass loss of sample due to the heat treatment was calculated according to the equation (1):

$$\text{Mass loss (\%)} = \frac{[M_0 - M_1]}{M_0} x100 \qquad , (1)$$

Where M_0 = weight of sample before treatment

M_1 = weight of sample after treatment

3.4.4 Oils for rubberwood treatment

Eight types of oil, including tung oil, linseed oil, sunflower oil, palm oil, epoxidized tung oil, epoxidized linseed oil, epoxidized sunflower oil and epoxidized palm oil were used for rubberwood treatment.

3.4.5 Cold soaking process

The rubberwood samples without preservatives were dried at 140°C for 6 h. Then the warm samples were suddenly soaked in an oil bath containing oil neat or in solution of oil 25%, 50% and 75% w/w in ethyl acetate for required hours and then dried in the oven at 100±3 °C for 1 h.

3.4.6 Impregnation process

The rubberwood samples without preservatives were dried at 140°C for 6 h before impregnation. Then rubberwood samples were placed in vacuum chamber. Pressure in chamber were reduced to 30 mbar and maintained at 30 mbar for 30 min. Then oil solution was introduced into the chamber. When the samples were covered by the oil solution, introduction of oil was stopped. The samples were then soaked for appropriate time. The treated wood was removed and the excess solution was wiped off followed by drying in the oven at 140°C for 6 h.

3.5. Characterizations of oil

3.5.1 Determination of peroxide value [12]

To a mixture of 2 volumes of chloroform and 3 volumes of glacial acetic acid (15 ml), saturated potassium iodide solution (0.25 ml) and deionized water (30 ml) were added into the 250 ml of Erlenmeyer flask oil samples (2.5 g) was added. The mixture was titrated with 0.01M sodium thiosulfate until the yellow color disappeared. 1% starch (5 mL) was added into the mixture and continued the titration until the colour disappeared. The volume of sodium thiosulfate used was recorded. The peroxide value value was calculated according to the equation (2):

$$\text{Peroxide value (meq peroxide/kg oil)} = \frac{[SxM]}{g}x1000 \qquad , (2)$$

Where S = mL of sodium thiosulfate (blank corrected)

M = molarity of sodium thiosulfate

g = gram of sample

3.5.2 Viscosity index [ASTM D 445]

7 ml of each sample was added into viscometer tube, and the tube was inserted into oil bath of viscometer. After insertion, the viscometer was allowed to reach bath temperature, Use suction to adjust the head level of the test sample to a position in the capillary arm of the instrument about 7 mm above the first timing mark. With the sample flowing freely, measure, in second to within 0.1 s, the time required for the meniscus to pass from the first to the second timing mark. The time of sample used was recorded. The viscosity was calculated according to the equation (3):

$$\text{Viscosity (cSt)} = Ct \qquad , (3)$$

Where C = Constant of viscometer tube (mm^2/s^2)

t = measured flow time (s)

3.6 Characterizations of rubberwood

3.6.1 Physical properties

3.6.1.1 Oil loading [15]

Before soaking, the samples were dried in an oven at the optimum temperature of heat treatment and weight of sample was determined. After soaking, the excess oil of the treated rubberwood samples were wiped out and dried in oven. Then the treated samples were weighed again and the oil loading was calculated according to the equation (4):

$$\text{Oil Loading (\%)} = \frac{\left[W_1 - W_0\right]}{W_0} x100 \qquad , (4)$$

Where W_1 = Weight of treated wood
W_0 = Weight of untreated wood (oven dried)

3.6.1.2 Water Absorption [15]

Rubberwood samples were cut into 2.5 x 10 x 2.5 cm and their weight and dimension in radial, tangential and longitudinal were measured. Then, they were placed in water container and distilled water was added until water covered the upper surface of samples. All samples were immersed in water at room temperature for 24 h. After the samples were taken out and dabbed dry with cloth, weight and dimension of the samples were measured. The water absorption was calculated according to the equation (5):

$$\text{Water Absorption (\%)} = \frac{[W_1 - W_0]}{W_0} x100 \qquad , (5)$$

Where W_1 = Weight of samples after soaking

W_0 = Weight of samples before soaking

The dimension of testing specimen is shown in Figure 3.1.

Figure 3.1 Dimension for water absorption and volumetric swelling testing sample.

Water desorption of rubberwood samples after water absorption test were examined everyday and water desorption was calculated according to the equation (6):

$$\text{Water desorption (\%)} = \frac{[W_1 - W_0]}{W_0} x100 \qquad , (6)$$

Where W_1 = Weight of samples after exposure in air

W_0 = Weight of samples after water absorption test

3.6.1.3 Volumetric swelling coefficient [26]

The dimensional stability of rubberwood samples was evaluated using volumetric swelling (%). Thus, the volume of rubberwood sample before and after soaking was measured. Dimension stability was reported as volume swelling according to the equation (7):

Volumetric swelling coefficient (S) = $\frac{[V_1 - V_0]}{V_0} x100$, (7)

Where V_1 = Wood volume after water soaking

V_0 = Wood volume before water soaking

The dimension of testing specimen is shown in Figure 3.1.

3.6.1.4 Water contact angle measurement

Rubberwood samples were cut into 2.5 x 2.5 x2.5 cm. These samples were rubbed by sandpaper. Water contact angle were determined by using the rame' hart instrument standard goniometer. In this study water contact angle measurements were performed in the radial, tangential and longitudinal direction. The dimension of testing specimen is shown in Figure 3.2.

Figure 3.2 Dimension of testing samples for water contact angle and decay test.

3.6.2 Mechanical Properties

3.6.2.1 Flexural strength and modulus of elasticity (MOE) [ASTM D143-94]

Rubberwood samples were cut into 2.5 x 2.5 x 410 cm. weight and dimensions, width and thickness, of the rubberwood samples were measured. The samples were placed on a beam support, which spanned 30 cm. The position of the wood tangential surface nearest to the pitch must be faced up when the load was applied downward. Firstly, the values read from the deflectometer and the testing machine was adjusted to zero. Then, the load was applied continuously at low speed of 1.3 mm/min. Meanwhile, the values from the deflectometer were collected simultaneously at each increment of load. After that, the curves of the relation between the applied load and the center of the deflections were plotted. Finally, the flexural stress and MOE were calculated according to the equation (8) and (9):

$$\text{Flexural Stress} = \frac{3L\Delta W}{b\mathrm{b}^2} \quad , (8)$$

$$\text{Modulus of Elasticity (MOE)} = \frac{L^3\Delta W}{4bd^3\Delta S} \quad , (9)$$

Where L = The span between the centers of supports (cm)
ΔW = The increment in load (kg)
b = The mean width (tangential direction) of the sample (cm)
d = The mean thickness (radial direction) of the sample (cm)
ΔS = The increment in deflection (cm)

The dimension of testing specimen is shown in Figure 3.3.

Figure 3.3 Dimension for flexures stress and MOE testing specimen.

3.6.3 Microstructure

Microstructure of treated rubberwoods was observed by scanning electron microscope and microstructure of untreated rubberwood and treated rubberwood was compared. The photographs were taken from the fractured surface of some selected samples after flexural strength test. The rubberwood was dried and then coated with gold before scanning.

3.6.4 Decay test [27]

Untreated and treated rubbwewood samples were exposed to white rot fungus (*Trametes versicolor*). The rubberwood samples were autoclaved. White rot fungus cultured on 3% malt agar for 2 weeks were cut (diameter 0.5 mm) by sterile corkborer to place in culture bottles with rubberwod samples. The rubberwood samples were exposed to the fungus grown on 3% malt agar in the culture bottles (Figure 3.4) and then incubated at 25 °C and 70-80 humidity for 8 weeks. The rubberwood samples were removed, autoclaved, removed mycelium and oven dried until the weight was constant. The samples were removed after the stipulated time and examined microscopically to access the growth of microorganisms. The dimension of testing specimen is shown in Figure 3.2.

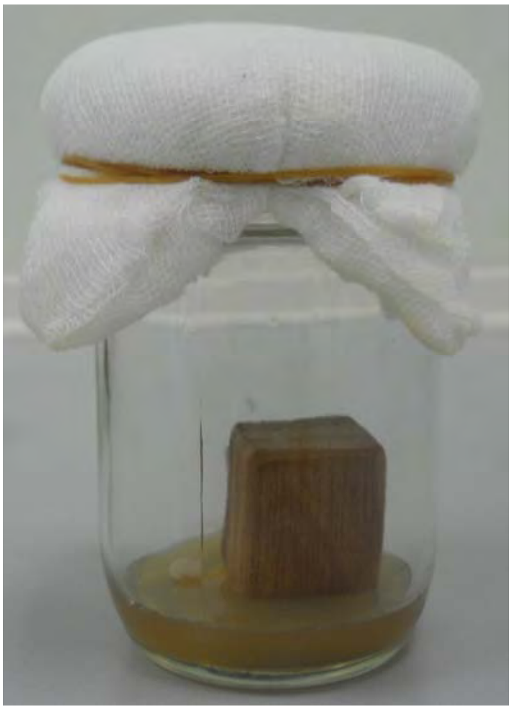


Figure 3.4 Rubberwood in culture bottle containing white rot fungus.

CHAPTER IV

RESULTS AND DISCUSSION

4.1 Characteristics of epoxidized oil

The H^1NMR spectrum of the oil and epoxidized oil were showed in Figures 4.1, 4.2, 4.3 and 4.4, respectively. For oil, a triplet signal at δ_H 0.75 ppm was corresponded to the ending methyl groups ($C\mathbf{H}_3$-(CH_2)n-) of fatty acid moiety. The signals at δ_H 1.2-1.4 ppm were corresponded to aliphatic methylene hydrogens (-$C\mathbf{H}_2$-). Beta hydrogens to the carbonyl group (-$C\mathbf{H}_2$-CH_2-C(O)-O-) were detected at δ_H 1.7 ppm. The signal at δ_H 2.05 ppm was corresponded to the allylic hydrogens (-$C\mathbf{H}_2$-CH=CH-). The alpha methylene hydrogens to carbonyl groups (-$C\mathbf{H}_2$-C(O)-O-) appear at δ_H 2.35 ppm. The signal of hydrogens between two double bonds (-CH=CH-$C\mathbf{H_2}$-CH=CH-) were detected at δ_H 2.7 ppm. The signals at δ_H 4.1-4.4 ppm were corresponded to methine and methylene hydrogens of the glyceride moiety. Finally vinyl hydrogens (-C**H**=C**H**-) of fatty acid moiety were detected at δ_H 5.3-5.5 ppm.

For epoxidized oil, hydrogens of oxirane moiety were detected at δ_H 2.7-3.2 ppm. Comparing oil and epoxidized oil the signals of hydrogens at δ_H 5.3-5.5 ppm were decreased. This result indicated that double bonds were converted to oxirane group. The percentage of epoxidation was calculated from the integrals of double bonds the signals at δ_H 5.3-5.5 ppm and epoxy ring hydrogen the signals at δ_H 2.7-3.2 ppm (see examples of calculation in appendix page 104). The percentage of epoxidized tung oil, epoxidized linseed oil, epoxidized sunflower oil and epoxidized palm oil were 30.39, 45.98, 49.89 and 78, respectively.

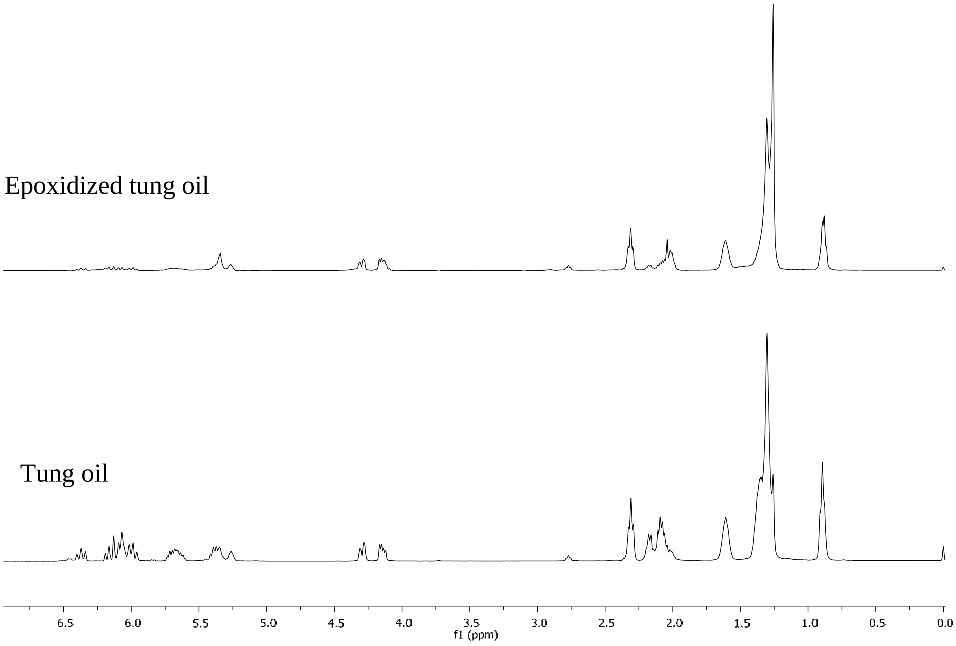


Figure 4.1 ^{1}H NMR spectrum of tung oil and epoxidized tung oil.

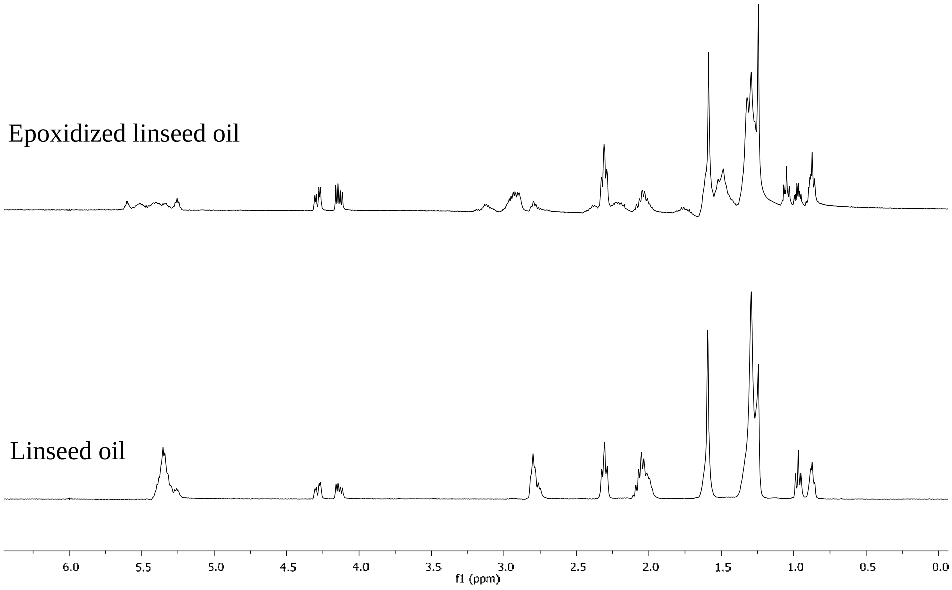


Figure 4.2 ^{1}H NMR spectrum of linseed oil and epoxidized linseed oil.

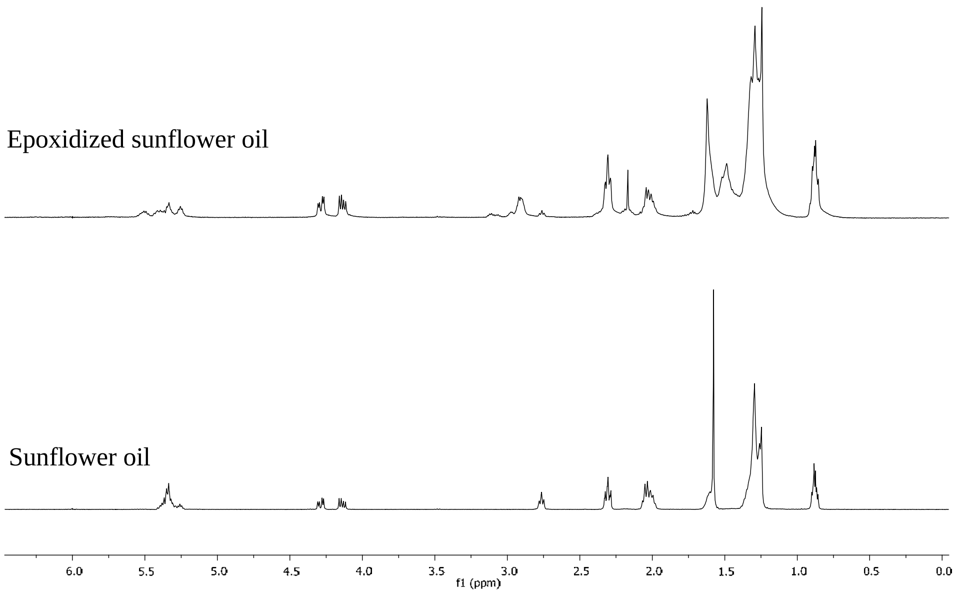


Figure 4.3 ^{1}H NMR spectrum of sunflower oil and epoxidized sunflower oil.

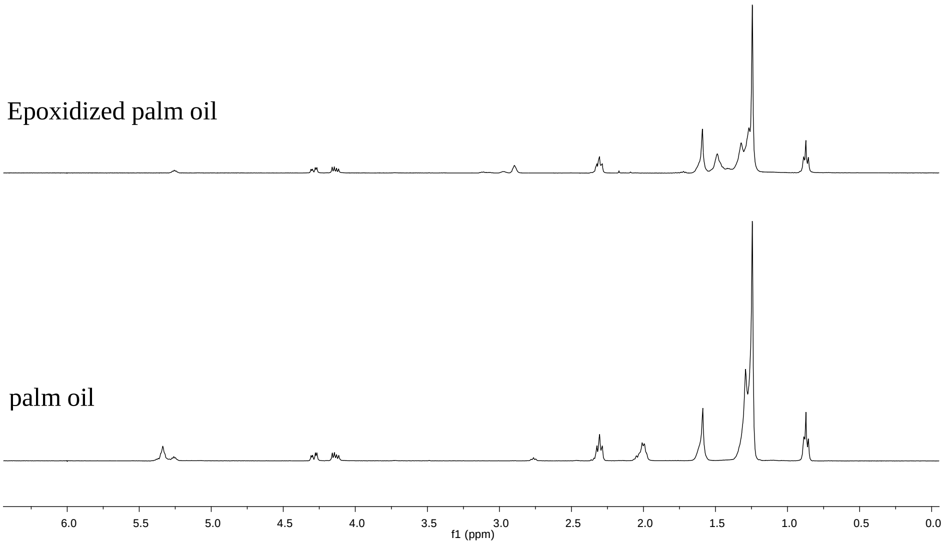


Figure 4.4 ^{1}H NMR spectrum of palm oil and epoxidized palm oil.

The FT-IR spectrum of oil and epoxidized oil as shown in Figures 4.5, 4.6, 4.7 and 4.8 were analyzed and crucial absorptions were summarized in Table 4.2. For oil, the absorption at 3026-3004 cm^{-1}, 1650 cm^{-1} and 724-717cm^{-1} were corresponded to the stretching vibration of the double bonds =C-H, C=C and *cis*-CH=CH, respectively. Absorption of C-O-C stretching of oxirane vibration was appeared at 1240-1230 cm^{-1} and 950-780 cm^{-1}. The most representative signal that evidences the oxirane group was the small intensity at 950-780 cm^{-1}. The identification of this absorption and the decreasing of the 3020 cm^{-1} double bonds band, indicated that epoxidation reaction was taken place. The absorption at 3400 cm^{-1} was attributed to the hydroxyl O-H stretching, indicated that some of epoxy group was opened.

Table 4.1 The IR absorption bands assignment of oils and epoxidized oils.

Functionality	Absorption band (cm^{-1})							
	TO	**ETO**	**LO**	**ELO**	**SFO**	**ESFO**	**PO**	**EPO**
C-O stretching peaks of ester	1156	1042-1234	1155-1237	1150-1237	1157-1240	1153-1240	1159-1237	1159-1237
C=O carbonyl of ester	1735	1735	1735	1735	1738	1738	1738	1738
C-H stretching of aliphatic	2847-2924	2847-2924	2850-2921	2850-2921	2850-2918	2850-2918	2854-2922	2854-2922
C-H vinyl	3023	-	3027	-	3005	-	3008	-
C-O-C stretching of oxirane	-	841-934	-	814-956	829-950	-	-	832-952

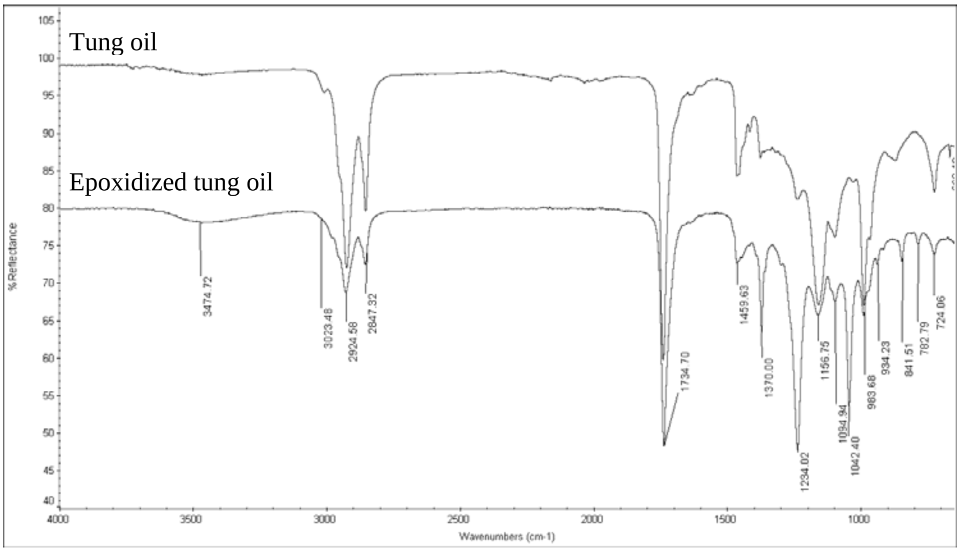


Figure 4.5 FT-IR spectrum of tung oil and epoxidized tung oil.

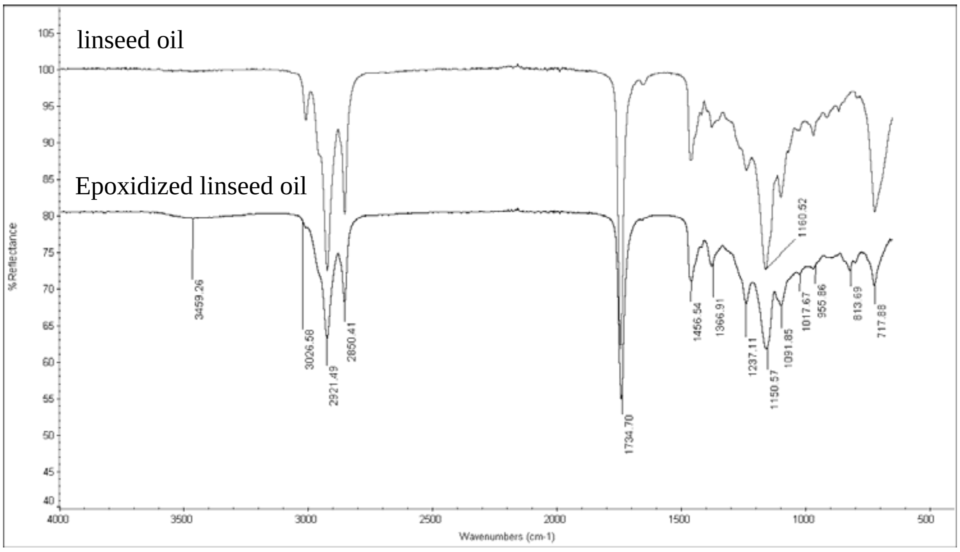


Figure 4.6 FT-IR spectrum of linseed oil and epoxidized linseed oil.

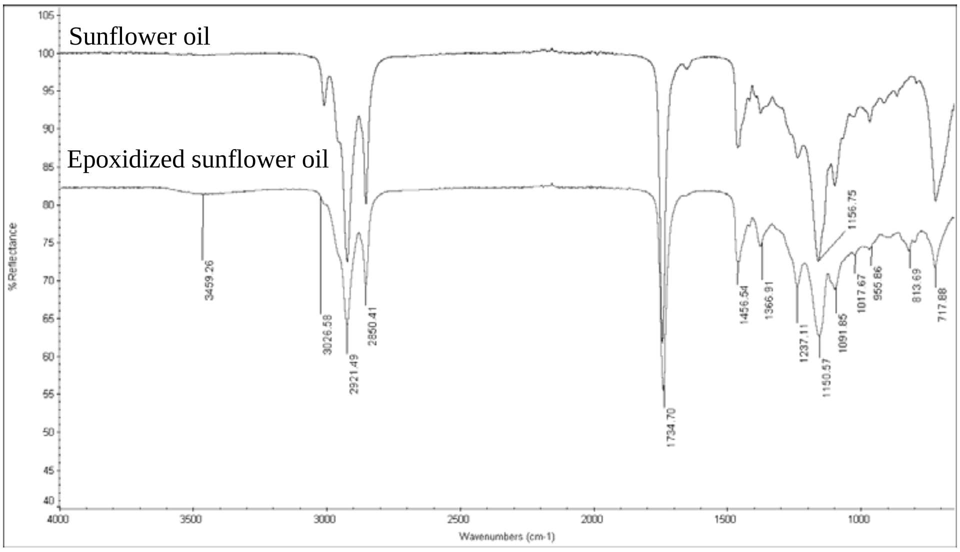


Figure 4.7 FT-IR spectrum of sunflower oil and epoxidized sunflower oil.

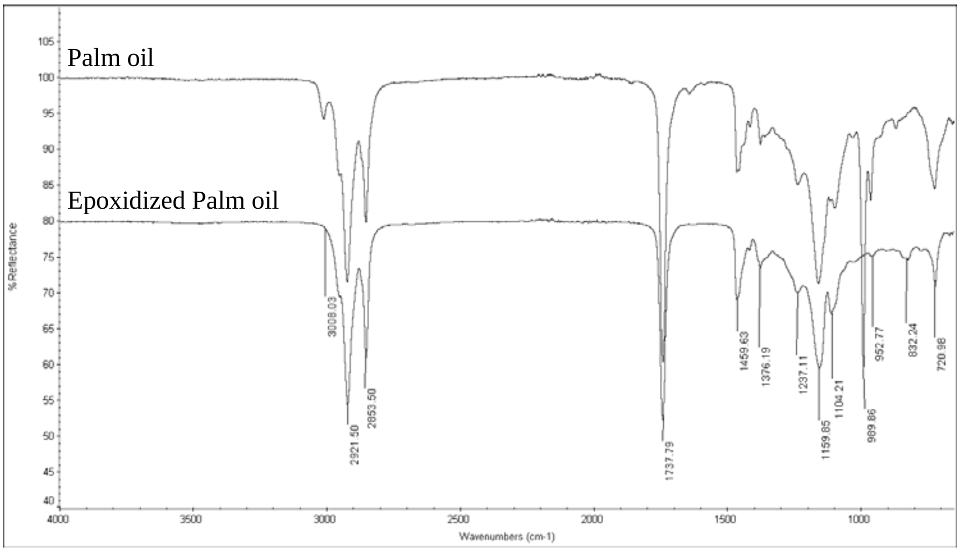


Figure 4.8 FT-IR spectrum of palm oil and epoxidized palm oil.

Table 4.2 shows viscosity of oil and epoxidized oil used in this study. The viscosity of oil and epoxidized oil were found to be differed. The viscosity of the epoxidized oils was higher than that of the corresponding oil.

Table 4.2 Viscosity of oil and epoxidized oil.

Oil	Viscosity (C*st*)
Palm	40.25
Sun flower	36.00
Linseed	28.50
Tung	166.75
Epoxidized Palm	82.50
Epoxidized Sunflower	108.50
Epoxidized Linseed	159.25
Epoxidized Tung	-

4.2 The effect of heat treatment on rubberwood

4.2.1 Mass loss

The effect of time and temperature on mass loss of commercial wood and fresh wood are shown in Figures 4.9 and 4.10, respectively. The treatment at high temperature had a significant effect on the mass loss of wood. In comparison between commercial wood and fresh wood, moisture in fresh wood was higher than commercial wood and mass loss rate of fresh wood was more than commercial wood. After heating both types of wood the appearance were similar. Increasing time and temperature, dark colour of wood was increased and shape of wood was distorted. The cause of colour changes after heat treatment was the degradation of hemicellulose [28]. According to the results obtained, the treated wood at 200ºC was found to be darker than other temperature. Figures 4.11 and 4.12 show photographs of rubberwood sample before and after heat treatment at 100-200ºC, respectively.

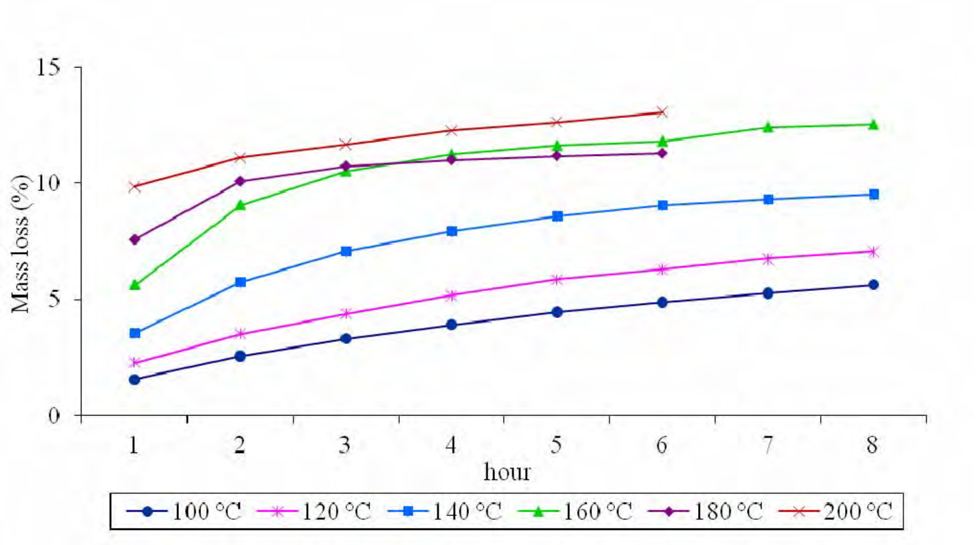


Figure 4.9 Effect of heat treatment on mass loss of commercial wood.

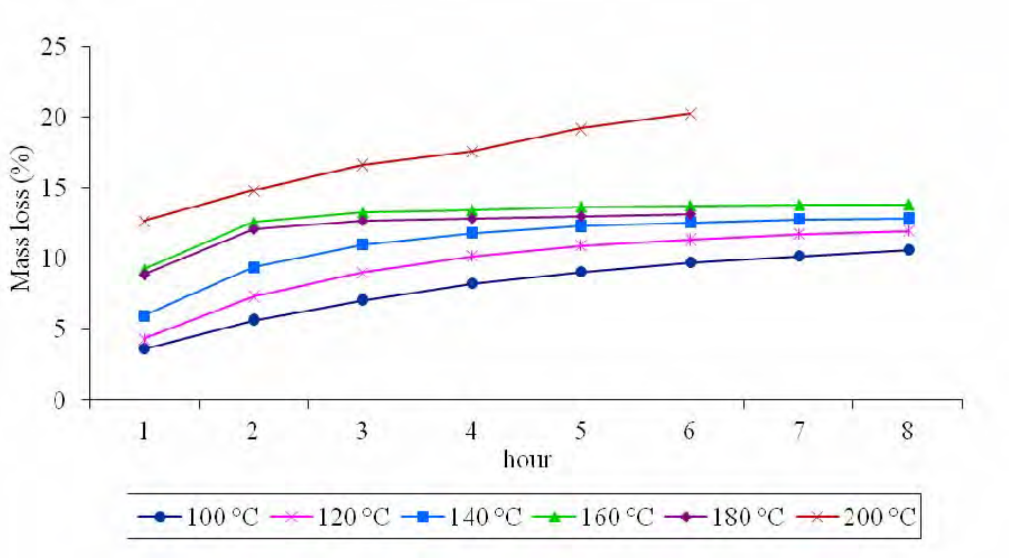
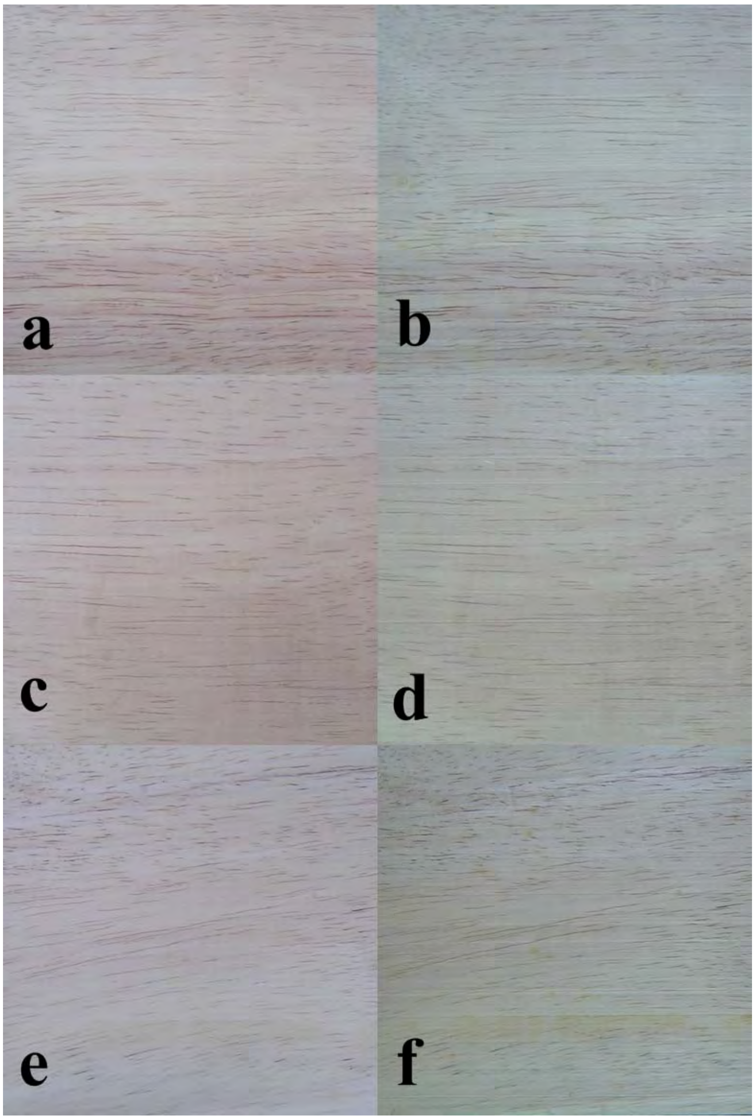


Figure 4.10 Effect of heat treatment on mass loss of fresh wood.



(a) Before heat treatment at 100°C

(b) After heat treatment at 100°C

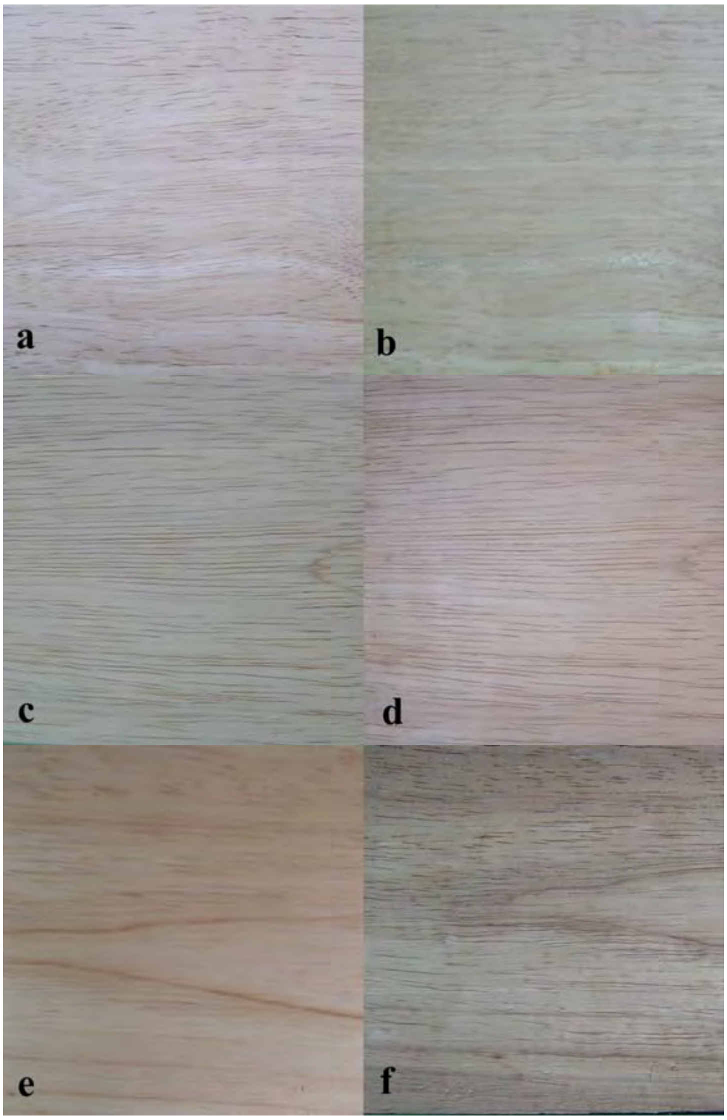
(c) Before heat treatment at 120°C

(d) After heat treatment at 120°C

(e) Before heat treatment at 140°C

(f) After heat treatment at 140°C

Figure 4.11 Comparison of rubberwood before and after heat treatment at 100-140°C.



(a) Before heat treatment at 160°C
(b) After heat treatment at 160°C
(c) Before heat treatment at 180°C
(d) After heat treatment at 180°C
(e) Before heat treatment at 200°C
(f) After heat treatment at 200°C

Figure 4.12 Comparison of rubberwood before and after heat treatment at 160-200°C

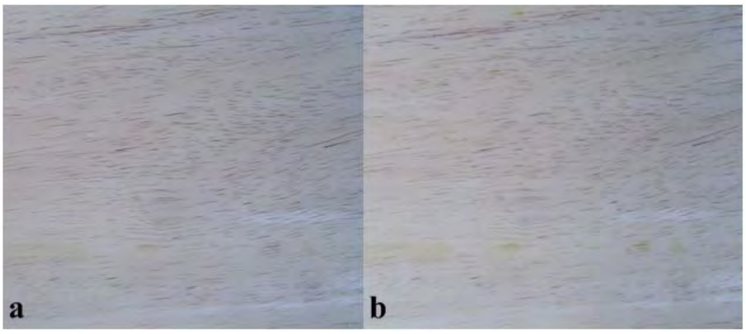


Figure 4.13 Comparison of rubberwood of (a) before (b) after heat treatment 140 °C for 6 h.

Figure 4.13 shows photographs of rubberwood sample before and after heat treatment 140°C for 6 h. The mass loss and appearance of treated wood at 140°C for 6 h were better than treated wood at other temperature. Thus, the rubberwood samples were dried at the 140°C for 6 h to reduce the moisture content in the part of oil treatment.

4.2.2 Hydrophobic property

The hydrophobic and hydrophilic properties of materials were measured by using water contact angle. The hydrophilic materials have a water contact angle (θ_a) 0-90° and hydrophobic materials have water contact angle (θ_a) 90-180°. Water contact angle of commercial wood and fresh wood are shown in Figures 4.14 and 4.15, respectively. Untreated wood showed hydrophilic property. Higher temperature of the treatment resulted in higher values of water contact angle. Commercial wood and fresh wood were hydrophobic when heat treatments were performed at above 140°C and above 160°C, respectively. This result was caused by moisture content of fresh wood that affected degradation of hemicellulose during the heat treatment process. In comparison of treated woods heat treatment of fresh wood gave more hydrophobic property than commercial wood. At the same temperature of treatment, commercial wood was less hydrophobic property than fresh wood. This should be caused by wood preservative containing in the commercial wood.

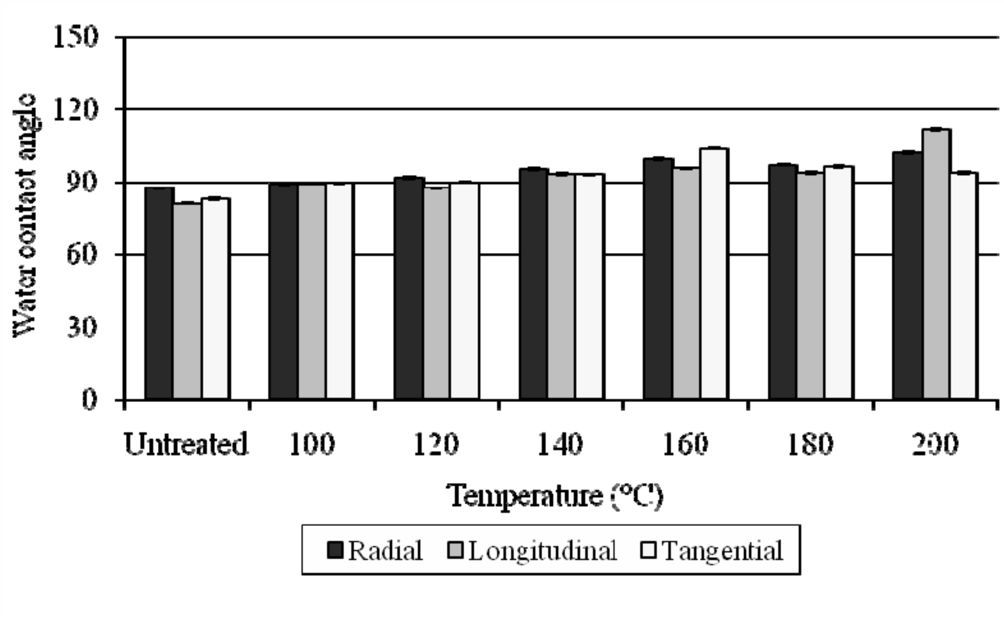


Figure 4.14 Effect of heat treatment on water contact angle of commercial wood.

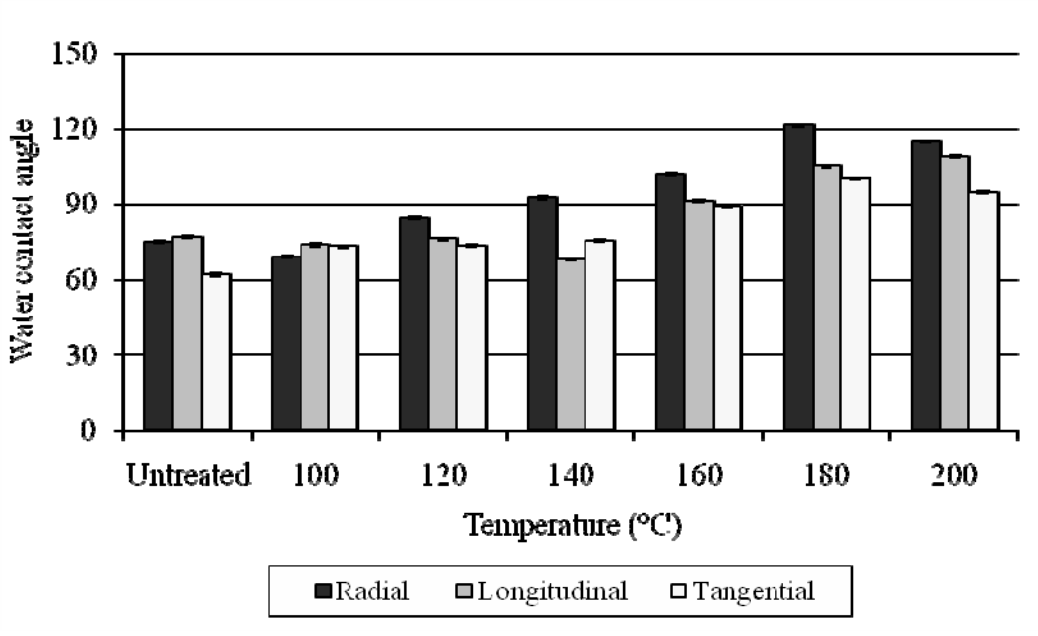


Figure 4.15 Effect of heat treatment on water contact angle of fresh wood.

4.2.3 Water absorption and volume swelling

In Figure 4.16 commercial wood and fresh wood treated at 160°C and 180°C showed the lowest water absorption. Overall water absorption of treated wood was not better than untreated wood but water desorption of treated wood (Figure 4.17 and 4.18) was faster than of the untreated. Also the treated wood at high temperature of treatment resulted in higher desorption rate of water. These could be explained that water absorption of wood was conducted by hydroxyl group while water desorption rate depended on hydrophobicity of the wood.

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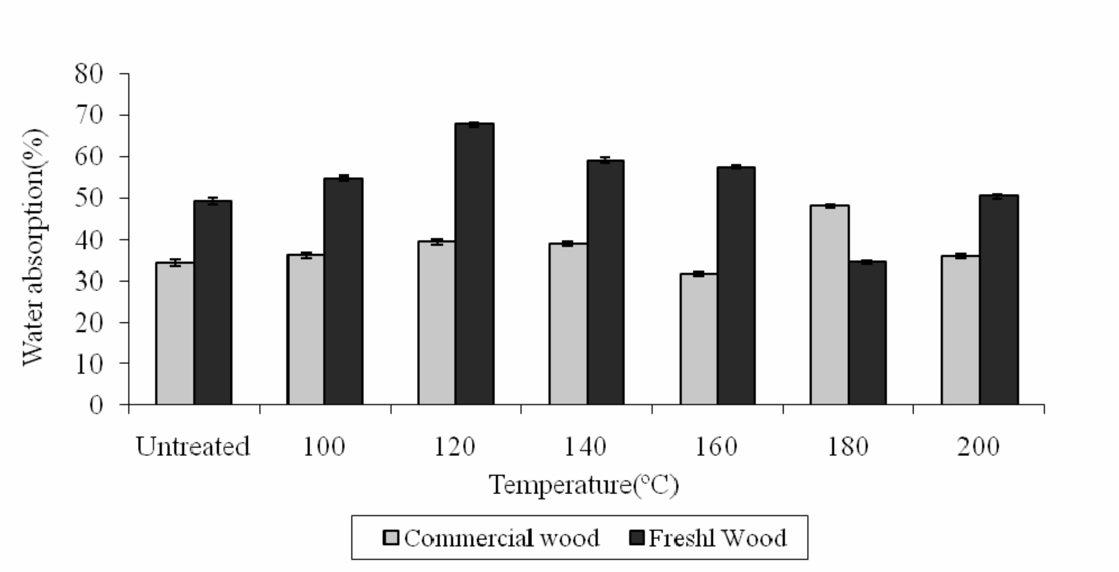


Figure 4.16 Effect of heat treatment on water absorption of wood.

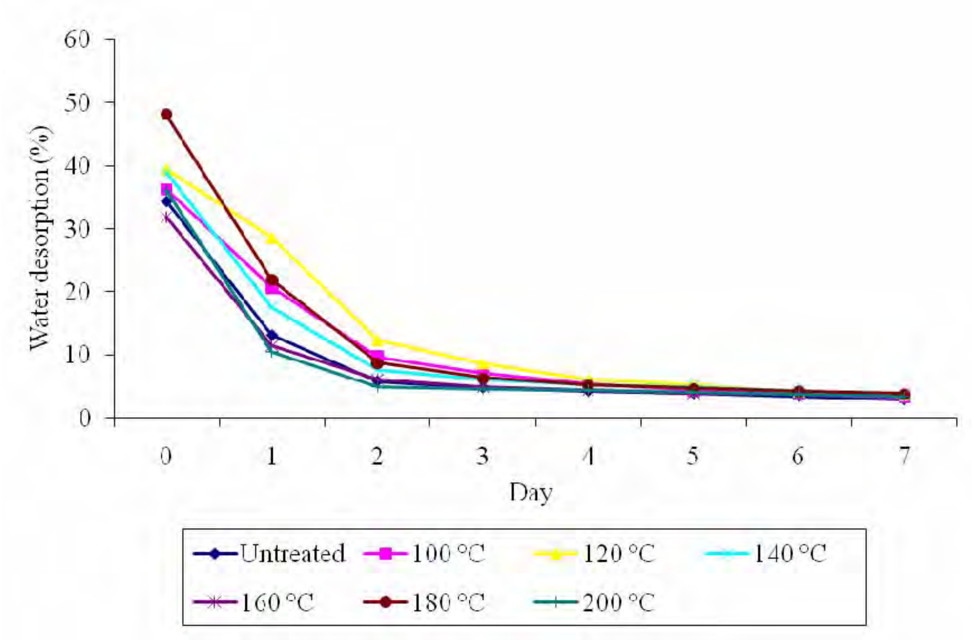


Figure 4.17 Effect of heat treatment on water desorption of commercial wood.

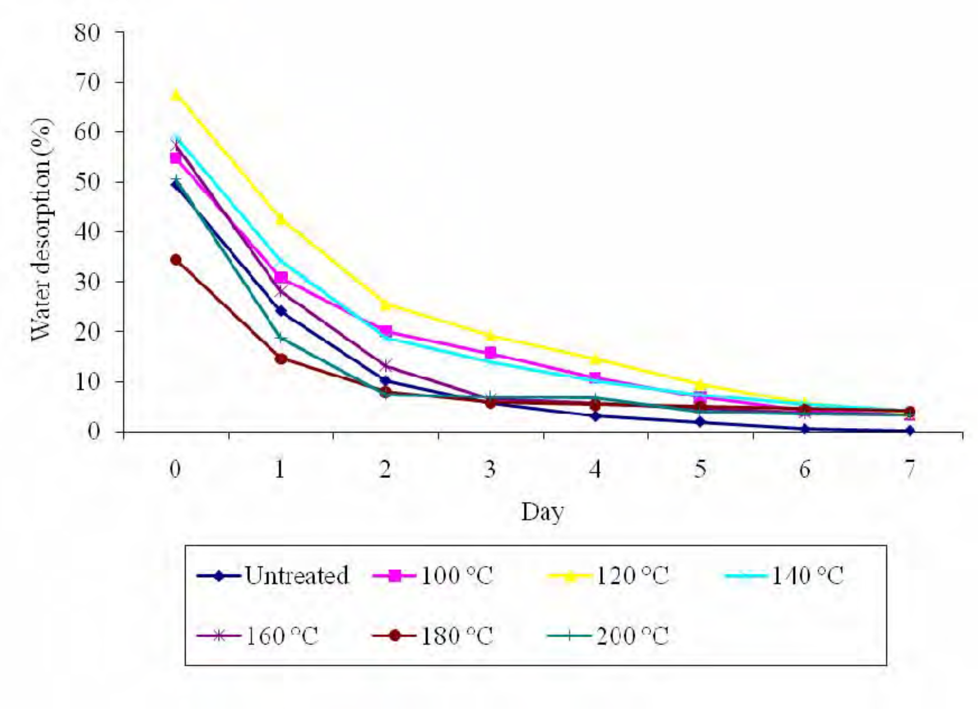


Figure 4.18 Effect of heat treatment on water desorption of fresh wood.

Figures 4.19 and 4.20 show the volume swelling of wood after 7 day of water desorption in atmosphere followed by oven drying at 103°C. The volume swelling of wood was similar except the volume swelling of 200°C treated wood was less volume swelling than other heat treatment. This may be caused by increased hydrophobic property of the wood.

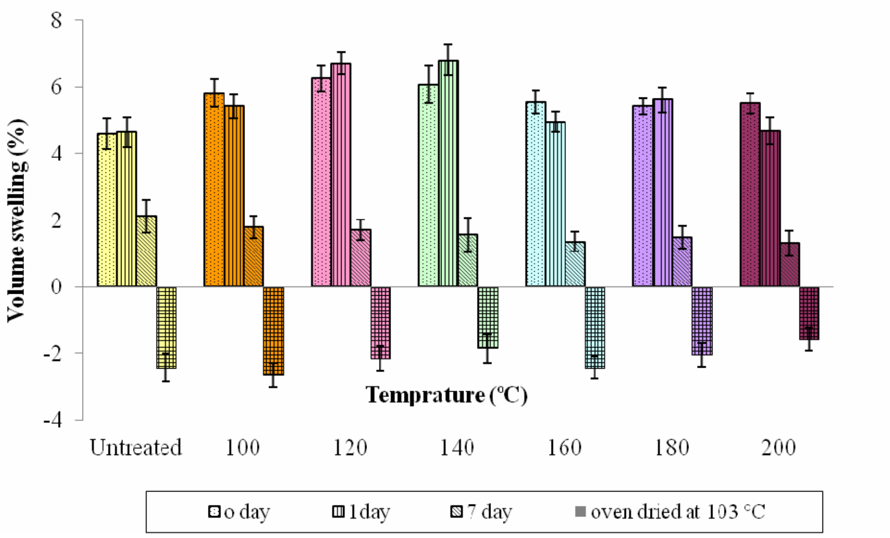


Figure 4.19 Effect of heat treatment on volume swelling of commercial wood.

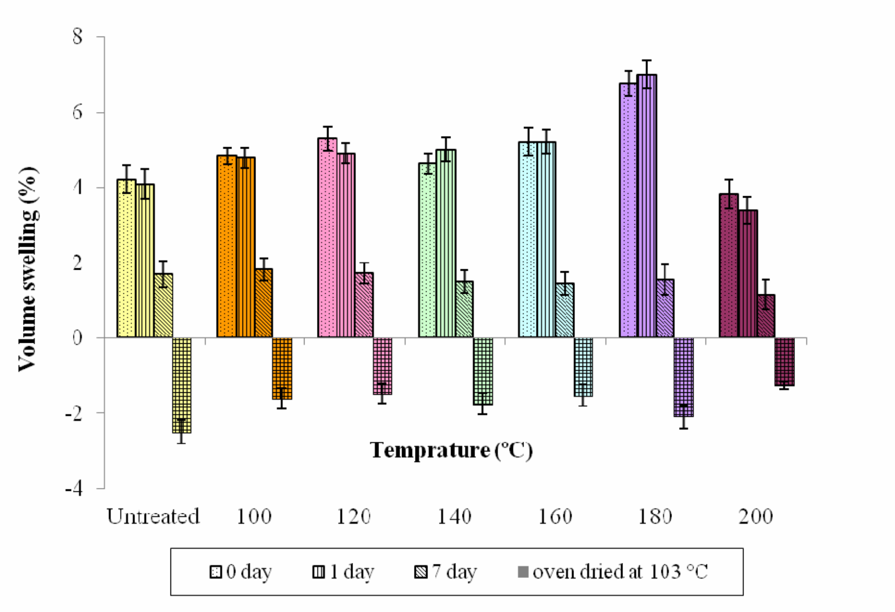


Figure 4.20 Effect of heat treatment on volume swelling of fresh wood.

4.3 Effect of oil treatment of rubberwood by cold soaking process

4.3.1 The effect of time on oil loading of rubberwood

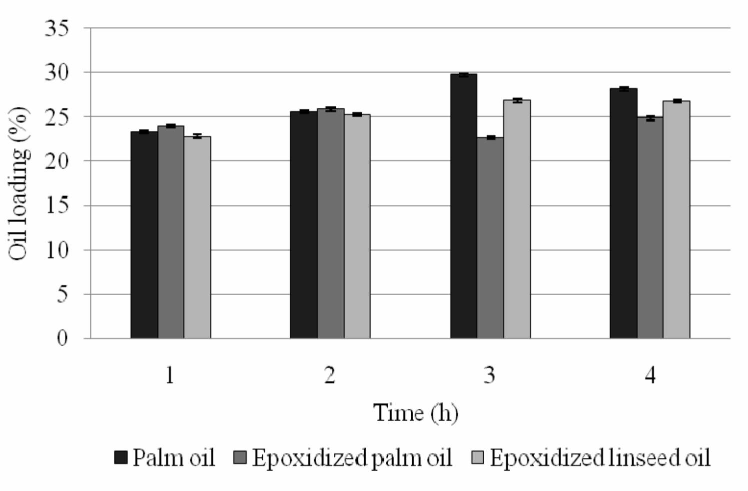


Figure 4.21 Effect of soaking time on oil loading of rubberwood with dimension of 2.5 x 10 x 2.5 cm.

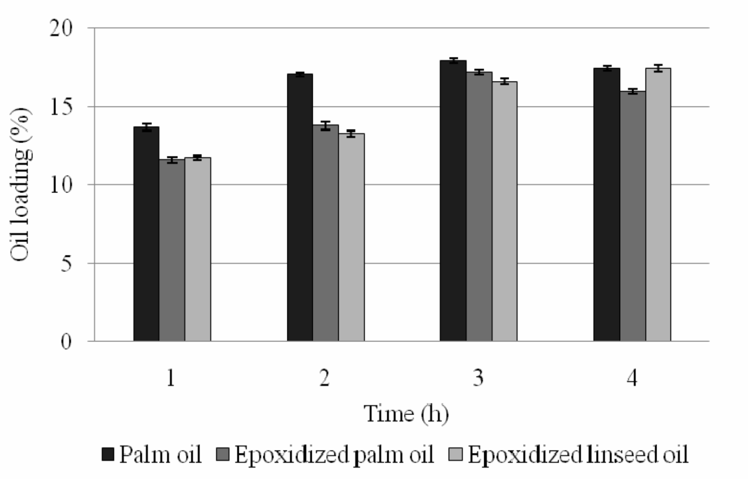


Figure 4.22 Effect of soaking time on oil loading of rubberwood with dimension of 2.5 x 2.5 x 410 cm.

In this experiment, rubberwoods were dried at 140°C for 6 h to reduce the moisture content of wood. Figures 4.21 and 4.22 show the effect of soaking time on oil loading of rubberwood at various sizes and type of oil. The soaking time was varied from 1, 2, 3 and 4 h. The results showed that oil loading obtained from 3 h and 4 h soaking time were not significantly different. Thus, the 3 h soaking time was used for the preparation of rubberwood in this study.

4.3.2 The effect of oil concentration on oil loading of rubberwood

Figure 4.23 shows the oil loading of the treated rubberwood. The rubberwood samples were treated by diluted oil in ethyl acetate at different concentrations, 25, 50, 75 and 100% w/w. At the same oil concentration, the results showed similar amount of the oil loading for all samples. Moreover, it can be seen that the oil loading was increased when using higher oil concentration. However, the oil loading of different sizes of rubberwood did not show significant difference.

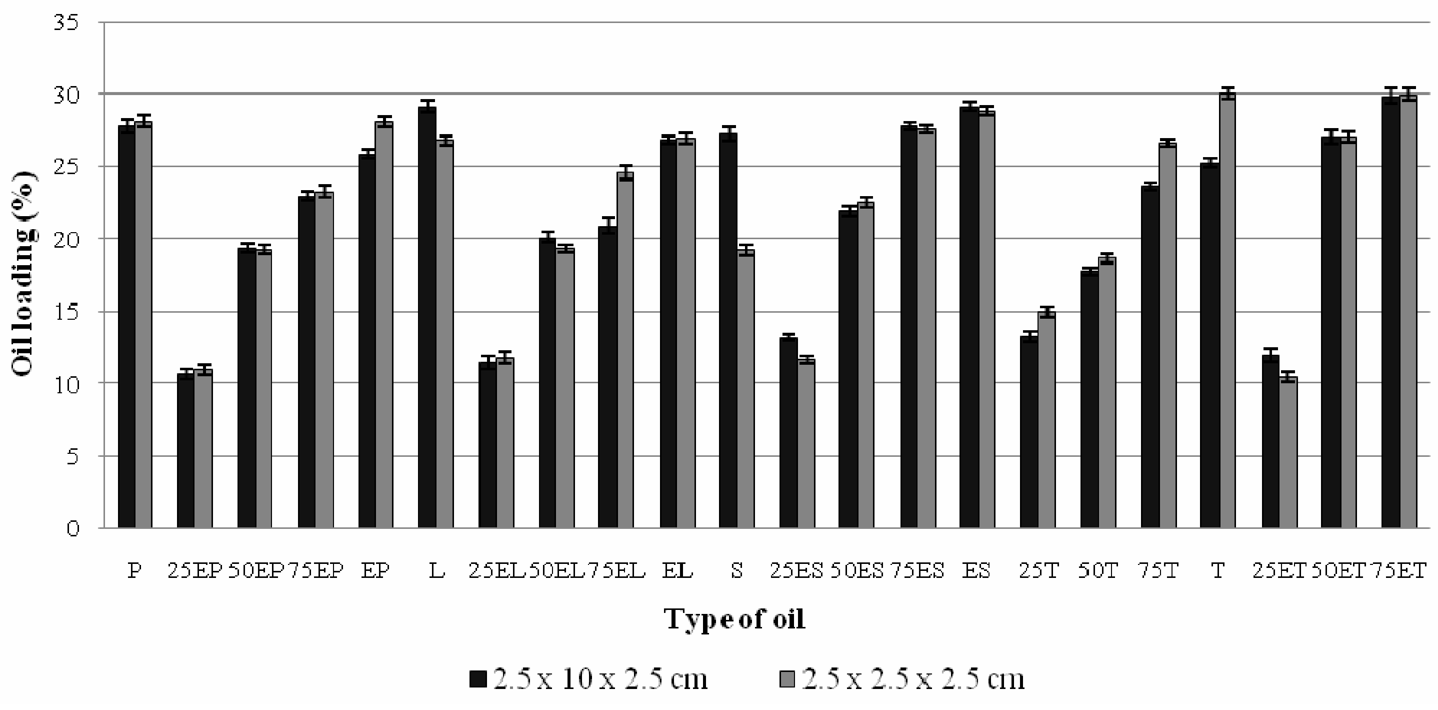
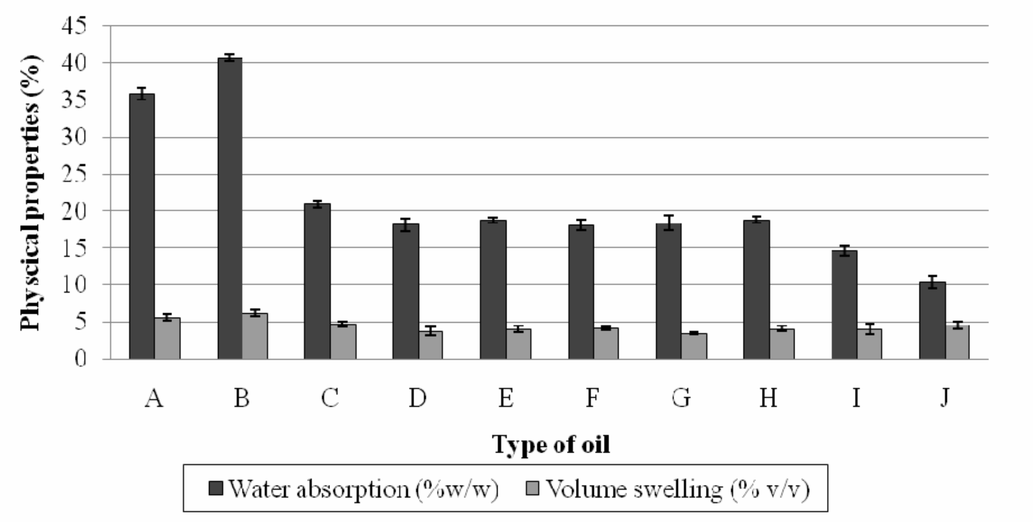


Figure 4.23 Effect of oil concentration on oil loading of rubberwood

4.3.3 Effect of oil type on physical properties of rubberwood



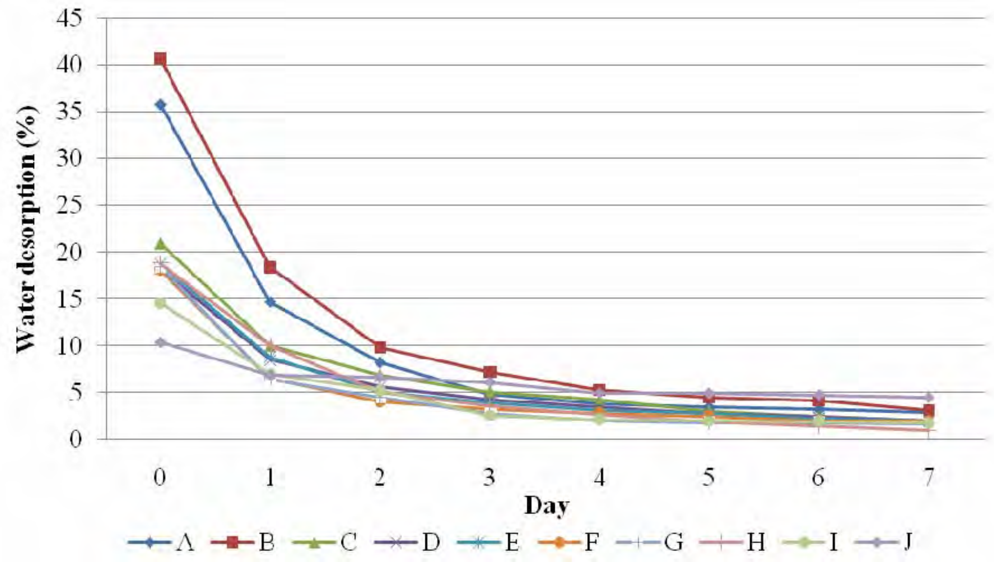
(A) Untreated; (B) Treated at 140°C for 6 h; (C) 28% loading of PO; (D) 26% loading of EPO; (E) 27% loading of SFO; (F) 29% loading of ESFO; (G) 29% loading of LO; (H) 27% loading of ELO; (I) 25% loading of TO; (J) 30% loading of ETO

Figure 4.24 Effect of oil type on physical properties of rubberwood.

In Figure 4.24, the water absorption of untreated rubberwood was higher than that of the treated rubberwood. The value of water absorption of untreated rubberwood and treated rubberwood absorbed water were 35.79% and lower 25%, respectively. For the effect of types of treating oil on water absorption, tung oil gave the lowest water absorption of rubberwood comparing with other oil, especially when rubberwood was treated with epoxidized tung oil. This should be caused by the conjugated double bonds of tung oil, which could be oxidized when exposed to oxygen in the air [29]. Compared with the oil and epoxidized oil, the water absorption of wood treated with epoxidized oil was lower than of oil treated wood. The lower water absorption of the epoxidized oil treated rubberwood might be caused by crosslinking of the fatty acid due to the epoxy group in epoxidized oil. Therefore, a thin layer was formed on the surface of the wood, resulting in a hindrance for the water absorption. The photographs of treated rubberwood with 11.98% loading of epoxidized tung oil sample before and after the water absorption test are shown in Figure 4.25.

Figure 4.25 Comparison of water absorption test of treated rubberwood with 12% loading of ETO (a) before test (b) after test (c) after test 24 h.

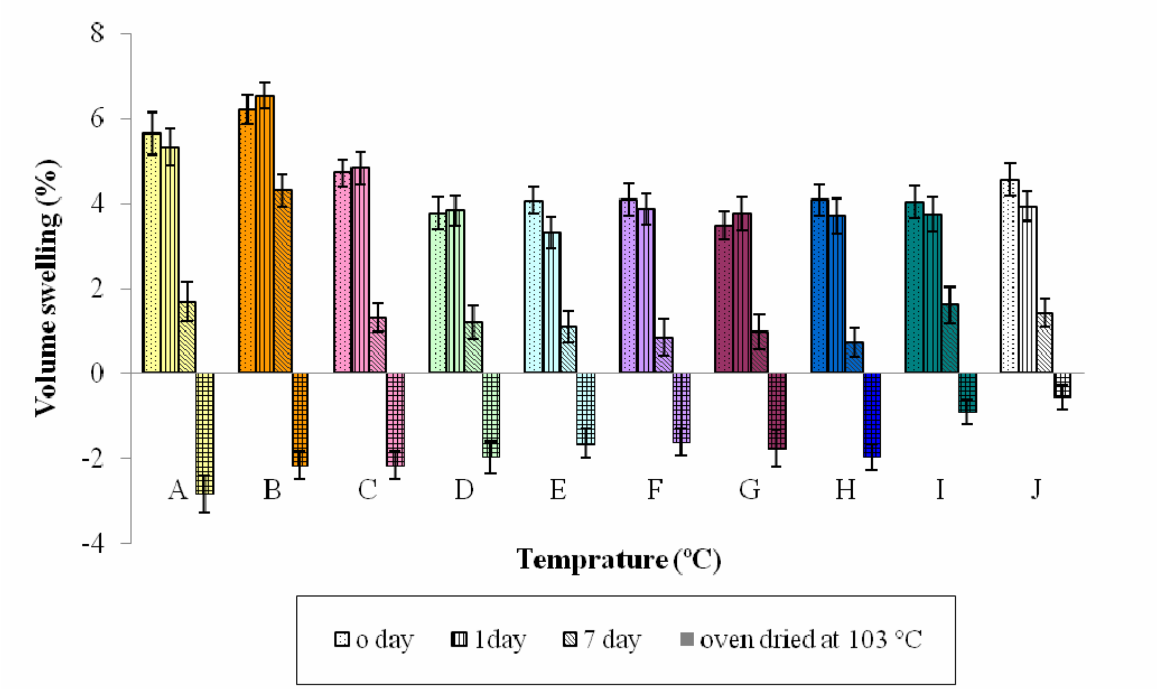
Comparing the untreated rubberwood and treated rubberwood, volume swelling of untreated rubberwood was higher than that the treated rubberwood. This showed that the oil treatment resulted in increasing hydrophobic property of the wood and then dimension of the treated woods was improved. Compared with the oil and epoxidized oil, the volume swelling of wood treated with epoxidized oil was lower than of oil treated wood. The lower volume swelling of the epoxidized oil treated rubberwood might be caused by crosslinking of the fatty acid moiety due to the epoxy group in epoxidized oil. The wood treated with tung oil had much higher volume swelling, especially when treated with epoxidized tung oil. However, moisture of wood treated with tung oil and epoxidized tung oil were rapidly evaporized than those of wood treated with other oil treatment and untreated rubberwood. According to the film of tung oil and epoxidized tung oil were swelled in water but it were rapidly evaporized water. Thus, wood treated with tung oil and epoxidized tung oil gave the lower water desorption rate than those of wood treated with other oil treatment. The water desorption of treated rubberwood after exposed to atmosphere for 7 days are shown in Figure 4.26.



(A) Untreated; (B) Treated at 140 °C for 6 h; (C) 28% loading of PO (D) 26% loading of EPO; (E) 27% loading of SFO; (F) 29% loading of ESFO (G) 29% loading of LO; (H) 27% loading of ELO; (I) 25% loading of TO; (J) 30% loading of ETO

Figure 4.26 Effect of oil type on water desorption of rubberwood.

After leaving all samples to environment under atmospheric pressure for 7 days to check for the water desorption, the samples were dried in the oven at 103ºC to measure the volume swelling of rubberwood as shown in Figure 4.27. The swelling of almost every sample was similar. However, the swelling of the treated rubberwood with tung oil and epoxidized tung oil were shrunk less than other treated rubberwood samples. This could be the result of the improved dimensional stability due to the water repellency property of the oil.



(A) Untreated; (B) Treated at 140°C for 6 h; (C) 28% loading of PO (D) 26% loading of EPO; (E) 27% loading of SFO; (F) 29% loading of ESFO (G) 29% loading of LO; (H) 27% loading of ELO; (I) 25% loading of TO; (J) 30% loading of ETO

Figure 4.27 Effect of oil type on volume swelling of rubberwood.

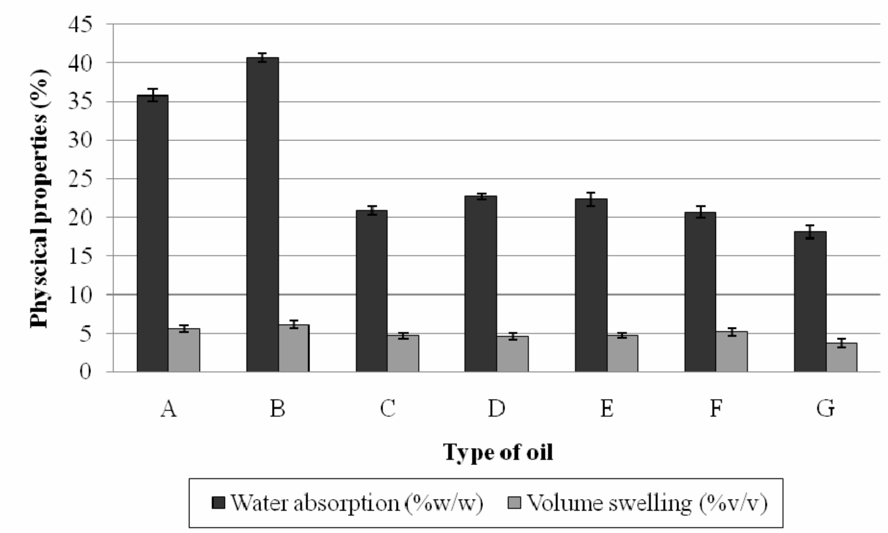
Table 4.3 The effect of methyl ester of tung oil and stearic acid on water absorption and volume swelling of rubberwood.

Test	Untreated	Treated at 140 °C	Methyl ester of tung oil	Stearic acid
Water absorption (%)	35.79	40.67	25.36	33.67
Volume swell (%)	5.64	6.21	5.79	5.92

Table 4.3 shows the volume swelling and the water absorption values of the rubberwood treated with methyl ester of tung oil and steric acid. It was found that the methyl ester of tung oil and steric acid could improve the water absorption, but it could not reduce the volume swelling. The value of volume swell of treated rubberwood with methyl ester of tung oil and steric acid were 5.79% and 5.92%, respectively. When the rubberwood was treated with methyl ester of tung oil, the methyl ester of tung oil could not diffuse into the wood. For the rubberwood treated with steric acid, the rubberwood would present hazy film in the presence of water. Therefore, the methyl ester of tung oil and the steric acid were not suitable for treating rubberwood in this study.

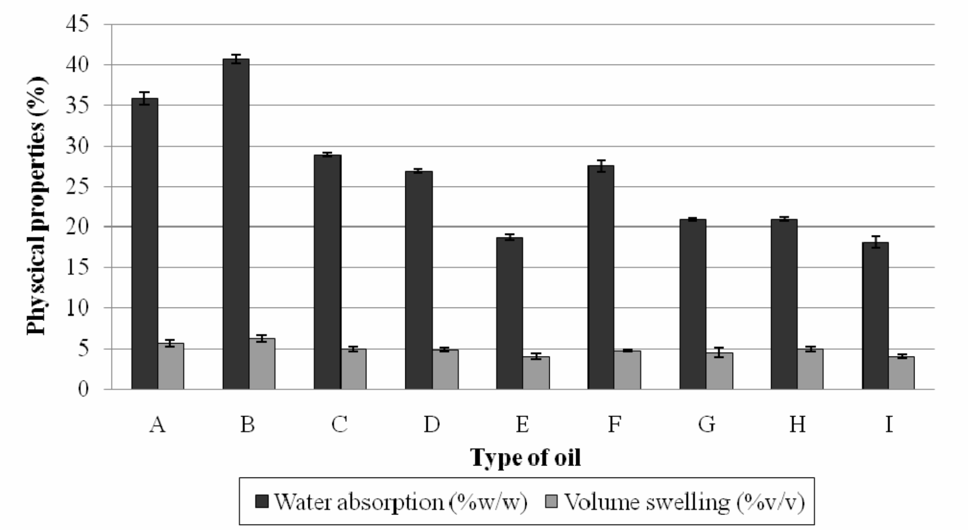
Figure 4.28 Comparison of water absorption test of treated rubberwood with methyl ester of tung oil (a) before test (b) after test.

4.3.4 Effect of oil loading on physical properties of rubberwood



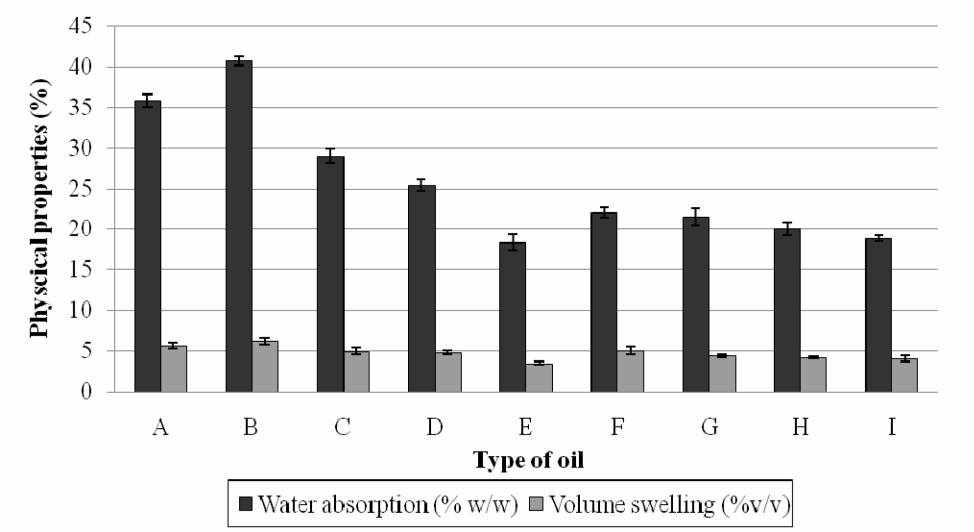
(A) Untreated; (B) Treated at 140°C; (C) 28% loading of PO; (D) 11% loading of EPO; **(**E) 19% loading of EPO; (F) 23% loading of EPO; (G) 26% loading of EPO

Figure 4.29 Effect of oil loading on physical properties of palm oil treated rubberwood.



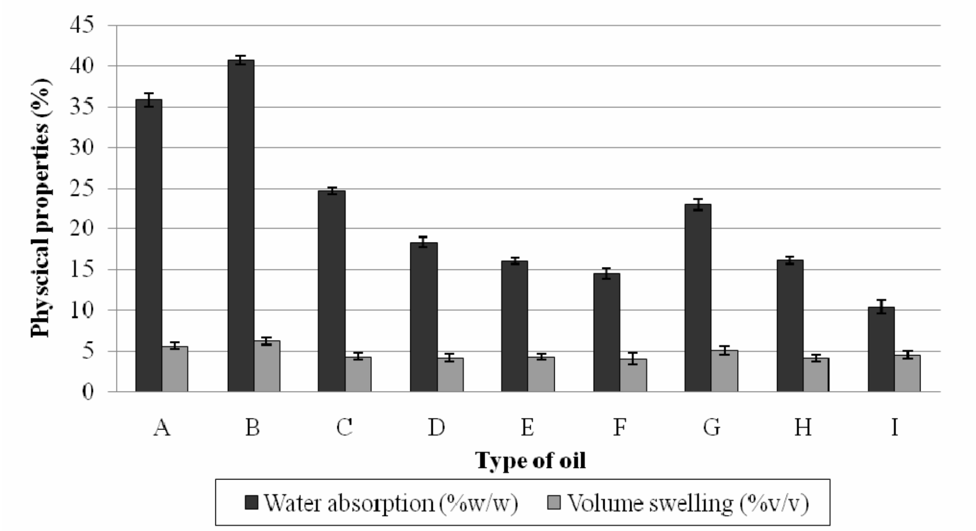
(A) Untreated; (B) Treated at 140°C; (C) 11% loading of SFO; (D) 19% loading of SFO; (E) 27% loading of SFO; (F) 13% loading of ESFO; (G) 21% loading of ESFO; (H) 27% loading of ESFO; (I) 29% loading of ESFO

Figure 4.30 Effect of oil loading on physical properties of sunflower oil treated rubberwood.



(A) Untreated; (B) Treated at 140°C; (C) 11% loading of LO; (D) 18% loading of LO; (E) 29% loading of LO; (F) 12% loading of ELO; (G) 20% loading of ELO; (H) 21% loading of ELO; (I) 27% loading of ELO

Figure 4.31 Effect of oil loading on physical properties of linseed oil treated rubberwood



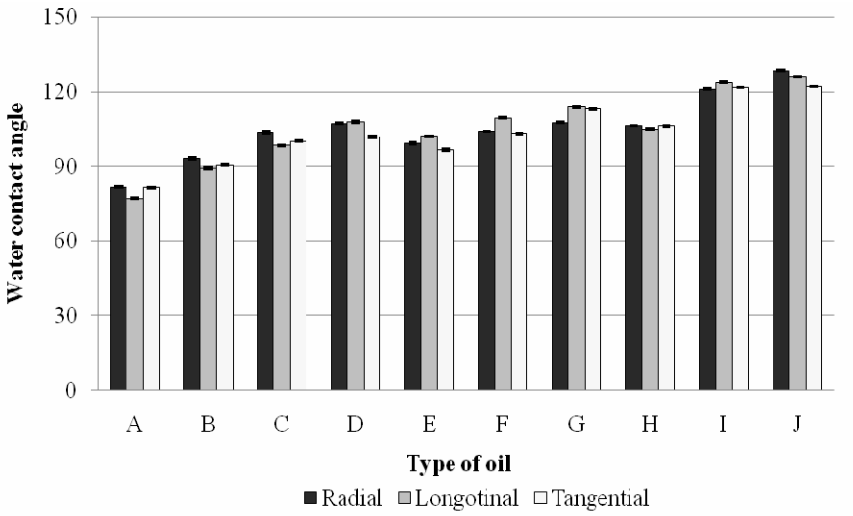
(A) Untreated; (B) Treated at 140°C; (C) 13% loading of TO; (D) 18% loading of TO; (E) 23% loading of TO; (F) 25% loading of TO; (G) 12% loading of ETO; (H) 27% loading of ETO; (I) 30% loading of ETO

Figure 4.32 Effect of oil loading on physical properties of tung oil treated rubberwood.

Figures 4.29-4.32 show the effect of oil loading on physical properties of rubberwood treated with palm oil, sunflower oil, linseed oil and tung oil, respectively. Compared with untreated rubberwood and treated rubberwood, the physical properties of treated rubberwood were improved. From graph, it was found the water absorption and volume swelling seemed to be increased by increasing concentration of oil.

4.3.5 Effect of oil type on hydrophobic property of rubberwood

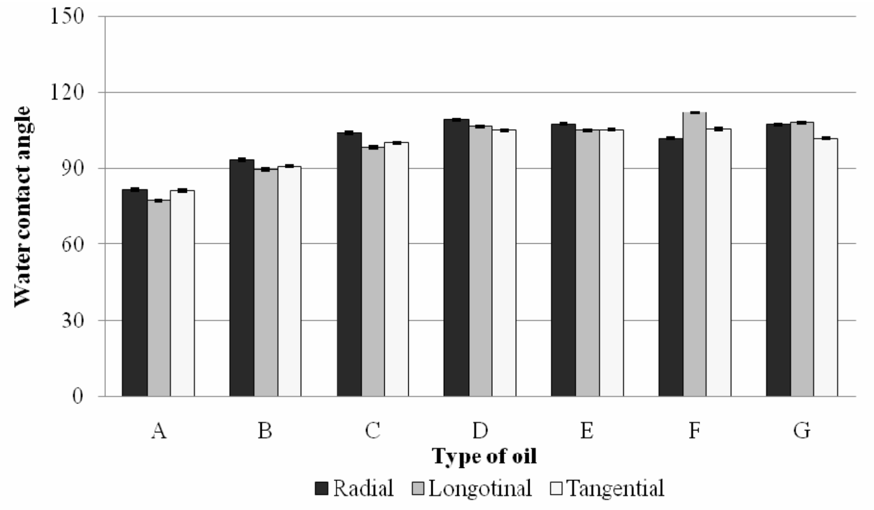
The water contact angle of untreated rubberwood and treated rubberwood are shown in Figure 4.33. Untreated wood showed hydrophilic property. Treated wood showed hydrophobic property. The hydrophobic property of the treated rubberwod could be explained by the water repellent property of the oil. The modification of wood may be caused by hydrophobic compounds coated on or reacted with cellulose [30].



(A) Untreated; (B) Treated at 140°C; (C) 28% loading of PO; (D) 26% loading of EPO; (E.) 27% loading of SFO; (F) 29% loading of ESFO (G) 29% loading of LO; (H.) 27% loading of ELO; (I) 25% loading of TO; (J) 30% loading of ETO

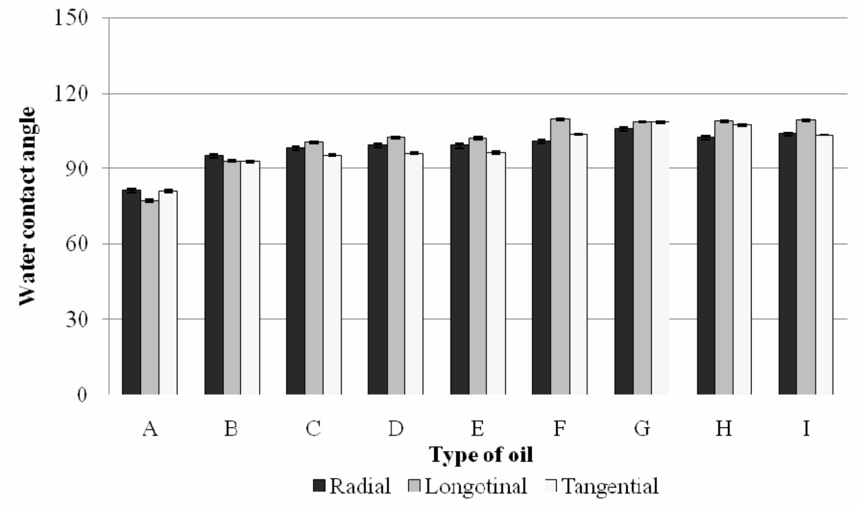
Figure 4.33 Effect of oil types on water contact angle of rubberwood.

4.3.6 Effect of oil loading on hydrophobic property of rubberwood



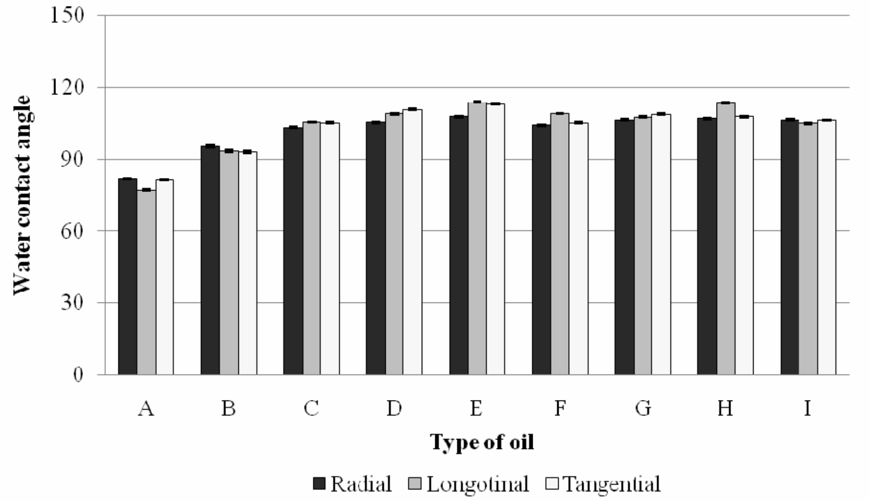
(A) Untreated; (B) Treated at 140°C; (C) 28% loading of PO; (D) 11% loading of EPO; **(**E) 19% loading of EPO; (F) 23% loading of EPO; (G) 26% loading of EPO

Figure 4.34 Effect of oil loading on water contact angle of palm oil treated rubberwood.



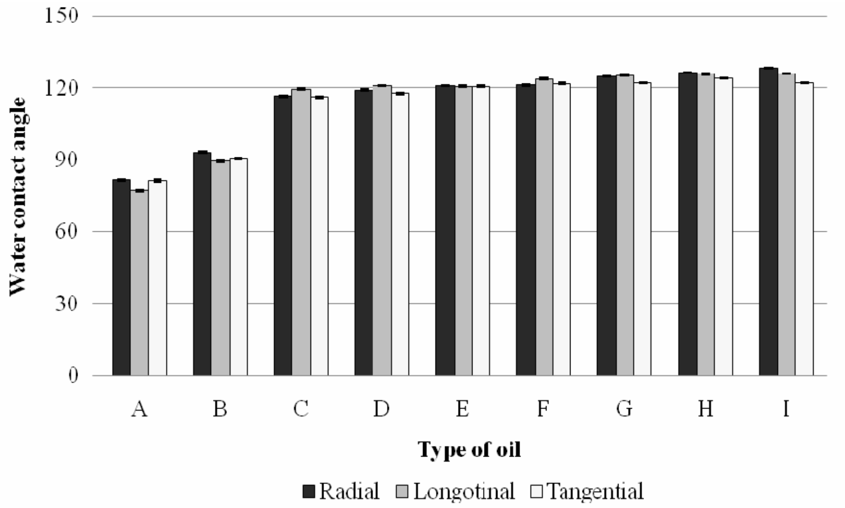
(A) Untreated; (B) Treated at 140°C; (C) 11% loading of SFO; (D) 19% loading of SFO; (E) 27% loading of SFO; (F) 13% loading of ESFO; (G) 21% loading of ESFO; (H) 27% loading of ESFO; (I) 29% loading of ESFO

Figure 4.35 Effect of oil loading on water contact angle of sunflower oil treated rubberwood.



(A) Untreated; (B) Treated at 140°C; (C) 11% loading of LO; (D) 18% loading of LO; (E) 29% loading of LO; (F) 12% loading of ELO; (G) 20% loading of ELO; (H) 21% loading of ELO; (I) 27% loading of ELO

Figure 4.36 Effect of oil loading on water contact angle of linseed oil treated rubberwood.



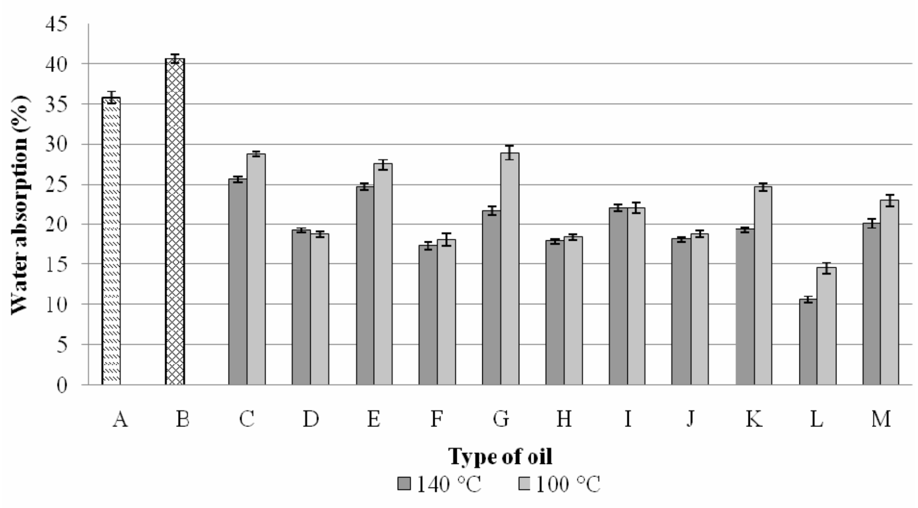
(A) Untreated; (B) Treated at 140°C; (C) 13% loading of TO; (D) 18% loading of TO; (E) 23% loading of TO; (F) 25% loading of TO; (G) 12% loading of ETO; (H) 27% loading of ETO; (I) 30% loading of ETO

Figure 4.37 Effect of oil loading on water contact angle of tung oil treated rubberwood.

Figures 4.34-4.37 show the effect of oil loading on water contact angle of rubberwood treated with palm oil sunflower oil, linseed oil and tung oil, respectively. From all of the results, the lower loading of epoxidized oil was improved water contact angle of rubberwood. This may be cause by epoxidized oil coated on the wood.

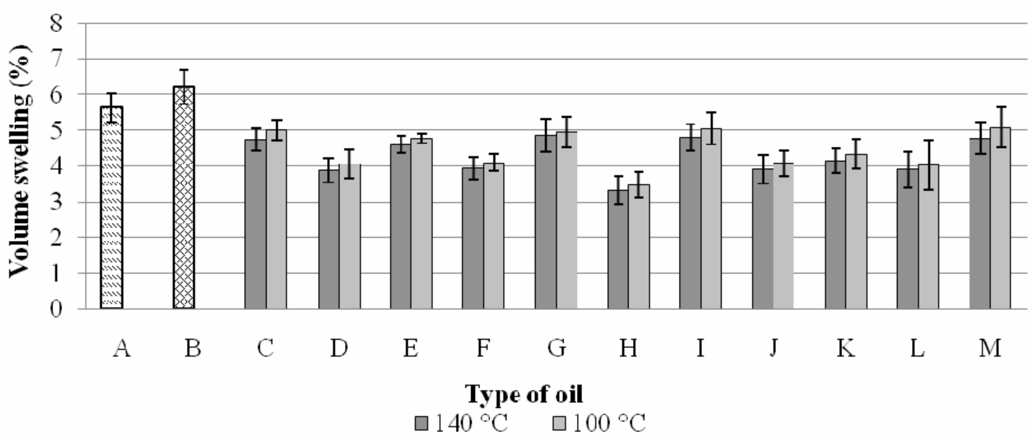
4.3.7 Effect of curing temperature on physical properties of rubberwood

Normally, the solid film of treating oil can be formed after drying at 140°C for 6 h. Thus, this experiment aimed to compare the physical properties of the treated rubberwood obtained from different curing temperature. The rubberwood sample was soaked in the oil until 10% loading was achieved. Then, each treated rubberwood sample was cured for 6 h at 100°C and 140°C. The types of oil treatment were SO, ESFO, LO, ELO, TO and ETO. The water absorption and the volume swelling of treated rubberwood are shown in Figures 4.37 and 4.38, respectively. The water contact angle of the treated rubberwood cured at 100°C and 140°C are shown in Figure 4.40. The results indicated that the treated rubberwood cured at 140°C had better water absorption, volume swelling and water contact angle.



(A) Untreated; (B) Treated at 140°C; (C) 10% loading of SO; (D) 27% loading of SO; (E) 10% loading of ESFO; (F) 29% loading of ESFO; (G) 10% loading of LO; (H) 29% loading of LO; (I) 10% loading of ELO; (J) 27% loading of ELO; (K) 10% loading of TO; (L) 25% loading of TO; (M) 10% loading of ETO

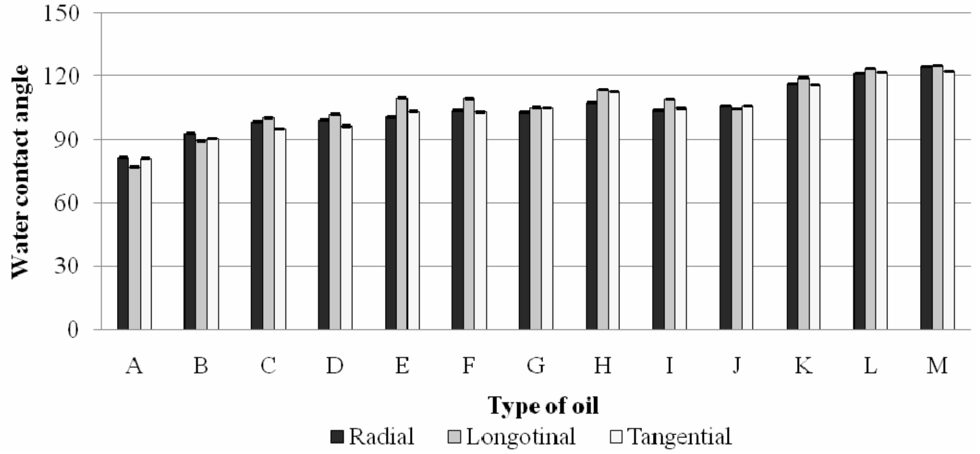
Figure 4.38 Effect of curing temperature on water absorption of rubberwood.



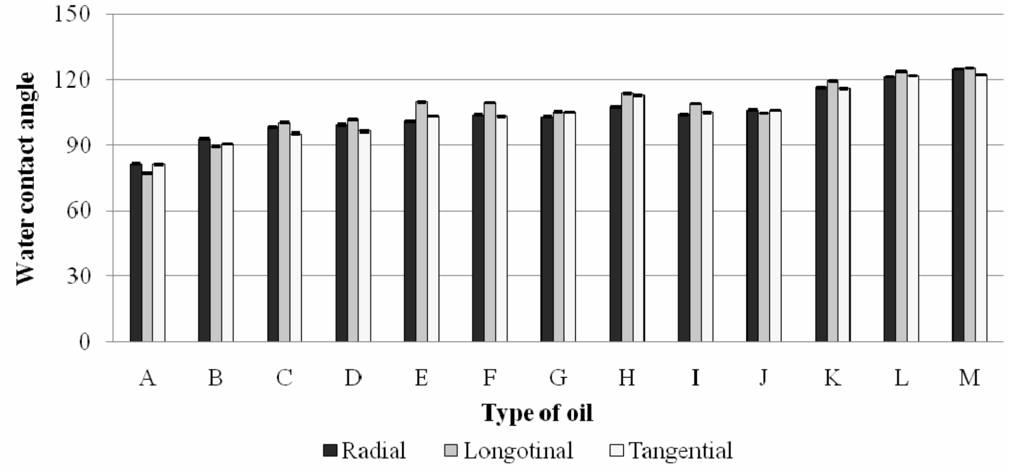
(A) Untreated; (B) Treated at 140°C; (C) 10% loading of SO; (D) 27% loading of SO; (E) 10% loading of ESFO; (F) 29% loading of ESFO; (G) 10% loading of LO; (H) 29% loading of LO; (I) 10% loading of ELO; (J) 27% loading of ELO; (K) 10% loading of TO; (L) 25% loading of TO; (M) 10% loading of ETO

Figure 4.39 Effect of curing temperature on volume swelling of rubberwood.

(a) Curing temperature at 100°C



(b) Curing temperature at 140°C

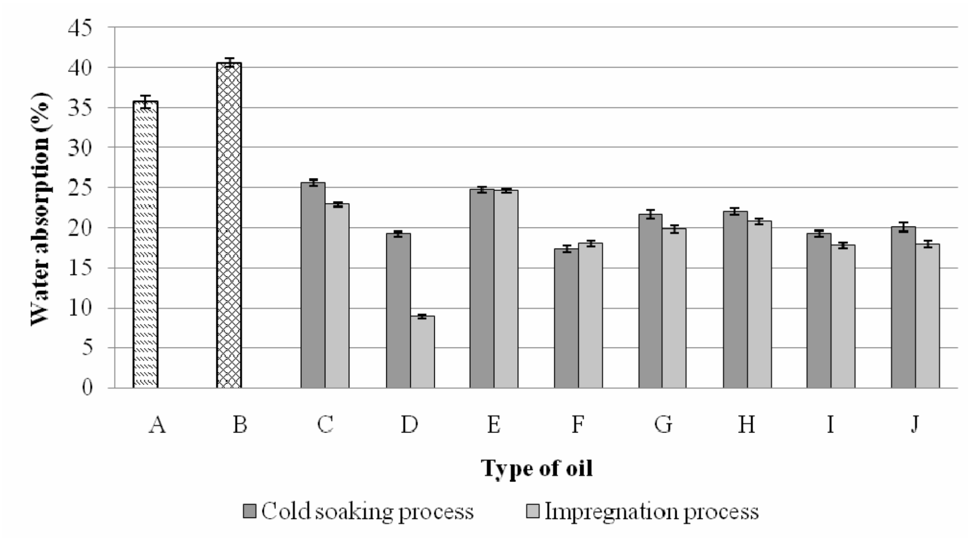


(A) Untreated; (B) Treated at 140°C; (C) 10% loading of SO; (D) 27% loading of SO; (E) 10% loading of ESFO; (F) 29% loading of ESFO; (G) 10% loading of LO; (H) 29% loading of LO (I) 10% loading of ELO; (J) 27% loading of ELO; (K) 10% loading of TO; (L) 25% loading of TO; (M) 10% loading of ETO

Figure 4.40 Effect of curing temperature at (a) 100°C and (b) 140°C on water contact angle of rubberwood.

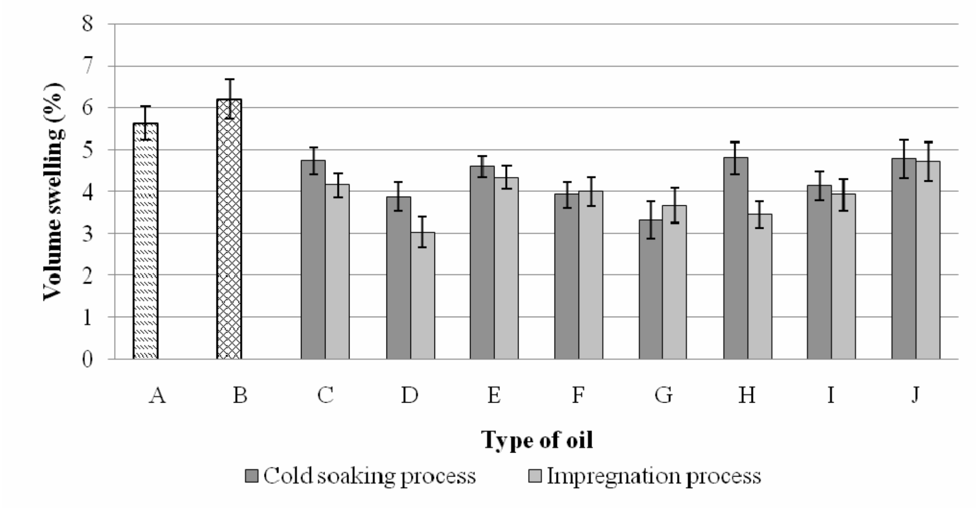
4.4 Effect of treatment process on physical properties of rubberwood

This experiment aimed to compare the process of the treated rubberwood obtained from different treatment process. The treating oil loading was 10% for all samples. The types of oil used in this experiment were SFO, ESFO, LO, ELO, TO and ETO. Using cold soaking process and impregnation process the water absorption, volume swelling and water contact angle of the treated rubberwood are shown in Figures 4.41-4.43. The results showed that better improvement of the water absorption, volume swelling and water contact angle of the treated rubberwood was obtained by impregnation process. Therefore, it can be concluded that the impregnation process could introduce more oil into vessel of wood comparing with the cold soaking process.



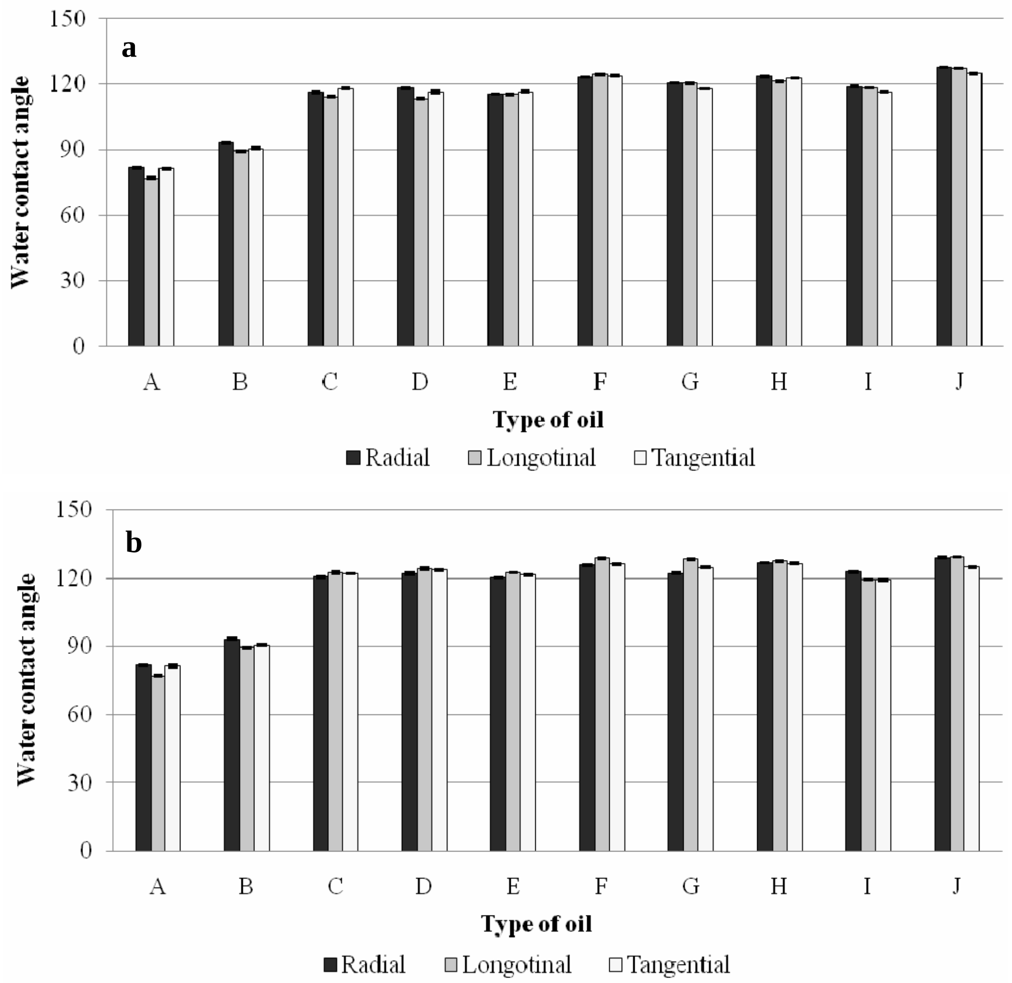
(A) Untreated; (B) Treated at 140°C; (C) 10% loading of SO; (D) 27% loading of SO; (E) 10% loading of ESFO; (F) 29% loading of ESFO; (G) 10% loading of LO; (H) 10% loading of ELO; (I) 10% loading of TO; (J) 10% loading of ETO

Figure 4.41 Effect of treatment process on water absorption of treated rubberwood.



(A) Untreated; (B) Treated at 140°C; (C) 10% loading of SO; (D) 27% loading of SO; (E) 10% loading of EFSO; (F) 29 % loading of EFSO; (G) 10% loading of LO; (H) 10% loading of ELO; (I) 10% loading of TO; (J) 10% loading of ETO

Figure 4.42 Effect of treatment process on volume swelling of treated rubberwood.



(A) Untreated; (B) Treated at 140 °C; (C) 10% loading of SO; (D) 27% loading of SO; (E) 10% loading of EFSO; (F) 29% loading of EFSO; (G) 10% loading of LO; (H) 10% loading of ELO; (I) 10% loading of TO; (J) 10% loading of ETO

Figure 4.43 Effect of (a) cold soaking process and (b) impregnation process on water contact angle of rubberwood.

4.5 Effect of oil treatment on mechanical properties of rubberwood

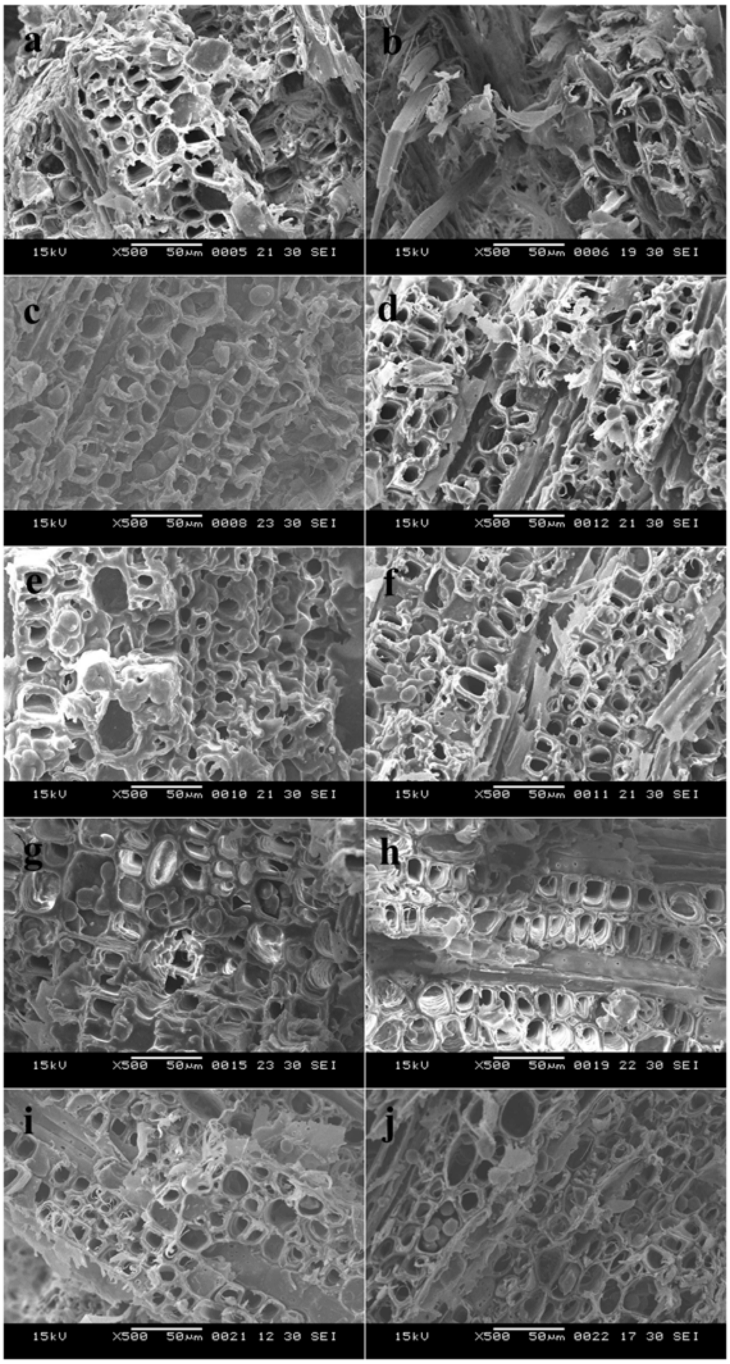
This experiment was conducted based on the fact that the mechanical properties of rubberwood are dependent on the types of oil. The oils used in this experiment were SFO, ESFO, LO, ELO, TO and ETO. Oil loading for all samples was 10%. However, higher loading of SFO and ESFO were also studied, as 23% and 20%, respectively. The impregnating process was applied as the wood treatment. The curing conditions were set at 140°C for 6 h. The mechanical properties of the treated rubberwood are shown in Table 4.4. It can be seen that the mechanical properties of each treated rubberwood are different. The flexure stress and modulus of elasticity were increased, when the oil loading was increased. By the way, for the effect of treating oil, rubberwood treated with tung oil provided the highest flexural stress and modulus of elasticity. This is contributed to better solid film formed within the wood that helped strengthen the rubberwood comparing other oil films. When comparing epoxidized oils, epoxidized linseed oil yielded the highest flexural stress and modulus of elasticity comparing with other epoxidized oil. These may be caused by crosslinking of the fatty acid moiety due to the epoxy group in epoxidized oil.

Table 4.4 Effect of oil treatment on flexure stress and modulus of elasticity of rubberwood.

Rubberwood	Flexure stress (kg/cm^2)	Modulus of elasticity (kg/cm^2)
Untreated rubberwood	230	39,764
10 % loading of SO	186.73	37,582
23 % loading of SO	242.35	54,830
10 % loading of ESFO	294.38	67,934
20 % loading of ESFO	368.46	212,572
10 % loading of LO	273.67	102,216
10 % loading of ELO	260.16	170,043
10 % loading of TO	437.56	267,894
10 % loading of ETO	350.67	134,295

4.6 Microstructure of rubberwood

The microstructure of untreated rubberwood and treated rubberwood from the fractured surface of samples after flexural test were examined by scanning electron microscopy (SEM). The oils used in this experiment were SFO, ESFO, LO, ELO, TO and ETO. The treating oil loading for all samples is 10%. However, higher loading of SFO and ESFO were also studied, as 23% and 20%, respectively. Figure 4.44 shows the photographs of untreated and treated rubberwood. Oil was deposited on surface of cell walls of rubberwood, which were important consequence on improvement of physical and mechanical properties of rubberwood.



(a) untreated rubberwood

(b) untreated rubberwood

(c) 10 % loading of SFO

(d) 10 % loading of ESFO

(e) 23 % loading of SFO

(f) 20 % loading of ESFO

(g) 10 % loading of LO

(h) 10 % loading of ELO

(i) 10 % loading of TO

(j) 10 % loading of ETO

Figure 4.44 SEM photographs of untreated and treated rubberwood.

4.7 Decay test

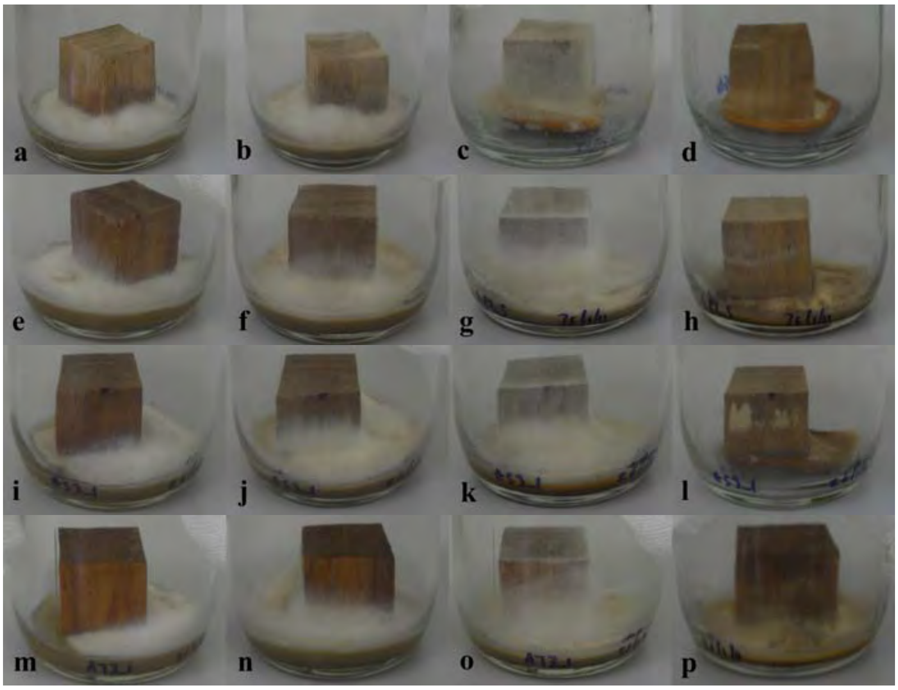


Figure 4.45 Comparison of biodegradation of (a) untreated 1 w; (b) untreated 2 w; (c) untreated 5 w; (d) untreated 8 w; (e) 28% loading of PO 1 w; (f) 28% loading of PO 2 w; (g) 28% loading of PO 5 w; (h) 28% loading of PO 8 w; (i) 27% loading of SFO 1 w; (j) 27% loading of SFO 2 w; (k) 27% loading of SFO 5 w; (l) 27% loading of SFO 8 w; (m) 25% loading of TO 1 w; (n) 25% loading of TO 2 w; (o) 25% loading of TO 5 w; (p) 25% loading of TO 8 w of rubberwood samples.

In the decay test, white rot fungus (*Trametes versicolor*) growth rate on untreated rubberwood and treated rubberwood samples were found to be differed. The untreated and treated rubberwood were inoculated within 8 week of exposure. The photographs of decay test on untreated rubberwood and treated rubberwood samples are shown in Figures 4.45-4.48. In comparison growth rate of fungus on untreated rubberwood and treated rubberwood samples, treated rubberwood took place more slowly than untreated rubberwood. The lower decay of treated rubberwood samples might have been due to the decrease in water uptake capacity of the treated rubberwood [31]. Compared with the palm oil, sunflower oil and tung oil, tung oil took place more slowly than untreated rubberwood. According to the film of tung oil was coated on the rubberwood.

Figure 4.46 shows the effect of treating oil on growth rate of fungus on rubberwood treated with epoxidized palm oil, epoxidized sunflower oil, epoxidized linseed oil and epoxidized tung oil, respectively. Compared with the growth rate of fungus on untreated rubberwood and epoxidized oil treated rubberwood, epoxidized oil treated rubberwood took place more slowly than untreated rubberwood. In comparison epoxidized the palm oil, epoxidized sunflower oil and epoxidized tung oil, epoxidized tung oil took place more slowly than the other epoxidized oil treated rubberwood. According to the film of epoxidized tung oil coated on the rubberwood could be decreased in water uptake capacity of the treated rubberwood.

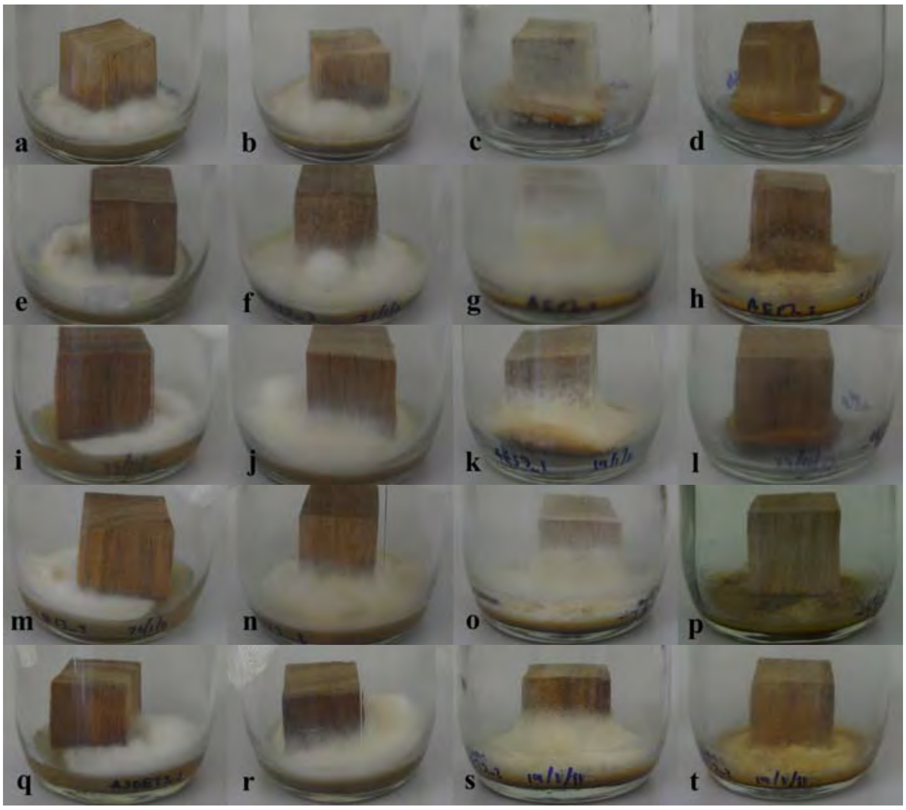


Figure 4.46 Comparison of biodegradation of (a) untreated 1 w; (b) untreated 2 w; (c) untreated 5 w; (d) untreated 8 w; (e) 28% loading of EPO 1 w; (f) 28% loading of EPO 2 w; (g) 28% loading of EPO 5 w; (h) 28% loading of EPO 8 w; (i) 29% loading of ESFO 1 w; (j) 29% loading of ESFO 2 w; (k) 29% loading of ESFO 5 w; (l) 29% loading of ESFO 8 w; (m) 27% loading of ELO 1 w; (n) 27% loading of ELO 2 w; (o) 27% loading of ELO 5 w; (p) 27% loading of ELO 8 w; (l) 30% loading of ETO 8 w; (m) 30% loading of ETO 1 w; (n) 30% loading of ETO 2 w; (o) 30% loading of ETO 5 w; (p) 30% loading of ETO 8 w of rubberwood samples.

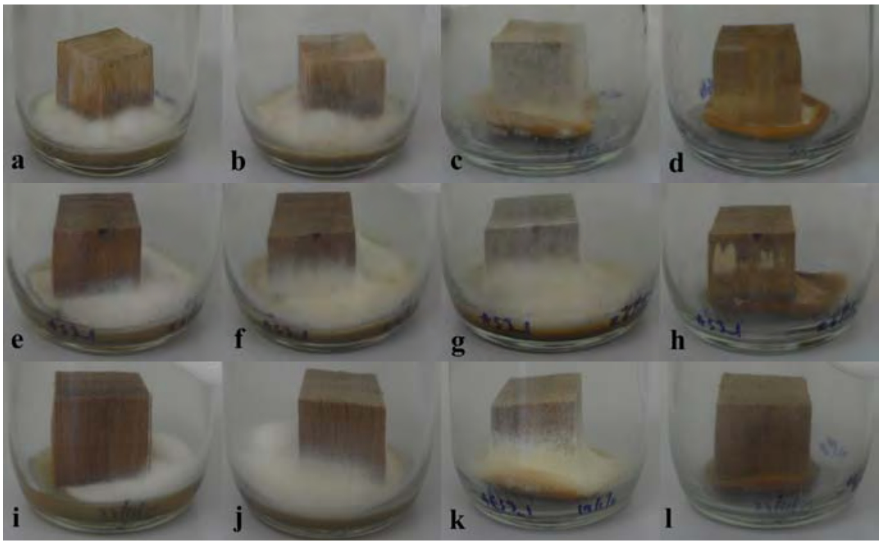


Figure 4.47 Comparison of biodegradation of (a) untreated 1 w; (b) untreated 2 w; (c) untreated 5 w; (d) untreated 8 w; (e) 27% loading of SFO 1 w; (f) 27% loading of SFO 2 w; (g) 27% loading of SFO 5 w; (h) 27% loading of SFO 8 w; (i) 29% loading of ESFO 1 w; (j) 29% loading of ESFO 2 w; (k) 29% loading of ESFO 5 w; (l) 29% loading of ESFO 8 w of rubberwood samples.

Figure 4.47 shows the effect of oil on growth rate of fungus on rubberwood treated with sunflower oil, epoxidized sunflower oil and untreated rubberwood, respectively. The result showed that, epoxidized sunflower oil treated rubberwood took place more slowly than untreated rubberwood. Figure 4.48 shows the effect of oil loading on growth rate of fungus on rubberwood treated with epoxidized sunflower oil. The result showed that, the higher loading of epoxidized oil was reduced the growth rate of fungus on rubberwood. The lower decay of epoxidized treated rubberwood samples might have been due to the decrease in water uptake capacity of the treated rubberwood [31].

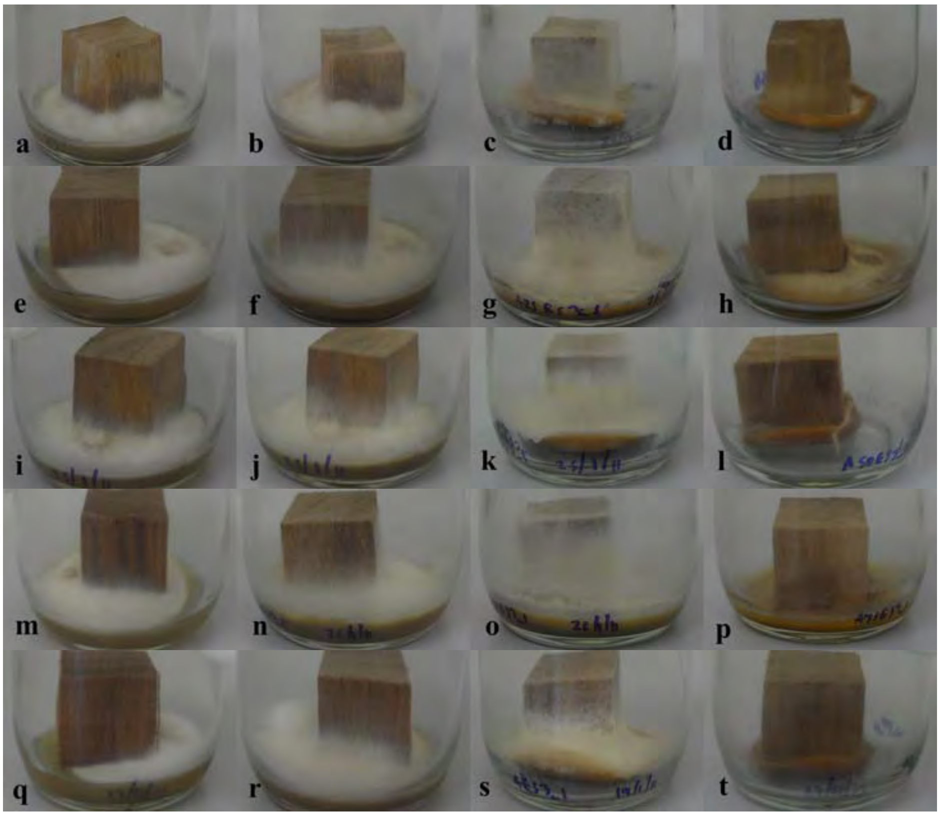


Figure 4.48 Comparison of biodegradation of (a) untreated 1 w; (b) untreated 2 w; (c) untreated 5 w; (d) untreated 8 w; (e) 13% loading of ESFO 1 w; (f) 13% loading of ESFO 2 w; (g) 13% loading of ESFO 5 w; (h) 13% loading of ESFO 8 w; (i) 21% loading of ESFO 1 w; (j) 21% loading of ESFO 2 w; (k) 21% loading of ESFO 5 w; (l) 21% loading of ESFO 8 w; (m) 27% loading of ESFO 1 w; (n) 27% loading of ESFO 2 w; (o) 27% loading of ESFO 5 w; (p) 27% loading of ESFO 8 w; (l) 29% loading of ESFO 8 w; (m) 29% loading of ESFO 1 w; (n) 29% loading of ESFO 2 w; (o) 29% loading of ESFO 5 w; (p) 29% loading of ESFO 8 w of rubberwood samples.

CHAPTER V

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The water absorption, volume swelling, water contact angle, flexure stress and modulusnof elasticity of rubberwood treated with heat treatment and oil treatment in this research were improve. In heat treatment, the treated commercial rubberwood at 160ºC and the fresh rubberwood at 180ºC gave water absorption better than other temperatures of the heat treatment and dimension stability of heat treated woods were improved. In oil treatment, the preparation method was carried out by impregnation process with tung oil, linseed oil, sunflower oil, palm oil and epoxidized oil. The optimum conditions for oil treatment were drying the rubberwood at 140 °C for 6 h, soaking in oil treatment for 3 h and curing treated rubberwood at 140 °C for 6 h. The oil treatment by impregnation procees with tung oil, linseed oil, sunflower oil, palm oil and epoxidized oil improved the water absorption, volume swelling, water contact angle, flexure stress and modulus of elasticity of rubberwood. Moreover, the treated epoxidized linseed oil was suitable for oil treatment.

5.2 Suggestion

The oil treatment should be further studied as the following

1. The effect of other oil types on mechanical and physical properties should be studied.
2. Modification of large scale impregnation process to prepare bigger specimens should be studied.

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APPENDIX

APPENDIX A

CHARACTERISTIC OF OILS

1.Characteristic of tung oil

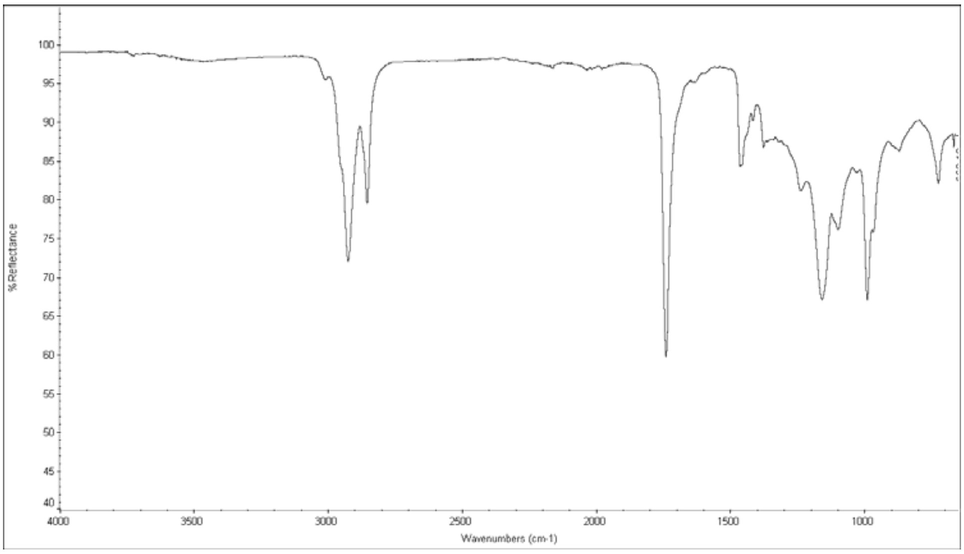

Figure A-1: FTIR spectrum of tung oil

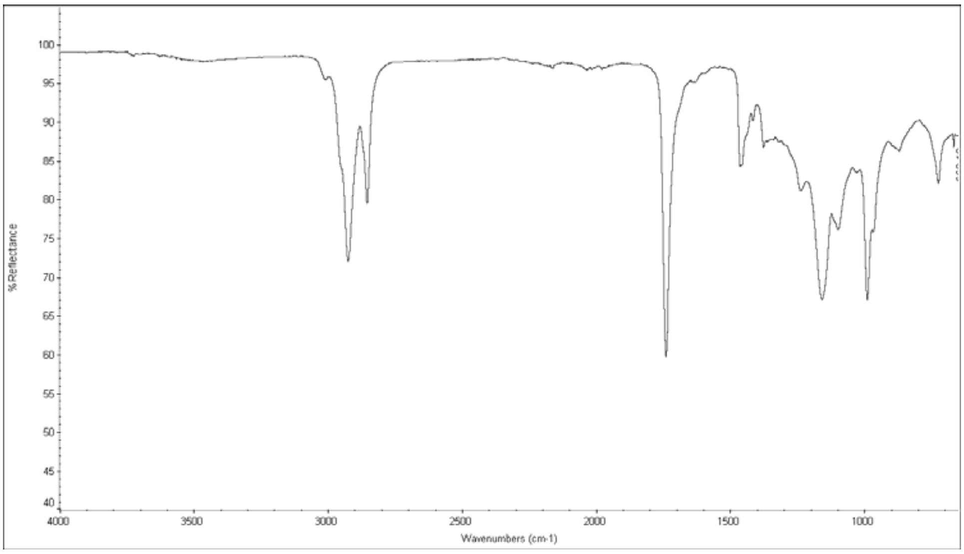

Figure A-2: FTIR spectrum of epoxidized tung oil

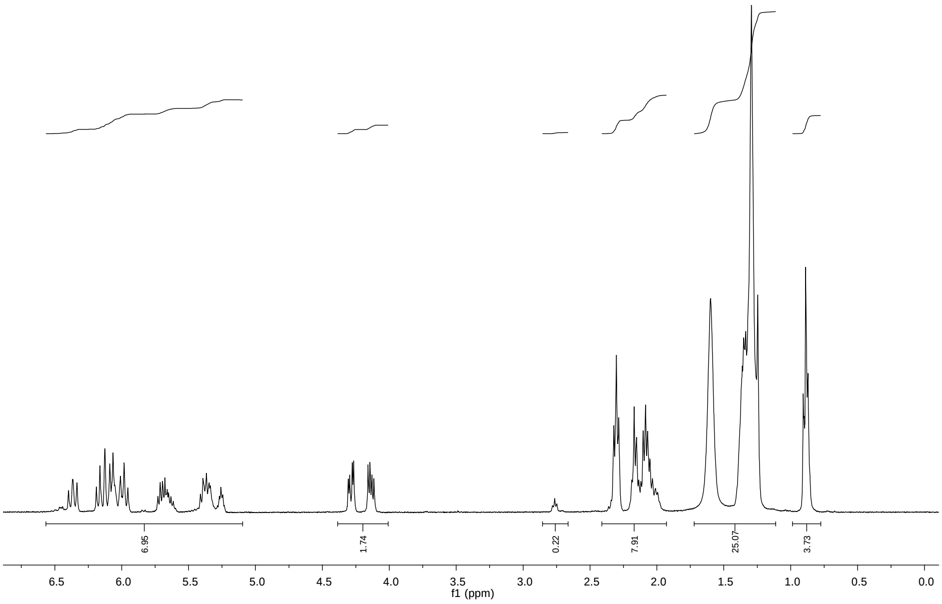

Figure A-3: ^{1}H NMR spectrum of tung oil

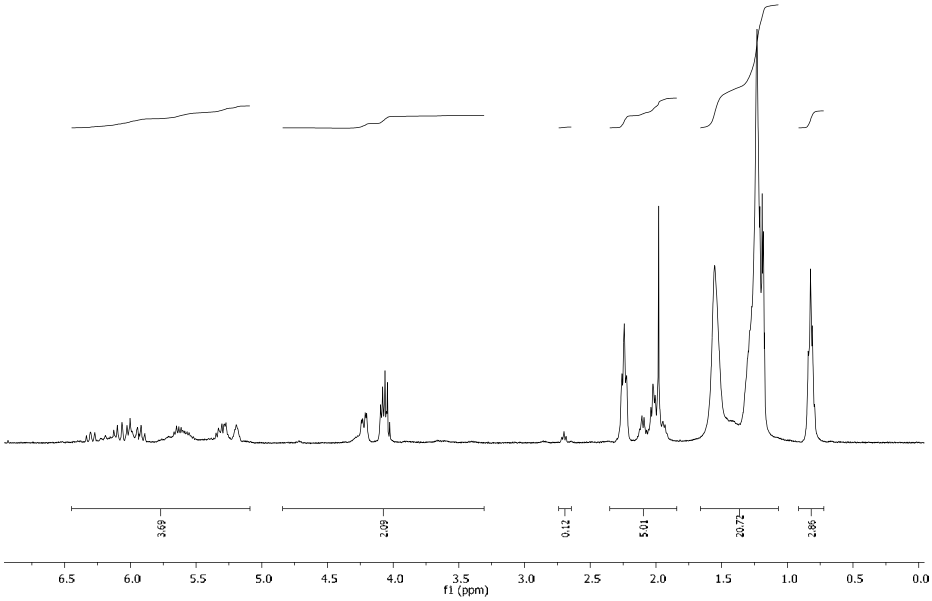

Figure A-4: ^{1}H NMR spectrum of epoxidized tungoil

2. Characteristic of linseed oil

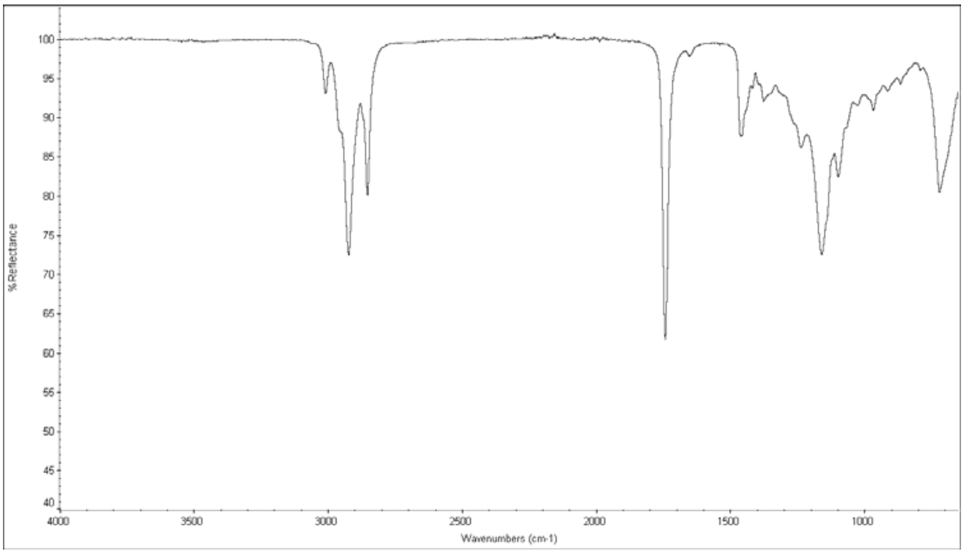

Figure A-5: FTIR spectrum of linseed oil

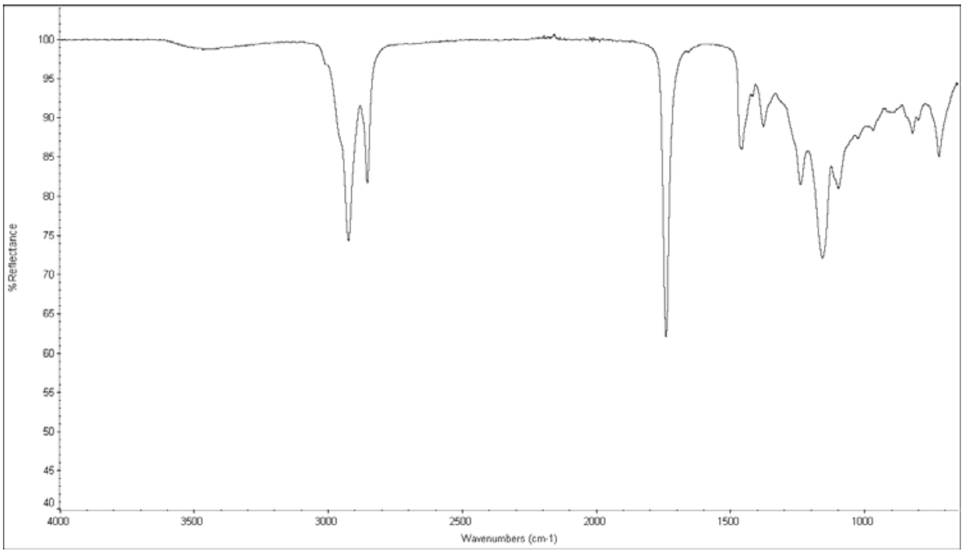

Figure A-6: FTIR spectrum of epoxidized linseed oil

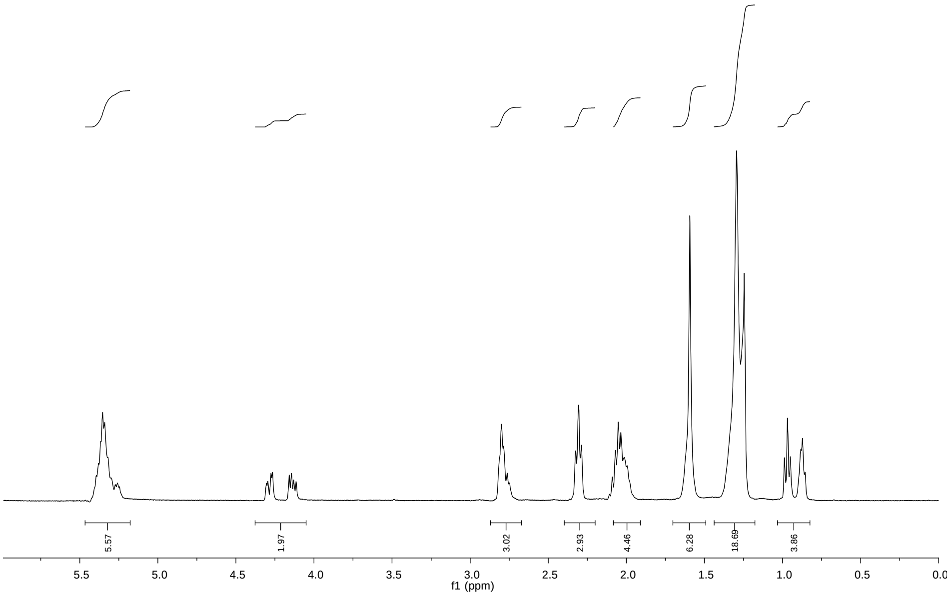

Figure A-7: ^{1}H NMR spectrum of linseed oil

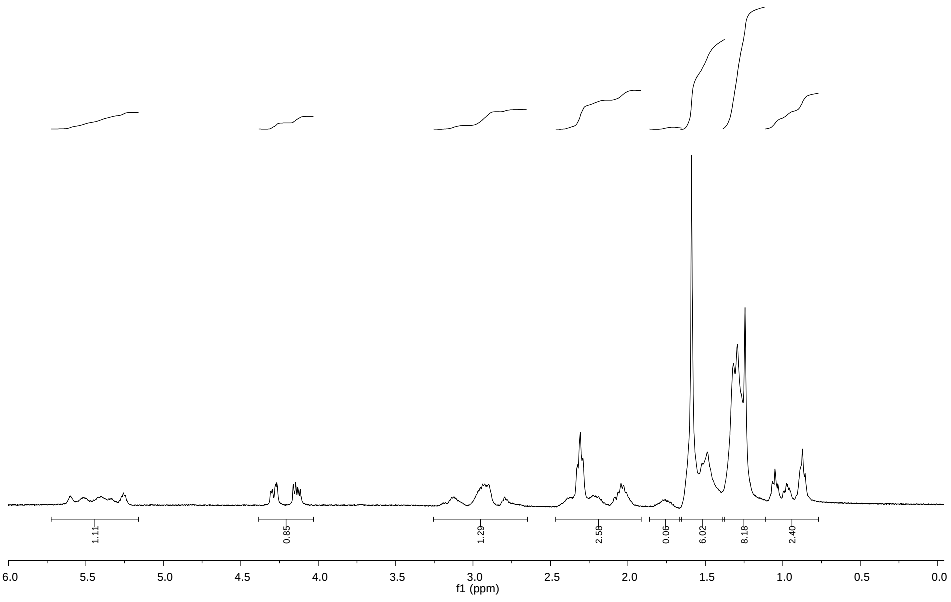

Figure A-8: ^{1}H NMR spectrum of epoxidized linseed

3. Characteristic of sunflower oil

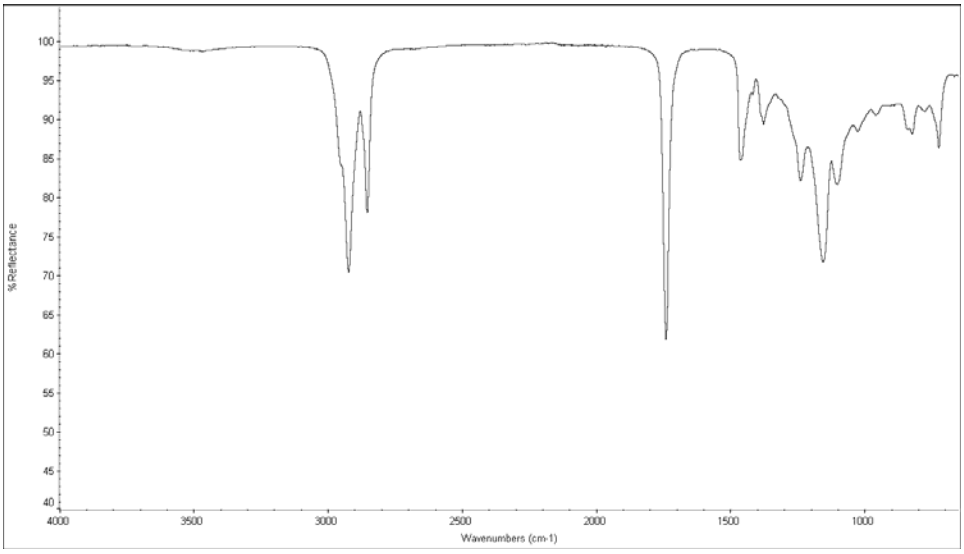

Figure A-9: FTIR spectrum of sunflower oil

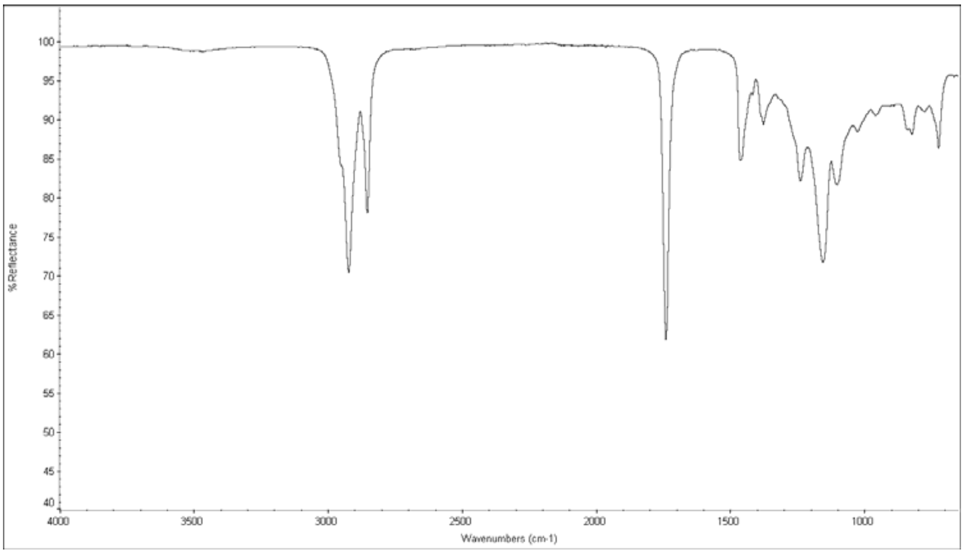

Figure A-10: FTIR spectrum of epoxidized sunflower oil

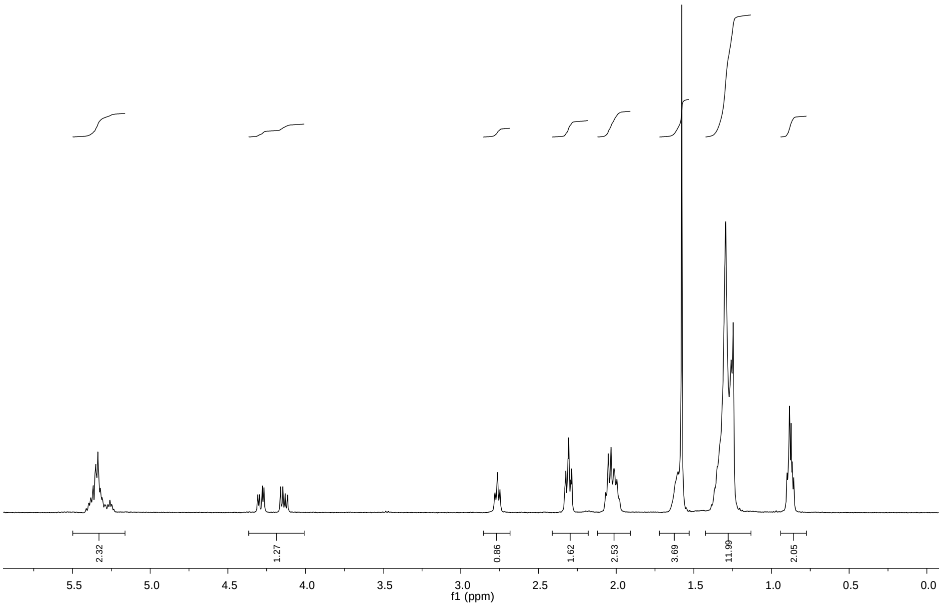

Figure A-11: ^{1}H NMR spectrum of sunflower oil

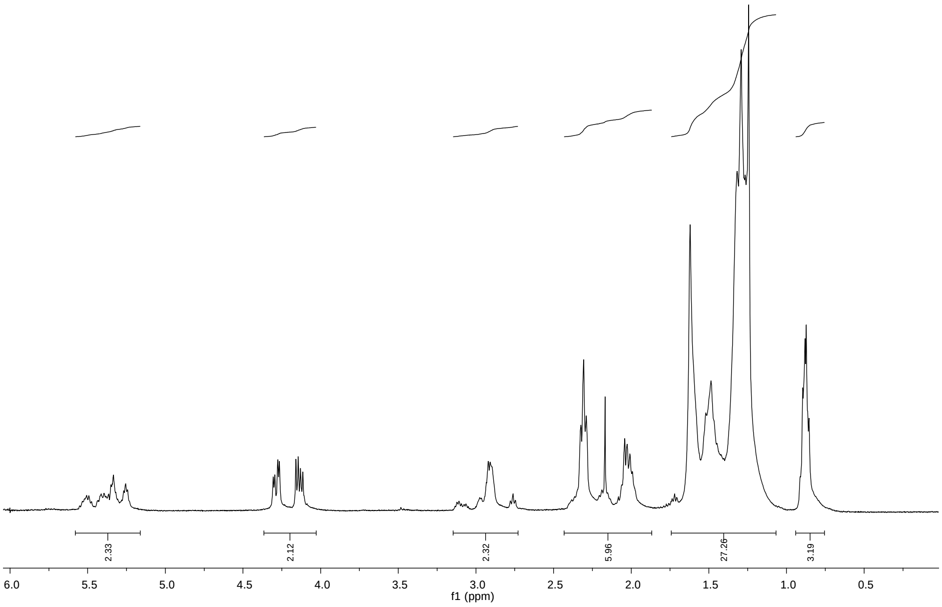

Figure A-12: ^{1}H NMR spectrum of epoxidized sunflower oil

4. Characteristic of palm oil

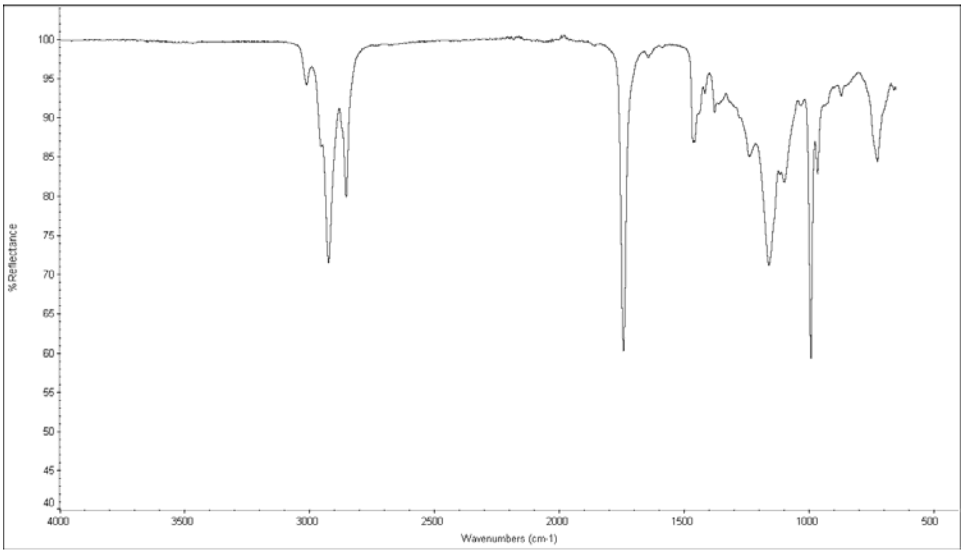

Figure A-13: FTIR spectrum of palm oil

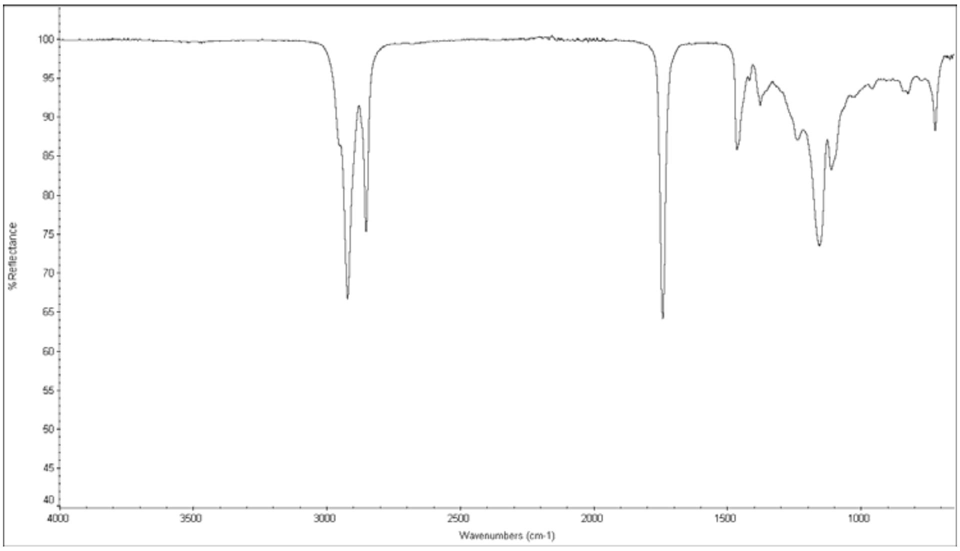

Figure A-14: FTIR spectrum of epoxidized palm oil

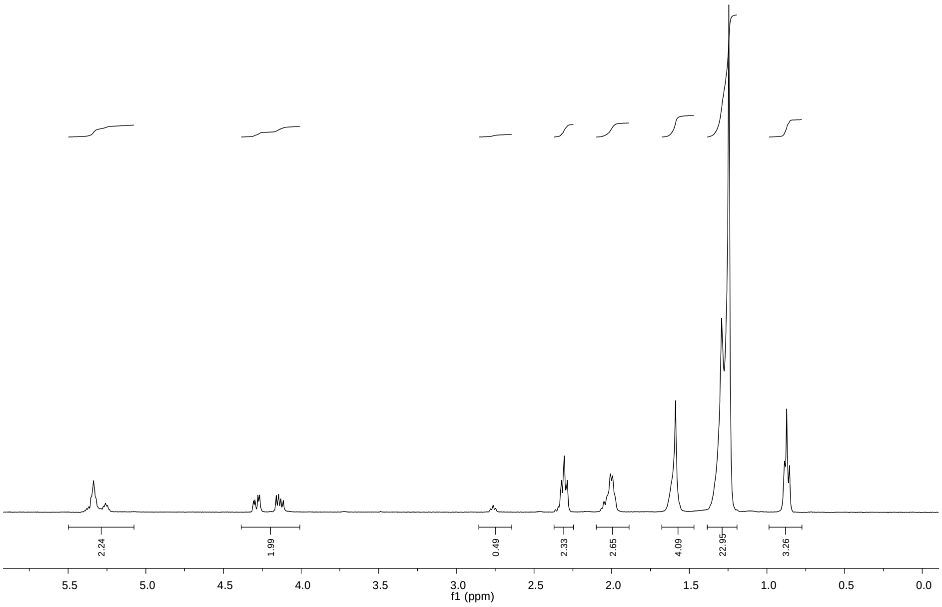

Figure A-15: ^{1}H NMR spectrum of palm oil

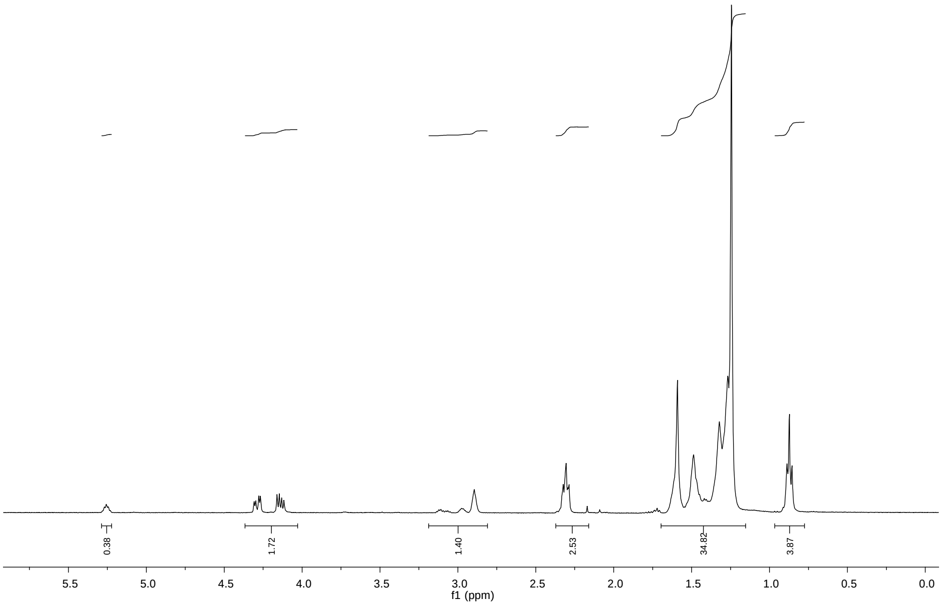

Figure A-16: ^{1}H NMR spectrum of epoxidized palm oil

5. Calculation of percentage of epoxidation in epoxidized oil

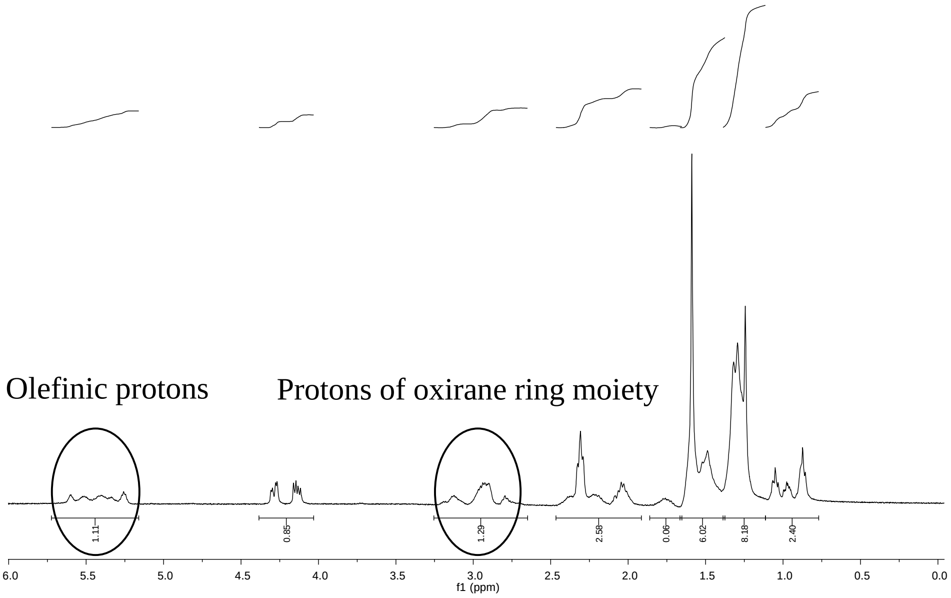

Figure A-17: ^{1}H NMR spectrum of epoxidized linseed oil for calculation % epoxidized oil

$$\% \text{ Epoxidation oil} = \frac{Ep}{Ep + Of} x100$$

Where Ep = Integrals of protons of oxirane ring moiety

Of = Integrals of olefinic protons

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