DEVELOPMENT OF NATURAL ADHESIVE USING LIGNIN AND SOY PROTEIN

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This thesis submitted in partial fulfillment
of the requirements for the award
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SUPERVISOR'S DECLARATION

"I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of the Bachelor of Chemical Engineering."

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STUDENT'S DECLARATION

I declare that this thesis entitled "**Development of Natural Adhesive Using Lignin and Soy Protein**" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ……

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DEDICATION

"I dedicate this thesis to my beloved mother, my late father and all my siblings, thanks for the never ending support. I love you all. To my supportive friends and my supervisor, thank you so much for the assist and guidance."

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DEVELOPMENT OF NATURAL ADHESIVE USING LIGNIN AND SOY PROTEIN

ABSTRACT

Adhesive are indispensable component in wood composite which is directly affect its physical, mechanical and chemical properties which are classified into two categories as synthetic and natural adhesive. By experiencing the emission of formaldehyde vapors which are carcinogenic of synthetic based adhesive, the natural adhesives in market be competitive, however they were weak in bonding and water resistance. As to overcome the problem this study attempts to develop natural adhesive that contain environmental friendly material by using lignin and soy protein, study its bonding strength and water resistant ability by chemically modify the protein to make the composite boards can pass the physical and mechanical tests of the medium density fiberboard (MDF). Therefore, the modification of soy flour with different concentration of lignin had been formulated by adding other chemical such as sodium hydroxide as alkali solution to the formulation to reach the required properties of strength and water resistance. As s conclusion, from this study it was found that only internal bonding give the good result and can be acceptable. In addition, high bonding strength and good water resistance of adhesive can be achieved by some additional modification of soy protein and lignin.

PENGHASILAN BAHAN PELAKAT ASLI MENGGUNAKAN LIGNIN DAN PROTIN SOYA

ABSTRAK

Bahan pelekat adalah komponen utama dalam komposit kayu kerana ia mempengaruhi cirri-ciri fizikal, mekanikal dan kimia dan terbahagi kepada dua kategori iaitu pelekat sintetik dan pelekat asli. Akibat mengalami pengeluaran gas formaldehid yang karsinogenik daripada bahan pelekat sintetik, bahan pelekat asli akan competetif dalam pasaran, walaubagaimanapun pelekat asli lemah dalam ikatan serat dan sifat kalis air. Untuk menyelesaikan masalah tersebut, kajian ini bertujuan untuk menghasilkan bahan pelekat asli yang mengandungi bahan mesra alam dengan menggunakan lignin dan protin soya, mengkaji kekuatan ikatan dan kebolehan kalis air dengan mengubahsuai protin secara kimia untuk memastikan komposit kayu boleh melepasi ujian fizikal dan mekanikal bagi papan serat kepadatan sederhana (MDF). Oleh itu, pengubahsuaian serbuk soya bersama lignin berlainan kepekatan telah diformulakan dengan menambah beberapa bahan kimia seperti natrium hidroksida sebagai larutan alkali untuk menjadikan formulasi mencapai cirri-ciri diperlukan bagi kekuatan dan sifat kalis air. Kesimpulannya, daripada kajian ini, ia mendapati bahawa hanya ikatan dalaman MDF menunjukkan keputusan yang baikdan boleh diterima. Tambahan lagi, kekuatan ikatan MDF dan sifat kalis air bahan pelekat boleh dicapai dengan penambahan pengubahsuaian protin soya dan lignin.

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LIST OF SYMBOL/ABBREVIATION

A	Absorbance value
C	Concentration of lignin
cm	Centimeter
D	Absorbtivity
FTIR	Fourier Transform Infrared Spectroscopy
g	Gram
H	Hour
kg	Kilogram
L	Liter
m	Meter
mL	Milliliter
mm	Millimeter
MPa	Megapascal
mPA	MiliPascal
N	Newton
ºC	Degree Celsius
V_{MDF}	MDF volume
λ	Wavelength
ρ_{MDF}	MDF density

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

In 2001, total sale of wood adhesive reach $ 6.1 billion as value the consumption of 13.3 million tones adhesive (Huang, 2007). Huang, 2007 also stated that adhesive are indispensable component in wood composite which is directly affect its physical, mechanical and chemical properties. Lei, 2009 also agreed with Huang by stated that wood adhesive play an important role in wood panel industry since the performance of the final wood panel depend on great degree of adhesive.

As the development of wood adhesive industry since 18^{th} century, it can be classified into two categories which are adhesives from petrochemical and natural material (Huang, 2007). Nihat and Nilgül, 2002 stated that synthetic polymer resin

based adhesive usually used in commercial wood composite product since it adhere well to wood and can form strong bond just like the wood. However, it is restricted because it is non-renewability and toxicity as well as highly cost since it was petroleum based product (Lei, 2009). Therefore, as a solution to the problem, natural adhesive being industrial interest lately since it was environmental friendly and ease to handle (Lei, 2009). Unfortunately, this type of adhesive is lack of bonding strength and almost no water resistance which mean it not a good adhesive to the wood or particle board.

1.2 Problem Statement

Formaldehyde based synthetic adhesive are restricted because it's non-renewability and emission of formaldehyde vapors which are carcinogenic. However, recent natural adhesives in market were weak in bonding and water resistance.

In order to overcome the environmental issue of synthetic resin and weakness of bonding strength with water resistance issue, this study had came with the new formulation to develop natural adhesive using lignin and soy protein based on it various properties.

1.3 Research Objectives

The objectives of this study is to develop natural adhesive that contain environmental friendly material by using lignin and protein and study its bonding strength as well as the water resistant ability of the natural adhesive. Besides this research also attempt to chemically modify the protein to make the composite boards can pass the physical and mechanical tests of adhesive.

1.4 Scope of Study

1.5.1 To study the development of natural adhesive that contains environmental friendly material by using lignin and soy protein.

1.5.2 To analyze the properties of the natural adhesive in the bonding strength and water resistance ability of medium density fiberboard (MDF).

1.5 Significance of Study

The high demand in the wood industrialized world had caused pollution problem due to the widespread use of formaldehyde in synthetic resins adhesive

which based on petrochemical. Therefore, it increasingly necessary to develop the natural adhesive with smaller environment impact which only used environmental friendly or non toxic material. The natural adhesive must be technically economically competitive, environmental acceptable, readily available and able to fully function as good adhesive which be the aim of this research. Natural adhesive also will be non-toxicity to human as well as environmental friendly.

1.6 Overview of the Thesis

As a conclusion, this study can improves the development of adhesive in wood composite industry which is more environmental friendly and give low cost of raw material (non petrochemical). For the next chapter, there will be the review of previous study about natural adhesive, deep review about lignin based adhesive and protein based adhesive as well as their properties. In third chapter, there will be the detail of the method that will be used in the development of natural adhesive using lignin and soy protein as well as the analysis to study its properties.

CHAPTER 2

LITERATURE REVIEW

This chapter consists of three subtopics that discuss about the previous study related to the developing natural adhesive using lignin and protein which are historical background of natural adhesive, development and properties of lignin based adhesive as well as development and properties of protein based adhesive.

2.1 Historical background of Natural Adhesive

A substance capable to hold material together by surfacing attachment was a definition of an adhesive by a dictionary stated by Nicholson (1991). Sun, Wang,

Zhong & Yang (2008) estimated that, the demand of adhesive in US will reach 15.2 billion pound in 2004 and most of these adhesive are from petroleum resources. In contrast, Frihart (2005) stated that protein glues have been used for thousand years as common bio-based adhesive and many early civilizations learned to make adhesive from plants and animals but today most of bio-diesel been replace by synthetic adhesive . Therefore, Lei (2009) had made a conclusion that the development of wood adhesive divided into two stages which were natural adhesive as the first stages and be replaced by synthetic thermosetting resin as the second stage in order to overcome the deficiency of natural adhesive in water resistance and bonding strength.

However, industrial interest lately was switched to natural adhesive due to the limited sources of petrochemical and environmental issue of toxicity emission as well as the availability of natural material as waste from other industry such as lignin and protein. In addition, Sun et al. (2008) stated that natural or environmentally friendly adhesive got lots of concerns recently since there were limited petroleum resources, environmental pollution issue and health problem cause by petroleum based adhesive. However, as to overcome the environment pressure, several investigation and research had been made in order to upgrade the weakness of most natural adhesive (Lei, 2009). Lei (2009) also stated that, those natural adhesive can be modified or cross-linked using formulation that not contain any toxicity material but still can improve the adhesive performance. Huang (2007) had been give similar opinion as Lei (2009), which stated that an urgent need to develop environmentally friendly wood adhesive to solve hazardous issue associated with formaldehyde based adhesive since formaldehyde emission will give health problem.

Frihart (2005) stated that many sources of protein adhesive such as animal bones and hides, milk (casein), blood, fish skin and soybean have been used as the raw material. Besides that, tree and bushes also provide several adhesive materials which include pitch, tannins and lignin (Frihart, 2005). Although carbohydrates not found much in wood industry adhesive, but usually starch used in many packaging application as adhesive (Frihart, 2005). Most of these materials usually can be found as the waste product in different industry such as animal blood from slaughterhouse, besides, those materials have renewability properties.

2.2 Development and Properties of Lignin Based Adhesive

As lignin were different type of waste and abundance product from pulp mills, the material could be other option for the preparation of adhesive as the natural material as an alternative to replace formaldehyde based adhesive (Lei, 2009). Nihat & Nilgül (2002) also stated that lignin is one most abundant, renewable natural product on earth as a by-product of the pulping process. Hüttermann, Mai & Kharazipour (2001) also introduce that the pulp and paper industry produce technical lignin as by-product in large quantities about 30-50 million ton per year. Other than that, lignin also the most important natural product coming from plants (Lei, 2009). Lignin is important factor for structural integrity for the cell wall and stiffness as well as strength of the stem (Boerjan, Ralph & Baucher, 2003). Boerjan et al. (2003) also

stated that lignin are complex racemic aromatic heteropolymers derived mainly from three hydroxycinnamyl alcohol monomers differing in their degree of methoxylation, p-coumaryl, coniferyl and sinapyl alcohol. In the other hand, Lei (2009) stated that in lignin natural form there is three dimensional polymers constituted of random polymerization of phenylpropane unit by ester and C-C bond.

Basically, lignin present in plant is quite variable and range about 20% to 40%. As lignin produce from pulping process (byproduct), the chemical pulping can be grouped into two classes which are sulfite and alkaline pulping (Lei, 2009). In sulfite pulping, the lignin fragment were called lignosulfate and give the lignin surface active, binding properties as well as hygroscopicity which lead to poor ability to co-crosslink with adhesive (Lei, 2009). Besides that, alkaline pulping was done with sodium hydroxide which third of it can be replace sodium sulfide and soda pulping as variation by adding catalytic quantities of anthraquinone (Lei, 2009). During the process, lignin extensively modified and cleavage of alkylaryl ether lingkage which drive the lignin fragment undergo condensation reaction (Lei, 2009). In the other hand, for the process of recovered sugar cane bagasse lignin, acidification of a black liquor produce by soda delignification of steam exploded sugar cane bagasse. However, this process produce low molecular weight lignin fragment (Guerra, Ferraz, Cotrim & Silva, 2000). In addition, Bourbonnais, Paice, Reid, Lantheir & Yaguchi (1995) found that kraft pulping delignified by combination of laccase and 3-ethylbenzthiazoline-6-sulfonate and it was reported that were more than 50% delignification of kraft pulp with laccase and another moderator or by repeated treatment following by alkaline extraction.

2.2.1 Chemical modification of lignin

Although, lignin present in plant act together with hemicelluloses as a perfect natural adhesive for cellulose fibers, isolated technical lignin generally are poor binders for wood composites compared to synthetics resins (Frihart, 2010). Frihart (2010) also stated that lignosulfonates were the most used technical lignin for making lignin-based adhesive, however in most cases they were isolated, purified and modified before used for producing adhesive since the required high pressure and temperature for resin curing. Frihart (2005) had stated that both tannin and lignin as adhesive tend to have good moisture resistance and are not readily attacked by microorganisms.

Mansouri, Pizzi and Salvadó (2007) found the following:

> *Lignin based wood adhesives prepared without formaldehyde substituted by non-volatile non-toxic aldehyde, namely glyoxal, were prepared and tested for application to wood panels such as particleboard. The adhesives not only yielded good internal bonding strength results of the board to comfortably pass relevant international standard specifications, but also showed sufficient reactivity to yield panels in press times comparable to those of formaldehyde-based commercial adhesive.* (p. 6)

Lei (2009) found that glyoxalated lignin gave low wood joint strength and cannot be used alone as wood adhesive until it be cross-linked furtherly. He also stated that, the addition of 4,4-diphenylmethane diisocyanate (pMDI) gave

improvement of the MOE on curing joint. Therefore, it can be conclude that improvement of lignin can be made by reaction with the glyoxal and addition of pMDI.

2.3 Development and Properties of Protein Based Adhesive

Protein had been used for long as wood adhesive and can be divided into two groups: one is based on plant protein adhesive; the other is based animal protein adhesive (Lei, 2009). Frihart (2005) stated that many sources have been used for protein based adhesive, including animal bones and hide, milk (casein), blood, fish skins and soybeans. As most other biomass material, protein also not uniform in composition and the process for using different sources of protein to make adhesives are varies (Frihart, 2005). In order to make a useful adhesive, the raw protein must be denatured to expose the polar group for solubilization and bonding purpose (Frihart, 2005).

Huang (2007) stated that, animal bones and hide contain high amount of amide group, free amino groups and carboxylic acid group which interact with the protein chains thus, provide the strength as adhesive toward the wood. This type of adhesive widely used in furniture, unfortunately, their many undesirable properties such as low moisture resistance, relatively high price and susceptibility to biological degradation make they had been replace by synthetic resins (Huang, 2007). In

addition, Huang (2007) also found that blood based adhesive can be improved by addition of formaldehyde and phenol formaldehyde resin in their properties of water resistance, strength and mold resistance. Frihart (2005) also stated that blood protein from beef and hogs gave more water resistance than others protein, however the availability for industrialize scale as well as composition were not consistent. Therefore, soy protein which have low cost, large supply, consistent composition as soybean flour and other special properties (Frihart, 2005) make most of the present research that work on protein adhesive was concentrate on soy protein and had been proposed industrially (Lei, 2009).

2.3.1 Soy protein based adhesive

Soy protein contains about 20% oil, 34% carbohydrates 40% protein and 4.9% ash (Huang, 2007). However, Lei (2009) stated that soy based protein contains 40-60% protein formerly and more that 90% were later. Huang (2007) also found that soy oil composed of saturated and unsaturated triglyceride, soybean carbohydrates consist polysaccharides such as cellulose, hemicelluloses and pectin, and another 18 amino acid in soy protein.

Huang (2007) stated that major commercial soybean product includes soy flour, soybean oil, defatted soybean meal, soy protein concentrate (SPC) and soy protein isolate (SPI). All of the products produced through several processes from

soybean powder. By removing soybean oil, defatted soybean meal and soy flour can be produce. Then, from defatted soy meal soy protein concentrate (SPC) and soy protein isolate (SPI) achieved. Table 2.1 shows the composition of different soy protein product.

Soy based adhesive were first developed around 1923 (Sun et. al., 2008). Huang (2007) also stated that soy based adhesive widely used in the production of wood composites since 1930s until 1960s. Unfortunately, as its poor water resistance, weak bonding strength and poor bio degradation resistance (Lei, 2009) as well as low gluing strength (Sun et al., 2008) make it been replace by synthetic resin. However, lots of research and investigation about this problem had been made to improve those properties since soybean is abundant, inexpensive, renewable and sustainable as well as environmental friendly.

Table 2.1: The composition of different soy protein products

g/100g product	Soy flour	Soy protein concentrate (SPC)	Soy protein isolate (SPI)
Protein	48	64	92
Fat	0.3	0.3	0.5
Moisture	10	10	<5
Fibers	3.0	4.5	<1
Ash	7	7	4
Carbohydrate	31-32	14-15	-

Source: Huang (2007)

2.3.1.1 Modification of soy protein

Because of the inferior properties of soy protein based adhesive, various new method have been investigated in order to improve the strength and water resistance wood composite by soy based adhesive (Huang, 2007). Lei (2009) stated that recently, soy protein been modified and combined with urea formaldehyde (UF), phenol formaldehyde (PF), isocynates or others to obtain more water resistance adhesive as well as stronger bonding. Unfortunately, those approaches contain high amount of synthetic material and maximization of natural component in the wood adhesive is demand (Lei, 2009). Soy protein can be chemically, physically or enzymatically modified in order to achieve desired properties of natural adhesive including hydrolysis, cleavage of disulphide bond, crosslinking, acylation, oxidation, reaction with alkoxy silane and copolymerization (Lei, 2009).

Huang (2007) had stated that soy flour (SF), maleic anhydride (MA), sodium hydroxide (NaOH) and polyethylenimine (PEI) can be combine as adhesive and superior to alkali-modified in term of enhancing strength and water resistance of particleboard. Huang (2007) also stated that by using curing agent such as sulfur containing compound, epoxy compound and aldehydes for cross linking of soy protein, will improve the strength and water resistance of soy based adhesive. As a conclusion, the soy protein needs to be modified by alkylation and adding of suitable curing agent in order to optimum strength and water resistance. Figure 2.1 illustrate the structure of denaturized soy protein.

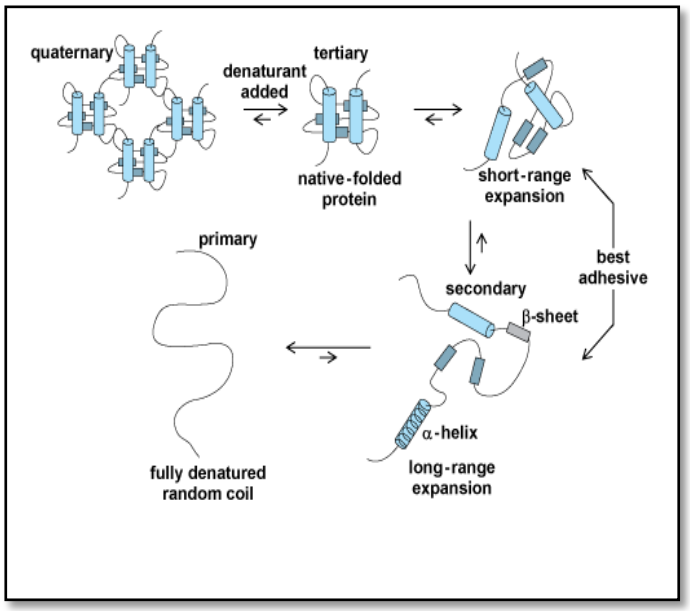


Figure 2.1: An illustration of the structure of protein chain during denaturized

Source: Frihart (2010)

"*Alkali-modified soy protein adhesive was reported to be stronger and more water resistant compared with adhesive containing unmodified soy protein.*" (Lei, 2009, pg 22). In the other hand, Sun et al. (2008) support modified soy protein adhesive gave more strength and water resistance compared to unmodified by experimenting several procedures of modification of soy protein in their research.

2.4 Wood-based Composite

Wood-based composite is terms present a group of product from wood material which was adhesively bonding together (Stark, Cai & Carll 2010). Table 2.2 indicates the classification of wood-based composite which reflect the latest product development.

Table 2.2: Classification of wood-based composites

Veneer-based material
Plywood
Laminated veneer lumber (LVL)
Parallel-strand lumber (PSL)
Laminates
Glue-laminated timbers
Overlayed materials
Laminated wood–nonwood composites[b]
Multiwood composites (COM-PLY[c])
Composite material
Fiberboard (low-, medium-, or high-density)
Cellulosic fiberboard
Hardboard
Particleboard
Waferboard
Flakeboard
Oriented strandboard (OSB)
Laminated strand lumber (LSL)
Oriented strand lumber (OSL)
Wood–nonwood composites
Wood fiber–polymer composites
Inorganic-bonded composites

Source: Maloney (1986)

Typical elements include fibers, particles, flakes, veneers, laminates, or lumber used in the production of wood-based composites and can be made in a variety of sizes and shapes (Stark, Cai & Carll 2010). Figure 2.2 shows the variation and relative size of wood elements.

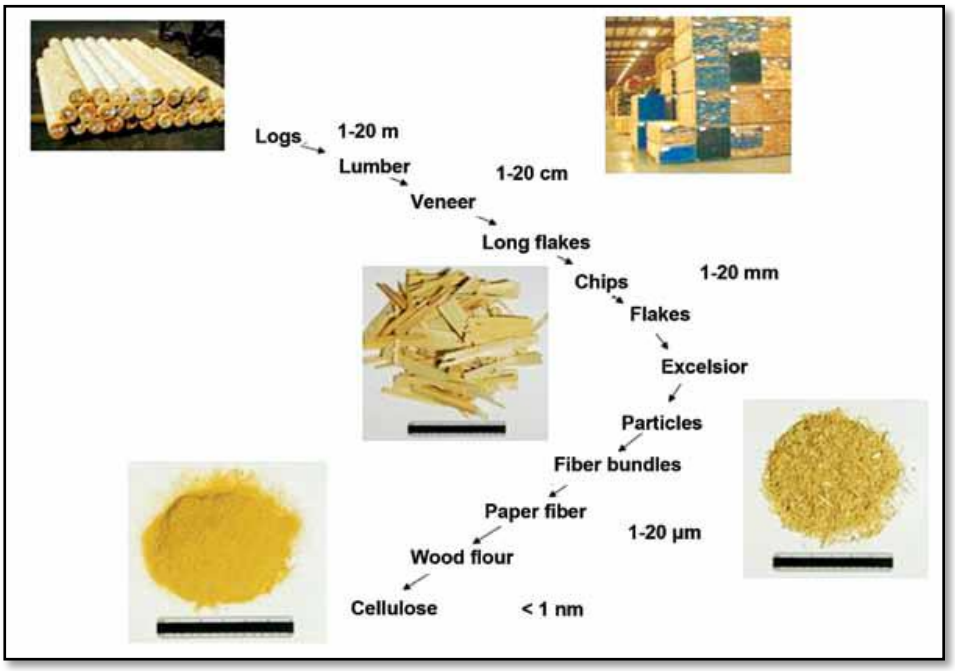


Figure 2.2: Basic wood elements, from largest to smallest

Source: Kretschmann (2007)

2.4.1 Medium Density Fiberboard (MDF)

MDF is frequently used in place of solid wood, plywood, and particleboard in many furniture applications. It is also used for interior door skins, moldings, and interior trim components. Fiberboard is normally classified by density and can be made by either dry or wet processes, however, dry processes are applicable for MDF manufacturing.

Dry-process fiberboard start with resin (UF or MF–UF) and other additives are applied to the fibers by spraying in short-retention blenders or introduced as wet fibers are fed from the refiner into a blow-line dryer. The adhesive-coated fibers are then air-laid into a mat for subsequent pressing, much the same as mat formation for particleboard. After the fiber mat is formed, it is typically pre-pressed in a band press. The densities mat is then trimmed by disk cutters and transferred to cauls plates for the hardboard pressing operation; for MDF, the trimmed mat is transferred directly to the press. Many dry-formed boards are pressed in multi-opening presses. Continuous pressing using large, high-pressure band presses is also gaining in popularity. Panel density is constantly monitored by moisture sensors using infrared light as an indicator of panel quality. (Stark, Cai & Carll 2010)

2.4.1.1 Mechanical Properties of MDF

A wide range of engineering properties are used to characterize the performance of wood-based composites. Mechanical properties are typically the most frequently used to evaluate wood-based composites for structural and nonstructural applications. The mechanical properties of wood composites depend upon a variety of factors, including wood species, forest management regimes (naturally regenerated, intensively managed), the type of adhesive used to bind the wood elements together, geometry of the wood elements (fibers, flakes, strands, particles, veneer, lumber), and density of the final product. (Cai & Ross, 2006)

Elastic and strength properties are the primary criteria to select materials or to establish design or product specifications. Elastic properties include modulus of elasticity (MOE) in bending, tension, and compression. Strength properties usually reported include modulus of rupture (MOR, bending strength), compression strength parallel to surface, tension strength parallel to surface, tension strength perpendicular to surface (internal bond strength), shear strength, fastener holding capacity, and hardness. (Cai & Ross, 2006)

2.5 Conclusion

As a conclusion, the developing of natural adhesive using lignin and protein will give less environment effect or harm to human health if the reactant or chemical use in the process of developing does not containing any toxicity material. In addition, the cost of adhesive manufacturing from lignin and soy protein will be less since the main materials were most abundant from other industries. Therefore, from the related sources of literature review, it is relevant that the developing natural adhesive using lignin and protein to industrialize.

CHAPTER 3

METHODOLOGY

There are several methods in developing natural adhesive. The most and preferred method is formulate the combination of modified lignin and modified protein. However, the modification of lignin was not conducted during preparation of this adhesive. In the experiment, protein was modified by conducting alkylation process. The experimental methodology consists of three basic sub chapters which are raw material, experimental procedures and sample analysis.

3.1 Raw Material

In this research, soy flour (SF) and lignin solution were the most important materials that were used. These materials preferred based on their lower price as abundant material from other industries. For soy protein modification, soy flour (SF) which is use as extruder cooking of textured vegetable protein. This material obtains from public store of groceries. In the other hand, lignin solution also important as the main material for lignin-soy protein adhesive which was obtains from other student. Finally, wood fiber (rubber wood) provided by Robin Resources (Malaysia) Sdn. Bhd, were used for board making.

3.2 Experimental Procedures

The experiment was divided into four sections which were modification of soy flour, lignin-soy protein adhesive making, and medium density fiberboard making. After this section done, the next steps were analysis of adhesive solution (analytical testing) and particle board (mechanical testing).

3.2.1 Adhesive Preparation

Lignin was prepared by evaporated water from dilute lignin solution (Lignin A) to two different concentrations. Three set of Lignin solution (500mL) were heated at 80ºC equipped with magnetic stirrer until one of the solution evaporated to 300mL (Lignin A), another two solution evaporated to 150 mL (Lignin C). All lignin solutions concentrations were analyzed by UV-Vis Spectrophotometer by obtaining the absorbance. The concentration was calculated using equation 3.1:

$$C = \frac{A}{\lambda \times D} \tag{3.1}$$

Where:

C = Concentration of lignin

A = Absorbance value

λ = Wavelength (cm) which is 280cm for lignin

D = Absorbtivity which is 20 L/g.cm for rubber fiber

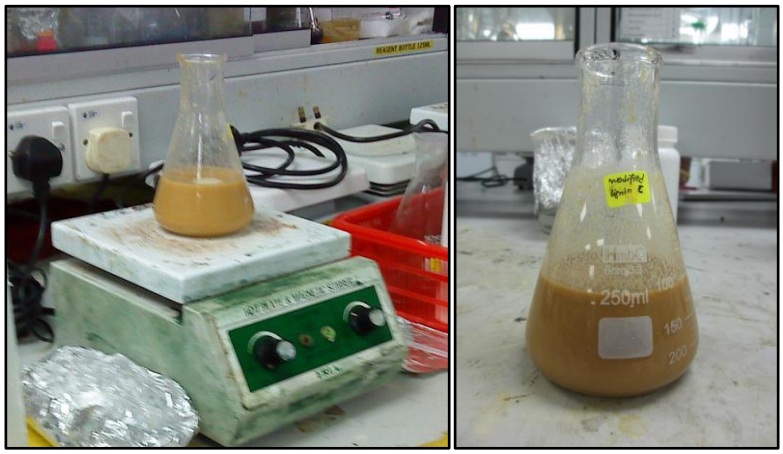
The next step was modification of soy flour. De-ionized water (50 mL) was stirred with maleic anhydride (2 g) and sodium hydroxide 50% solution (10-12mL) until the solution homogenous (approximately 5 minute) in room temperature. After the solution completely homogenous, soy flour (28 g) was added little by little into the solution with the same condition. Mixing process was continuously applied to the solution until the mixture completely mixed.

(a) (b)

Figure 3.1: Illustration of (a) Soy flour as raw material, (b) Modified soy flour

Lignin solution (100mL) immediately poured into the solution in the same condition for 30 minutes to make sure the solution was well mixed. The adhesive were ready to use for board manufacturing. There are three different adhesive were prepared with three different concentration of lignin.

All the mixing and heating process of the solution for adhesive preparation were charge in appropriate volume of conical flask equipped with magnetic stirrer and hot plate. Thermometer also used to observe the temperature during preparing lignin solution.



(a) (b)

Figure 3.2: Photos of (a) Experimental setup, (b) Adhesive C

3.3 Board Manufacturing

From the dried fibers, a fluffed mat of dimension 200 x 200 mm was produced and then pre-pressed to a desired thickness. The mat was then hot-pressed between aluminum cauls plates with a steel screen of 6 mm for a total time of four minutes under 5 MPa of pressure and 180ºC plates temperatures to give a 6 mm board with a target density of 800 kg/m^3 (MDF) . To obtain this desired board, 202 g of dried fiber needed to blend with 20.2 g of adhesive in a blending machine. The mass of wood fiber and adhesive were calculated based on the dimension and desired density by using equations 3.2 and 3.3 respectively:

$$\text{Fiber Mass} = (V_{MDF} \times \rho_{MDF}) + 5\% (V_{MDF} \times \rho_{MDF}) \quad (3.2)$$

$$\text{Adhesive Mass} = 10\% \text{ (Fiber Mass)} \quad (3.3)$$

Four replicates of board were manufactured for the three types of adhesives with the same hot-pressed condition, dimension and density. For each type of board, eight samples was prepared for bending test or modulus of ruptured (MOR) with average dimension 200 x 51 x 6 mm, five sample for thickness test with average dimension 51 x 51 x 6 mm , and seven samples for internal bonding test with average dimension of 51 x 51 x 6 mm.

3.4 Adhesive and Board Analysis

Chemical modification of lignin-soy based adhesive was analyzed by comparing the lignin solution before modified with the adhesive produced by adding modified soy flour to the lignin. The analysis was conducted by FTIR Spectrometer and peak of functional group were collected. Other properties of adhesive also collected such as solution pH and solution viscosity.

For board analysis, there are three important testing were conducted in order to prove that the board passes the board requirement for medium fiber board. There are thickness tests, internal bonding test and modulus of rupture test.

The thickness test is testing procedure to define water resistance requirement. Five particleboard specimens (51 x 51 x 6 mm) were cut from each particleboard and soak in water at 24 $\pm$ 3 ºC for 2 hours, and continue to complete 24 hour soak duration. Dimension of each specimens were taken after 2 hours and 24 hours duration. All specimens are inspecting to see whether they are delaminating

Bonding strength was statistically analyze using SYSTAT 9.0 software after modulus of rupture (MOR) and modulus of elasticity (MOE) data were determined from internal bonding and static bending test.

(a) (b) (c)

Figure 3.3: Illustration of mechanical board testing (a) Bending testing, (b) Internal bonding testing, (c) Thickness testing

CHAPTER 4

RESULT AND DISCUSSION

In this chapter, several result and some discussion will be introduce. There will be result of the analysis for adhesive and medium density fiberboard (MDF) and some discussion of the result appeared.

4.1 Lignin Properties

Only two properties of lignin were analyzed for this study which was concentration and viscosity. Concentration of lignin must be analyze since it was the manipulation variable for this study. It had been calculated by using equation 3.1 by substituting the value from UV-Vis Spectrophotometer analysis for lignin. In the

other hand, the viscosity of the lignin was measured by viscometer. Both data for concentration and viscosity of each lignin was tabulated in table 4.1.

Table 4.1: Concentration and Viscosity data for lignin

Lignin	Concentration (kg/L)	Viscosity (mPa)
Lignin A	37.143	2.62
Lignin B	64.643	2.62
Lignin C	132.321	2.50

From the data shows that, lignin C have the highest concentration among the lignin while lignin A has the lowest concentration.

4.2 Adhesive Properties

In this study, only two physical properties of adhesive were check. First of all, the pH of the three adhesives was checked to proof that the modification of the soy protein was in alkaline. And the other properties were viscosity of the adhesive. All the data was tabulated in table 4.2.

Table 4.2: Viscosity and pH data for natural adhesive

Natural Adhesive	Viscosity (mPa)	pH
Adhesive A	74.72	13.56
Adhesive B	57.90	13.62
Adhesive C	41.60	13.68

By comparing the viscosity of lignin and adhesive, it shows that adhesive which contain lignin and soy protein give higher viscosity than lignin. Therefore, it proof that, development of natural adhesive change the physical properties of the solution.

4.3 Chemical Composition of Adhesive

The analysis of composition (functional group) of adhesive was conducted by using FTIR Spectrometer. This analysis was conducted to compare the functional group consist before and after the adhesive was developed. Besides that, from the peak of the analysis, the functional group of the solution can be found by comparing the wavelength with spectra wavelength. The result of this analysis will shows in figure 4.1 until figure 4.3.

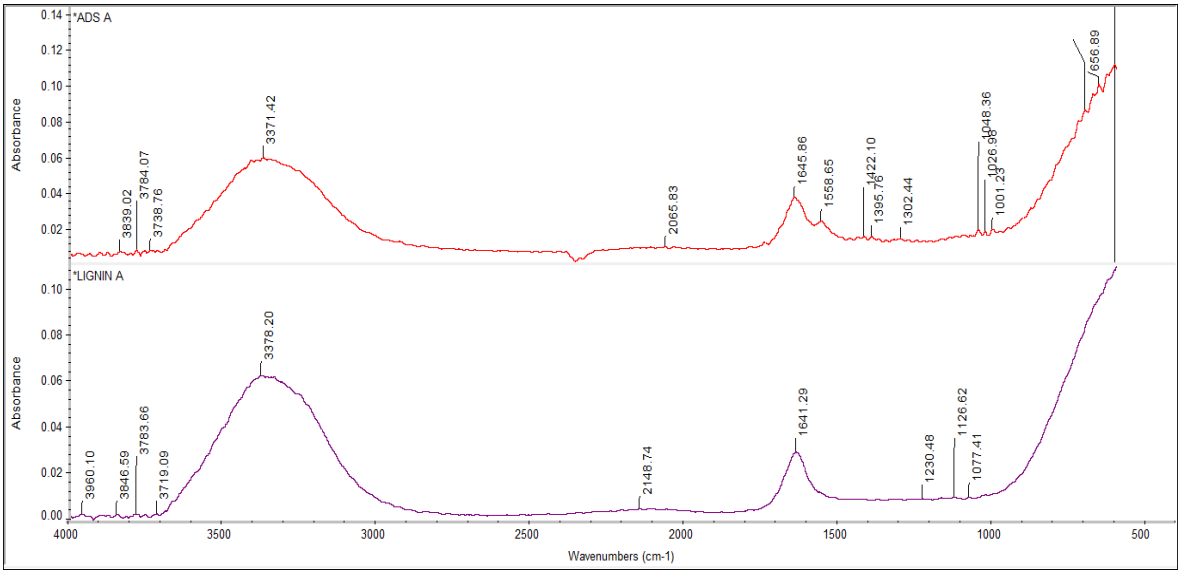


Figure 4.1: Comparison of lignin A (Purple line) with adhesive A (Red line)

Figure above show the slightly different peaks in adhesive A (red line) which is additional peaks at wavelength 1558 and 1442. Others peaks are similarly to each other such as at wavelength 3371 and 1645 but a bit different in absorbance value. In table 4.3, the functional group and molecular motion of the peaks will showed.

Table 4.3: Peaks description of adhesive A

Wavelength	Functional Group	Molecular motion
3371	Alcohol	O-H stretch
1645	alkenes	C=C stretch (isolated)
1558	Nitro group	NO_2 (aliphatic)
1422	Carboxylic acids	O-H bend
1048	sulfoxides	S=O stretch

Figure 4.2 show the slightly different peaks in adhesive B (blue line). There is missing and additional peaks. However, through this analysis, it shows that the adhesive B chemically modified from the lignin B. Table 4.4 will describe five higher peaks illustrated in figure 4.2.

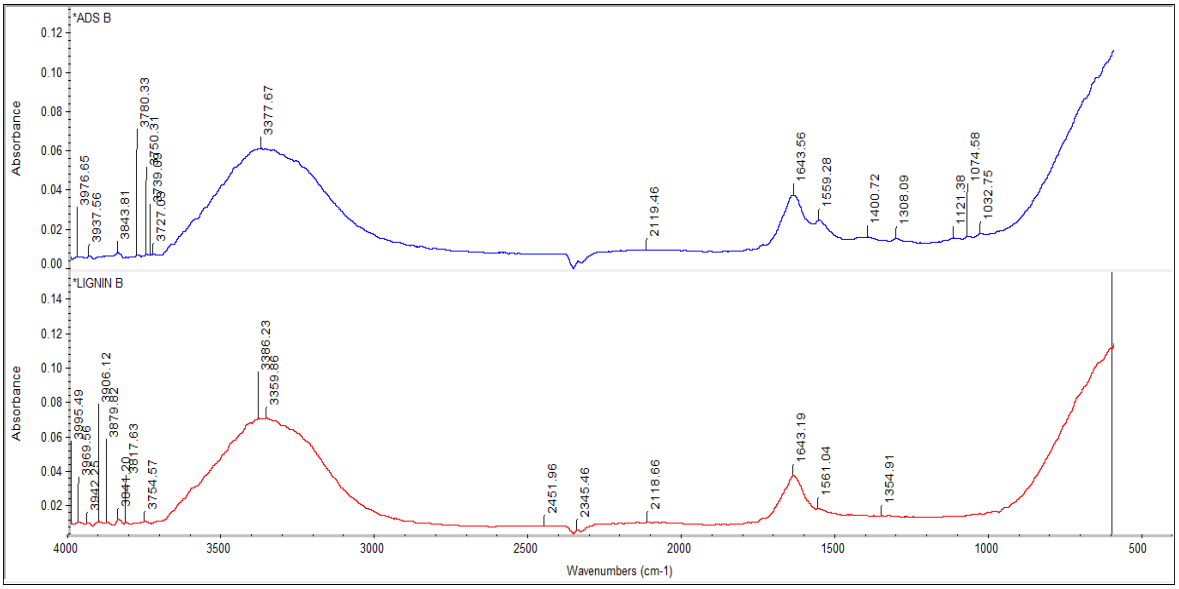


Figure 4.2: Comparison of lignin B (red line) with adhesive B (blue line)

Table 4.4: Peaks description of adhesive B

Wavelength	Functional Group	Molecular motion
3377	Alcohol	O-H stretch
1643	alkenes	C=C stretch (isolated)
1559	Nitro group	NO_2 (aliphatic)
1400	Carboxylic acids	O-H bend
1308	ketones	C-C stretch

Figure above show the different curve in adhesive C (blue line) with lignin C (red line). The curve of lignin C gives many peaks of functional group if compared to adhesive C curve. Unfortunately, actually the peaks are similarly to each other but slightly different at the absorbance value there always are missing peaks and additional peaks at adhesive C curve. However, through this analysis, it shows that the adhesive C chemically modified from the lignin C. Table 4.5 will describe five higher peaks for adhesive C.

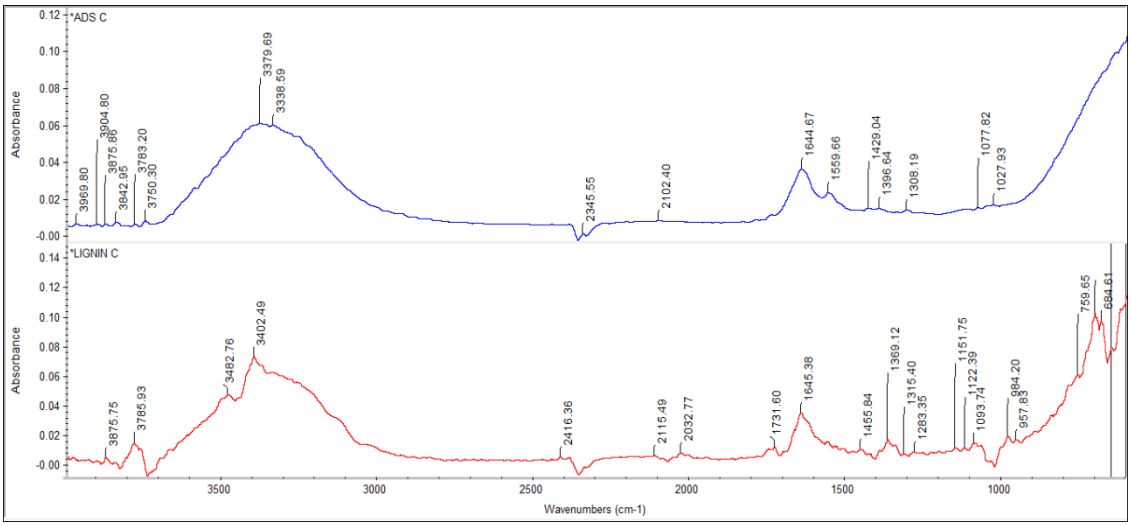


Figure 4.3: Comparison of lignin C (blue line) with adhesive C (red line)

Table 4.5: Peaks description of adhesive C

Wavelength	Functional Group	Molecular motion
3379	Alcohol	O-H stretch
3338	Amines/ amide	N-H stretch
1643	Alkenes	C=C stretch (isolated)
1559	Nitro group	NO_2 (aliphatic)
1429	Carboxylic acids	O-H bend

4.4 Modulus of Rupture Test (MOR)

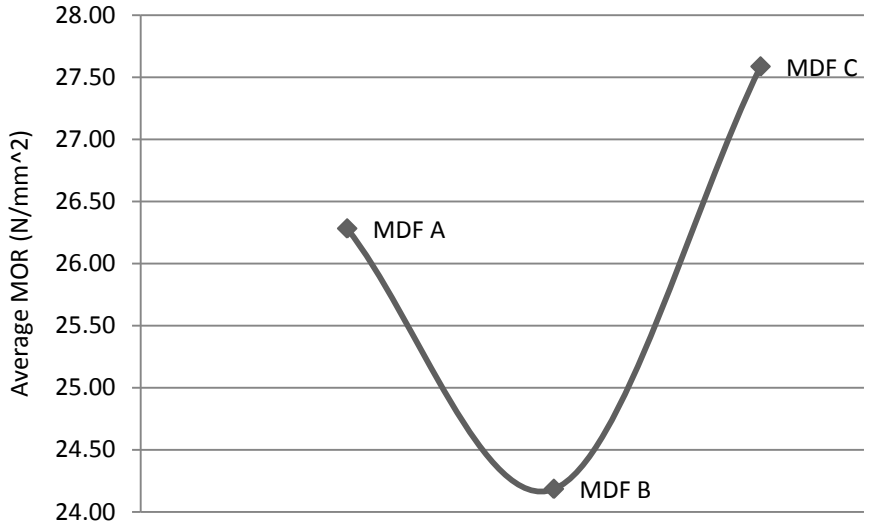


Figure 4.4: Average MOR of each type of Medium Density Fiberboard

Based on the figure 4.4, it shows that MDF C had the highest MOR value compared to MDF A and MDF B. Unfortunately, the lowest average MOR is owned by MDF B, in this case, it can be cause by the pressing temperature influence since during the board making of MDF B, there is some problem with the thermocouple wire connected with bottom plate temperature which make the temperature cannot reaches the set temperature (180 ^{o}C).

However, it was expected that the board will reach value of 35 N/mm^2 to compete the petroleum-based adhesive in the market. But then, the value of the MOR still makes the MDF reasonable to consider as good board and can be useful to the industry since it is environmental friendly.

4.5 Internal Bonding Strength Test (IB)

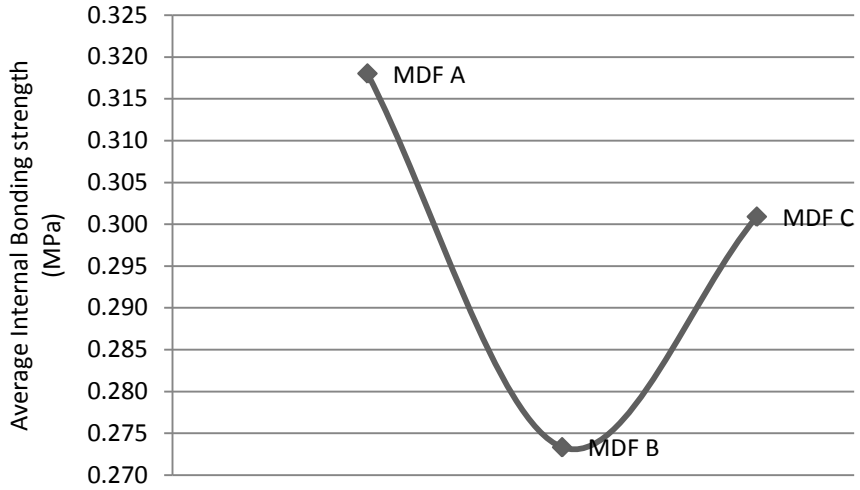


Figure 4.5: Average internal bonding value of medium density fiberboard

Based on the figure 4.5, it shows that the average IB strength value of MDF A is the higher one and the lowest value is MDF B. Unfortunately, based on the data gained from the testing (Appendix G), most of the specimens were experience glue failure which means the breaking part of maximum force was at the glue attachment (Figure 4.6). Therefore, the value of average IB strength will not consider the reading. As stated previously, MDF B give the lowest value of average IB strength since there is some problem with the thermocouple wire connected with bottom plate temperature during board making.

In the other hand, based on the average value, MDF C had IB strength of 0.301 MPa. However, one of the samples from MDF C gave the highest value which sample C3 internal bonding strength of 0.411 MPa. Therefore, it indicates that, the inconstant dispersion of adhesive to the fiber influence the internal bonding strength

of the board. Based on the observation also, there was no glue failure for MDF C since the precaution was applied.

(a) (b)

Figure 4.6: Illustration of internal bonding test result (a) Glue attachment failure (b) MDF break at middle (pass)

4.6 Thickness Test (water absorption test)

Based on observation of the structure of the sample after the test, it can be assume all three type of board are fail for this thickness test since the sample thickening double from their initial thickness. The increments of the thickness of each type of the sample were illustrated in table 4.6. However, some parts of the sample are not thickening as much as other part (figure 4.7). It may cause by higher volume of adhesive to the small part of fiber. Therefore, it indicates that the percentage of adhesive used for board making effect the water resistance ability of the board. In the other hand, the board is highly absorb water can be cause from the weakness of soy protein it self as hydrophilic substance.

Table 4.6: Average thickness test result

MDF Sample	Thickness Swelling (%)		Water Absorption (%)	
	2 h	24 h	2 h	24 h
MDF A	116	133	239	261
MDF B	150	150	350	380
MDF C	100	133	274	299

Figure 4.7: Thickness test sample

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

As a conclusion, natural adhesive that contain environmental friendly material had been develop by using lignin and soy protein and some other non-toxic material. Table 5.1 show the formulation used to develop the natural adhesive. Unfortunately, the result of board testing shows that, the adhesive cannot compete with the formaldehyde-based adhesive. However, high internal bonding strength and good water resistance of adhesive can be achieved by some additional modification of soy protein and lignin. In the other hand, from this study it was found that only bending strength gives the good result and can be acceptable.

Table 5.1 Formulation of natural adhesive

Component	Percentage (%)
Lignin	53
Soy Flour	15
Sodium Hydroxide (50%)	5
Di-ionized Water	26
Maleic Anhydride	1

5.2 Recommendation

From the conclusion, it shows that an additional modification of soy protein need to be add in order to increase the water resistance ability of the board. The soy can be treated to separate the hydrophilic from the hydrophobic structure of substance. In the other hand, modification of lignin also will help in increase the strength of the binding properties of adhesive, however, must remember to avoid using of toxic chemical.

Furthermore, during manufacturing the board, some parameters also affect the mechanical and water resistance ability of the board. It also suggested to blenders the fiber with more percentage of adhesive other than 10% as in this study. Besides that, the parameter such as temperature and pressing time also will affect the mechanical testing result. Therefore, it is recommended to wait until the temperature reaches the set temperature value before start the hot pressing process.

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Appendix A

Gantt Chart

Project Title (PSM): Development of Natural Adhesive Using Lignin and Soy Protein										
Year	PSM I: 2012					PSM II: 2012-2013				
Month	Feb	Mac	Apr	May	Jun	Sep	Oct	Nov	Dec	Jan
Project Tasks / Weeks	1 2 3 4	1 2 3 4	1 2 3 4	1 2 3 4	1 2 3 4	1 2 3 4	1 2 3 4	1 2 3 4	1 2 3 4	1 2 3 4
Identify project (problem to investigate) and scope of research	x									
Plan work schedule		x								
Review related literature		x x x x								
Determine methodology			x x							
Write proposal and abstract (summary of proposal)			x x							
Present and defend proposal in oral presentation				x						
Submit written research proposal and abstract					x					
Collect and analyse data						x x x x	x			
Interpret results							x x	x		
Evaluate results**:**										
Achieve research objectives/ milestones								x x		
Draw conclusions and **suggest** recommendations								x	x	
Revise and edit first draft of Introduction, Literature Review and Methodology (from proposal)									x	
Write first draft of Results & Discussions, Conclusions & Recommendations									x x	x
Revise and edit abstract (from proposal)										x
Compile entire final report and revised abstract										x
Present and defend final report in oral presentation										x
Submit written final report and abstract										x

Appendix B

UV-Vis Spectrophotometer Data

Table B1: Data for UV-Vis Spectrophotometer

Solution	Wavelength (nm)	Dilution factor	Absorbance	Absorptivity (L/g.cm)
Lignin A	280	100	0.208	20
Lignin B	280	100	0.362	20
Lignin C	280	100	0.741	20

Appendix C

Viscometer Data

Table C1: Data collection for Viscometer

Solution	Spindel type	RPM	τ (%)	Viscosity (mPa)	S STR (N/m^2)	S Rate (s^{-1})
Lignin A	31-SC4	240	2.3	2.62	0.22	81.6
Lignin B	31-SC4	240	2	2.62	0.21	81.6
Lignin C	31-SC4	240	2	2.5	0.21	81.6
Adhesive A	73	50	74.4	747.2	-	-
Adhesive B	73	200	23.2	57.9	-	-
Adhesive C	73	200	16.6	41.6	-	-

Appendix D

FTIR Result

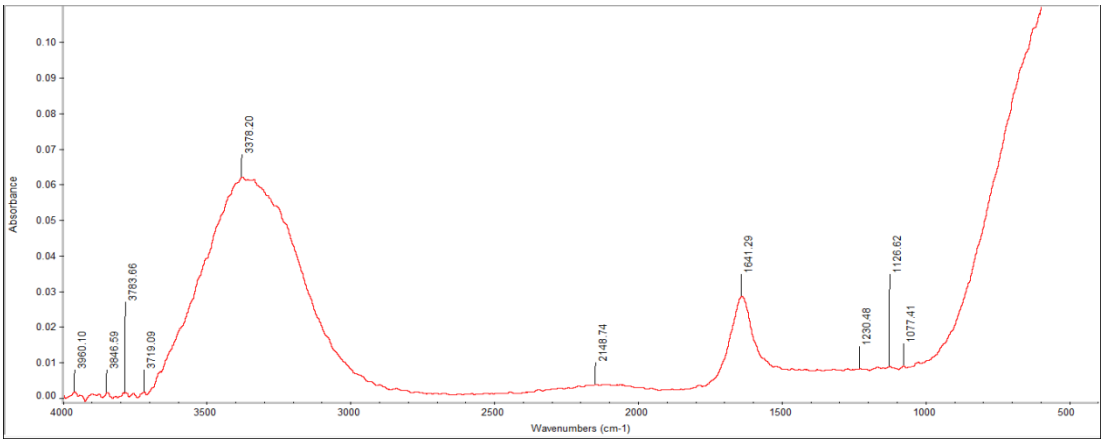

Figure D1: FTIR peaks for lignin A

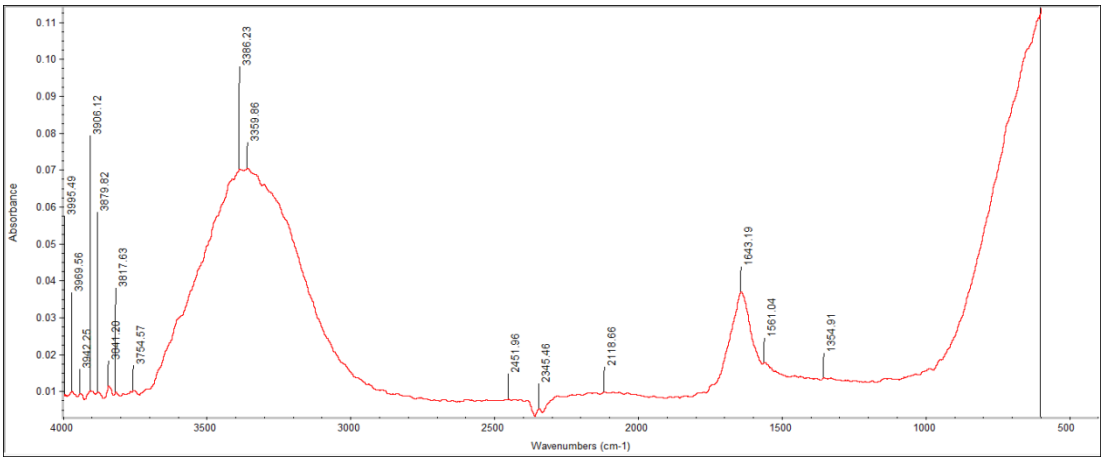

Figure D2: FTIR peaks for lignin B

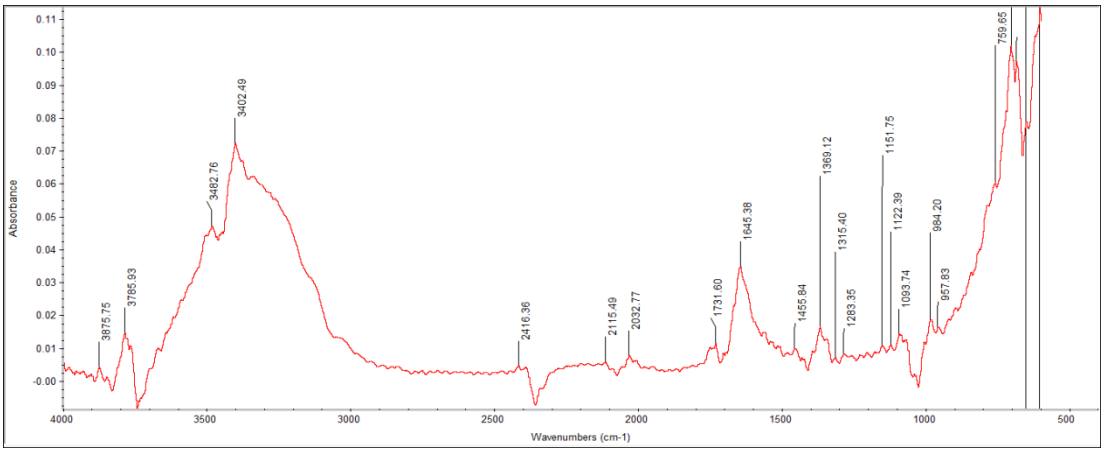

Figure D3: FTIR peaks for lignin C

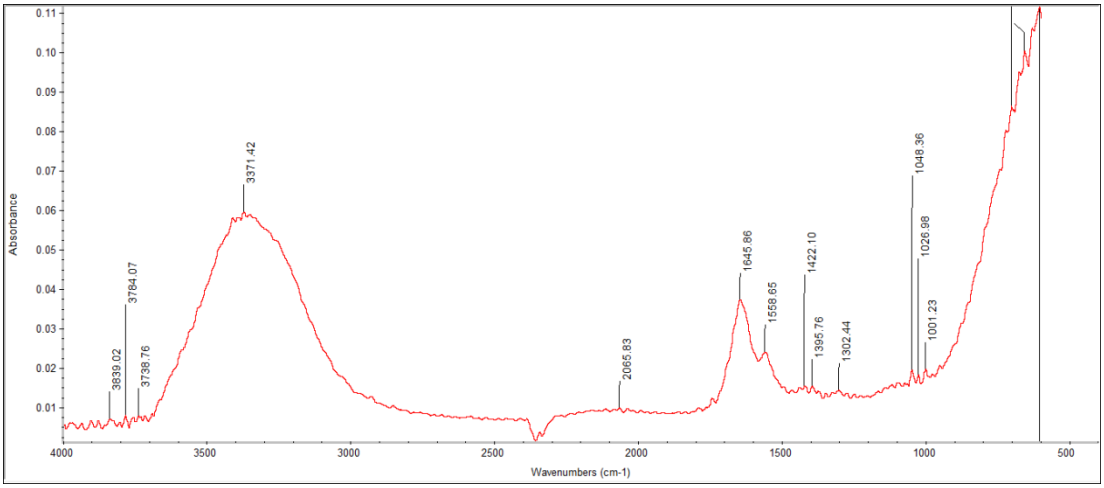

Figure D4: FTIR peaks for adhesive A

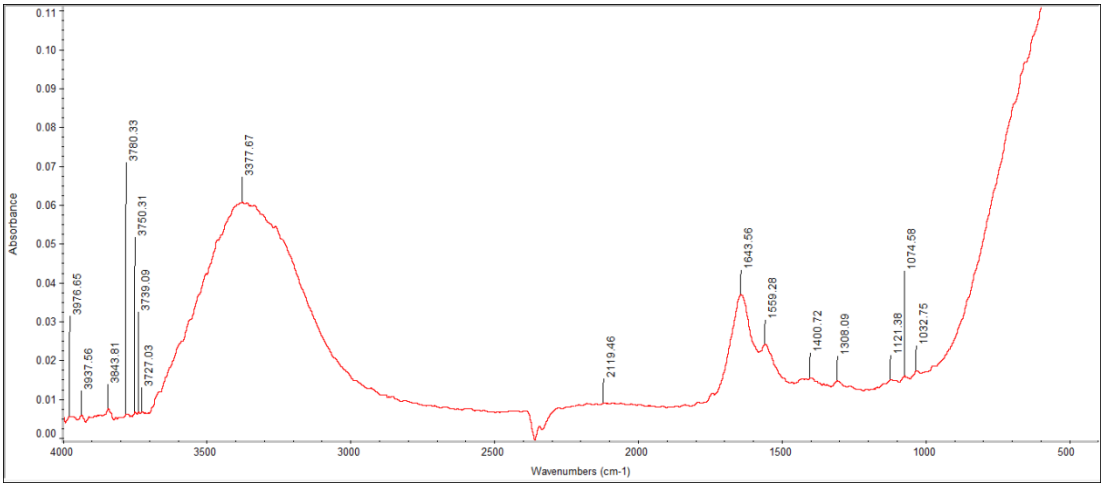

Figure D5: FTIR peaks for adhesive B

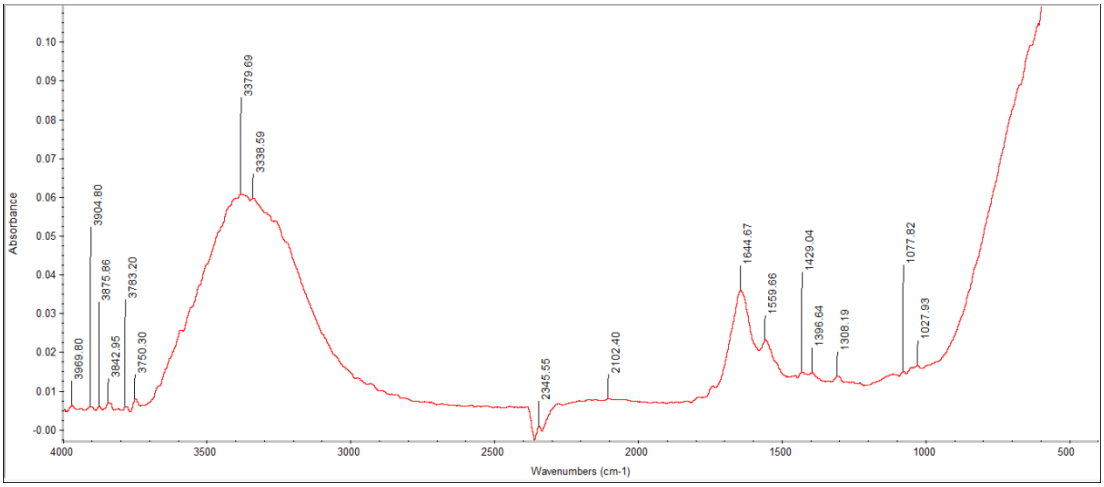

Figure D6: FTIR peaks for adhesive C

Appendix E

Summary of FTIR Spectra Table

Simplified Table of Main IR Frequencies

Wave number, cm^{-1}	Functional Group	Peak Description
3300 – 3600	O-H (alcohol)	Strong and broad
2500 – 3000 can reach	O-H (carboxylic acids)	Very broad (over ~ 500 cm^{-1}), often looks like distorted baseline, above 3000 cm^{-1}.
3200 – 3500	N-H	Doublet in case of NH_2 group of a primary amine or amide
3300	$\equiv$ C—H terminal alkyne	Usually sharp and strong
3000 - 3100	= C—H alkene or arene	Often weak, overlaps with CH alkane absorption
2800 – 3000	C-H (sp^3 carbon)	Strong, broad and multi-banded
2250 - 2220	C$\equiv$N	Medium intensity
2100 - 2260	C$\equiv$C alkyne	Medium intensity for terminal alkynes, very weak for internal
1680 – 1820	C=O (amides, ketones, aldehydes carboxylic acid, esters)	Very strong; lower frequency for amides and when C=O is conjugated
1600 – 1650	C=C alkene, aromatic ring	Check to see if you have C-H unsaturated >3000 cm^{-1} (if not, it's completely substituted)
~ 1600	$-NH_2$ (bending) 1° amines and amides	Only if you have corresponding N-H peak at 3200-3500 cm^{-1} (this peak may be mistaken for C=C otherwise)
1200	Ar-O	Strong (look for =C-H & C=C first)
1050-1150	C-O	
690 and 750	phenyl group	Strong (look for =C-H & C=C first)

Appendix F

Bending Test Data Table

Sample	Volume (mm^3)	Density (kg/m^3)	Force (N)	MOR (N/mm^2)
A1	61200	658.350	173.72	21.289
A2	60000	683.700	195.97	24.496
A3	61200	651.422	181.23	22.210
A4	61200	709.395	287.27	35.205
A5	61200	648.105	186.14	22.811
A6	61200	700.572	232.34	28.473
A7	61200	652.467	182.75	22.396
A8	61200	730.016	272.33	33.374
Average	61050.000	679.253	213.969	26.282
B1	61200	610.327	169.10	20.723
B2	61200	720.065	236.80	29.020
B3	61200	664.673	147.30	18.051
B4	61200	714.167	258.16	31.637
B5	61200	656.078	194.18	23.797
B6	61200	709.706	261.60	32.059
B7	61200	644.837	126.19	15.464
B8	61200	643.971	185.45	22.727
Average	61200.000	670.478	197.348	24.185
C1	61200	657.582	174.89	21.433
C2	61200	679.510	279.26	34.223
C3	61200	689.739	209.22	25.640
C4	61200	698.219	250.05	30.643
C5	61200	630.343	176.39	21.616
C6	61200	678.546	226.57	27.766
C7	61200	650.850	206.61	25.320
C8	61200	725.065	277.84	34.049
Average	61200.000	676.232	225.104	27.586

Appendix G

Internal Bonding (IB) Strength Data Table

Sample	Weight (g)	Volume (mm^3)	Density (kg/m^3)	Force (N)	IB (MPa)
A1	11.06	15606	708.702	865.90	0.333
A2	9.98	15606	639.498	-.-	-.-
A3	11.48	15606	735.615	814.21	0.313
A4	11.01	15606	705.498	113.97	0.044
A5	11.41	15606	731.129	801.28	0.308
A6	11.34	15606	726.644	198.19	0.076
A7	11.57	15606	741.382	270.92	0.104
Average	11.121	15606.000	712.638	437.781	0.168
B1	11.85	15606	759.323	542.64	0.209
B2	11.92	15606	763.809	360.70	0.139
B3	10.76	15606	689.478	202.13	0.078
B4	10.82	15606	693.323	392.89	0.151
B5	12.41	15606	795.207	593.47	0.228
B6	10.18	15606	652.313	80.70	0.031
B7	11.60	15606	743.304	828.50	0.319
Average	11.363	15606.000	728.108	428.719	0.165
C1	11.07	15606	709.343	584.76	0.225
C2	11.43	15606	732.411	434.10	0.167
C3	12.02	15606	770.217	1068.13	0.411
C4	11.59	15606	742.663	908.91	0.349
C5	11.16	15606	715.110	958.44	0.368
C6	12.29	15606	787.518	713.72	0.274
C7	11.77	15606	754.197	810.44	0.312
Average	11.619	15606.000	744.494	782.643	0.301

Appendix H

Thickness Test Data

Table H1: Initial measurement of MDF at time 0 hour

Sample	Length (cm)	Width (cm)	Thickness (cm)	Weight (kg)
A1	5.0	5.1	0.6	10.282
A2	5.1	5.1	0.6	11.880
A3	5.1	5.1	0.6	11.118
A4	5.1	5.1	0.6	11.190
A5	5.1	5.1	0.6	11.986
Average	5.1	5.1	0.6	11.3
B1	5.1	5.1	0.6	9.030
B2	5.1	5.1	0.6	9.537
B3	5.1	5.1	0.6	11.190
B4	5.2	5.1	0.6	9.547
B5	5.2	5.1	0.6	10.786
Average	5.1	5.1	0.6	10.0
C1	5.1	5.1	0.6	10.280
C2	5.1	5.1	0.6	11.880
C3	5.1	5	0.6	8.508
C4	5.1	5.1	0.6	10.190
C5	5.1	5.1	0.6	10.160
Average	5.1	5.1	0.6	10.2

Table H2: Measurement of MDF after 2 hours soaks in water

Sample	Length (cm)	Width (cm)	Thickness (cm)	Weight (kg)
A1	5.2	5.2	1.3	38.114
A2	5.2	5.3	1.3	37.083
A3	5.2	5.2	1.5	42.486
A4	5.2	5.1	1.2	37.932
A5	5.2	5.1	1.3	36.387
Average	5.2	5.2	1.3	38.4
B1	5.2	5.2	1.7	45.718
B2	5.2	5.2	2	54.175
B3	5.1	5.2	1.4	43.975
B4	5.2	5.2	1.2	39.201
B5	5.2	5.2	1.3	41.996
Average	5.2	5.2	1.5	45.0
C1	5.2	5.2	1.3	37.149
C2	5.2	5.2	1.2	38.239
C3	5.2	5.1	1.1	37.982
C4	5.3	5.2	1.2	38.27
C5	5.2	5.2	1.3	39.448
Average	5.2	5.2	1.2	38.2

Table H3: Measurement of MDF after 24 hours soaks in water

Sample	Length (cm)	Width (cm)	Thickness (cm)	Weight (kg)
A1	5.2	5.2	1.5	41.07
A2	5.2	5.2	1.4	39.143
A3	5.2	5.2	1.3	44.901
A4	5.2	5.2	1.5	40.078
A5	5.2	5.1	1.3	38.971
Average	5.2	5.2	1.4	40.8
B1	5.3	5.2	1.4	49.298
B2	5.4	5.2	1.9	58.446
B3	5.2	5.1	1.5	46.173
B4	5.3	5.3	1.4	41.172
B5	5.3	5.2	1.4	44.693
Average	5.3	5.2	1.5	48.0
C1	5.3	5.2	1.5	39.94
C2	5.2	5.2	1.4	40.951
C3	5.3	5.1	1.3	40.307
C4	5.3	5.3	1.4	40.785
C5	5.3	5.2	1.5	41.38
Average	5.3	5.2	1.4	40.7

Appendix I

Picture of Equipments

Figure I1: FTIR Spectrometer

Figure I2: Viscometer

Figure I3: Saw Cutter

Figure I4: Universal Mechanical Testing Machine

Figure I5: Blender Mixer

Figure I6: UV-Vis Spectrophotometer